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An Investigation of Chinese Historical Grey Bricks of Soochow, Jiangsu and the Effect of Tung Oil Treatment

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Abstract
The grey brick is one of the key materials to Chinese traditional architecture. While brick-making in Europe and North America is well documented in sufficient literature, the kiln, firing and properties of the Chinese grey brick is to be explored more in detail. The process gives the bricks a different character and color. Bunches of Chinese literature and informal records show the outstanding character of Chinese grey bricks. And it is why historical grey bricks were commonly used in architectural buildings, city walls, mausoleum. This thesis is aimed to verify the good properties of Chinese grey brick through experiments, and investigate the effect of Tung oil in the treatment of brick materials, especially grey bricks.

Keywords
China, grey brick, Tung oil, microstructure, durability

Disciplines
Architectural History and Criticism | Chinese Studies | Historic Preservation and Conservation

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AN INVESTIGATION OF CHINESE HISTORICAL GREY BRICKS OF SOOCHOW, JIANGSU AND THE EFFECT OF TUNG OIL TREATMENT

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Dedicated to my father and mother…

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CHAPTER 1: Introduction

Like wood, earth material is essential in ancient China. “Civil engineering” is interpreted as “work of earth and wood” (“土木”) in the language of Chinese mandarin. Traditional clay bricks in China, were made of earth, mixed with water and then put into dedicated molds. Under some circumstances, they were firstly sun-dried before firing in the kiln and becoming durable and firm.

According to Institute of Archaeology, Chinese Academy of Social Science, the oldest known bricks were building blocks in early Western Zhou (西周, 1111-771 B.C.)[1]. Solid architectural ceramic walling material was introduced around 3 century B.C. The glazing layer dated from 260 A.D. was the earliest from such ceramics known in China[2].

The grey brick is one of the key materials to Chinese traditional architecture. While brick-making in Europe and North America is well documented in sufficient literature, the kiln, firing and properties of the Chinese grey brick is to be explored more in detail. The process gives the bricks a different character and color. Bunches of Chinese literature and informal records show the outstanding character of Chinese grey bricks. And it is why historical grey bricks were commonly used in architectural buildings, city walls, mausoleum constructions and other important historical structures, which can be dated back to Han Dynasty (漢朝: 206 B.C.-220 A.D.).

[1] Chinese Silicate Society, *History of Chinese Pottery and Porcelain*. Beijing: 1982: 81

[2] Qinghua Guo, Tile and Brick Making in China: a Study of the Yingzao Fashi. (營造法式). *Construction History* 16, 2000: 3

To summarize the outstanding points of grey bricks that have been stated in Chinese literature:

- Good performance in compressive strength;
- More resistant to chemicals;
- Lower to mediate fired, but not more porous.

The type of clay really matters to the properties of the brick. The historical bricks of best quality were originated from Soochow, Jiangsu Province (a. k. a *Wujiang* brick, *吳江* 磚), which were applied in Forbidden City, Beijing and still can be seen today. Additionally, from the relevant chapters of *Yingzao Fashi* (*營造法式*) : Official building guide book of Northern Song Dynasty (北宋), the *Wujiang* bricks were treated after being fired and promoted in quality. It is also called *Jinzhuan* (*金磚*) in Chinese, which means gold brick. The Chinese name of this historical brick gives people some hints of its high density as well as top quality.

In general, durability of architectural ceramics is considered to be closely related to the firing and vitrification degree. However, confined with the technological factors, some of the early bricks were not highly vitrified (871℃ -1316℃). Actually, the vitrification degree alone proved to be insufficient to predict weathering behavior: both porosity and pore size distribution must be considered as well.

Previous researches show there is a general scarcity in the data of the porosity, pore size distribution and hygric behavior of Chinese historical bricks. The first aim of this thesis is to complete the basic research and record the data for reference. The next is to verify the superiority of grey bricks to red ones, which was questionable and not scientifically proved

before. The third issue, as a plus, is to test the effect of ancient treatments to the brick samples- both grey and red, historical and modern. (According to a Ming book on brick making, the *Zaozhuan Tushuo*[3] *(造磚圖說)*, the floor brick tiles used in the yards of imperial palaces were soaked in Tung oil). Also brick carving pieces tended to be treated in Tung oil before being carved. This is actually a way to carry special finishing out. Hence, Tung oil must have its specific effect on the grey bricks, making them even more durable and applicable.

The research is based on the understanding of the theory of building conservation of architectural ceramics. Within the historical and technical spectra of the common bricks in Philadelphia, United States, as well as the knowledge of the building history of ancient China, the research was proposed and carried on.

In the first phase of the research, the literature review in English is done. The abstract of methodology came together as it is generally applied to the architectural ceramic building materials. In the second phase, I traced back to the Chinese literature, such as he Ming Dyndasty book *Tiangong Kaiwu* (*天工开物*) and the Song Dynasty official building guide book *Yingzao Fashi* (*營造法式*), and other useful local literature to check the records and data of Chinese traditional recipe of brick-making. In the third phase, the experimental designs are done about the physical and chemical properties. The final phase includes the Tung oil treatment procedure and a set of tests and examination on the original samples and Tung-oil-treated samples including SEM.

[3] This issue written by Wenzhi Zhang, Zaozhuan Tushuo (The Illustrated Essay on Brickmaking) (Ming Dynasty) was part of Imperial Manuscript Library (四庫全書).

CHAPTER 2: Chinese Traditional Grey Brick

2.1 Historical Use

Some newly emerged evidence indicates Chinese people started to make bricks about 7,000 years ago. Indeed, the history of brick-making in Neolithic China still remains extremely sketchy and unclear. The reliable literature reflects Chinese brick-making dated back before Han Dynasty. Specimens of brickwork have been found in the tombs: the brick walls were double-layered. Some were even stamped with specific emblems. Another finding was that there was no mortar used but red pottery tiles were there to fill the gaps in the vaulting[4].

To have an overview upon Chinese architecture, although many important buildings are yet wholly of wood or timberwork, brick might still be the principal building material in China. In building the city walls, the bricks being about twelve inches long were used. However, for some imperial palaces, temples and other fine building work, grey bricks of smaller size were used frequently. The typical American brick is reddish brown in color, while the traditional vernacular Chinese brick is greyish, slightly green. In Chinese, this color is called “caesious” (青, 青色). Traditional grey brick is used throughout mainland China in typical Chinese architecture, with the finishes of lime wash, creating the basic image of architectural style around the *Yangtze* Plain (a. k. a *Jiangnan* area, 江南). Grey brick

[4] J. Trevor Holmes. “The Traditional Buildings of the Pearl River Delta, China in the Ching Dynasty (清朝) 1644-1912.” PhD Dissertation. Institute of Advanced Architectural Studies, University of York. March 1994: 156

buildings can always be found in the orthodox, wealthy The term, *Feng Qiang Dai Wa* "*粉墙黛瓦*" (white wall and dark greenish tiles) is used to describe this dwelling style.

Figure 2.1.1 The Surging Waves Pavilion (滄浪亭), one of the four greatest classical private gardens of Soochow: a grey brick structure. (Photo by Wenwen Xia)

Another distinctive character of grey brick is its ornamental properties, also showing the preference in decoration and craftsmanship. Brick carving of grey bricks is unique in China. As is known, the brickwork of Western style is more relevant to the design of the pattern and the precision of bricklayers. Also, there are different types of bonding in different places in different historical periods. Unlike the Western style brickwork, as far as Chinese brick carving concerned, the carved bricks, usually can be pulled out and even displayed

as an individual artful piece. Before being carved, usually the grey bricks need to be soaked in water so that the hardness is appropriate for the next process. In fact, for higher quality work, they were also soaked in “Tung oil” instead of water.

Figure 2.1.2 A historical building on Pingjiang Rd (平江路), Soochow,
originally built with grey bricks, renovated with red bricks (Photo by Wenwen Xia)

Besides the fine building structures, grey bricks are still common to be seen as an indispensable building material in ordinary buildings. Fortunately, the crafts of brick carving is also alive, too. Craftsmen have inherited the discipline and methodology from their ancestors. There are still a number of craftsmen and workshops making these

decorative brick pieces and building units. In historical district of Soochow, bungalows are still being built but mimicking the antique buildings as well as the brick carving pieces.

Figure 2.1.3 Contemporary Soochow made grey brick carving piece (Photo by Wenwen Xia)

2.2 Manufacturing History

In North America, it is universally accepted that brick masonry is as an inferior alternative to stone masonry material. The overall situation is the most celebrated or monumental buildings are of stone. Few fine examples of brick buildings are left except for some earliest buildings back to Independence era. Still the historical significance and spirituality weighs far more than the materiality in such monumental structures like Carpenter's Hall

(1775, Philadelphia, PA), President's House (1767, Philadelphia, PA) and other early brick buildings.

While in China, there is a tradition of making ceramic products. Although the timbered structure buildings take a prevailing position in Chinese ancient architectural history, the architectural ceramics are not compromises in the material-using history, belonging to a high-class category, among which the glazed tiles of various colors（a. k. a"琉璃瓦"）used in imperial palaces are remarkable.

According to Institute of the History of Natural Science, Chinese Academy of Science, the brick making in China is divided into three periods[5]----the Pre- Qin (先秦, 2100 B.C.- 1100 A.D.) period, the Han Dynasty period, and the Post- Southern and Northern Dynasty (魏晋南北朝, 220 - 589 A.D.) period. Up to the last period, with the advancement of burning of load bearing brick, stupas built in brick came into existence. Furthermore, in Song Dynasty (宋, 920- 1279 A.D.), with the completion of *Yingzao Fashi* (*營造法式*), the brick-making specification was officially standardized.

In the Ming Dynasty book *Tiangong Kaiwu* (*天工开物*), even the narrative illustrations and interpretation about grey bricks could be found. These visuals and interpretation are vivid and simple to understand. For instance, in Figure 2.2.1, the man is pouring water from the top of the kiln, while the other man is adding fuel to the kiln. The introduction of water into the hot kiln produces a large amount of water vapor, meanwhile, keeping off the fresh air from entering the kiln during cooling down, as well as helping to speed up the cooling[6]. In Figure 2.2.2, the illustration provides information about the type of the fuel used in the

[5] Institute of the History of Natural Science, *Chinese Academy of Science. History and Development of Ancient Chinese Architecture*. (in Chinese) Beijing: Science Press. 2000:253-257

[6] Guo, Tile and Brick Making in China: a Study of the Yingzao Fashi. 4

firing process, confirming the use of coal at that period. These illustrations and interpretation are precious and valuable to the coming researchers within the field of Chinese manufacturing history of architectural ceramics.

Figure 2.2.1 Illustration from Tiangong Kaiwu, 1st Edition. No. 49

Adding water from the top of the kiln.

初刻本插图五〇：煤炭烧砖。

Figure 2.2.2 Illustration from Tiangong Kaiwu, 1st Edition. No. 50

The use of coal as fuel of firing process.

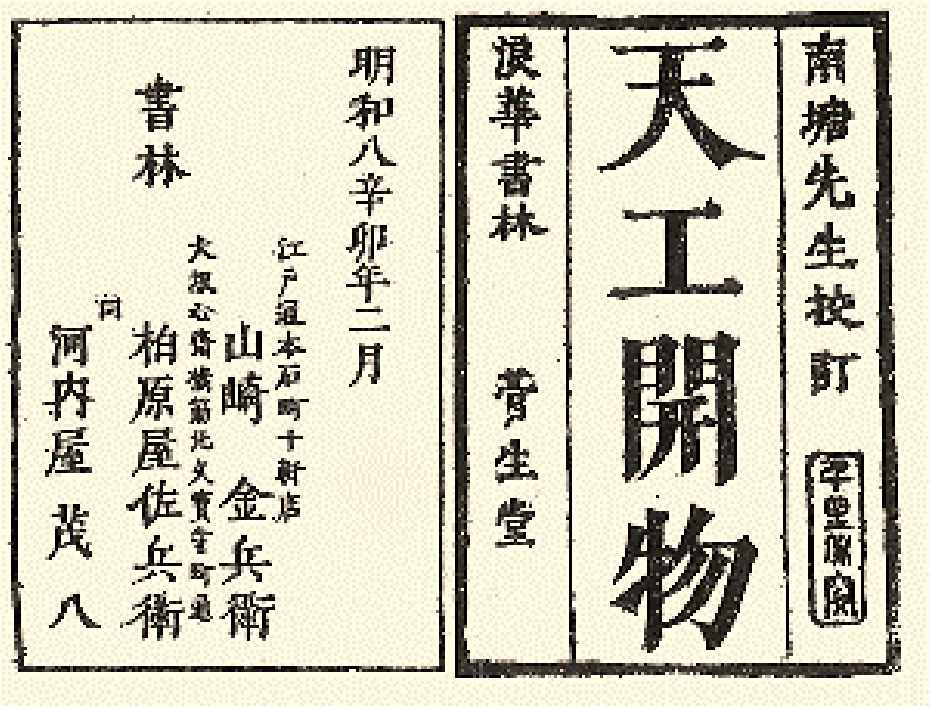


Figure 2.2.3 Cover of Tiangong Kaiwu

2.3 Manufacturing Technology and Principles

In the making procedures of grey brick, the design of kiln is no doubt the key point to be noticed. As is shown in Figure 2.2.1, the construction of kiln is especially designed for grey bricks. Once the clay bricks are properly air dried, they have to be placed into the kiln for firing. Even if the types of kiln vary from place to place in China, they still share a basic design philosophy and prototype. Most are dug into the earth near where the bricks are formed and are faced with rough stones.

Basically, the color of bricks reflects the types of soil from which they are made, spraying water on them while the cooling stage in the closed kiln produces a grey color, which is typical in spotted places in China including Soochow area. In essence, oxidation and

reduction are chemical processes, often deliberately invoked during firing, usually by adjusting the amount of air that is burning the fuel. With plenty of air the kiln atmosphere is described as oxidizing, with the main products being carbon dioxide and water.

Despite granite and quartzite, the clay of Soochow area, actually most natural clays contain iron minerals, either oxides or hydroxides. Iron can exist as ferrous iron in the reduced state Fe (II), in which case it forms dark grey oxides and hydroxides, and this causes some sediments to be dark in color. After firing, however, this iron may be oxidized to the Fe (III) state, or ferric state, which forms the red-brown iron oxide hematite[7].

Nevertheless, no matter in North America or China, the most essential for the architectural ceramic products is the quality of clay. So the characters of the clay are worth studying. As the bricks used in royal palatial places, what features make Wujiang brick outstanding is to be explored.

[7] According to http://www.ucl.ac.uk/earth-sciences/impact/geology/london/ucl/materials/brick

CHAPTER 3: Literature Review

3.1 Durability and Microstructure

The same as red brick, the deterioration of grey brick is mainly due to human effect and natural weathering. Human factors range from the deficiency in the making of materials to the failed treatment. Natural weathering occurs like corrosion owing to acid rain, efflorescence, etc.

To see the definition of durability in building materials: “the quality of being durable; ability to last in spite of frequent use or hard wearing”[8].Durability is to be a criterion for selecting building materials or components. Hence, some quantitative terms are brought in to help in to judge the performance of building materials. General approaches are provided to test durability. The ASTM Recommended Practice for Developing Short- Term Accelerated Tests for Prediction of the Service Life of Building Components and Materials outlines basic involved factors.

There has been a paper[9] touched the correlation between the durability of bricks used in the conservation of historic buildings and their composition and microstructure. Differences in mineralogical and textural evolution during firing of calcareous and non-calcareous bricks are studied and correlated with their behavior in hygric and weathering tests. Results reveal significant differences in the evolution of vitrification degree, porosity and pore size distribution. Such evolution depends mostly on raw clay composition and

[8] Geoffrey Frohnsdorff and L. W. Masters, The Meaning of Durability and Durability Prediction, *Durability of Building Materials and Components*. 1980.

[9] Kerstin Elert, et al, Durability of Bricks Used in the Conservation of Historic Buildings—Influence of Composition and Microstructure, *Journal of Cultural Heritage* 4(2003): 91–99

firing temperatures. A higher degree of vitrification and of compressive strength is displayed by calcareous rather than non-calcareous bricks at lower firing temperatures of between 700 °C and 900 °C.

Through this study of the effect of vitrification to bricks, it can be inferred that in ordinary cases, higher vitrification degree means less porosity and more durability to bricks. However, from the general description of grey bricks, we cannot find any statement about its inferiority originated from its low-fired producing process. Furthermore, it is said to be of better performance in acid/alkaline resistance, compressive strength, and or so. The mentioned properties will be covered in my tests, to see if it is contradicting to this general research as an exception.

Also, their resistance to salt crystallization and freezing is not notably improved by high degree vitrification because of unfavorable pore size distribution and crack development. The latter are caused by the transformation of the calcite in prepared samples for this test into calcium oxide at around 800 °C, which reacts readily with moisture to form calcium hydroxide, thus leading to a volume increase (lime blowing). This problem can be avoided by closely controlling grain size and content of carbonates in the raw clays. High firing temperatures of 1100 °C in the case of calcareous clay and 1000 °C in the case of non-calcareous clay are required to produce durable bricks that remain unaltered upon weathering. The improved durability appears to be due to a more favorable pore size distribution and a reduction in porosity.

Results from textural and hygric studies of the brick samples indicate that these parameters can to a significant extent be controlled by varying raw clay composition and firing temperature, thus making it possible to fabricate replacement bricks for particular conservation purposes. Otherwise, it could not completely deny the potential correlation

between the type of raw clay and firing temperature through this study. As a variable, the composition of raw clay, or possible compounds can have important effect on the effect of vitrification degree, including the porosity and pore distribution.

This paper also addresses limitations regarding the interpretation of test results, as well as the lack of a systematic application of existing standards for evaluating the state of conservation of historic bricks and for establishing specifications for replacement bricks.

As for the testing of the durability of historical material, the accelerated weathering test is applied to mimic the process of natural weathering. Typically, the accelerated weathering test is part of my experimental study of in the thesis. About the parameters/factors of this process, and justification of using weatherometer in the tests need some criteria. For example, the impact of UVA/UVB impact on aging of the material is not the major issue as far as my thesis concerned while the factor of moisture is crucial to the accelerated weathering. Also, the period for weatherometer process is up to 20 days for the samples. For these limitations, some secondary factors will be excluded in the tests. Therefore, salt test and acid resistance test will take the place of weatherometer process in the experimental design.

The article[10] by A.J. Lewry and L. F. E Crewdson can be used as a working document for the subcommittee on 'Design of short term test methods' of the RILEM 140-TSL/CIB W80 on the design life of buildings.

The recommended approach would enable the response of materials to any environment to be modelled, a consequence of which would be more accurate service-life prediction.

[10] A.J. Lewry and L.F.E. Crewdson, Approaches to Test the Durability of Materials Used in the Construction and Maintenance of Buildings, *Construction and Building Materials*. 8(4) (1994): 211–222.

In addition this would provide a better understanding for improving material quality. Critical steps for this approach include:

(i) Consideration of the factors which might affect the durability;

(ii) Accelerated testing to define the most significant factors causing degradation, and the mechanism of that degradation;

(iii) Evaluation of the significant causes of degradation in the proposed end use environment;

(iv) Construction of models and damage functions for the material's response to the environment based on the accelerated test results and the environmental measurements.

3.2 Properties of Chinese Traditional Grey Brick

It was not until 1982 that the central government recognized the identity crisis and launched laws and regulations concerned with urban conservation and regeneration.[11] The grey brick is the key material to Chinese traditional architecture. Bunches of unofficial records and informal citations show the less possibility of efflorescence on Chinese grey bricks. And it is why historical grey bricks are preferred to be used in building city walls, mausoleums and other important historical structure, which can be dated back to Han Dynasty.

In a previous Master thesis[12], of which the author is from Tongji University, Shanghai, the

[11] H. Jia, *Zhongguo Lishi Wenhua Mingcheng Tonglun 中國歷史文化名城通論 [The cultural and historical cities in China]* Southeast University Press, Nanjing. 2007.

[12] Haiao, Zhang. "Primary Research of Shanghai Simple Brick Buildings in Modern and Contemporary Times." Master thesis in History and Theory, Tongji University, 2008.

use of grey bricks is concluded in detail. The simple grey brick buildings in Shanghai, in modern and contemporary times, is the epitome of Shanghai, which can show the change of the time, the progress of technology and the transformation of people's conception. The dissertation concentrates on the basic context and the summary of previous studies the material properties, constructional way and artistic characters. Also, it affirms the significance and unique value of bricks, including commonly used early grey bricks, as a key material to Chinese architecture. These statements can justify the value of the study and prospected research of preserving prototype of historical brick structures. Meanwhile, the dissertation also has discussed about the origin of the material and the influence of architectural style. The first part of this dissertation talks about the history and technology development of brick buildings; this part provide a basic knowledge and prototype of brick as an iconic building material.

3.3 Factors Affect the Quality of the Bricks

In a previous literature of late Qing and the 1980s' grey bricks in Wujiang, Soochow, China, and the isothermal sorption properties were studied[13]. The isothermal sorption curves of grey bricks are one of the basic moisture characteristics for studying the heat transfer of brick building enclosure structures and are also a fundamental parameter of research into the degradation mechanisms of Chinese traditional architecture. In this study, the isothermal sorption curves of *Wujiang* bricks from the 1980s and the late Qing Dynasty

[13] Yonghui Lin, et al, Experimental Study of the Isothermal Sorption Properties of Late Qing and 1980s Grey Bricks in Wujiang, Suzhou, China, *Frontiers of Architectural Research* (2013) 2: 483–487

period have been measured and plotted, by applying constant relative humidity controlled by saturated salt-water solutions under isothermal conditions. Their fitting formulas for humidity bound ranges are presented of 5-92%. The results indicate that samples of the same area traditional Soochow grey bricks from different historic periods show relatively large differences in their isothermal sorption properties, with the isothermal sorption property values of the late Qing grey bricks about three times those of the 1980s grey bricks.

This research paper is regarded to be an informative reference for my proposed thesis. It adds more justification on the selection of the study area, Soochow, Jiangsu Province as the origin place for the grey bricks to be studied while the places of production of grey bricks scatter all over the country. Also, as is stated, Soochow, Jiangsu area is of relatively high yearly precipitation and average temperature. In this climatological context, this paper gives more useful information about the differentiation of historical grey bricks and contemporary grey bricks.

The experimental study is based on comparative analysis and control variable method. The diagrams showing the results of two different types of samples simultaneously is also a fundamental method to present the differentiation in samples of different periods or types. The results of the experiments can be contributive to the research of degradation mechanism of moisture analyses of grey bricks; they can also be referred for the relevant studies.

Contemporary manufacture of brick and other architectural ceramics follows the traditional procedures used for thousands of years and passed down to decedents as a form of

cultural heritage. For clay bricks, the clay was selected and kneaded with water, worked into the desired shape and left to be dry before being fired in a designated kiln.

Although the type of clay has much to do with the finished pieces of ceramics. The firing temperature and other factors are still very important. Physical and chemical properties of the bricks will be greatly affected[14]. The aim of this paper is to compare the physical changes that take place when ceramic samples molded and fired in the laboratory are treated with different treatment products. It is an investigation seeking to evaluate to what extent these product improve the quality of ceramics and suggest possible new solutions for the conservation of the ceramics used in our architectural heritage.

Also, the conclusion drawn out from a set of experiments are supportive to my proposed study, such as:

- No evidence of vitrification was observed in ceramic samples fired at 900°C. Few mineralogical change were detected: these included the disappearance of kaolinite and microcline and the crystallization of mullite and sanidine.
- The application of any of the clay bricks being tested led to changes in the pore system and a reduction in open porosity.
- The application of laboratory firing process led to a change in the color of the sample.

  These conclusions are to be reference to the proposed study.

[14] Giuseppe Cultrone, Evaluation of the Effectiveness of Treatment Products in Improving the Quality of Ceramics Used in New and Historical Buildings, J*ournal of Cultural Heritage.* 14(4)(2013): 304–310

From the viewpoint of petrology, the microstructure as well as mineralogy can be distributive to the performance and quality of the bricks. The article[15] reviewed in this scope is mainly about the mineralogy and phycio-chemical properties of a certain type of clay. Moreover, the physical and chemical properties allow it to be an ideal raw material of fired earth and fine ceramic product.

Alluvial clays from four localities along the Sanaga River (Center Cameroon) were studied by physico-chemical, mineralogical and technological characterization in order to assess their suitability as ceramic raw materials. The chemical compositions indicated that SiO_2 (65–70%) and Al_2O_3 (12–15%) are major elements while Fe_2O_3 is less (4–7%). Kaolinite, quartz and feldspar are the main minerals. These results were from XRD (X-Ray Powder Diffraction). Crystalline structure and chemical composition in this way are subjective to be fully studied with the help of this technique. Also the particle distribution (clay~silt~sand), together with liquid limit, plastic limit and plastic index, as a vital physical property was also tested. Both these chemical and physical tested results were expressed in a percentage.

Particle size distribution and chemical composition are indicative of “plastic red clays” belonging to heavy sandy clays group. Their medium to high plasticity is suitable for fired earth and fine ceramics products. Pressed samples were fired at temperatures ranging between 900 and 1100 °C for coarse ceramic products. Linear shrinkage, flexural strength and water absorption indicated that the clays from one site (Mbandjock) are good for brick making. Clays from the three other localities present poor technological properties (higher shrinkage and cracks), they need degreasers before use as ceramic raw materials.

[15] Nzeugang Nzeukoua, et al, Mineralogy and Physico-chemical Properties of Alluvial Clay from Sanaga Valley (Center, Cameroon): Suitability for Ceramic Application. *Applied Clay Science*. 83–84(2013): 238–243

Although water absorption and flexural strength parameters are good for all the studied samples, firing shrinkage needs to be improved.

It is noticeable that SEM (Scanning Electronic Microscopy) is applied in a stage of the research of this case. It shows stacks of pseudo-hexagonal platelets for kaolinite in alluvial clays. According to the previous conclusions on the morphology and classification, the mineral can be defined as poorly crystallized kaolinite.

As for the results of this study, Semi-quantitative mineralogical analysis results of the clay fraction confirmed that quartz and kaolinite are the major minerals present with significant amounts of smectite and illite.

The presence of quartz, kaolinite, illite, feldspar and smectite associated with the physico-chemical properties of all the studied samples, categorized as “plastic red clay”, is generally suitable for various red ceramic bodies. Characterization of some specimens firing at 900 °C, 1000 °C and 1100 °C in order to make structural ceramic for building construction helped to know that water absorption and flexural strength parameters fell within the usual industrial required values for fired bricks (BS 3921-1985, China.). This may give a reference about the standardization of the chemical and physical properties for ceramic production as building material.

3.4 Ceramic Artifacts Deterioration

The use of clay bricks can be dated back to over two thousand years as artwork and cultural heritage. For example, the Han Dynasty bricks used have been buried underground and deteriorated by the humid environment in the cemetery, as well as

interfering factors as groundwater and chemical constituents of soil. Unavoidable, the bricks have been seriously damaged in essence when they are brought to light.

In a Master thesis of Applied Chemistry, the degradation mechanism and protection of these bricks were discussed[16]. Study from the microscopic to learn the degradation mechanism, and made a reasonable measure of protection is the aim of this thesis. X-Ray Powder Refraction (XRD), Scanning Electron Microscope (SEM), energy spectrum analysis methods had been used to analyze the Han Dynasty brick's microstructure and mineralogical properties.

Some Chinese clay brick standardization measures are used to analyze the hydraulics, anti-salt capability, anti-freeze capability, and contrasted with the modern bricks performance. The mechanism of damage from the microcosmic of the bricks is studied. The experiments results show that, the Han Dynasty brick after more than two thousand years burial experience, it was destroyed by the humid environment in the cemetery, micro-organisms and some other factors. On the surface of the brick there is much gypsum, this is the most important reason of the brick's degradation. In addition the destruction of micro-organisms and the migration of soluble salts within the pore size, made the water absorption increase, its resistance, salt resistance, anti-freeze turned to more poor than before.

This dissertation has discussed the basic experimental methods and analytical techniques involved in the investigation of the properties of clay brick. While it focuses on the degradation mechanism of Han Dynasty bricks of a history of over two thousand years, the study of historical grey bricks would be more relevant to the comparative analysis of

[16] Xiaoli, Zhu. "The Degradation Mechanism and Protection of the Han Dynasty Brick". Master dissertation in Applied Chemistry. Zhengzhou University. 2009

the effect of firing process, vitrification degree, and the elemental composition and relevant properties of designated types of grey and red bricks as contemporary building materials.

It is also necessary to look after the alteration of the mechanical properties of brick masonry due to aging. The variation of the mechanical characteristics of brick and masonry: strength and deformability has been measured through compression tests carried out on altered and unaltered prisms. In the test, the influence of absorption characteristics of brick and adhesion of mortar has been taken into account. Bond strength tests and measurement of dimensional variation of brick and mortar have been accomplished in the research paper published on 7th International Brick Masonry Conference, 1985[17].

It is natural that the efflorescence phenomenon is prevalent in coastal region due to the ubiquity of sodium chloride. In other regions calcium, sodium and/or magnesium sulfate can be present in the soil or generated by chemical attack due to atmospheric pollution. The authors of this paper had a previous study about such situation in northern Italy. The durability of bricks did have a high influence on resistance to aggressive agents. Also, some hygric properties of material have to be taken into account.

One aim of this paper was to point out the importance of choice of mortars to the durability of brick-masonry works. Also the mechanical characteristics of brick and masonry would be affected by the phenomenon of salt crystallization. The influence of porosity of bricks are studied. It indicates that highly absorbent bricks, even when joined with high strength

[17] L. Binda and G. Baronio. "Alteration of the Mechanical Properties of Masonry Prisms due to Aging", In Proceeding of the 7th International Brick Masonry Conference. Melbourne, Australia, 605-616. Feb, 1985.

and rigid mortars will allow water to penetrate into the structure. It will cause a deep alteration in bricks.

It has some comparability in the producing area of samples concerned in this study and the ones in mine. Efflorescence has more chance to happen to the buildings nearby coastal area. Soochow, Jiangsu area is also coastal area adjacent to Taihu Lake (太湖). Despite of the interference of mortar selection, the brick with high absorption will also be subjected to cycles of crystallization and altered in mechanical characteristics. This alteration can be an important indicator of weathering and degradation.

3.5 Non-Destructive Porosimetry for Ceramics

From a 2004 journal article[18], a latest method to test porosity was carried out. It is non-destructive, efficient and precise. In the field of building engineering, it is essential to choose building envelope material and to optimize its hydrothermal performance[19]. The various strength parameters (compression, flexure, impact, shear, tension) of materials can be adversely affected by moisture[20]. The porosimetry of architectural ceramics is also an important issue within the study of the basic properties of building bricks.

The archaeologists have borrowed well-established methods from the material science for their study while these methods can have drawbacks for archaeological applications

[18] Karen G. Harry, Allen Johnson. A Non-destructive Technique for Measuring Ceramic Porosity Using Liquid Nitrogen, *Journal of Archaeological Science* 31 (2004):1567-1575

[19] O.F Osanyintola and C.J Simonson. Moisture Buffering Capacity of Hygrosopic Building Materials: Experimental Facilities and Energy Impact. *Energy Build* 38 (10) (2006): 1270-1282.

[20] Heinz R. Trechsel (ed) Manual on moisture control in buildings. ASTM manual series: MNL 18. Library of Congress. 1994

including the potential to damage archaeological residues contained within the sherd, the need for expensive equipment, and/or the use of hazardous materials.

The non-destructive method introduced in the paper for measuring porosity based on the infusion of liquid nitrogen into the sherd. A comparison of results obtained from this method with those obtained from other approaches indicates that the technique produces reliable reference.

The rationality and fundamental of this method are straightforward. If liquid nitrogen, which will turn to gas state in room temperature, can be fully absorbed by the samples to be tested, the porosity of the sample can be measured through calculating the change of the weight. The gasified nitrogen can be quantitated in this way. The density of liquid nitrogen is known, the volume of the porosity can be obtained through simple calculation.

Referring to ASTM C830[21], the liquid porosimetry test is based on the liquid immersion techniques. This method has been applied to the art conservation and museum since it was carried out and the accuracy was confirmed.

3.6 Tung Oil and Repair of Ancient Architecture

There was a paper[22] focusing on the characteristics of Tung oil and investigating the use of Tung oil in the repair of ancient architecture. Tung oil is a drying oil obtained by pressing the seed from the nut of the Tung tree (Vernicia fordii). The color of the liquid is dark

[21] American Society for Testing and Materials, C830-00: Standard Test Methods for Apparent Porosity, Liquid Absorption, Apparent Specific Gravity and Bulk Density of Refractory Shapes by Vacuum Pressure, ASTM Annual Book of Standards, Volume 15.01: Refractories; Activated Carbon; Advanced Ceramics (2002)
[22] Ibid, 27-31.

golden yellow, thick and sticky. The relative density is 0.936-0.940 (20° C). The liquid is neutral (pH=7).

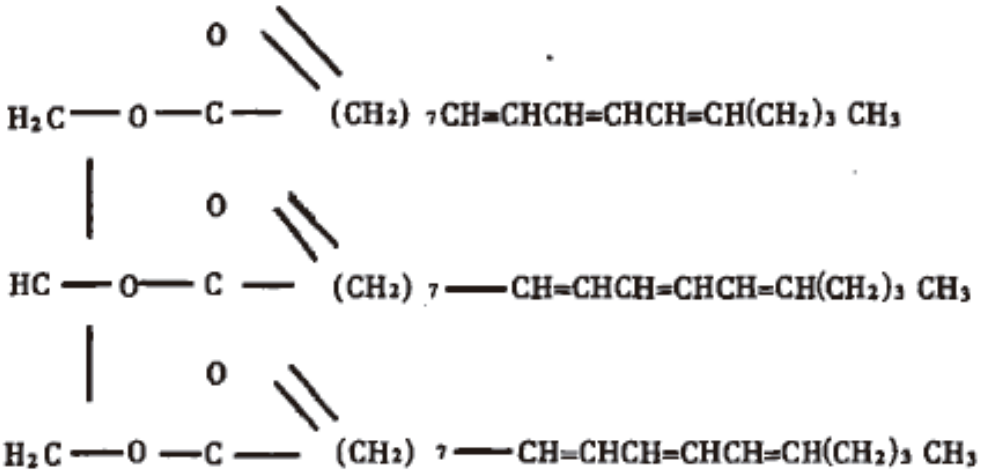


Figure 3.6.1 Chemical structural formula of Tung oil[23]

Since the Tung oil tend to be uneven as a coating on architectural ceramics like floor tiles, even having peeling layer, which looks indecent. So the researchers attempted to make the Tung oil diluted, reducing the viscosity. In this way, the penetration of liquid would be improved.

In the experiment on grey brick treatment, the procedure is as follows:

- Dilute the Tung oil with ethyl acetate at a ratio of 1:1.
- Flatten the surface of brick and polished

[23] Guangquan Zhen and Chanjuan Li. Modified Tung oil soak experiment in the repair of tiled floor in ancient architecute. Wenwu Baohu Yu Kaogu Kexue, 文物保护与科学 7(1) (2005):27-31. Shanghai Shi Wenwu Guanli Xiehui, 上海市文物管理协会 Shanghai, China.

- Apply the 50% Tung oil onto the surface, repeated several times

All the treated samples went to a water absorption test which lasted for 28 days. In this test, there was also a control group of untreated grey bricks so as to compare the effect of Tung oil treatment.

The original dry grey brick is 815 g in weight, and 820 g after Tung oil treatment.

The average of control group (untreated samples) is 22.2%, which shows a dramatic difference on data. We can draw a conclusion that Tung oil treatment can greatly impact the water penetration upon the grey bricks.

Table 3.6.1 Grey brick water absorption results[24]

	24-Hour Immersion	48-Hour Immersion	3-day Immersion	7-day Immersion
Weight (g)	825	830	840	865
Water absorbed (g)	5	10	20	45
Absorption rate (%)	0.6	1.2	2.4	5.5

[24] The experimental results in this Chapter are from the paper published in 2005 article by Zhen and Li. Ibid

And the researchers also did a boiling test on different types of grey bricks, which provides a useful and reliable source for the quality of the grey bricks. Five types were tested: old grey bricks, contemporary grey bricks from *Hebei* Province (*河北*), contemporary grey bricks from Soochow, Jiangsu Province, weathered old bricks, golden brick (金磚, treated by Tung oil).

Table 3.6.2 Five types of grey bricks water absorption results

	Old grey brick	Contemporary grey brick	Hebei grey brick	Weathered old brick	Golden brick
24-hour water absorption (%)	16.9	20.1	15.1	16.6	12.6
5-hour water boiled water absorption (%)	22.7	25.2	16.6	22.4	14.1

From the table above, we can see the golden bricks have a significant advantage over the other four types of grey bricks. Moreover, through the comparison, we can confirm the effect of Tung oil in promoting the durability and water resistance in the repair of grey bricks as architectural ceramics.

CHAPTER 4: Samples and Preliminary Testing

4.1 Samples and Sample Preparation

Type I- Historical grey brick, of early 20th century, Soochow, Jiangsu Province, China. (HG). These samples are obtained from the collection of historical stock products of Kan family workshop in Soochow. They are dated back to the early 20th century.

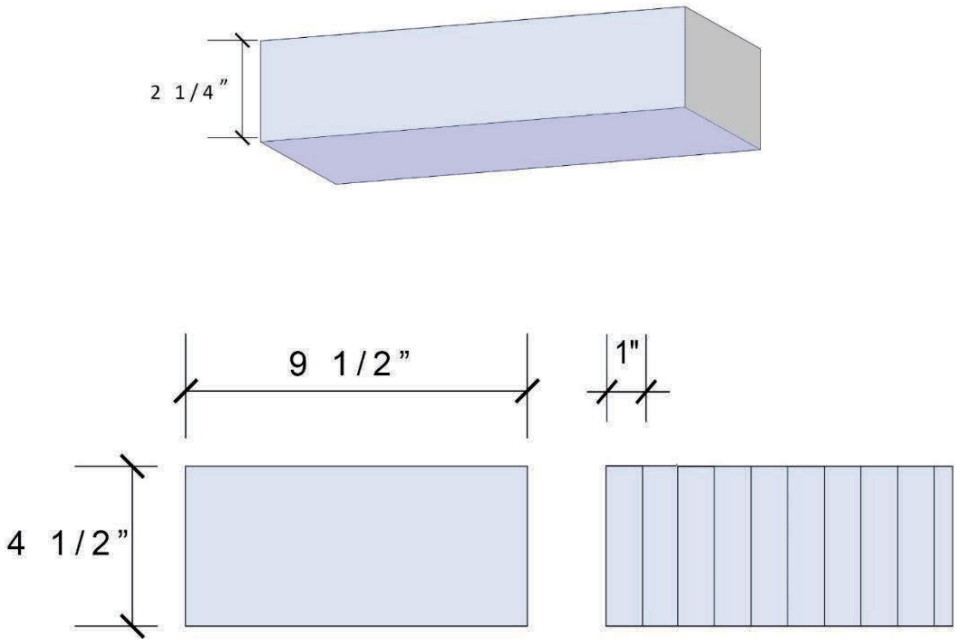


Figure 4.1.1 Schematic diagram of the cutting of the HG samples- drawn in AutoCAD

The system of naming is HG.01 (/02/03…).T (N), among which, 01, 02, 03… is standing for the sequential order of the whole brick. For example, 01 is for the piece consisting the

face of “head”. “T” is marked for Tung oil soaking process. “N” is non-Tung oil treatment group.

Type II- Contemporary grey brick, provided by the Kan (闞) family brick workshop, Soochow, Jiangsu Province, China. (CG). This type is currently a regular product of the workshop. This group of samples are named as CG.01 (/02/03…). T (N).

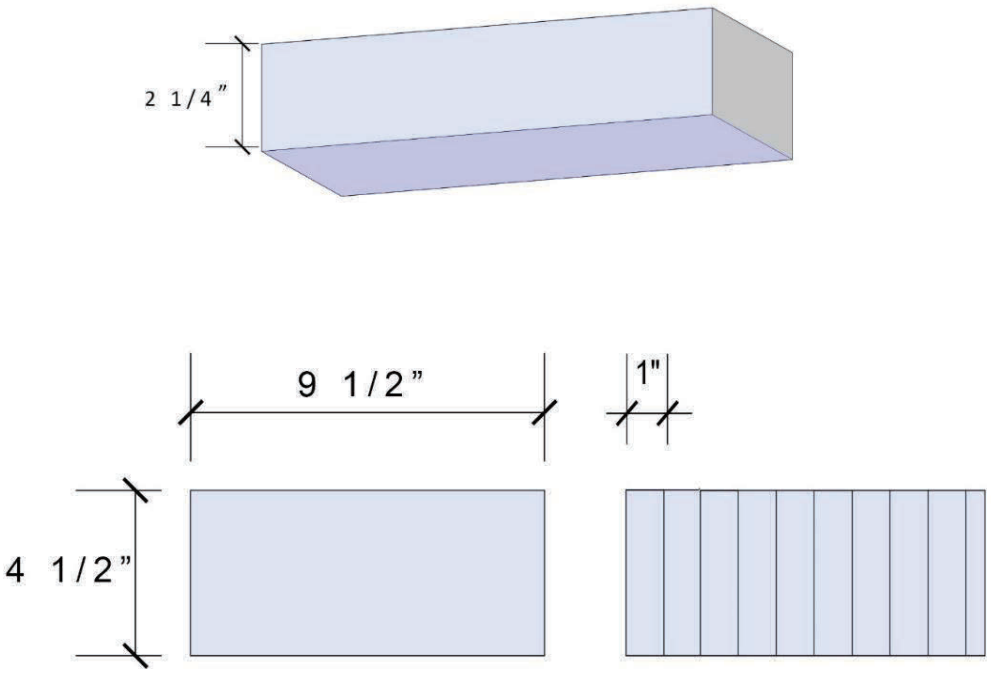


Figure 4.1.2 Schematic diagram of the cutting of the CG-samples- drawn in AutoCAD

Type III- Contemporary red brick, taken from a demolished bungalow dwelling of late 1980s, Soochow, Jiangsu Province, China. (CR). This group of samples are named as CR.01 (/02/03…). T (N).

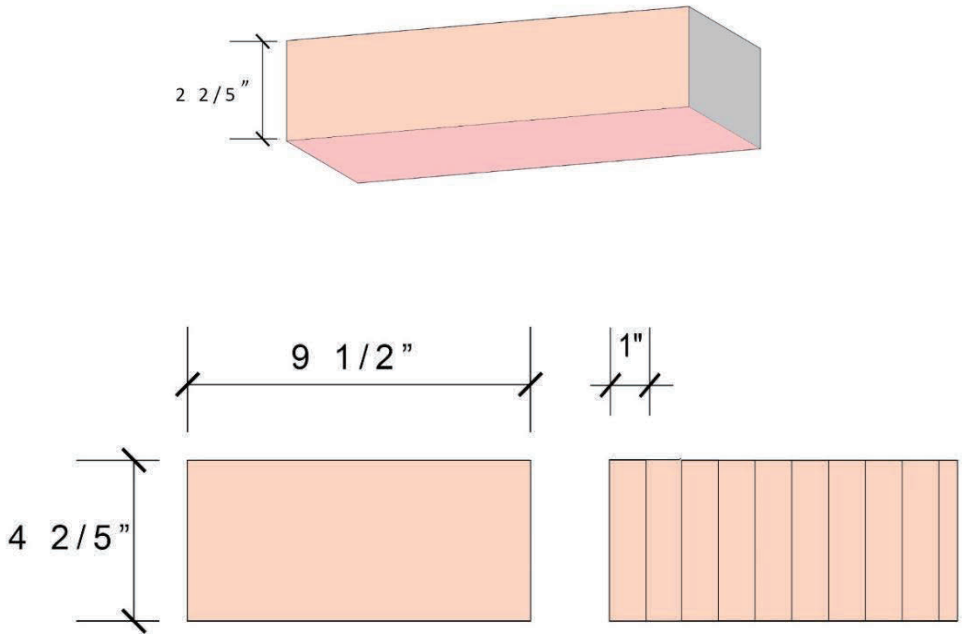


Figure 4.1.3 Schematic diagram of the cutting of the CR-samples- drawn in AutoCAD

A Felker diamond blade stone saw was used in the Fabrication Laboratory at the School of Design of the University of Pennsylvania. The manager of the Fabrication Laboratory, Dennis Pierattini helped with the operation of the sample cutting process.

Additionally, the hardness of three types of samples were tested through Mohs hardness test. The historic grey brick is 6; the contemporary grey brick is 7; the contemporary red brick is 4.

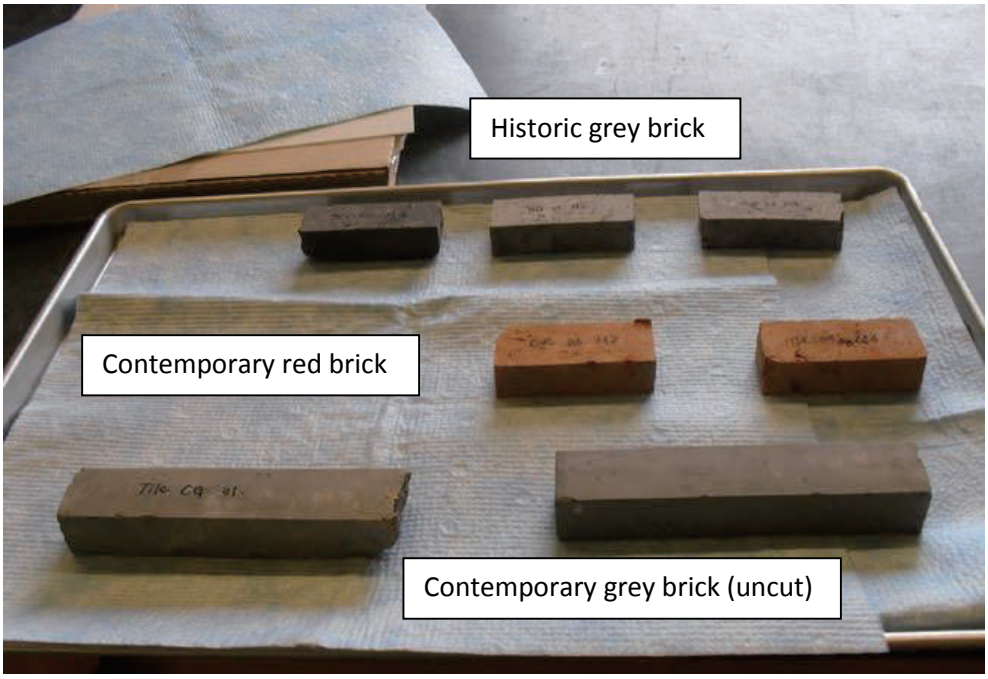


Figure 4.1.4 Three types of the samples

4.2 Liquid Nitrogen Porosimetry

This experimental test was applied to measure the original porosity of the three types of samples.

Figure 4.2.1 The experimental setup of liquid nitrogen porosimetry test[25]

[25] This experimental set was first used in Irene Matteini's thesis: AN ASSESSMENT AND EVALUATION OF ACIDIC CLEANING METHODS ON UNGLAZED TERRACOTTA USING ACCELERATED WEATHERING TEST PROTOCOLS. University of Pennsylvania

According to the article mentioned in Chapter 3.5, liquid nitrogen was used to measure porosity. This method is based on the liquid immersion technique ASTM C830 77 and proved useful as applied to cultural material (ceramic sherds) at the University of Las Vegas[26].

The percentage porosity can be calculated as follows: $P = \frac{W-D}{W-S} \times 100\%$

P is percent porosity. **W** is the weight of the saturated sample in grams. **D** is the weight of the dry sample in grams. **S** is the weight of saturated sample suspended in liquid nitrogen.

HG Group

Table 4.2.1 HG group liquid nitrogen porosity test results

SAMPLE	**D (g)**	**W (g)**	**S (g)**	**P%**
HG.01.N	212.62	243.73	142.99	25.74
HG.02.N	194.57	223.01	98.77	22.89
HG.03.N	202.38	227.21	119.87	23.13
HG.04.N	194.87	223.64	106.32	24.52
$\overline{P}$ %=24.07%		Standard deviation: 0.01325		

[26] American Society for Testing and Materials, C830-00: standard test methods for apparent porosity, liquid absorption, apparent specific gravity and bulk density of refractory shapes by vacuum pressure, ASTM Annual Book of Standards, Volume 15.01: Refractories; Activated Carbon; Advanced Ceramics (2002)

CG Group

Table 4.2.2 CG group liquid nitrogen porosity test results

SAMPLE	D (g)	W (g)	S (g)	P%
CG.01.N	56.82	61.40	30.12	14.64
CG.02.N	52.39	56.86	26.09	14.53
CG.03.N	61.01	66.82	31.01	16.22
CG.04.N	59.45	63.97	34.12	15.31
$\overline{P}$ %=15.13%		Standard deviation: 0.0078		

CR Group

Table 4.2.3 CR group liquid nitrogen porosity test results

SAMPLE	D (g)	W (g)	S (g)	P%
CR.01.N	171.26	199.70	92.26	26.47
CR.02.N	166.10	192.06	94.40	26.58
CR.03.N	157.00	186.78	91.45	31.24
CR.04.N	159.84	187.20	88.28	27.66
$\overline{P}$ %=27.99%		Standard deviation: 0.02234		

From the experiments and set of data, several observations were obtained:

- There are porosity difference in between grey bricks and red bricks, however, it is not totally relevant to the age of the materials.
- As the samples of CG group are much lighter than the other two groups, it should have been more likely to gain deviation for the same error factors in the real operation. But, from the data and the calculated standard deviation we can see the results are just the opposite. That is to say, the experimental data may be more reliable on less porous samples.
- The general data reflect that cumulated tests can slightly decrease the porosity results.

4.3 Electronic Scanning Microscopy Observations

The series of test and examination were done at Singh Center for Nanotechnology at University of Pennsylvania. The type of the electronic scanning microscope was FEI Quanta 600. The Quanta line of scanning electron microscopes are versatile, high-performance instruments with three modes (high vacuum, low vacuum and ESEM) to accommodate the widest range of samples of any SEM system[27].

[27] Refer to the website of the product: http://www.fei.com/products/sem/quanta-sem/

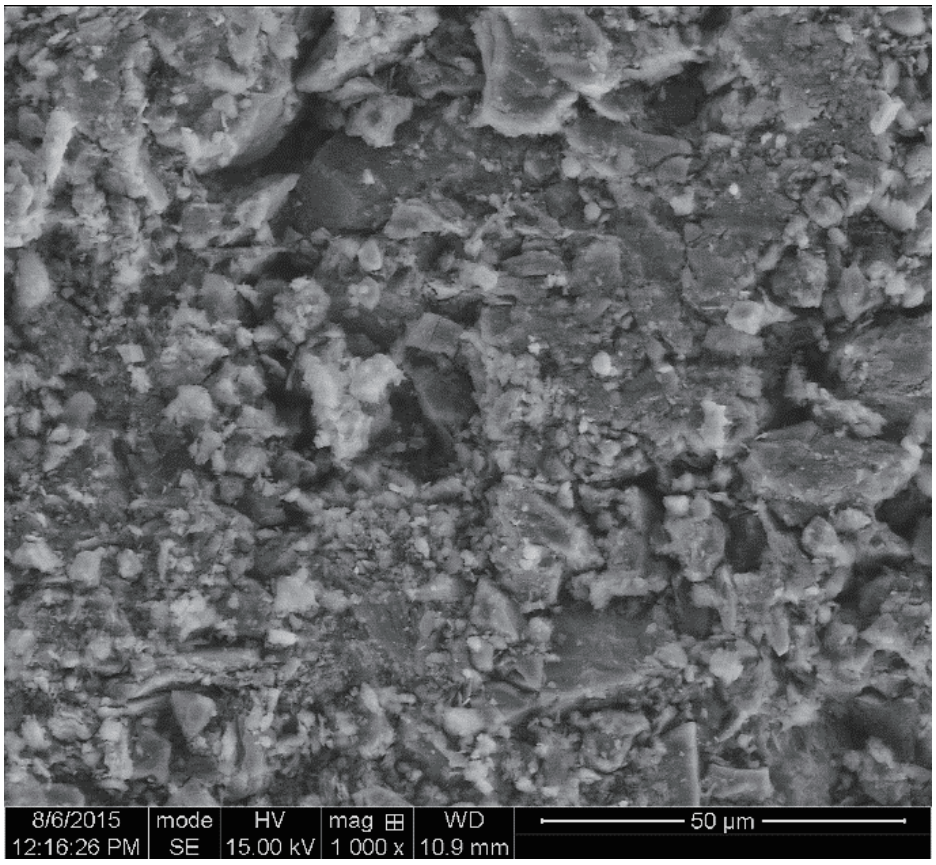


Figure 4.3.1 The SEM micrograph of an untreated HG sample

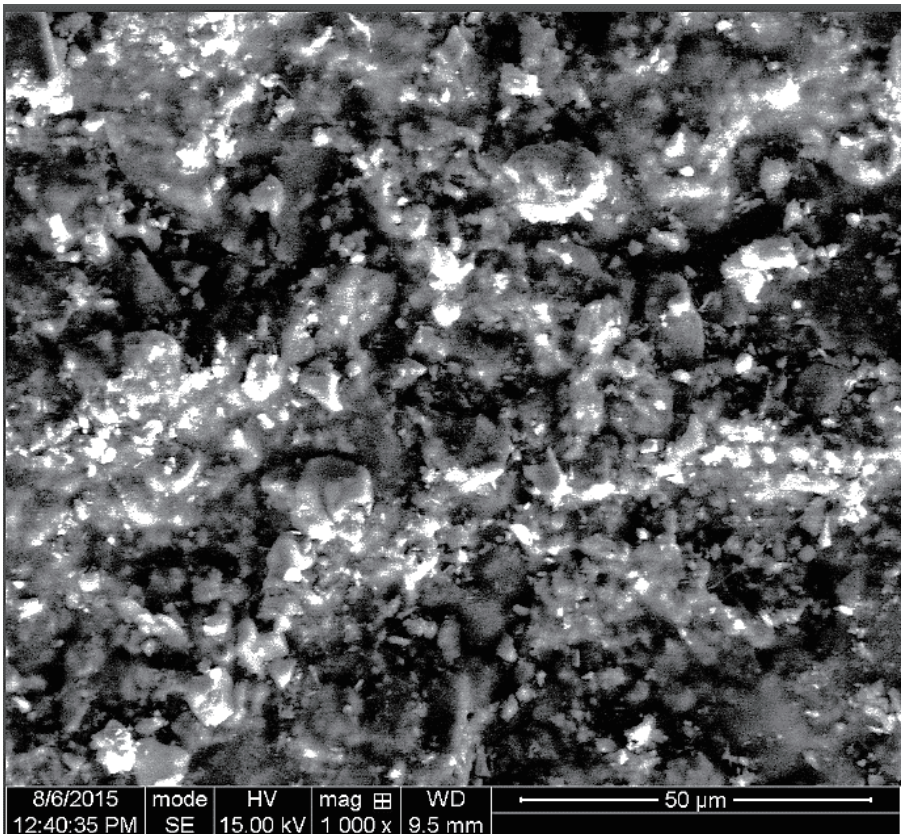


Figure 4.3.2 The SEM micrograph of an untreated CG sample

The grey brick series are more homogeneous and smoother. The contemporary grey brick looks more compressed, with finer particles. Otherwise, the morphology of the contemporary red brick surface looks more rugged and rough, showing a more complicated microstructure and composition.

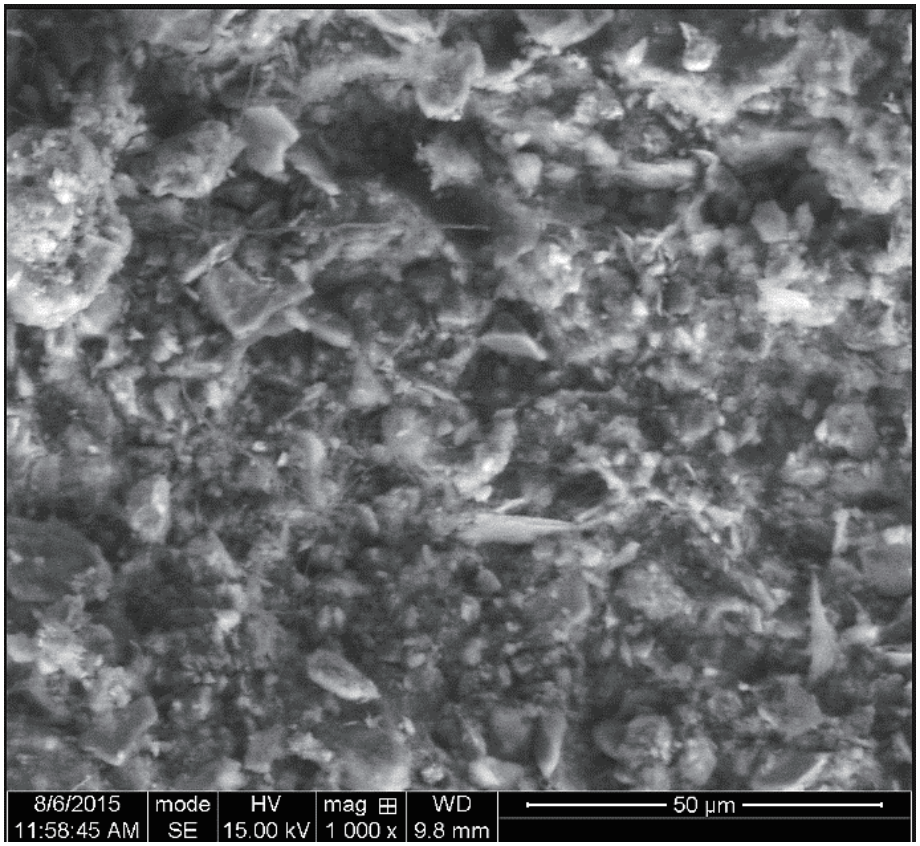


Figure 4.3.3 The SEM micrograph of an untreated CR sample

CHAPTER 5: Petrographic Study of Three Types of Chinese Clay Bricks

5.1 Introduction

The grey bricks do not show inferiority in the durability and performance though in different color and firing temperature. It may be the difference in the porosity and microstructure. In fact, not only the porosity but also the pore distribution and connectivity will affect the strength of the material. The mineral composition and clay type is essential as the contributors to the property and the quality. Hence, petrographic study is a valuable forensic tool in knowing the characteristics of architectural ceramics.

Mineralogy is the systematic study that extensively covers description, crystallography, physical, chemical and environmental features of all minerals. The internal structure and crystal system of individual minerals are unique and form under certain natural, physical and in a narrow range of chemical conditions. It normally occurs as group of associated minerals such as chromium, osmiridium, platinum and nickel. The physical properties like color, streak, luster, hardness, cleavage, fracture and brittleness are descriptive aspects and help in identification. Minerals are classified under main their forming groups and chemistries, including autochthonous elements, sulfides, oxides and hydroxides, carbonates, halides, sulfates, phosphates and silicates with subclasses and other kind of rock forming groups. All major minerals have been described in this chapter in detail, as individual and group, illustrating standard physical and chemical properties and crystal system.

The essential work principle and the properties of classified minerals are in an introductive and structural organization. It could be a general guidance and reference chapter during the experimental study of mineralogy and petrography of samples[28].

5.2 Sampling

- All the samples are from Soochow, Jiangsu Province, China. The grey bricks, HG (Historic Grey Bricks) and CG (Contemporary Grey Bricks) are provided by Kan (闞) workshop, the local brick-maker family in Soochow. HG were made in early 20th Century while CG were recently made grey bricks.
- The red brick CR (Contemporary Red Bricks) were from demolished traditional bungalow dwellings of lower middle class built in 1980-1985, in Soochow city.

Dennis Pierattini, the manager of PennDesign Fablab helped to cut the brick flakes. Felker diamond blade wet stone saw was used. Dr. Marie-Claude Boileau got them made into thin section for further petrographic study. The blue colored poxy was used in making the thin sections, which helped the identification of voids and white transparent minerals/ inclusions.

[28] S.K. Haldar, *Chapter 2 – Basic Mineralogy*, Introduction to Mineralogy and Petrology, Elsevier 2014: 39–79

5.3 Geological Scope of the Study Area

Soochow city, Jiangsu province is regarded as the paradise city in China, in the South-east of China, adjacent to Taihu Lake. Founded in 514 B.C., Soochow city is almost the oldest towns in Yangtze Basin, even the richest area in the Basin.

Figure. 5.3.1 Location map of Suzhou (Soochow) City in China

The geology of the clay products produced in Soochow is mainly characterized by Soochow granite, micrite, and dioritic porphyries. Also, there is coal deposits because The *Renlou* (*任楼*) coal mine is located South-west to Soochow city. In this area, unconsolidated materials, about 3000 meters thick, of alternating marine and continental facies were deposited on the bedrock during the Quaternary Period. The deformed

Neoproterozoic to late Paleozoic strata in the region constitute a *Xu (zhou)-Huai (nan)* (*徐州-淮南*) structural nappe[29].

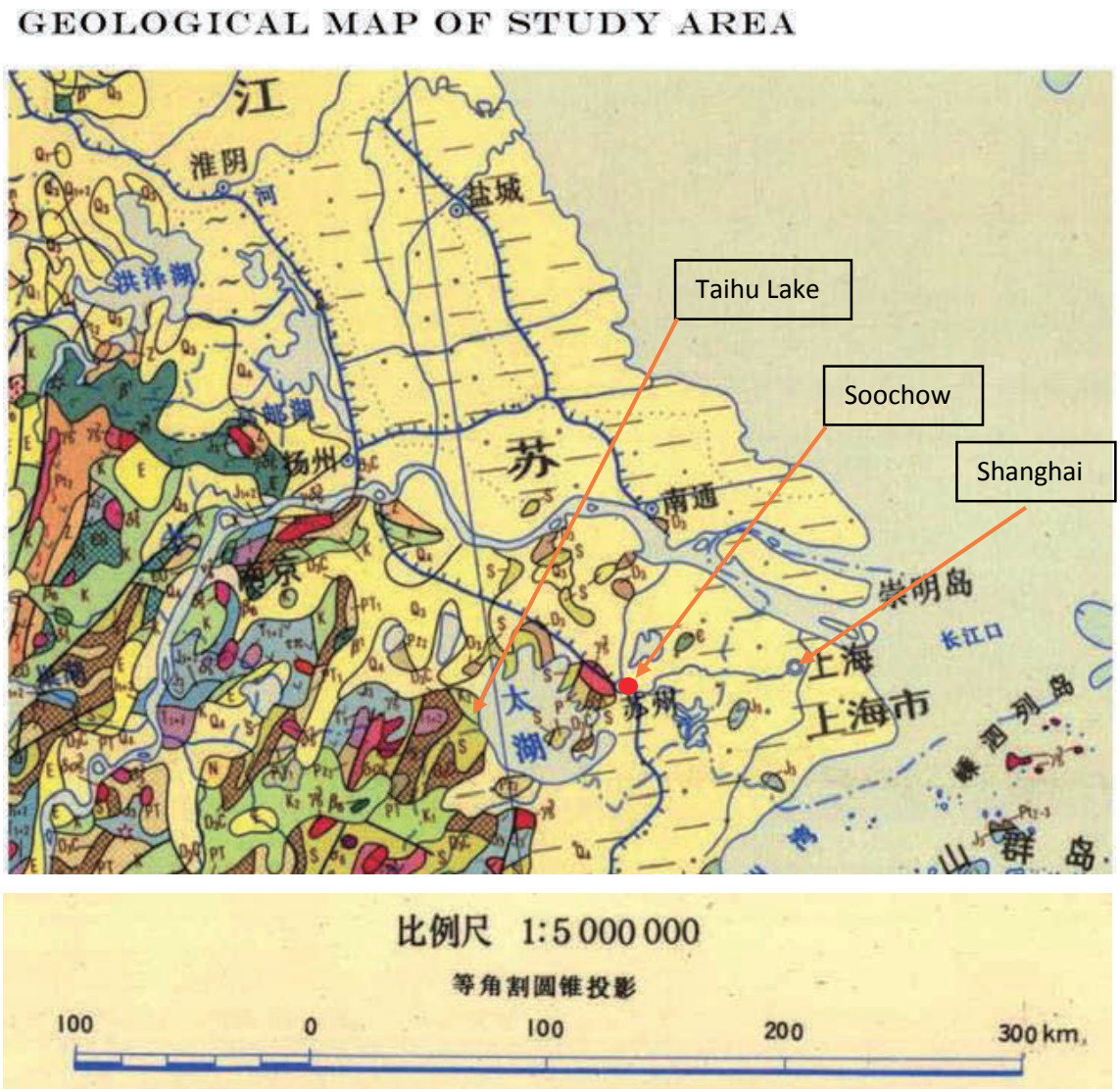


Figure. 5.3.2 Geological Map of Study Area[30]

[29] G Wang,et al, 1998. On the Xuzhou-Suzhou arcuate duplex-imbricate fan thrust system. Acta Geologica Sinica 72, 228–236 (in Chinese with English abstract).

[30] Cited from the map: Department of Geology and Mineral Resources, People's Republic of China, Geological Map of China [map], 1:5 000 000. 1990

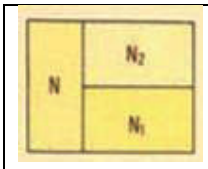
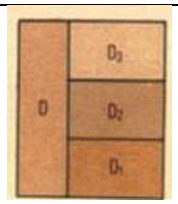
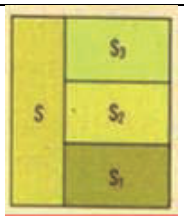
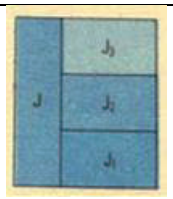
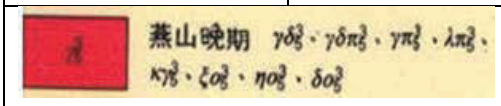
 The Upper Tertiary	 Devonian	 Silurian	 Jurassic	 Cambrian
 Intrusive rocks, granite		 Marine debris and clay		

Table. 5.3.1 Legend of Geological Map

The dominant geologic symbols shown in the legend are representing for Alluvium, Loess, Sandstone Shale Limestone and Coal, Diorite and Granite, Laterite...

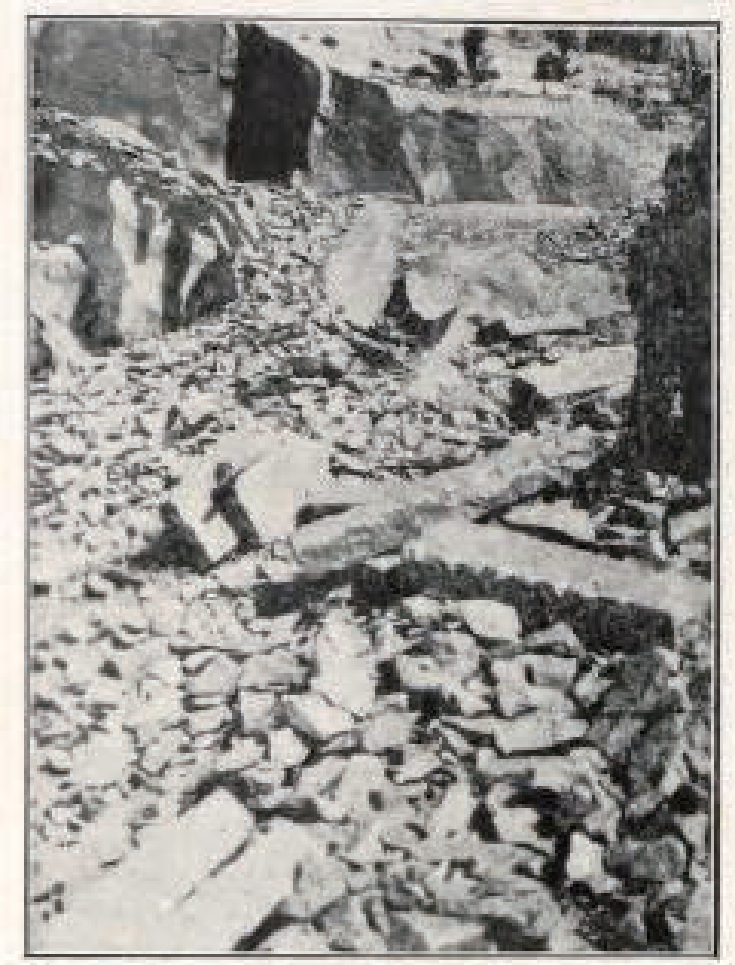


Figure 5.3.3 Granite quarry of Chinshan, Soochow

5.4 Petrographic Results

<u>Fabric GROUP 1:</u> Very fine-grained dark caesious fabric with quartz-dominant inclusions

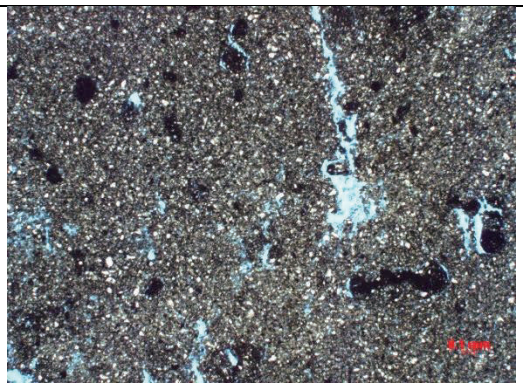
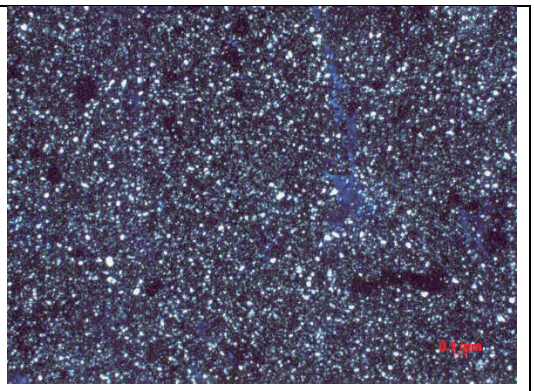
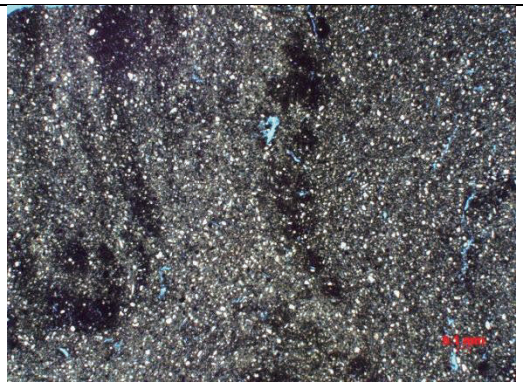
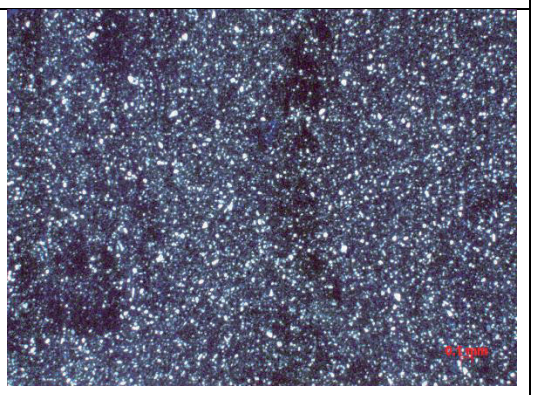
HG Left: PPL, 25X Right: XPL, 25X		
CG Left: PPL, 25X Right: XPL, 25X		

Figure 5.4.1 Optical microscope photographs of Fabric Group-1

HG and CG

<u>Fabric GROUP 1:</u> Very fine-grained dark caesious fabric with quartz-dominant inclusions

Microstructure

Silt size monocrystalline quartz inclusions are frequent in the very fine dark caesious fabric. The porosity is low. The quartz-feldspar dominant mineral composition fit with the local geology for the abundance of granite/granodiorite.

Groundmass

It is homogeneous with fine inclusions set in a very fine groundmass. It is optically inactive. The dark caesioius color (in PPL, X25; even darker in XPL, X25) suggests the reducing atmosphere of the production. (Color of Fe (ii))

Inclusions

C: f: v0.125mm= ca 5: 90: 5 to 3: 90: 7

Measurements of the inclusions in sample HG (in CG which is similar):

The average particle size is 0.022mm<0.0625mm which is silt size. And it can be seen from the measurements that inclusions are well sorted, sa-sr.

Fine Fraction (<0.125mm)

Predominant	Micrite (clay size, matrix)
Frequent	Monocrystalline quartz
Very Few	Plagioclase feldspar
	Muscovite
	Opaque (iron oxides, mottled in the groundmass)
Very Rare	Amphibole (? slight pleochroism observed, high relief)
	Epidote (? Pale yellow in PPL, 2nd or 3rd order colored blue in XPL, high relief)

Coarse Fraction (>0.125mm)

Almost absent.

Concentration Feature

There are a few dark (opaque) clay pellets of “amorphous” concentration feature (mottled opaques, Fe-oxide nodules are also probable.), with sharp to merging boundaries, (Whitbread; 1986, 80). <2.4mm, mode 0.7mm sr-r. ; And depletion features around voids.

In CG thin section, besides the clay pellets, there are visible black streaks owing to the mixing of clay.

<u>Fabric GROUP 2:</u> Very fine-grained reddish brown micrite fabric with quartz-dominant inclusions

Groundmass

The groundmass is in reddish brown in both PPL (X25) and XPL (darker in XPL, X25), optically inactive. It is in an uneven color and streak-like texture which may be caused by the mixing of clay or pressing process of the brick. It is heterogeneous with poorly sorted inclusions set in a very fine and even groundmass.

Inclusions

C: f: v0.125mm= ca 10: 78: 12

In coarse fraction, the inclusions are poorly sorted within few multi-chamber microfossils. < 2.4mm, of various shapes.

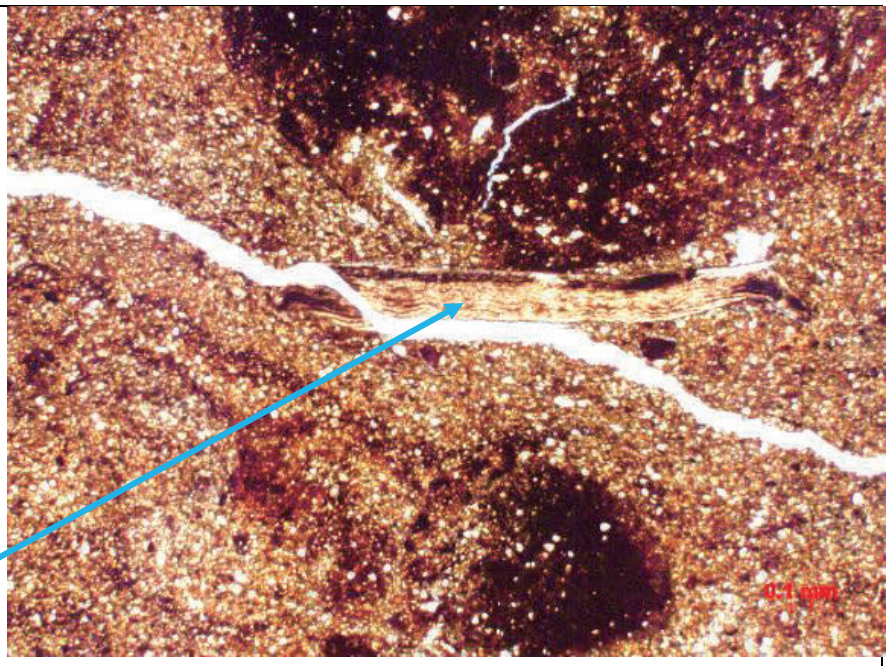
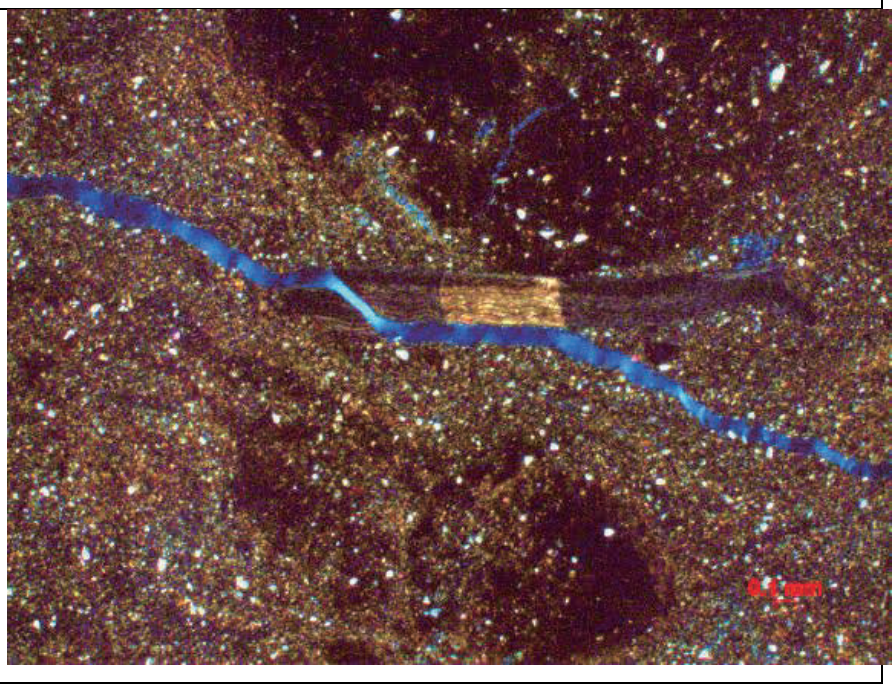
CR PPL, 25X Calcium carbonate	
XPL, 25X	

Figure 5.4.2 Optical microscope photographs of Fabric Group-2

In coarse fraction, the inclusions are poorly sorted within few multi-chamber microfossils.

< 2.4mm, of various shapes.

In fine fraction, the inclusions are well sorted, the average size of inclusions are 0.03mm, sr-r.

Fine Fraction (<0.125mm)

Dominant	Micrite (clay size, matrix)
Frequent	Monocrystalline quartz
Few	Plagioclase feldspar
Very Few	Orthoclase
	Opaques (iron oxides)
Rare	Muscovite
	Microfossils,foraminiferar

Coarse Fraction (>0.125mm)

Very Few	Calcium carbonate

1) Calcium carbonate (algae-like), <2.4mm, cream in color, elongated, with wavy layered texture inside.

2) Calcium Carbonate (marine (benthic) organism), cellular structure is visible, 0.15mm to 0.25mm

Concentration Feature

Dark (opaque) clay pellets with sharp to merging boundaries, <2.7mm, mode 1.1mm, sr-r. The fabric also contains a few dark brown and light orange streaks in the groundmass, probably as a result of clay mixing. And depletion features around voids.

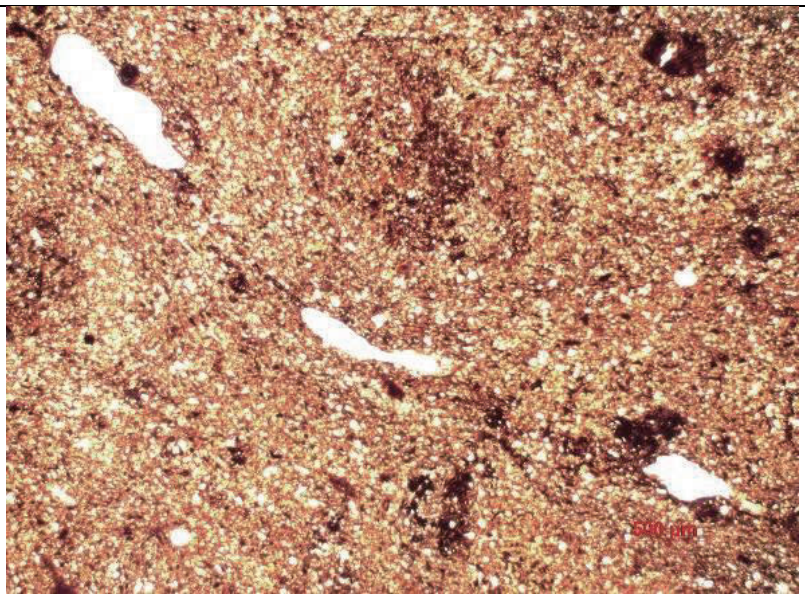
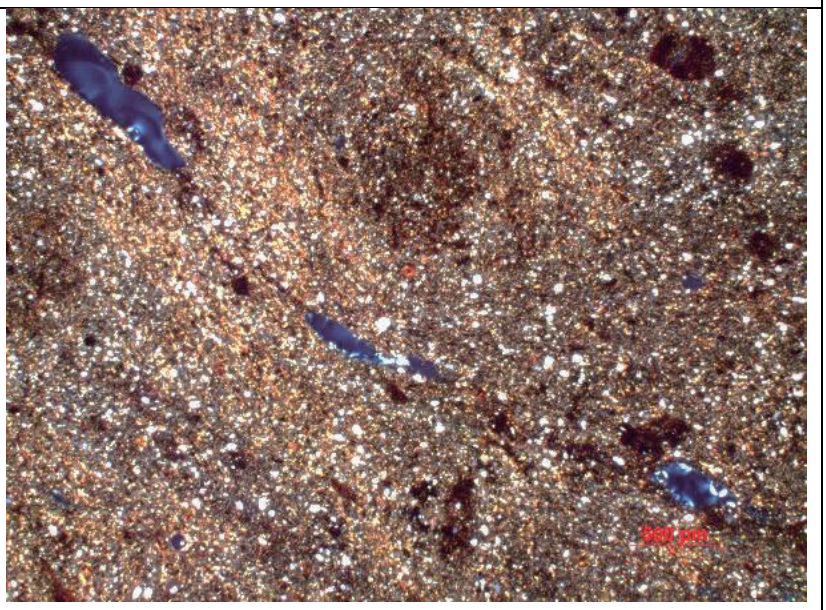
CR. PPL, 25X Channel voids with preferred orientation (Quinn, 2013, p.98[31])	
CR. XPL 25X The streaks and concentration texture.	

Figure. 5.4.3 Petrographic features in CR

[31] Patrick Quinn, Ceramic Petrography: The Interpretation of Archaeological Pottery and Related Artefacts in Thin Section. Oxford. 2013

5.5 Discussion and Analysis

All the samples in the two fabric groups feature frequent silt size monocrystalline quartz inclusions in very fine groundmass. The porosity is from low to moderate. Fabric group 1 is of a pretty low porosity. The quartz-feldspar dominant mineral composition fit with the local geology for rich in granite/granodiorite, quartzite.

From the observation of the two fabric groups, three thin sections, the frequent quartz inclusions are well-sorted. The mineral composition is relatively simple and even. The historical grey brick (HG) is of the lowest porosity, mostly mesovughs. Otherwise there are planar voids present in CG (seldom) and CR (frequent). They have a certain orientation.

Porosity, distribution of pores, and mineral composition can be the reasons for the outstanding performance of Chinese traditional grey bricks. Quartz of a hardness of 7 in Mohs hardness. As a frequent mineral in the thin sections, it may contribute to the hardness and mechanical strength of the building materials.

This Fabric group 1 is a very typical machine-made grey brick in Soochow area, very fine-grained. HG is dated as no later than 1920s and CR is a contemporary one. According to the manufacture data, the firing temperature is about 700℃. The dark *caesious* color fabric suggests the bricks were cooled in the reducing atmosphere in which the kilns were particularly designed.

The Fabric group 2 comprises coarse-grained inclusions, namely various shape of calcium carbonate. There are still many calcium-based microfossils, since calcite breaks down at about 850℃, their firing temperatures must not have exceeded this level.

Also the concentration feature in Fabric group 1 is more typical. The clay pellets inclusions reflect the depositional environment of the clay which is in accord with the geological context and other evidence in petrographic results. The darker brown and light orange streaks in the groundmass is probably resulted from the mixing of clay.

Besides, the voids are generally larger in size than the ones in Fabric group 1. Planar voids are common, parallel to the vessel wall. Channels and mesovughs also present. The relative lower porosity -the c: f: v ratio of Fabric group 1 and Fabric group 2 may also account for its inferiority in structure strength.

And there are more abundant microcrystalline quartz in Fabric group 1 than in Fabric group 2. The micrite in clay size of the groundmass, together with the coarse fraction calcium carbonate do not work as a strong enough structural aggregate in the sample CR.

CHAPTER 6: Chemical Resistance and Tung Oil Treatment

6.1 Introduction

Microstructure and composition of the structural clay material are primary contributing factors to the resistance to chemicals. The area of clay production, the mineralogical composition of the clays used, the method adopted to treat the brick, the temperature at which the clay is fired, and the location of the bricks in the kiln will determine the properties of the final product.

The chemical resistance is of importance because corrosion negatively affects mechanical properties, resulting a loss of functional performance. In the previous study of Grimshaw[32], the factors relevant to resistance of ceramic products were listed as follows:

a. the nature of the material, and especially its texture and hardness;

b. the mode of preparation;

c. the nature of the bond (if any);

d. the amount of the bond (if any);

e. the extent of vitrification;

f. the temperature of the material when it is examined.

[32], Rex W. Grimshaw, The Chemistry and Physics of Clays and Allied Ceramic Materials, 4th Edition, New York: Wiley Interscience, 197. 1997

Among the above factors a., b., e., f should be considered in designing the test to measure the quantitative effects of chemical resistance.

6.2 Acid Resistance Test

New mineral processing techniques being investigated by the Bureau of Mines and others, such as processing at elevated temperatures, leaching with acids and bases, chloride leaching, and dissolution in fused-salt baths, may require the use of construction materials that have superior corrosion resistance. One example is the construction material needed to line leaching vessels used in the extraction of alumina from clay using HCl (hydrochloric acid), HNO_3 (nitric acid), or H_2S0_4 (sulfuric acid)[33].

In the praxis of brickwork cleaning, acid and alkaline cleansing products are broadly applied in treating the dirt on the surface of building material. Whereas, acid and alkaline also result in decay of the architectural ceramics. According to ASTM C279-88[34]: Standard Specification for Chemical-Resistant Masonry Units, the acid resistance test on brick samples is designed.

It was indicated in a 1981 literature review[35] that corrosion data on other industrial ceramic products are scarce and based primarily upon supplier information, which often does not cover the specific conditions of interest. This lack of definitive data is partly due to the wide variation in industrial products and their applications. Hence, the test on the physical ceramic products is necessary.

[33] J. A. Eisele, Producing Alumina Clay by the Hydrochloric Acid ProA Bench-Scale Study. BuMines RI 1980:21
[34] ASTM Standard C279-88, "Specification for Concrete Aggregates," ASTM International." Reapproved 2007.
[35] T. A Clancy, T. A. High-Temperature Corrosion Resistance of Ceramic Materials. BuMines IC 8843, 1981:31

This test is based on the sulfuric absorption and solubility test. The preparation of samples and procedures are referred to the ASTM C279-88 (See appendix). The results of experiment are as follows:

Table 6.2.1 Sulfuric Acid Solubility Test

Sample	**HG. 05. N**	**HG. 06. N**	**HG. 07. N**	**Average**
Chemical Resistance **(Sulfuric Acid Solubility Test)** **Max Weight Loss %**	3.0	3.0	3.1	3.0
Sample	**CG. 05. N**	**CG. 06. N**	**CG. 07. N**	**Average**
Chemical Resistance **(Sulfuric Acid Solubility Test)** **Max Weight Loss %**	1.2	1.3	1.7	1.4
Sample	**CR. 05. N**	**CR. 06. N**	**CR. 07. N**	**Average**
Chemical Resistance **(Sulfuric Acid Solubility Test)** **Max Weight Loss %**	4.4	4.6	4.9	4.6

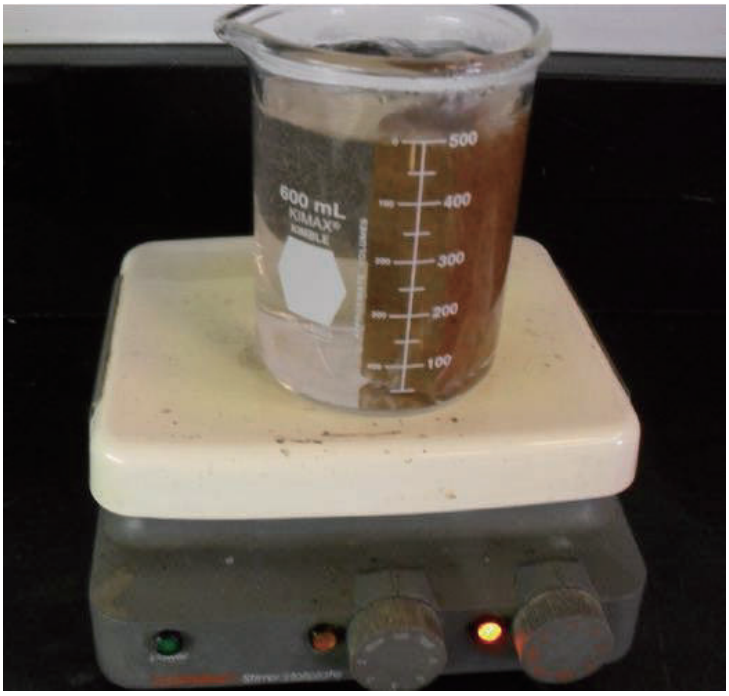


Figure 6.2.1 10-min boiling procedure in the Acid Resistance Test

It is noticeable that the drying process have to be done in an oven over 240° F (120° C) for at least 16 hours. The measurements should be done until the samples are totally cooled down in a desiccator.

6.3 Tung Oil Treatment

Tung oil is made from pressed seeds from the nut of the Tung tree (a. k. a Tung oil tree). The Tung tree is native to China. In the 14th century, Chinese merchants were noted for using Tung oil to waterproof and protect wooden ships from the eroding powers of the sea. There are even mentions of Tung oil appearing in the writings of Confucius in around 400 B.C. For these reasons, it is also sometimes referred to as “China wood oil.”

Figure 6.3.1 The Tung Oil Used in the Treatment

The Tung tree is valued for Tung oil, which is derived from the seeds of the tree. Tung oil, also called China wood oil or nut oil, has traditionally been used in lamps in China. In modern times, it is used as an ingredient in paint, varnish, and caulk. It is also used as a wood finish for furniture and other wooden objects. After processing to remove gums in the oil, it was also used as a motor fuel[36].

Marco Polo wrote in the 13th century "The Chinese take some lime and chopped hemp, and these they knead together with a certain wood oil; and when the three are thoroughly amalgamated they hold like any glue, and with this mixture they paint their ships"[37].

It has been introduced to Argentina, Paraguay, Thailand, and the United States for oil production. Just prior to World War I, a number of seeds received from the United States Ambassador to China were planted in California, but the young trees could not take hold

[36] "Aleurites fordii Hemsl. Handbook of Energy Crops. Purdue University. 1997.
[37] Marco Polo., translated by Henry Yule, The Travels of Marco Polo, Book 3, Chapter 1. Unabridge Edition. 1903.

in the dry climate. David Fairchild of the Department of Agriculture successfully introduced the tree in 1905 in the U.S. Gulf States from Florida west to eastern Texas[38].

The liquid is clear, dark golden yellow in color, thick and shiny. (As is shown in Figure 6.3.1 Right)

Figure 6.3.2 Tung tree nuts-source for Tung oil[39]

Since it is a sort of drying oil, Tung oil hardens upon exposure to air. The resulting coating is pale yellow and transparent, a plastic-like thin layer. Tung oil is property exploited in

[38] K Keeler W Brown, "The History of Tung Oil" Wildland Weeds 9 (1): 4–6.2005
[39] https://www.flickr.com/photos/tgerus/4457194458

most of its applications such as wood finishing, as well as in the composition of oil paints and making of printing inks.

In the making of floor tiles of Forbidden City, the clay tiles were soaked in the Tung oil for a period of time so as to be more wearable. I have immersed the three types of brick samples concerned in my experiment, marking them as HG. 001. T, CG. 001. T, CR.001. T. The letter “T” is standing for “Tung oil treatment”, as opposed to the letter “N”, which is standing for “No Tung oil treatment”.

The samples treated under 22° C, room temperature and pressures, for over 24 hours are shown in the Figure 6.3.3-6.3.5:

Figure 6.3.3 HG brick immersed in Tung oil for over 24 hours

Figure 6.3.4 CG brick immersed in Tung oil for over 24 hours

Figure 6.3.5 CR brick immersed in Tung oil for over 24 hours

It is noticeable that after setting still isolated from Tung oil, the luster of the surface faded. The surface turned to matte.

6.4 Salt Test

The pressure generated by salt crystal growth in confined spaces in porous building materials such as terracotta, brick, and stone is fully acknowledged to be a major cause of damage and an important factor in their durability[40].

Sodium sulfate (Na_2SO4) is the salt of choice for the RILEM VB Salt Test, for the following reasons:

- Availability: in modern buildings, highways and civil work, soluble salts as sodium and calcium sulfates are commonly released by Portland cement.
- Its destructive nature: sodium sulfate is very damaging because it undergoes a high degree of volume change when hydrated[41].

The testing protocol selected for this project is RILEM VB Salt Test according to the standard. (See the Appendix)

The RILEM test consists of 15 cycles of 2 hour immersion in a 10% solution of sodium sulfate. For fifteen days, a daily cycle was completed which included: two hours of immersion, followed by 19 hours oven-dried at 60 degree Celsius and then cooling within 3 hours.

[40] A. S. Goudie and H Viles, Salt Weathering Hazards. Chichester: John Wiley and Sons. 1997.
[41] R. U. Cooke, Salt Weathering in Desserts, Proc Geol Assoc London 1981: 1-16

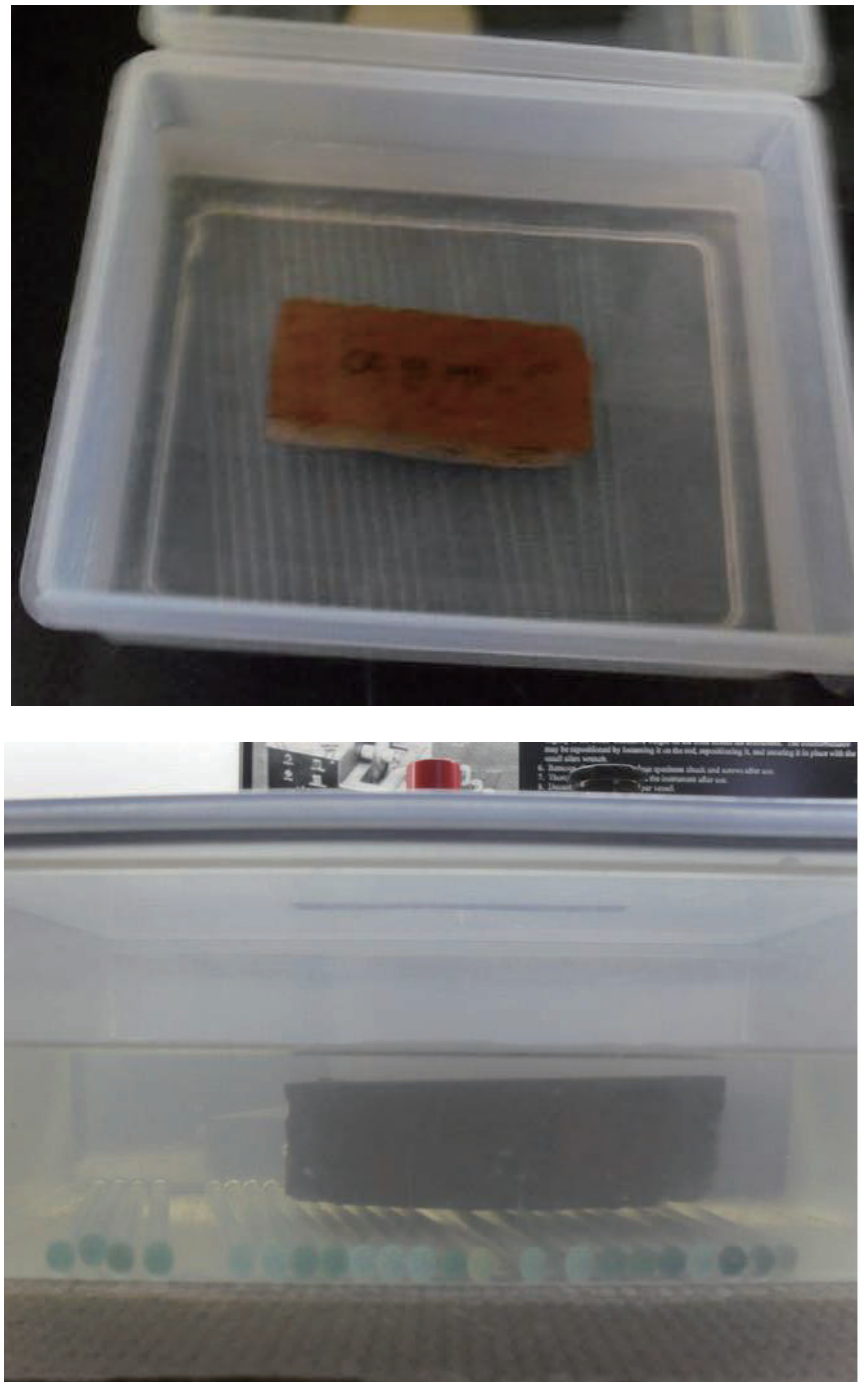


Figure 6.4.1 Samples during the Salt Test

The test was run on 3 samples for each group. The 10% solution of sodium sulfate (Na_2SO_4) was prepared in the lab prior to the test. After completing 15 cycles, the samples were immersed for 7 days in distilled water; and the water was changed daily.

6.5 After Treatment Test

Liquid Nitrogen Porosity

Following the methodology and standard in Chapter 4.2, the liquid nitrogen porosity test was duplicated on the treated samples. The experimental results are as follows:

HG Group

Table 6.5.1 HG-Treated group liquid nitrogen porosity test results

SAMPLE	D (g)	W (g)	S (g)	P%
HG.001.T	197.35	210.43	126.40	15.57
HG.002.T	201.78	217.36	120.72	15.79
HG.003.T	241.10	259.69	143.88	16.05
HG.004.T	217.31	231.34	138.13	15.05
$\overline{P}$ %=15.615%		Standard deviation: 0.00425		

CG Group

Table 6.5.2 CG-Treated group liquid nitrogen porosity test results

SAMPLE	D (g)	W (g)	S (g)	P%
CG.001.T	55.79	58.42	25.32	7.95
CG.002.T	56.85	59.57	25.70	8.03
CG.003.T	52.61	55.01	23.17	7.54
CG.004.T	52.32	54.92	22.80	8.09
$\overline{P}$ %=7.903%		Standard deviation: 0.00248		

CR Group

Table 6.5.3 CR-Treated group liquid nitrogen porosity test results

SAMPLE	D (g)	W (g)	S (g)	P%
CR.001.T	162.28	177.42	88.62	17.05
CR.002.T	160.52	178.04	85.29	18.89
CR.003.T	161.48	176.89	84.97	16.76
CR.004.T	155.26	172.24	79.28	18.27
$\overline{P}$ %=17.743%		Standard deviation: 0.01007		

Acid Resistance Test

As the brick was completely soaked in the Tung oil, and, we can also judge from the section, it can be infer that the brick samples are fully soaked with Tung oil. Therefore, the after treatment acid resistance test can basically follow the methodology in Chapter 6.2. The result data of the experiments are as follows:

Table 6.5.4 Sulfuric Acid Solubility Test

Sample	**HG. 005. T**	**HG. 006. T**	**HG. 007. T**	**Average**
Chemical Resistance (Sulfuric Acid Solubility Test) Max Weight Loss %	1.4	1.7	1.8	1.6
Sample	**CG. 005. T**	**CG. 006. T**	**CG. 007. T**	**Average**
Chemical Resistance (Sulfuric Acid Solubility Test) Max Weight Loss %	0.8	0.4	0.6	0.6
Sample	**CG. 005. T**	**CG. 006. T**	**CG. 007. T**	**Average**
Chemical Resistance (Sulfuric Acid Solubility Test) Max Weight Loss %	3.3	3.6	2.9	3.3

Salt Test

For the reason that the untreated brick samples did not show apparent signs resulting from the salt test. So this test is omitted in this section

6.6 Microstructure and Observations

The surfaces being examined are all face surfaces, not cutting surfaces. In this way, the results are comparable.

From the SEM micrographs shown below, we may have several observations:

- The grey brick series are less porous and rugged;
- The treated samples have much finer surface

On one hand, we can confirm that the historic grey brick (HG) and contemporary grey brick (CG) have a higher firing temperature. Therefore, the cellular structure (see arrows) is detectable[42]. In the micrographs of Tung oil treated historic grey brick (HG) and contemporary grey brick (CG), we can see the bubbles and a vague coating layer, which shows the state of the Tung oil applied: partly absorbed but there is still residue on the surface. The Tung oil attaching to the surface is not easily washable and it covers the whole brick surface and fills the pores. In this way, the surface is smoother.

[42] Kerstin Elert, et al. Durability of bricks used in the conservation of historic buildings—influence of composition and microstructure. Journal of Cultural Heritage 4 (2003) 91–99: 94

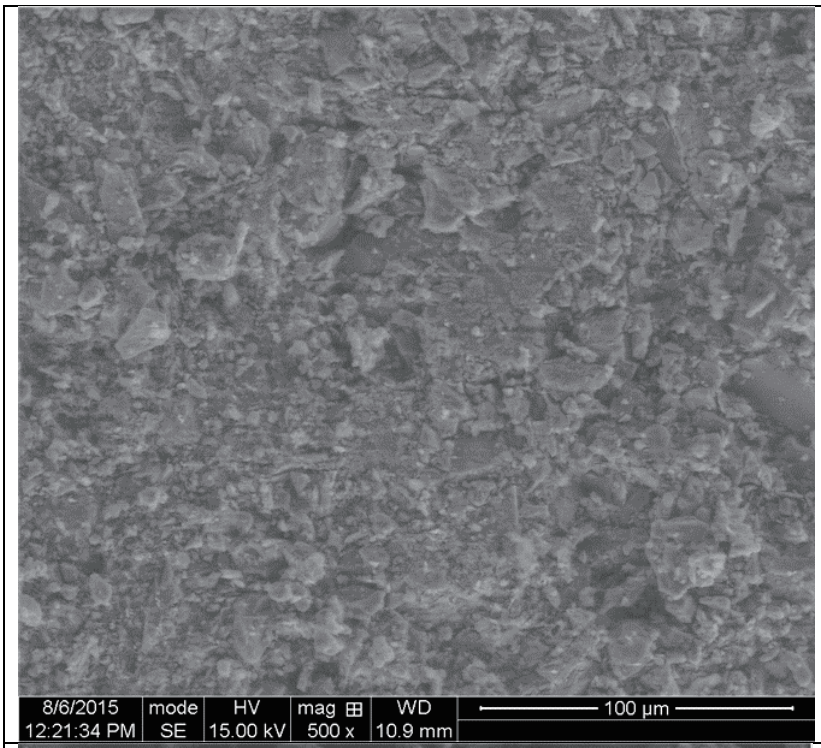
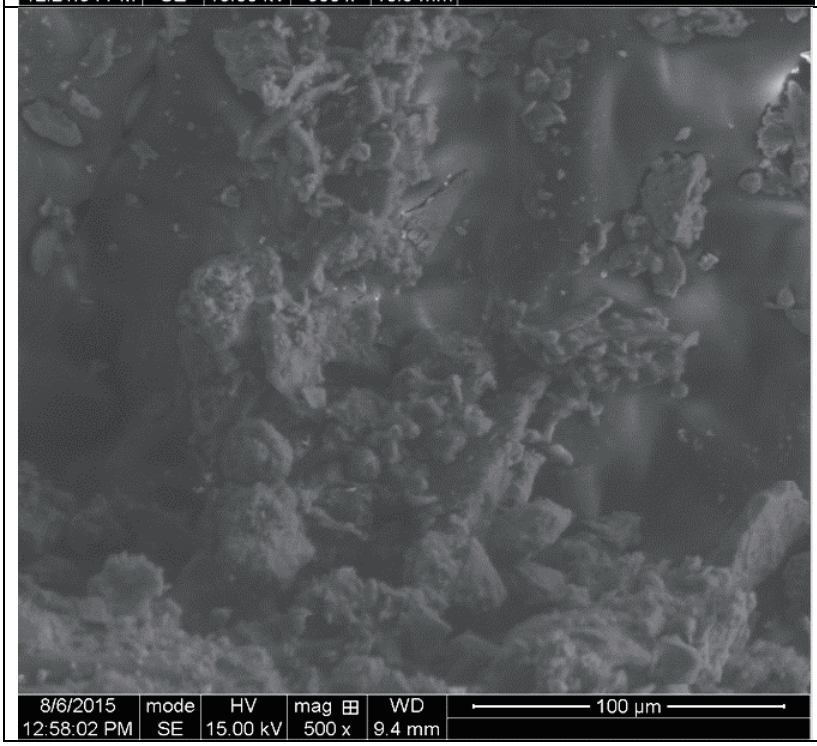
	**SEM Micrograph** **500X** **Untreated, historic grey brick**
	**SEM Micrograph** **500X** **Tung oil treated, historic grey brick**

Figure 6.6.1 SEM micrographs of untreated/Tung oil treated historic grey brick

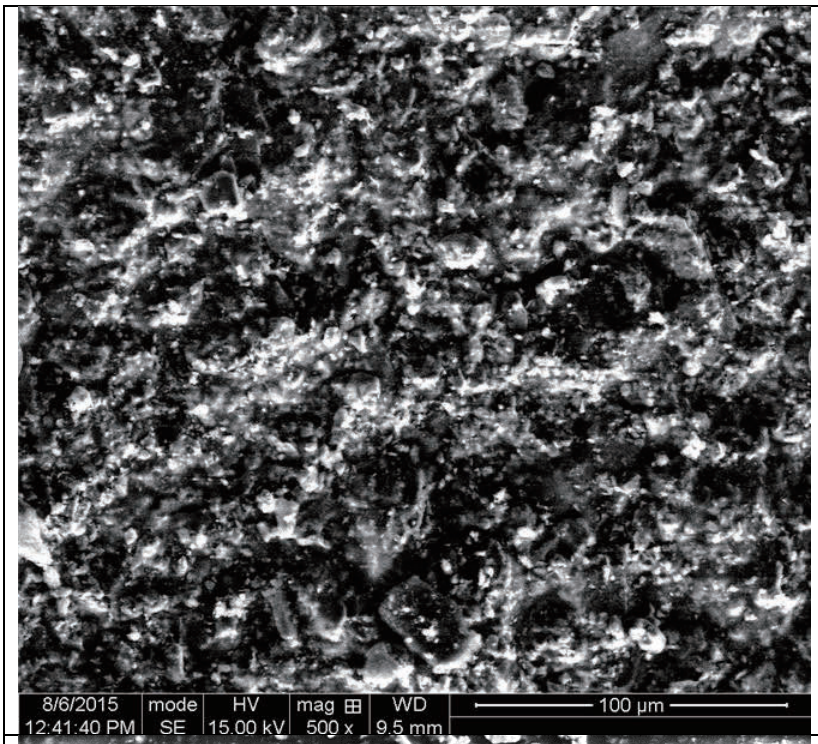
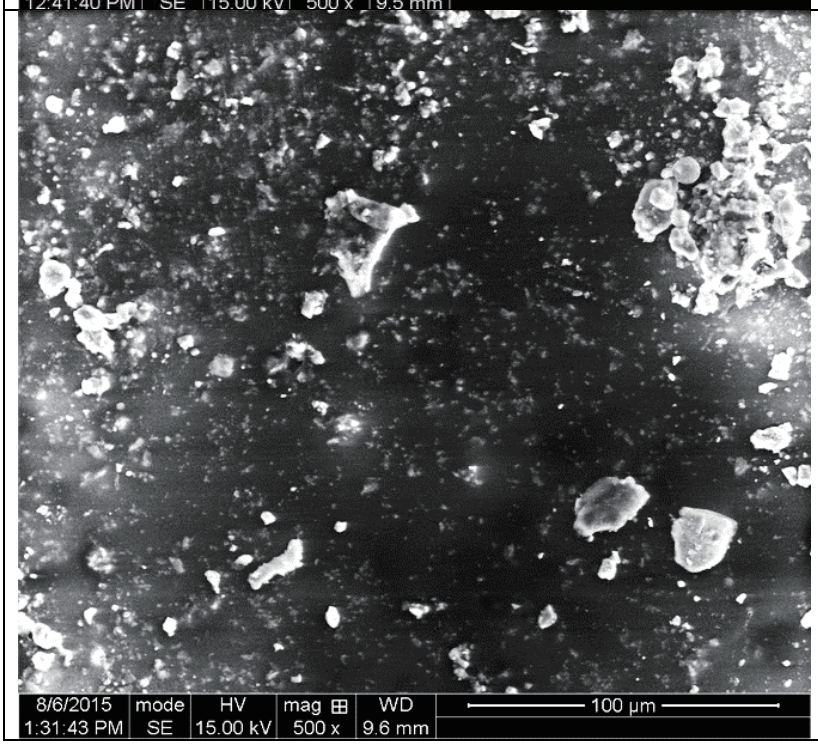
	**SEM Micrograph** **500X** **Untreated, cotemporary grey brick**
	**SEM Micrograph** **500X** **Tung oil treated, contemporary grey brick**

Figure 6.6.2 SEM micrographs of untreated/Tung oil treated contemporary grey brick

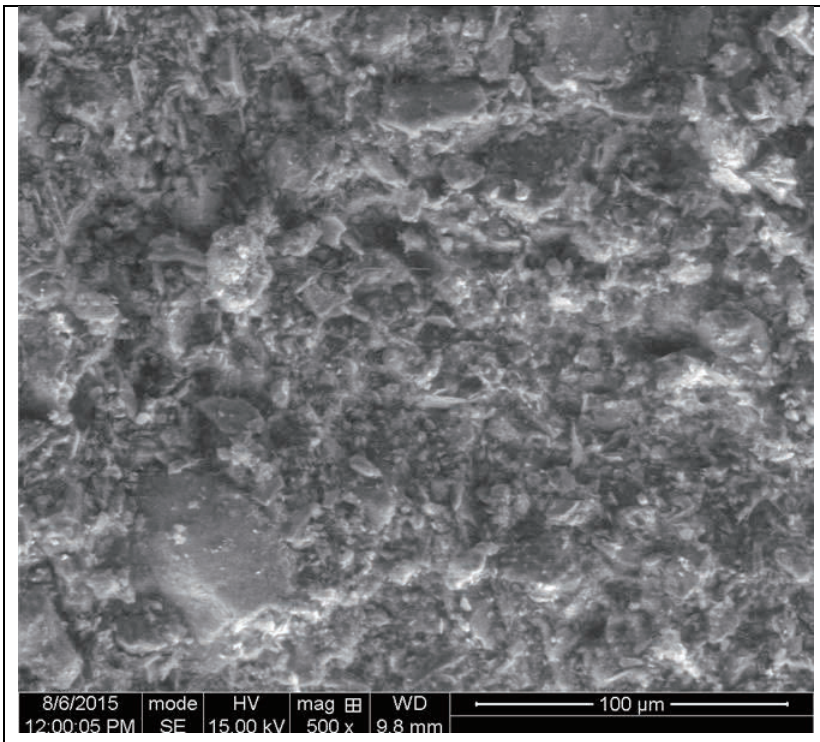
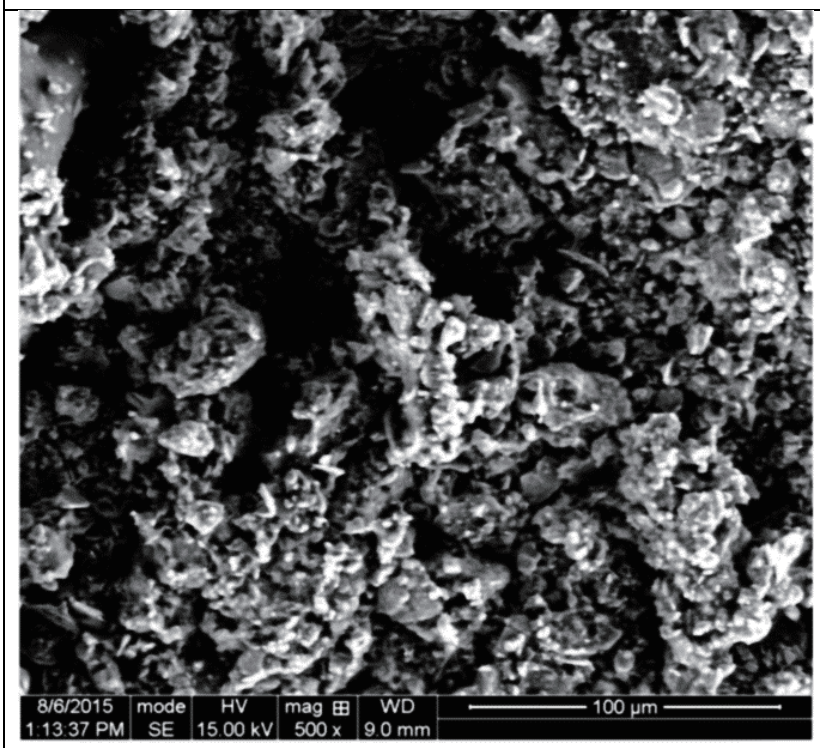
	**SEM Micrograph** **500X** **Untreated,** **contemporary** **red brick**
	**SEM Micrograph** **500X** **Tung oil** **treated,** **contemporary** **red brick**

Figure 6.6.3 SEM micrographs of untreated/Tung oil treated contemporary red brick

On the other hand, the micrographs of contemporary red brick (CR) show the microstructure of the pores has been altered after soaking the Tung oil. The lower image shows an advanced stage of coalescence of phyllosilicate than the upper one, which reflects evident laminar habit of phyllosilicates as it was not highly fired. (Figure 6.6.3)

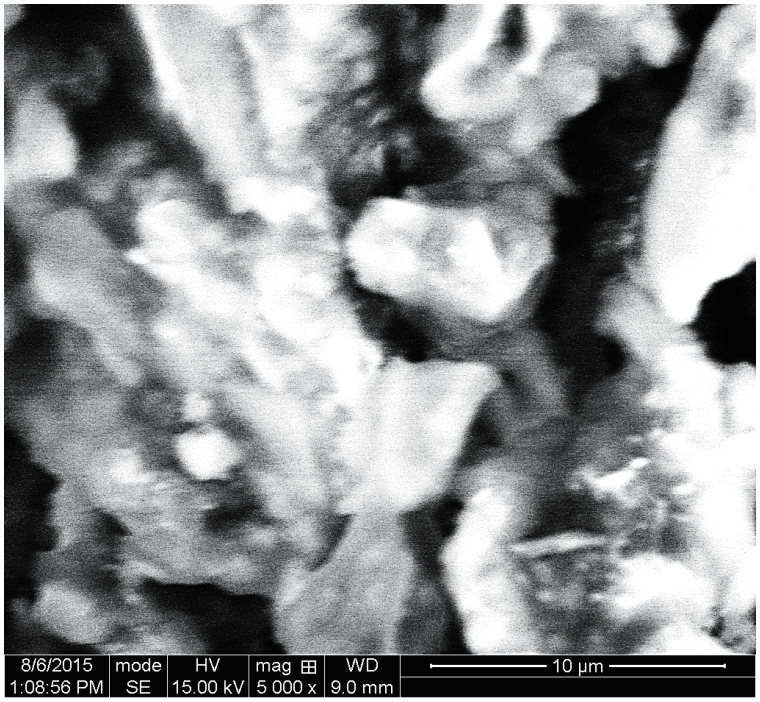


Figure 6.6.4 SEM micrograph of Tung oil treated contemporary red brick,

5000X magnification

CHAPTER 7: Data Analysis and Conclusions

7.1 Summary of Results

From a set of experiments and observation, we may sum up the results in this section:

(i) Historic grey bricks and contemporary grey bricks have a similar mineral composition and microstructure. While the contemporary red bricks show different features other than the grey ones. From the petrographic results, we can tell that the contemporary red bricks were not highly vitrified. Also the clay type of the contemporary red is not the same as the grey ones.

(ii) In the porosity test, the average results of three types of bricks are 24.07%, 15.13% and 27.99%, from which we can see, the contemporary grey bricks not only are the highest in Mohs hardness, but also are the least porous.

(iii) The after treatment group of samples also have undergone the porosity test, the average results of three types of bricks are 15.62%, 7.90% and 17.74%. Compared with the results shown in (ii). The porosity of treated samples is significantly reduced.

(iv) In the chemical resistance test, the weight loss (%) can be seen as an indicator of this system. The weight loss of historic grey (HG), contemporary grey (CG) and contemporary red (CR) are 3.0%, 1.4%, 4.6% respectively.

(v) The chemical resistance test done on the Tung oil treated group of samples are more resistant to sulfuric acid solution. The weight loss of treated historic grey (HG), contemporary grey (CG) and contemporary red (CR) are 1.6%, 0.6%, 3.3%.

(vi) Tung oil as a coating material, when applied to the brick surface, it will form an oily, shiny layer. However, when the brick is set still for over 24 hours, this layer will turn to a matte texture.

(vii) From the SEM micrographs, we can see grey brick series are more homogeneous and smoother in microstructure and texture. Actually, the contemporary grey brick (CG) looks more compressed, composed with finer particles. And the morphology of the contemporary red brick (CR) surface looks more rugged and rough, showing a more complicated microstructure and composition.

(viii) From the results of electronic scanning microscopy, we may find that the grey brick series are less porous and compressed. The texture is much finer. The connectivity of pores of grey brick is also poor. From the micrographs of contemporary red brick, we may find that it is much more porous. Furthermore, the connectivity of the pores is much better than the grey ones. The particles are less compressed and coarser.

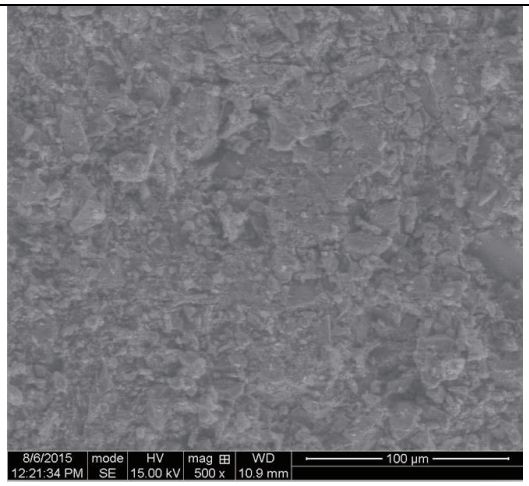
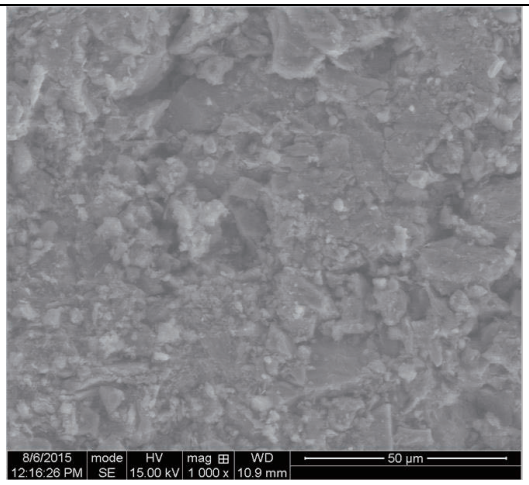
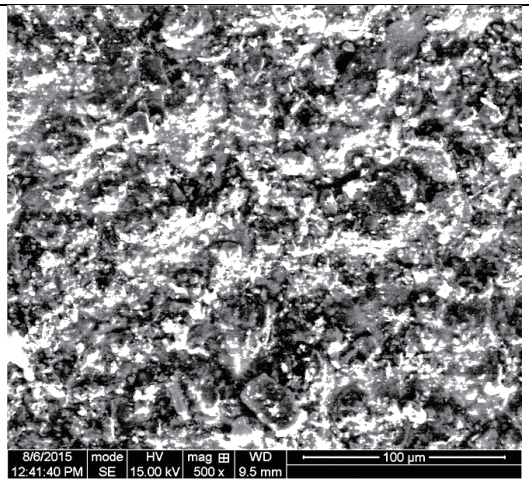
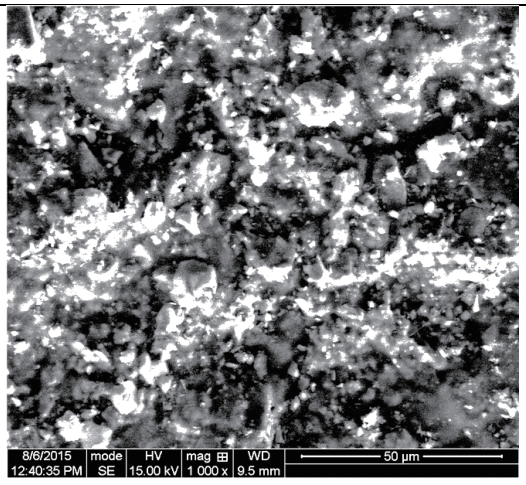
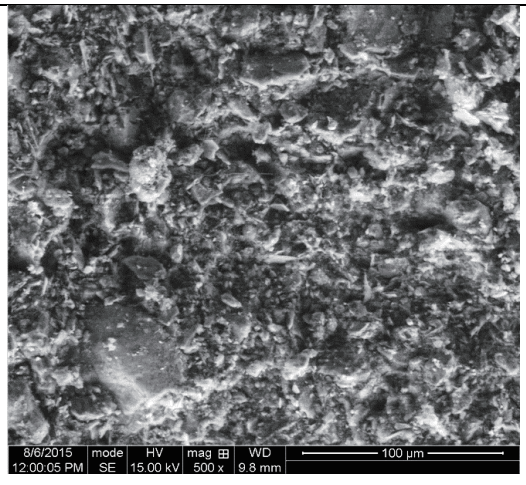
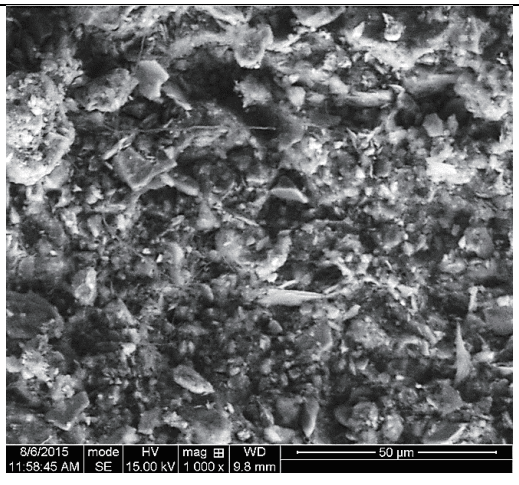
	500x	1000x
HG		
CG		
CR		

Figure 7.1.1 SEM micrographs of three types of brick samples of 500X and 1000X magnification

7.2 Data Analysis

The overall situation is the grey brick series have a better performance than the red ones as far as concerned in this thesis. And the contemporary grey bricks score best in the previous test. Compared with the standardization in China[43], in which it is clearly pointed out that the standard porosity is about 30%, the three types of bricks, even though untreated, live up to the standard. Relatively speaking, the grey brick series show a good quality from the experimental data.

It is apparent from the data listed in Table 7.2.1 and Table 7.2.2, to see the improvement in porosity and chemical resistance. Tung oil has improved all the types of bricks in this property. Also, we can find that in both two sets of experiments, Tung oil was most effective on the samples of CG group.

Table 7.2.1 The porosity of untreated/Tung oil treated samples

	Average porosity (%)		
Brick type	HG	CG	CR
Untreated	24.07%	15.13%	27.99%
Tung oil treated	15.62%	7.90%	17.74%
Improvement rate (%)	35.11%	47.79%	36.62%

[43] GB 5101-2003, Standardization, China

Table 7.2.2 The weight loss of untreated/Tung oil treated samples

undergone the Chemical Resistance Test

	Weight Loss after Chemical Resistance Test (%)		
Brick type	HG	CG	CR
Untreated	3.0%	1.4%	4.6%
Tung oil treated	1.6%	0.6%	3.3%
Improvement rate (%)	46.67%	57.14%	28.26%

7.3 Conclusions

In the Local History of Jiangsu Province- Building History[44], Chapter 5, there is some description and narrative about the 1980s Soochow vernacular brick buildings. First, the grey brick never lost its premiere position in the vernacular building history. It is symbolic and featuring the image of architectural style. Second, during the 1980s, the facing brick was becoming more and more popular in the decoration of building. Mosaics, various architectural ceramics, glazing tiles were frequently applied in the buildings. Third, traditional building was still mostly in brick-timber structure before the 1990s.

[44] The Commission of Local History, Jiangsu Province (ed). Local History of Jiangsu Province- Building History. 2011: 34 建筑志 / 江苏省地方志编纂委员会 [编]. -- 江苏古籍出版社, -- (江苏省志 / 江苏省地方志编纂委员会编).

In fact, as early as late 19th century, the decorative carved grey bricks were common for the public and private buildings. However, such brick carving craftsmanship hardly appeared in red bricks. As the petrographic study indicates, the grey brick and the red brick could be unnecessarily to be made of a same type of clay, though they were originated from the same place. The type for the grey bricks is simpler in mineral composition and more homogeneous. While the type for the red brick contains more organism, which means it has constituent of marine clay. Additionally, the firing temperature for the contemporary red bricks of 1980s is not necessarily higher than that for historic grey bricks in early 20th century. The residues of micro-organism in the micrographs are as evidence in supporting this conclusion.

A specific group of red bricks of 1980s tend to be of poor quality, since they were likely to act as walling material behind various facing bricks, mosaics, artificial masonry facing materials. In this context, even if the contemporary red bricks are more modern than the historic grey ones, they are not necessarily better in quality.

The porosity test and acid resistance test successfully verified the superiority of historic grey brick (HG) and contemporary grey brick (CG) over the contemporary red brick (CR). The historic grey bricks were typically used without any plastering or paint, to create a neat work of façade. Nevertheless, the red bricks were commonly used as walling material.

During the process of production the grey bricks are cooled under a reduced atmosphere, for this reason, the color of these bricks turns to grey instead of red. However, even cooled off by water spray, the water vapor works to enclose the pores of the surface. According to the SEM micrographs, we can see the connectivity of historic grey brick (HG) and contemporary grey brick (CG) is much poorer than the contemporary red one (CR).

Historic grey bricks (HG) and contemporary grey bricks (CG) have a better performance in porosity and chemical resistance. Contemporary grey bricks are recently machine-made products, which are tightly compressed, in a very fine texture. The crystalline microstructure is fine. The hardness in Mohs of contemporary grey brick (CG) is also the highest.

As for the Tung oil treatment, from the Chapter 7.2, it is obvious that the effect of Tung oil treatment is manifest. Since the historic grey bricks (HG) and contemporary grey bricks (CG) are less porous, the penetration of Tung oil throughout the grey series samples is not as high as the contemporary red brick (CR). From the SEM micrographs, it is easy to see the existence of Tung oil attached to the surface of grey series samples. The treatment helps in reducing the porosity and isolate the chemicals or other factors that result in weathering. However, the penetration of Tung oil into the contemporary red brick is much higher. The microstructure of contemporary red brick (CR) is dramatically altered after the treatment. The particles of contemporary red brick have gone through a bio-mineralization process. Because of the regulation function of Tung oil, the particles are gathered more closely to each other.

To sum up, the Tung oil works to the architectural ceramic materials. Among the three types of bricks in question, the contemporary grey brick gains the greatest improvement from the treatment. From the petrographic study, we can confirm that the grey bricks and red bricks are at different firing temperatures. On the terms of SEM observation, the cellular structure indicates the grey bricks were more vitrified and less porous, while the red bricks shows a laminar habit. Tung oil covers the surface and fills the pores of both historic grey bricks (HG) and contemporary grey bricks (CG) as a coating protection. Otherwise, the Tung oil treated contemporary red bricks (CR) appear to be in a more

advanced stage of coalescence of phyllosilicate as a result of taking in Tung oil instead of having it attached to the surface. It may be protected but it is by no means less porous in this way. Therefore, Tung oil treatment is not as effective as it is on the grey bricks to this lower fired red bricks.

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APPENDIX

I. ASTM C67-03a

Designation: C 67 – 03a

American Association State Highway and Transportation Officials Standard
AASHTO No.: T 32-70

Standard Test Methods for Sampling and Testing Brick and Structural Clay Tile[1]

This standard is issued under the fixed designation C 67; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ε) indicates an editorial change since the last revision or reapproval.

This standard has been approved for use by agencies of the Department of Defense.

1. Scope*

1.1 These test methods cover procedures for the sampling and testing of brick and structural clay tile. Although not necessarily applicable to all types of units, tests include modulus of rupture, compressive strength, absorption, saturation coefficient, effect of freezing and thawing, efflorescence, initial rate of absorption and determination of weight, size, warpage, length change, and void area. (Additional methods of test pertinent to ceramic glazed facing tile are included in Specification C 126.)

1.2 The text of this standard references notes and footnotes which provide explanatory material. These notes and footnotes (excluding those in tables and figures) shall not be considered as requirements of the standard.

NOTE 1—The testing laboratory performing this test method should be evaluated in accordance with Practice C 1093.

1.3 Unless otherwise indicated, the values stated in inch-pound units are to be regarded as the standard. The values given in parentheses are for information only.

1.4 *This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

2. Referenced Documents

2.1 *ASTM Standards:*

C 43 Terminology of Structural Clay Products[2]

C 126 Specification for Ceramic Glazed Structural Clay Facing Tile, Facing Brick, and Solid Masonry Units[2]

C 150 Specification for Portland Cement[3]

C 1093 Practice for Accreditation of Testing Agencies for Unit Masonry[2]

E 4 Practices for Force Verification of Testing Machines[4]

E 6 Terminology Relating to Methods of Mechanical Testing[4]

3. Terminology

3.1 *Definitions*—Terminology E 6 and Terminology C 43 shall be considered as applying to the terms used in these test methods.

4. Sampling

4.1 *Selection and Preparation of Test Specimens*—For the purpose of these tests, full-size brick, tile, or solid masonry units shall be selected by the purchaser or by the purchaser's authorized representative. Specimens shall be representative of the lot of units from which they are selected and shall include specimens representative of the complete range of colors, textures, and sizes and shall be free of or brushed to remove dirt, mud, mortar, or other foreign materials unassociated with the manufacturing process.

4.2 *Number of Specimens*:

4.2.1 *Brick*—For the modulus of rupture, compressive strength, abrasion resistance, and absorption determinations, at least ten individual brick shall be selected for lots of 1 000 000 brick or fraction thereof. For larger lots, five additional specimens shall be selected from each additional 500 000 brick or fraction thereof. Additional specimens are taken at the discretion of the purchaser.

4.2.2 *Structural Clay Tile*—For the weight determination and for compressive strength and absorption tests, at least five tile shall be selected from each lot of 250 tons (226.8 Mg) or fraction thereof. For larger lots, five additional specimens shall be tested for each 500 tons (453.6 Mg) or fraction thereof. In no case shall less than five tile be taken. Additional specimens are taken at the discretion of the purchaser.

4.3 *Identification*—Each specimen shall be marked so that it may be identified at any time. Markings shall cover not more than 5 % of the superficial area of the specimen.

5. Specimen Preparation

5.1 *Weight Determination*:

[1] These test methods are under the jurisdiction of Committee C15 on Manufactured Masonry Units and is the direct responsibility of Subcommittee C15.02 on Brick and Structural Clay Tile.

Current edition approved June 10, 2003. Published August 2003. Originally published as C 67 – 37T and C 112 – 34T. Last previous edition approved in 2003 as C 67 – 03.

[2] *Annual Book of ASTM Standards*, Vol 04.05.

[3] *Annual Book of ASTM Standards*, Vol 04.01.

[4] *Annual Book of ASTM Standards*, Vol 03.01.

***A Summary of Changes section appears at the end of this standard.**

5.1.1 *Drying*—Dry the test specimens in a ventilated oven at 230 to 239°F (110 to 115°C) for not less than 24 h and until two successive weighings at intervals of 2 h show an increment of loss not greater than 0.2 % of the last previously determined weight of the specimen.

5.1.2 *Cooling*—After drying, cool the specimens in a drying room maintained at a temperature of 75 ± 15°F (24 ± 8°C), with a relative humidity between 30 and 70 %. Store the units free from drafts, unstacked, with separate placement, for a period of at least 4 h and until the surface temperature is within 5°F (2.8°C) of the drying room temperature. Do not use specimens noticeably warm to the touch for any test requiring dry units. The specimens shall be stored in the drying room with the required temperature and humidity maintained until tested.

5.1.2.1 An alternative method of cooling the specimens to approximate room temperature shall be permitted as follows: Store units, unstacked, with separate placement, in a ventilated room maintained at a temperature of 75 ± 15°F (24 ± 8°C), with a relative humidity between 30 and 70 % for a period of 4 h and until the surface temperature is within 5°F (2.8°C) of the ventilated room temperature, with a current of air from an electric fan passing over them for a period of at least 2 h. The specimens shall be stored in the ventilated room with the required temperature and humidity maintained until tested.

5.1.3 *Weighing and Report*:

5.1.3.1 Weigh five dry full size specimens. The scale or balance used shall have a capacity of not less than 3000 g and shall be sensitive to 0.5 g.

5.1.3.2 Report results separately for each specimen to the nearest 0.1 g, with the average of all specimens tested to the nearest 0.1 g.

5.2 *Removal of Silicone Coatings from Brick Units*—The silicone coatings intended to be removed by this process are any of the various polymeric organic silicone compounds used for water-resistant coatings of brick units. Heat the brick at 950 ± 50°F (510 ± 28°C) in an oxidizing atmosphere for a period of not less than 3 h. The rate of heating and cooling shall not exceed 300°F (149°C) per h.

NOTE 2—Where indicated for specific individual tests, additional specimen preparation may be required.

6. Modulus of Rupture (Flexure Test)

6.1 *Test Specimens*—The test specimens shall consist of whole dry full-size units (see 5.1.1). Five such specimens shall be tested.

6.2 *Procedure*:

6.2.1 Support the test specimen flatwise unless specified and reported otherwise (that is, apply the load in the direction of the depth of the unit) on a span approximately 1 in. (25.4 mm) less than the basic unit length and loaded at midspan. If the specimens have recesses (panels or depressions) place them so that such recesses are on the compression side. Apply the load to the upper surface of the specimen through a steel bearing plate ¼ in. (6.35 mm) in thickness and 1½ in. (38.10 mm) in width and of a length at least equal to the width of the specimen.

6.2.2 Make sure the supports for the test specimen are free to rotate in the longitudinal and transverse directions of the test specimen and adjust them so that they will exert no force in these directions.

6.2.3 *Speed of Testing*—The rate of loading shall not exceed 2000 lbf (8896 N)/min. but this requirement is considered as being met if the speed of the moving head of the testing machine immediately prior to application of the load is not more than 0.05 in. (1.27 mm)/min.

6.3 *Calculation and Report*:

6.3.1 Calculate and report the modulus of rupture of each specimen to the nearest 1 psi (0.01 MPa) as follows:

$$S = 3W(l/2 - x)/bd^2 \quad (1)$$

where:

S = modulus of rupture of the specimen at the plane of failure, lb/in.^{2} (Pa),
W = maximum load indicated by the testing machine, lbf (N),
l = distance between the supports, in. (mm),
b = net width, (face to face minus voids), of the specimen at the plane of failure, in. (mm),
d = depth, (bed surface to bed surface), of the specimen at the plane of failure, in. (mm), and
x = average distance from the midspan of the specimen to the plane of failure measured in the direction of the span along the centerline of the bed surface subjected to tension, in. (mm).

6.3.2 Calculate and report the average of the modulus of rupture determinations to the nearest 1 psi (0.01 MPa).

7. Compressive Strength

7.1 *Test Specimens*:

7.1.1 *Brick*—The test specimens shall consist of dry half brick (see 5.1.1), the full height and width of the unit, with a length equal to one half the full length of the unit ±1 in. (25.4 mm), except as described below. If the test specimen, described above, exceeds the testing machine capacity, the test specimens shall consist of dry pieces of brick, the full height and width of the unit, with a length not less than one quarter of the full length of the unit, and with a gross cross-sectional area perpendicular to bearing not less than 14 in.^{2} (90.3 cm^2). Test specimens shall be obtained by any method that will produce, without shattering or cracking, a specimen with approximately plane and parallel ends. Five specimens shall be tested.

7.1.2 *Structural Clay Tile*—Test five dry tile specimens in a bearing bed length equal to the width ±1 in. (25.4 mm); or test full-size units.

7.2 *Capping Test Specimens*:

7.2.1 All specimens shall be dry and cool within the meaning of 5.1.1 and 5.1.2 before any portion of the capping procedure is carried out.

7.2.2 If the surface which will become bearing surfaces during the compression test are recessed or paneled, fill the depressions with a mortar composed of 1 part by weight of quick-hardening cement conforming to the requirements for Type III cement of Specification C 150, and 2 parts by weight of sand. Age the specimens at least 48 h before capping them. Where the recess exceeds ½ in. (12.7 mm), use a brick or tile

slab section or metal plate as a core fill. Cap the test specimens using one of the two procedures described in 7.2.3 and 7.2.4.

7.2.3 *Gypsum Capping*—Coat the two opposite bearing surfaces of each specimen with shellac and allow to dry thoroughly. Bed one of the dry shellacked surfaces of the specimen in a thin coat of neat paste of calcined gypsum (plaster of paris) that has been spread on an oiled nonabsorbent plate, such as glass or machined metal. The casting surface plate shall be plane within 0.003 in. (0.076 mm) in 16 in. (406.4 mm) and sufficiently rigid; and so supported that it will not be measurably deflected during the capping operation. Lightly coat it with oil or other suitable material. Repeat this procedure with the other shellacked surface. Take care that the opposite bearing surfaces so formed will be approximately parallel and perpendicular to the vertical axis of the specimen and the thickness of the caps will be approximately the same and not exceeding ⅛ in. (3.18 mm). Age the caps at least 24 h before testing the specimens.

NOTE 3—A rapid-setting industrial type gypsum is frequently used for capping.

7.2.4 *Sulfur-Filler Capping*—Use a mixture containing 40 to 60 weight % sulfur, the remainder being ground fire clay or other suitable inert material passing a No. 100 (150-µm) sieve with or without plasticizer. The casting surface plate requirements shall be as described in 7.2.3. Place four 1-in. (25.4-mm) square steel bars on the surface plate to form a rectangular mold approximately ½ in. (12.7 mm) greater in either inside dimension than the specimen. Heat the sulfur mixture in a thermostatically controlled heating pot to a temperature sufficient to maintain fluidity for a reasonable period of time after contact with the surface being capped. Take care to prevent overheating, and stir the liquid in the pot just before use. Fill the mold to a depth of ¼ in. (6.35 mm) with molten sulfur material. Place the surface of the unit to be capped quickly in the liquid, and hold the specimen so that its vertical axis is at right angles to the capping surface. The thickness of the caps shall be approximately the same. Allow the unit to remain undisturbed until solidification is complete. Allow the caps to cool for a minimum of 2 h before testing the specimens.

7.3 *Procedure*:

7.3.1 Test brick specimens flatwise (that is, the load shall be applied in the direction of the depth of the brick). Test structural clay tile specimens in a position such that the load is applied in the same direction as in service. Center the specimens under the spherical upper bearing within 1/16 in. (1.59 mm).

7.3.2 The testing machine shall conform to the requirements of Practices E 4.

7.3.3 The upper bearing shall be a spherically seated, hardened metal block firmly attached at the center of the upper head of the machine. The center of the sphere shall lie at the center of the surface of the block in contact with the specimen. The block shall be closely held in its spherical seat, but shall be free to turn in any direction, and its perimeter shall have at least ¼ in. (6.35 mm) clearance from the head to allow for specimens whose bearing surfaces are not exactly parallel. The diameter of the bearing surface shall be at least 5 in. (127.00 mm). Use a hardened metal bearing block beneath the specimen to minimize wear of the lower platen of the machine. The bearing block surfaces intended for contact with the specimen shall have a hardness not less than HRC60 (HB 620). These surfaces shall not depart from plane surfaces by more than 0.001 in. (0.03 mm). When the bearing area of the spherical bearing block is not sufficient to cover the area of the specimen, place a steel plate with surfaces machined to true planes within ± 0.001 in. (0.03 mm), and with a thickness equal to at least one third of the distance from the edge of the spherical bearing to the most distant corner between the spherical bearing block and the capped specimen.

7.3.4 *Speed of Testing*—Apply the load, up to one half of the expected maximum load, at any convenient rate, after which, adjust the controls of the machine so that the remaining load is applied at a uniform rate in not less than 1 nor more than 2 min.

7.4 *Calculation and Report*:

7.4.1 Calculate and report the compressive strength of each specimen to the nearest 10 psi (0.01 MPa) as follows:

$$\text{Compressive strength, } C = W / A \quad (2)$$

where:

C = compressive strength of the specimen, lb/in.^{2} (or kg/cm^2) (or Pa·10^4),
W = maximum load, lbf, (or kgf) (or N), indicated by the testing machine, and
A = average of the gross areas of the upper and lower bearing surfaces of the specimen, in.^{2} (or cm^2).

NOTE 4—When compressive strength is to be based on net area (example: clay floor tile), substitute for A in the above formula the net area, in in.^{2} (or cm^2), of the fired clay in the section of minimum area perpendicular to the direction of the load.

7.4.2 Calculate and report the average of the compressive strength determinations to the nearest 10 psi (0.1 MPa).

8. Absorption

8.1 *Accuracy of Weighings*:

8.1.1 *Brick*—The scale or balance used shall have a capacity of not less than 2000 g, and shall be sensitive to 0.5 g.

8.1.2 *Tile*—The balance used shall be sensitive to within 0.2 % of the weight of the smallest specimen tested.

8.2 *Test Specimens*:

8.2.1 *Brick*—The test specimens shall consist of half brick conforming to the requirements of 7.1.1. Five specimens shall be tested.

8.2.2 *Tile*—The specimens for the absorption test shall consist of five tile or three representative pieces from each of these five tile. If small pieces are used, take two from the shell and one from an interior web, the weight of each piece being not less than 227 g. The specimens shall have had their rough edges or loose particles ground off and, if taken from tile that have been subjected to compressive strength tests, specimens shall be free of cracks due to failure in compression.

8.3 *5-h and 24-h Submersion Tests*:

8.3.1 *Procedure*:

8.3.1.1 Dry and cool the test specimens in accordance with 5.1.1 and 5.1.2 and weigh each one.

8.3.1.2 *Saturation*—Submerge the dry, cooled specimen, without preliminary partial immersion, in clean water (soft, distilled or rain water) at 60 to 86°F (15.5 to 30°C) for the

specified time. Remove the specimen, wipe off the surface water with a damp cloth and weigh the specimen. Complete weighing of each specimen within 5 min after removing the specimen from the bath.

8.3.2 *Calculation and Report*:

8.3.2.1 Calculate and report the cold water absorption of each specimen to the nearest 0.1 % as follows:

$$\text{Absorption, \%} = 100(W_s - W_d) / W_d \quad (3)$$

where:

W_d = dry weight of the specimen, and
W_s = saturated weight of the specimen after submersion in cold water.

8.3.2.2 Calculate and report the average cold water absorption of all specimens to the nearest 0.1 %.

8.4 *1-h, 2-h, and 5-h Boiling Tests*:

8.4.1 *Test Specimens*—The test specimens shall be the same five specimens used in the 5-h or 24-h cold-water submersion test where required and shall be used in the state of saturation existing at the completion of that test.

8.4.2 *Procedure*:

8.4.2.1 Return the specimen that has been subjected to the cold-water submersion to the bath, and subject it to the boiling test as described in 8.4.2.2.

8.4.2.2 Submerge the specimen in clean water (soft, distilled or rain water) at 60 to 86°F (15.5 to 30°C) in such a manner that water circulates freely on all sides of the specimen. Heat the water to boiling, within 1 h, boil continuously for specified time, and then allow to cool to 60 to 86°F (15.5 to 30°C) by natural loss of heat. Remove the specimen, wipe off the surface water with a damp cloth, and weigh the specimen. Complete weighing of each specimen within 5 min after removing the specimen from the bath.

8.4.2.3 If the tank is equipped with a drain so that water at 60 to 86°F (15.5 to 30°C) passes through the tank continuously and at such a rate that a complete change of water takes place in not more than 2 min, make weighings at the end of 1 h.

8.4.3 *Calculation and Report*:

8.4.3.1 Calculate and report the boiling water absorption of each specimen to the nearest 0.1 % as follows:

$$\text{Absorption, \%} = 100(W_b - W_d) / W_d \quad (4)$$

where:

W_d = dry weight of the specimen, and
W_b = saturated weight of the specimen after submersion in boiling water.

8.4.3.2 Calculate and report the average boiling water absorption of all specimens to the nearest 0.1 %.

8.5 *Saturation Coefficient*:

8.5.1 Calculate and report the saturation coefficient of each specimen to the nearest 0.01 as follows:

$$\text{Saturation coefficient} = W_s^{\,2} - W_d / W_b^{\,5} - W_d \quad (5)$$

where:

W_d = dry weight of the specimen,
$W_s^{\,2}$ = saturated weight of the specimen after 24-h submersion in cold water, and
$W_b^{\,5}$ = saturated weight of the specimen after 5-h submersion in boiling water.

8.5.2 Calculate and report the average saturation coefficient of all specimens to the nearest 0.01.

9. Freezing and Thawing

9.1 *Apparatus*:

9.1.1 *Compressor, Freezing Chamber, and Circulator* of such design and capacity that the temperature of the air in the freezing chamber will not exceed 16°F (−9°C) 1 h after introducing the maximum charge of units, initially at a temperature not exceeding 90°F (32°C).

9.1.2 *Trays and Containers*, shallow, metal, having an inside depth of 1½ ± ½ in. (38.1 ± 12.7 mm), and of suitable strength and size so that the tray with a charge of frozen units can be removed from the freezing chamber by one man.

9.1.3 *Balance*, having a capacity of not less than 2000 g and sensitive to 0.5 g.

9.1.4 *Drying Oven* that provides a free circulation of air through the oven and is capable of maintaining a temperature between 230 and 239°F (110 and 115°C).

9.1.5 *Thawing Tank* of such dimensions as to permit complete submersion of the specimens in their trays. Adequate means shall be provided so that the water in the tank may be kept at a temperature of 75 ± 10°F (24 ± 5.5°C).

9.1.6 *Drying Room*, maintained at a temperature of 75 ± 15°F (24 ± 8°C), with a relative humidity between 30 and 70 %, and free from drafts.

9.2 *Test Specimens*:

9.2.1 *Brick*—The test specimens shall consist of half brick with approximately plane and parallel ends. If necessary, the rough ends may be smoothed by trimming off a thin section with a masonry saw. The specimens shall be free from shattering or unsoundness, visually observed, resulting from the flexure or from the absorption tests. Additionally, prepare specimens by removing all loosely adhering particles, sand or edge shards from the surface or cores. Test five specimens.

9.2.2 *Structural Clay Tile*—The test specimens shall consist of five tile or of a cell not less than 4 in. (101.6 mm) in length sawed from each of the five tile.

9.3 *Procedure*:

9.3.1 Dry and cool the test specimens as prescribed in 5.1.1 and 5.1.2 and weigh and record the dry weight of each.

9.3.2 Carefully examine each specimen for cracks. A crack is defined as a fissure or separation visible to a person with normal vision from a distance of one foot under an illumination of not less than 50 fc. Mark each crack its full length with an indelible felt marking pen.

9.3.3 Submerge the test specimens in the water of the thawing tank for 4 ± ½ h.

9.3.4 Remove the specimens from the thawing tank and stand them in the freezing trays with one of their head faces down. Head face is defined as the end surfaces of a whole rectangular brick (which have the smallest area). A space of at least ½ in. (12.7 mm) shall separate the specimens as placed in the tray. Pour sufficient water into the trays so that each specimen stands in ½ in. depth of water and then place the trays and their contents in the freezing chamber for 20 ± 1 h.

9.3.5 Remove the trays from the freezing chamber after 20 ± 1 h and totally immerse them and their contents in the water of the thawing tank for 4 ± ½ h.

9.3.6 Freeze the test specimens by the procedure in 9.3.4 one cycle each day of the normal work week. Following the 4 ± ½ h thawing after the last freeze-thaw cycle of the normal work week, remove the specimens from the trays and store them for 44 ± 1 h in the drying room. Do not stack or pile units. Provide a space of at least 1 in. (25.4 mm) between all specimens. Following this period of air drying, inspect the specimens, submerge them in the water of the thawing tank for 4 ± ½ h, and again subject them to a normal week of freezing and thawing cycles in accordance with 9.3.4 and 9.3.5. When a normal 5-day work week is interrupted, put specimens into a drying cycle which may extend past the 44 ± 1 h drying time outlined in the procedures of this section.

9.3.7 Continue the alternations of drying and submersion in water for 4 ± ½ h, followed by 5 cycles of freezing and thawing or the number of cycles needed to complete a normal work week, until a total of 50 cycles of freezing and thawing has been completed. Stop the test if the test specimen develops a crack as defined in 9.4.3, breaks, or appears to have lost more than 3 % of its original weight by disintegration as judged by visual inspection.

9.3.8 After completion of 50 cycles, or when the test specimen has been withdrawn from test as a result of disintegration, dry and weigh the specimen as prescribed in 9.3.1.

9.4 *Calculations, Examination, Rating and Report*:

9.4.1 *Calculation*—Calculate the loss in weight as a percentage of the original weight of the dried specimen.

9.4.2 *Examination*—Re-examine the surface of the specimens for cracks (see 9.3.2) and record the presence of any new cracks developed during the freezing-thawing testing procedure. Measure and record the length of the new cracks. Examine the specimen for gradual disintegration during the freeze-thaw process.

9.4.3 *Rating*—A specimen is considered to fail the freezing and thawing test under any one of three circumstances:

9.4.3.1 *Weight Loss*—A gradual disintegration resulting in a weight loss of greater than that required by the referenced unit specification for the appropriate classification.

9.4.3.2 *Breakage*—The specimen separates into two or more pieces, or

9.4.3.3 *Cracking*—A specimen develops a crack during the freezing and thawing procedure that exceeds in length the minimum dimension of the specimen.

If none of the above circumstances occur, the specimens are considered to pass the freezing and thawing test.

9.4.4 *Report*—The report shall state whether the sample passed or failed the test. Any failures shall include the rating and the reason for classification as a failure and the number of cycles causing failure in the event failure occurs prior to 50 cycles.

10. Initial Rate of Absorption (Suction) (Laboratory Test)

10.1 *Apparatus*:

10.1.1 *Trays or Containers*—Watertight trays or containers, having an inside depth of not less than ½ in. (12.7 mm), and of such length and width that an area of not less than 300 in.^{2} (1935.5 cm^2) of water surface is provided. The bottom of the tray shall provide a plane, horizontal upper surface, when suitably supported, so that an area not less than 8 in. (203.2 mm) in length by 6 in. (152.4 mm) in width will be level when tested by a spirit level.

10.1.2 *Supports for Brick*—Two noncorrodible metal supports consisting of bars between 5 and 6 in. (127.00 and 152.5 mm) in length, having triangular, half-round, or rectangular cross sections such that the thickness (height) will be approximately ¼ in. (6.35 mm). The thickness of the two bars shall agree within 0.001 in. (0.03 mm) and, if the bars are rectangular in cross section, their width shall not exceed 5⁄16 in. (1.94 mm).

10.1.3 *Means for Maintaining Constant Water Level*—Suitable means for controlling the water level above the upper surface of the supports for the brick within ±0.01 in. (0.25 mm) (see Note 5), including means for adding water to the tray at a rate corresponding to the rate of removal by the brick undergoing test (see Note 6). For use in checking the adequacy of the method of controlling the rate of flow of the added water, a reference brick or half brick shall be provided whose displacement in ⅛ in. (3.18 mm) of water corresponds to the brick or half brick to be tested within ±2.5 %. Completely submerge the reference brick in water for not less than 3 h preceding its use.

NOTE 5—A suitable means for obtaining accuracy in control of the water level may be provided by attaching to the end of one of the bars two stiff metal wires that project upward and return, terminating in points; one of which is ⅛ − 0.01 in. (3.18 − 0.25 mm) and the other ⅛ + 0.01 in. (3.18 + 0.25 mm) above the upper surface or edge of the bar. Such precise adjustment is obtainable by the use of depth plates or a micrometer microscope. When the water level with respect to the upper surface or edge of the bar is adjusted so that the lower point dimples the water surface when viewed by reflected light and the upper point is not in contact with the water, the water level is within the limits specified. Any other suitable means for fixing an maintaining a constant depth of immersion may be used if equivalent accuracy is obtained. As an example of such other suitable means, there may be mentioned the use of rigid supports movable with respect to the water level.

NOTE 6—A rubber tube leading from a siphon or gravity feed and closed by a spring clip will provide a suitable manual control. The so-called "chicken-feed" devices as a rule lack sensitivity and do not operate with the very small changes in water level permissible in this test.

10.1.4 *Balance*, having a capacity of not less than 3000 g, and sensitive to 0.5 g.

10.1.5 *Drying Oven*, conforming to the requirements of 9.1.4.

10.1.6 *Constant-Temperature Room*, maintained at a temperature of 70 ± 2.5°F (21 ± 1.4°C).

10.1.7 *Timing Device*—A suitable timing device, preferably a stop watch or stop clock, which shall indicate a time of 1 min to the nearest 1 s.

10.2 *Test Specimens*, consisting of whole brick. Five specimens shall be tested.

10.3 *Procedure*:

10.3.1 The initial rate of absorption shall be determined for the test specimen as specified, either oven-dried or ambient air-dried. If not specified, the initial rate of absorption shall be determined for the test specimens oven-dried. Dry and cool the test specimens in accordance with the applicable procedures

10.3.1.1 or 10.3.1.2. Complete the test procedure in accordance with 10.3.2, 10.3.3, and 10.3.4.

NOTE 7—There is no correlated relationship between the value of initial rate of absorption for ambient air-dried and oven-dried units. The test methods provide different information.

10.3.1.1 *Oven-dried Procedure*—Dry and cool the test specimens in accordance with 5.1.1 and 5.1.2.

10.3.1.2 *Ambient Air-dried Procedure*—Store units unstacked, with separate placement in a ventilated room maintained at a temperature of 75 ± 15°F (24 ± 8°C) with a relative humidity between 30 % and 70 % for a period of 4 h, with a current of air from an electric fan passing over them for a period of at least 2 h. Continue until two successive weighings at intervals of 2 h show an increment of loss not greater than 0.2 % of the last previously determined weight of the specimen.

10.3.2 Measure to the nearest 0.05 in. (1.27 mm) the length and width of the flatwise surface of the test specimen of rectangular units or determine the area of other shapes to similar accuracy that will be in contact with the water. Weigh the specimen to the nearest 0.5 g.

10.3.3 Adjust the position of the tray for the absorption test so that the upper surface of its bottom will be level when tested by a spirit level, and set the saturated reference brick (10.1.3) in place on top of the supports. Add water until the water level is ⅛ ± 0.01 in. (3.18 ± 0.25 mm) above the top of the supports. When testing tile with scored bed surfaces, the depth of water level is ⅛ ± 0.01 in. plus the depth of scores.

10.3.4 After removal of the reference brick, set the test brick in place flatwise, counting zero time as the moment of contact of the brick with the water. During the period of contact (1 min ± 1 s) keep the water level within the prescribed limits by adding water as required. At the end of 1 min ± 1 s, lift the brick from contact with the water, wipe off the surface water with a damp cloth, and reweigh the brick to the nearest 0.5 g. Wiping shall be completed within 10 s of removal from contact with the water, and weighing shall be completed within 2 min.

NOTE 8—Place the brick in contact with the water quickly, but without splashing. Set the brick in position with a rocking motion to avoid the entrapping of air on its under surface. Test brick with frogs or depressions in one flatwise surface with the frog or depression uppermost. Test molded brick with the struck face down.

10.4 *Calculation and Report*:

10.4.1 The difference in weight in grams between the initial and final weighings is the weight in grams of water absorbed by the brick during 1-min contact with the water. If the area of its flatwise surface (length times width) does not differ more than ±0.75 in.^{2} (4.84 cm^2) (±2.5 %) from 30 in.^{2} (193.55 cm^2), report the gain in weight of each specimen to the nearest 0.1 g, as its initial rate of absorption in 1 min.

10.4.2 If the area of its flatwise surface differs more than ± 0.75 in.^{2} (4.84 cm^2) (±2.5 %) from 30 in.^{2} (193.55 cm^2), calculate the equivalent gain in weight from 30 in.^{2} (193.55 cm^2) of each specimen to the nearest 0.1 g as follows:

$$X = 30\ W / LB \text{ (metric } X = 193.55\ W / LB) \quad (6)$$

where:

X = gain in weight corrected to basis of 30 in.^{2} (193.55 cm^2) flatwise area,
W = actual gain in weight of specimen, g,
L = length of specimen, in., (cm), and
B = width of specimen, in., (cm).

10.4.3 Report the corrected gain in weight, X, of each specimen to the nearest 0.1 g, as the initial rate of absorption in 1 min.

10.4.4 If the test specimen is a cored brick, calculate the net area and substitute for LB in the equation given in 10.4.2. Report the corrected gain in weight, X, of each specimen to the nearest 0.1 g, as the initial rate of absorption in 1 min.

10.4.5 If specimen is non-prismatic, calculate the net area by suitable geometric means and substitute for LB in the equation given in 10.4.2.

10.5 Calculate and report the average initial rate of absorption of all specimens tested to the nearest 0.1 g/min/30 in.^{2} (193.55 cm^2).

10.6 Report the method of drying as oven-dried (in accordance with 10.3.1.1) or ambient air-dried (in accordance with 10.3.1.2).

11. Efflorescence

11.1 *Apparatus*:

11.1.1 *Trays and Containers*—Watertight shallow pans or trays made of corrosion-resistant metal or other material that will not provide soluble salts when in contact with distilled water containing leachings from brick. The pan shall be of such dimensions that it will provide not less than a 1-in. (25.4-mm) depth of water. Unless the pan provides an area such that the total volume of water is large in comparison with the amount evaporated each day, suitable apparatus shall be provided for keeping a constant level of water in the pan.

11.1.2 *Drying Room*, conforming to the requirements of 9.1.6.

11.1.3 *Drying Oven*, conforming to the requirements of 9.1.4.

11.2 *Test Specimens*:

11.2.1 The sample shall consist of ten full-size brick.

11.2.2 The ten specimens shall be sorted into five pairs so that both specimens of each pair will have the same appearance as nearly as possible.

11.3 *Preparation of Specimens*—Remove by brushing any adhering dirt that might be mistaken for efflorescence. Dry the specimens and cool them as prescribed in 5.1.1 and 5.1.2.

11.3.1 Calculate the weight per unit area of each specimen as follows:

$$W_a = \frac{nW_d}{A_{fa1} + A_{fa2}} \quad (7)$$

Where:
W_a = weight per unit area of the specimen, lb/sq. ft. (kg. m^2),
n = number of faces of the specimen (1 for split tile units or 2 for all other units)
Wd = dry weight of the specimen, lb (kg),
A_{fa1} = area (height × length) of finished face of specimen, sq. ft. (m^2), and
A_{fa2} = area (height × length) of back face of specimen, sq. ft. (m^2).

11.3.2 Report the results of Eq. 7 in 11.3.1 separately for each unit and the average for all specimens tested.

11.4 *Procedure*:

11.4.1 Set one specimen from each of the five pairs, on end, partially immersed in distilled water to a depth of approximately 1 in. (25.4 mm) for 7 days in the drying room. When several specimens are tested in the same container, separate the individual specimens by a spacing of at least 2 in. (50.8 mm).

NOTE 9—Do not test specimens from different sources simultaneously in the same container, because specimens with a considerable content of soluble salts may contaminate salt-free specimens.

NOTE 10—Empty and clean the pans or trays after each test.

11.4.2 Store the second specimen from each of the five pairs in the drying room without contact with water.

11.4.3 At the end of 7 days, inspect the first set of specimens and then dry both sets in the drying oven for 24 h.

11.5 *Examination and Rating*—After drying, examine and compare each pair of specimens, observing the top and all four faces of each specimen from a distance of 10 ft. (3 m) under an illumination of not less than 50 footcandles (538.2 lm/m^2) by an observer with normal vision. If under these conditions no difference is noted, report the rating as "not effloresced." If a perceptible difference due to efflorescence is noted under these conditions, report the rating as "effloresced." Report the appearance and distribution of the efflorescence.

11.6 *Precision and Bias*—No information is presented about either the precision or bias of the test method for efflorescence because the test result is nonquantitative.

12. Weight per Unit Area

12.1 *Apparatus*—A scale or balance sensitive to within 0.2 % of the weight of the smallest specimen.

12.2 *Test Specimens*—Weigh five dry full size structural clay tile units (see 5.1.1).

12.3 *Calculation and Report*:

12.3.1 Calculate the weight per unit area of a specimen by dividing the total weight in pounds by the average area in square feet of the two faces of the unit as normally laid in a wall.

12.3.2 Report results separately for each specimen to the nearest 1 g, with the average to the nearest 1 g.

13. Measurement of Size

13.1 *Apparatus*—Either a 1-ft (or metric) steel rule, graduated in 1/32-in. (or 1-mm) divisions, or a gage or caliper having a scale ranging from 1 to 12 in. (25 to 300 mm), and having parallel jaws, shall be used for measuring the individual units. Steel rules or calipers of corresponding accuracy and size required shall be used for measurement of larger brick, solid masonry units, and tile.

13.2 *Test Specimens*—Measure ten whole dry full-size units. These units shall be representative of the lot and shall include the extremes of color range and size as determined by visual inspection. (The same samples may be used for determining efflorescence and other properties.)

13.3 *Individual Measurements of Width, Length, and Height*—Measure the width across both ends and both beds from the midpoints of the edges bounding the faces. Record these four measurements to the nearest 1/32 in. (1 mm) and record the average to the nearest 1/64 in. (0.5 mm) as the width. Measure the length along both beds and along both faces from the midpoints of the edges bounding the ends. Record these four measurements to the nearest 1/32 in. (1 mm) and record the average to the nearest 1/64 in. (0.5 mm) as the length. Measure the height across both faces and both ends from the midpoints of the edges bounding the beds. Record these four measurements to the nearest 1/32 in. (1 mm) and record the average to the nearest 1/64 in. (0.5 mm) as the height. Use the apparatus described in 13.1. Retest by the same method when required.

13.4 *Report*—Report the average width, length, and height of each specimen tested to the nearest 1/32 in. (0.8 mm).

14. Measurement of Warpage

14.1 *Apparatus*:

14.1.1 *Steel Straightedge*:

14.1.2 *Rule or Measuring Wedge*—A steel rule graduated from one end in 1/32-in. (or 1-mm) divisions, or alternatively, a steel measuring wedge 2.5 in. (60 mm) in length by 0.5 in. (12.5 mm) in width by 0.5 in. (12.5 mm) in thickness at one end and tapered, starting at a line 0.5 in. (12.5 mm) from one end, to zero thickness at the other end. The wedge shall be graduated in 1/32-in. (or 1-mm) divisions and numbered to show the thickness of the wedge between the base, *AB*, and the slope, *AC*, Fig. 1.

14.1.3 *Flat Surface*, of steel or glass, not less than 12 by 12 in. (305 by 305 mm) and plane to within 0.001 in. (0.025 mm).

14.2 *Sampling*—Use the sample of ten units selected for determination of size.

14.3 *Preparation of Samples*—Test the specimens as received, except remove any adhering dirt by brushing.

14.4 *Procedure*:

14.4.1 *Concave Surfaces*—Where the warpage to be measured is of a surface and is concave, place the straightedge lengthwise or diagonally along the surface to be measured, selecting the location that gives the greatest departure from straightness. Select the greatest distance from the unit surface to the straightedge. Using the steel rule or wedge, measure this distance to the nearest 1/32 in. (1 mm), and record as the concave warpage of the surface.

14.4.2 *Concave Edges*—Where the warpage to be measured is of an edge and is concave, place the straightedge between the ends of the concave edge to be measured. Select the greatest

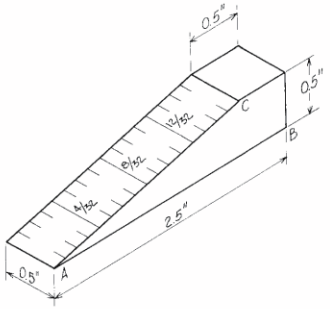

FIG. 1 Measuring Wedge

distance from the unit edge to the straightedge. Using the steel rule or wedge, measure this distance to the nearest 1/32 in. (1 mm), and record as the concave warpage of the edge.

14.4.3 *Convex Surfaces*—When the warpage to be measured is of a surface and is convex, place the unit with the convex surface in contact with a plane surface and with the corners approximately equidistant from the plane surface. Using the steel rule or wedge, measure the distance to the nearest 1/32 in. (1 mm) of each of the four corners from the plane surface. Record the average of the four measurements as the convex warpage of the unit.

14.4.4 *Convex Edges*—Where the warpage to be measured is of an edge and is convex, place the straightedge between the ends of the convex edge. Select the greatest distance from the unit edge to the straightedge. Using the steel rule or wedge, measure this distance to the nearest 1/32 in. (1 mm) and record as the convex warpage of the edge.

14.5 *Report*—Report all recorded warpage measurements of each specimen tested to the nearest 1/32 in. (0.8 mm).

15. Measurement of Length Change

15.1 *Apparatus*—A dial micrometer or other suitable measuring device graduated to read in 0.0001-in. (or 0.001-mm) increments, mounted on a stand suitable for holding the specimen in such a manner that reproducible results can be obtained, shall be used for measuring specimen length. Provisions shall be made to permit changing the position of the dial micrometer on its mounting rod so as to accommodate large variations in specimen size. The base of the stand and the tip of the dial micrometer shall have a conical depression to accept a 1/4-in. (6.35-mm) steel ball. A suitable reference instrument shall be provided for checking the measuring device.

15.2 *Preparation of Specimen*—Remove the ends of deeply textured specimens to the depth of the texture by cutting perpendicular to the length and parallel to each other. Drill a hole in each end of the specimen with a 1/4-in. (6.35-mm) carbide drill. Drill these holes at the intersection of the two diagonals from the corners. Place 1/4-in. (6.35-mm) steel balls in these depressions by cementing in place with a calcium aluminate cement. Any equivalent method for establishing the reference length is permissible.

15.3 *Procedure*—Mark the specimen for identification and measure to the nearest 0.0001 in. (or 0.001 mm) in a controlled environment and make subsequent measurements in the same controlled environment, ±2°F and ±5 % relative humidity. Record the temperature and relative humidity. Apply a reference mark to the specimen for orientation in the measuring device. Check the measuring device with the reference instrument before each series of measurements.

15.4 *Report*—When more than one specimen is tested, calculate and report the average length change of all specimens to the nearest 0.0001 in. (0.001 mm). The report shall include all individual recordings as well as the recorded laboratory temperature and relative humidity.

16. Initial Rate of Absorption (Suction)—Field Test

16.1 *Scope*—This test method is intended to serve as a volumetric means of determining the initial rate of absorption (IRA) of any size brick when weighing determination, described in Section 10 of these test methods, is impractical. This test method is applicable to assess the need for wetting the brick. This test method is performed on specimens taken from the field with no modification of moisture content, therefore, the IRA determined by this test method may differ from the IRA determined by the laboratory test method in Section 10, which requires drying the specimens.

16.2 *Apparatus*:

16.2.1 *Absorption Test Pan*—A watertight, rectangular pan, constructed of noncorroding material, with a flat, rigid bottom and inside depth of about 1½ in. (38.1 mm). The inside length and width of the pan shall exceed the length and width of the tested brick by a minimum of 3 in. (76.2 mm) but not more than 5 in. (127.0 mm).

16.2.2 *Brick Supports*—Two noncorroding rectangular bars, 1/4 in. (6.4 mm) in height and width and 1 in. (25.4 mm) shorter than the inside width of the pan in length. The brick supports can be placed on the bottom of the pan just before the test or permanently affixed to the bottom of the pan. The space between the supports should be about 4 in. (101.6 mm) shorter than the length of the tested brick. A device indicating the desired water level can be permanently attached to the end of one of the brick supports or suspended from the top of the pan (see Fig. 2 (a) and (b)). Any other device of equivalent accuracy for controlling the required water level, 1/8 in. (3.2 mm) above the brick supports, can be used in place of that depicted in Fig. 2.

16.2.3 *Timing Device*—A suitable timing device that shall indicate a time of 1 min to the nearest 1 s.

16.2.4 *Squeeze Bottle*—A plastic squeeze bottle, 100 mL capacity.

16.2.5 *Graduated Cylinder*—A plastic or glass graduated measuring cylinder, 100 mL capacity.

16.3 *Test Specimens*—Select six whole brick in accordance with the requirements of Paragraph 4.1.

16.4 *Procedure*:

16.4.1 Completely immerse one brick specimen in a container of water for 2 h.

16.4.2 Measure to the nearest 1/16 in. (1.6 mm) the length and width of the five remaining specimens at the surface that will be in contact with water. If the test specimens are cored, determine the area of the cores at the same surface.

16.4.3 Pre-wet and drain the absorption pan and place it on a flat, level surface.

16.4.4 Remove the pre-wetted specimen from the container, shake off the surface water, and place the specimen on brick

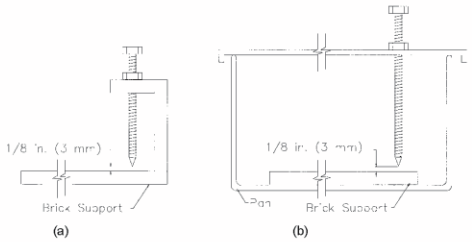

FIG. 2 Water Level Indicators

supports in the pan. Pour water into the pan until the water reaches a level ⅛ in. (3.2 mm) above the brick supports. (If using a pointed level water indicator, pour water into the pan until the water makes a minimum contact (dimpling effect).) Remove the pre-wetted brick, and tilt the brick sharply so that one corner serves as a drip point for clinging surface water to return to the pan. A gentle shake of the brick may be necessary to make the last drop fall. Put the pre-wetted brick back into the container of water.

16.4.5 Using the graduated cylinder, fill the squeeze bottle with exactly 100 mL of water.

16.4.6 Set the first test specimen squarely on the brick supports, counting zero time as the moment the brick contacts the water. At the end of 1 min ± 1 s lift the test specimen from water and tilt the brick sharply so that one corner serves as a drip point for clinging surface water to return to the pan. A gentle shake of the brick may be necessary to make the last drop fall.

16.4.6.1 Continue setting the remaining test specimens into the pan in the same way until all five specimens are tested. During the test add water to the pan, using the squeeze bottle, to keep the water level approximately constant at the ⅛ in. depth. Refill the squeeze bottle with 100 mL of water when empty, recording each refill.

16.4.6.2 After the last specimen is tested, place the pre-wetted brick back in the pan and restore the original level with water from the squeeze bottle.

NOTE 11—Place the brick in contact with the water quickly, but without splashing. Set the brick in position with a rocking motion to avoid the entrapping of air on its under surface. Test brick with frogs or depressions in one flatwise surface with the frog or depression uppermost. Test molded brick with the struck face down.

16.4.7 Using the graduated cylinder, measure the volume of water remaining in the squeeze bottle.

16.5 *Calculation and Report*:

16.5.1 The number of refills plus the first full bottle, times 100 mL, minus the volume of water remaining in the squeeze bottle, is the total measured volume of water in millilitres absorbed by the five specimens.

$$V_t = 100\,(n + 1) - V_r \tag{8}$$

where:

V_t = total measured volume of water absorbed by all tested specimens, mL,
n = the number of squeeze bottle refills, and
V_r = the volume of water remaining in the squeeze bottle, mL.

16.5.2 When the average net surface area in contact with water of a single specimen (sum of net surface areas divided by the number of specimens) differs by ±0.75 in.^{2} (4.84 cm^2) or less from 30 in.^{2} (193.5 cm^2), report the total measured absorbed volume of water divided by five, the number of tested specimens, as the IRA (Field) in g/min/30 in.^{2}

$$\text{IRA (Field)} = \frac{V_t}{5} \tag{9}$$

16.5.3 If the average net surface area in contact with water differs by more than ±0.75 in.^{2} (4.84 cm^2) from 30 in.^{2} (193.5 cm^2), calculate the equivalent volume in 1 min for 30 in.^{2} (193.5 cm^2) of surface as follows:

$$V_c = \frac{30\,V_t}{A_n}\left(\text{metric } V_c = \frac{193.5\,V_t}{A_n}\right) \tag{10}$$

where:

V_c = average volume of absorbed water by a specimen, corrected to basis of 30 in.^{2} (193.5 cm^2) of surface, mL, and
A_n = sum of net surface areas in contact with water of all tested specimens, in.^{2} (cm^2).

16.5.4 *Report*—Report the corrected volume (V_c) as the IRA (Field) in g/l min/30 in.^{2}

16.6 *Precision and Bias*—Insufficient data is currently available for a precision and bias statement.

17. Measurement of Void Area in Cored Units

17.1 *Apparatus*:

17.1.1 *Steel Rule or Calipers*—As described in 13.1.

17.1.2 *Graduated Cylinder*—A glass cylinder with a capacity of 500 mL.

17.1.3 *Paper*—A sheet of smooth, hard-finish paper not less than 24 by 24 in. (610 by 610 mm).

17.1.4 *Sand*—500 mL of clean, dry sand.

17.1.5 *Steel Straightedge*.

17.1.6 *Flat Surface*—A level, flat, smooth, clean dry surface.

17.1.7 *Brush*—A soft-bristle brush.

17.1.8 *Neoprene Mat*—24 by 24 in. (610 by 610 mm) open-cell neoprene sponge ¼ in. (6.4 mm) in thickness.

17.1.9 *Balance*—See 10.1.4.

17.2 *Test Specimens*—Use of a sample of ten units selected as described for the determination of size (The samples taken for the determination of size may be used).

17.3 *Preparation of Samples*—Test the specimens as received, except remove any adhering dirt by brushing.

17.4 *Procedure*:

17.4.1 Measure and record the length, width, and depth of the unit as described for the determination of size.

17.4.2 Place the unit to be tested bed down (cores vertical) on the sheet of paper that has been spread over the neoprene mat on the flat surface.

17.4.3 Fill the cores with sand, allowing the sand to fall naturally. Do not work the sand into the cores. Using the steel straightedge, bring the level of the sand in the cores down to the top of the unit. With the brush, remove all excess sand from the top of the unit and from the paper sheet.

17.4.4 Lifting the unit up, allow all of the sand in the cores to fall on the sheet of paper.

17.4.5 Transfer the sand from the sheet of paper to the balance, weighing and recording to the nearest 0.5 g.

17.4.6 With a separate portion of the sand, fill a 500 mL cylinder to the exact 500 mL graduation by allowing the sand to fall naturally and without shaking or vibrating the cylinder. Transfer this sand to the balance, weighing and recording to the nearest 0.5 g.

17.5 *Calculation and Report*:

17.5.1 Determine the volume of sand held in the test unit as follows:

$$V_s = \frac{500\ \text{mL}}{S_c} \times S_u \quad (11)$$

where:
V_s = volume of sand held in test unit,
S_c = weight, in grams, of 500 mL sand contained in graduated cylinder, and
S_u = weight in grams of sand held in test unit.

17.5.2 Determine the percentage of void as follows:

$$\%\ \text{Void area} = \frac{V_s}{V_u} \times \frac{1}{16.4} \times 100 \quad (12)$$

where:
V_s = volume of sand determined in 17.5.1, mL, and
V_u = length × width × depth recorded in 17.4.1, in.^{3}

17.5.3 Report the results of Eq 12 in 17.5.2 for each specimen to the nearest 1 %, as the unit's percentage of void area.

18. Measurement of Void Area In Deep Frogged Units

NOTE 12—The area measured corresponds to a section located ⅜ in. (9.5 mm) distant from the voided bed of the units.

18.1 *Apparatus*:

18.1.1 *Steel Rule or Gage or Calipers* (inside and outside)— as described in 13.1.

18.1.2 *Steel Straightedge*.

18.1.3 *Marking Pen or Scribe*.

18.2 *Test Specimens*—Use a sample of 10 units selected as described for the determination of size. (The samples taken for the determination of size may be used.)

18.3 *Preparation of Sample*—Test the specimens as received except remove any adhering dirt by brushing.

18.4 *Procedure*:

18.4.1 Measure the length along both faces and the width along both ends at a distance of ⅜ in. (9.5 mm) down from the bed containing the deep frogs. Record the measurements to the nearest 1/32 in. (1 mm). Record the average of the two length measurements to the nearest 1/32 in. (1 mm) as the length of the unit and the average of the two width measurements to the nearest 1/32 in. (1 mm) as the width of the unit.

18.4.2 With the steel straightedge parallel to the length of the unit and centered over the deep frog or frogs, inscribe a mark on both faces of the frog ⅜ in. (9.5 mm) below the underside of the steel straightedge (mark 1 on Fig. 3). With the steel straightedge parallel to the width of the unit and centered over the deep frog, inscribe a mark on both faces of each frog ⅜ in. (9.5 mm) below the underside of the steel straightedge (mark 2 on Fig. 3).

18.4.3 Measure and record to the nearest 1/32 in. (1 mm) the distance between the inscribed marks on a line parallel to the length of the unit for each frog, and measure and record to the nearest 1/32 in. (1 mm) the distance between the inscribed marks on a line parallel to the width of the unit for each frog.

18.5 *Calculations and Report*:

18.5.1 Using the recorded length and width measurements calculate the gross area of the unit (A_u) in the plane of the unit ⅜ in. (9.5 mm) down from the frogged bed.

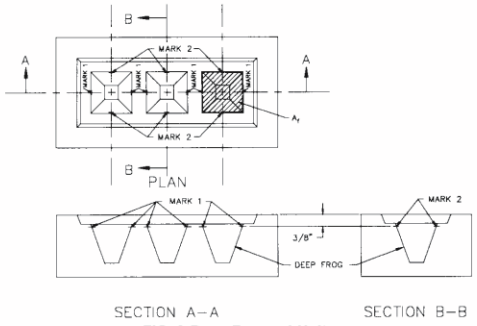

FIG. 3 Deep Frogged Units

18.5.2 Using the distance between the inscribed marks calculate the inside area of each deep frog (A_f) in the plane of the unit ⅜ in. (9.5 mm) down from the frogged bed (see Fig. 3).

18.5.3 Determine the percentage of void as follows:

$$\%\ \text{Void area} = \frac{\Sigma A_f \times 100}{A_u} \quad (13)$$

where:
ΣA_f = sum of the inside area of the deep frogs, and
A_u = gross area of unit.

18.5.4 Report the results of the equation in 18.5.3 for each specimen to the nearest 1 %, as the unit's percentage of void area.

19. Measurement of Out of Square

19.1 *Apparatus*:

19.1.1 *Steel Rule or Calipers*, as described in 13.1.

19.1.2 *Steel Carpenter's Square*.

19.2 *Procedure*:

19.2.1 Place one leg of a carpenter's square adjacent to the length of the unit when laid as a stretcher. Align the leg of the square parallel to the length of the unit by having the corners of the face of the unit in contact with the leg of the square.

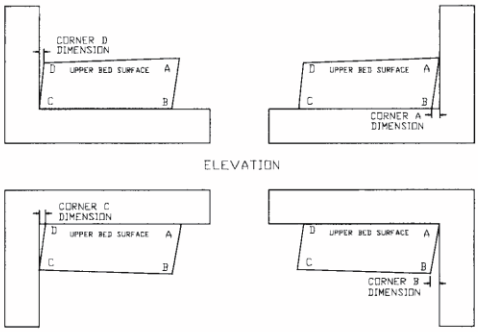

FIG. 4 Out-of-Square Measurements

Locate the square parallel to and at or within ¼ in. (6.4 mm) of the face to be exposed. See Fig. 5.

19.2.2 Measure the deviation due to the departure from the 90° angle at each corner of the exposed face of the unit. Record the measurement to the nearest 1/32 in. (0.8 mm) for each corner. See Fig. 4.

19.3 *Report*—Report the recorded measurements for each specimen tested to the nearest 1/32 in. (0.8 mm) as the unit's deviation from square.

20. Measurement of Shell and Web Thickness

20.1 *Apparatus*—a caliper rule graduated in not more than 1/64 in. (0.4 mm) divisions and having parallel jaws not less than ½ in. (12.7 mm) in length.

20.2 *Test Specimens*—Use a sample of five units as described for the measurement of size (samples taken for the determination of size are permitted to be used).

20.3 *Preparation of Samples*—Remove any shards or other projections interfering with measurement of the minimum parallel distance of two surfaces.

20.4 *Procedure*—For each unit, measure the shell thicknesses and, when required, the web thicknesses at the thinnest point of each element ½ in. (12.7 mm) into the unit from either direction and record to the nearest division of the caliper.

NOTE 13—Current ASTM specifications for solid masonry units from clay or shale do not include minimum web thickness requirements.

21. Breaking Load

21.1 *Test Specimens*—The test specimens shall consist of whole full-size units (see 5.1.1). Five such specimens shall be tested.

21.2 *Procedure*:

21.2.1 Test units that have been dried according to 5.1.1.

21.2.2 Unless specified and reported otherwise, support the test specimen flatwise (that is, apply the load in the direction of the height of the unit). The load shall be placed at the midspan, within 1/16 in. (2 mm) of the center. If the specimens have frogs or depressions, place the specimen so that the frogs or depressions are on the underside of the specimen. The supports for the specimen shall be solid steel rods 1 ± ⅜ in. (25.4 ± 10 mm) in diameter placed ½ ± 1/16 in. (12.7 ± 2 mm) from each end. The length of each support shall be at least equal to the width of the specimen. See Fig. 6.

21.2.3 Apply the load to the upper surface of the specimen through a steel bearing plate ¼ in. (6.4 mm) in thickness and 1½ in. (38.1 mm) in width and of a length at least equal to the width of the specimen.

21.2.4 *Speed of Testing*—The rate of loading shall not exceed 2000 lbf (8896 N)/min. This requirement shall be considered as being met if the speed of the moving head of the testing machine immediately prior to application of the load is not more than 0.05 in. (1.27 mm)/min.

21.3 *Report*:

21.3.1 Record the unit dimensions and span length.

21.3.2 Record the transverse breaking load, P, of each unit to the nearest lb (N).

21.3.3 Calculate and record the breaking load per width of unit as $p = P/w$ for each unit, lb/in. (N/mm). Report the average of the breaking loads per width of all the specimens tested as the breaking load of the lot.

22. Keywords

22.1 absorption; compressive strength; efflorescence; freezing and thawing; initial rate of absorption; length change; modulus of rupture; out-of-square; sampling; size; void area; warpage

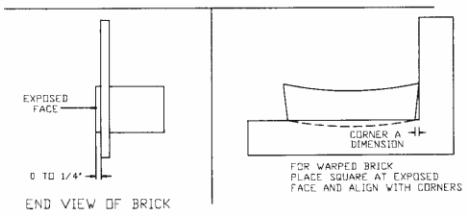

FIG. 5 Location of Carpenter's Square

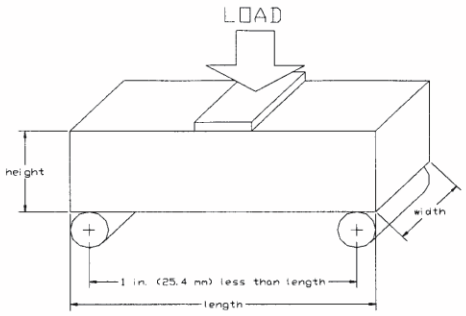

FIG. 6 Breaking Load Configuration

SUMMARY OF CHANGES

Committee C15 has identified the location of selected changes to this standard since C 67 – 03 that may impact the use of this standard. (Approved June 10, 2003)

(1) Section 4.1 was revised to require removal of foreign materials from all test specimens.

Committee C15 has identified the location of selected changes to this standard since C 67 – 02c that may impact the use of this standard.

(1) Section 8.3.6 was revised to remove the alternate test procedure of conducting the test over 50 consecutive days.
(2) Sections 11.3.1 and 11.3.2 were changed to include an equation for calculating the weight per unit area.
(3) Section 4.1 was modified to provide clarity of specimen selection.

II. Rilem VB Salt Test

T E S T Nº V.1b

CRYSTALLISATION TEST BY TOTAL IMMERSION (FOR TREATED STONE)

1.- DEFINITION OF MEASURED PROPERTY

Resistance to a test that closely simulates the crystallisation phenomena occuring under natural environmental conditions.

2.- AIM

The destruction effect of sodium sulphate, filling the pore space of porous material, is used in the test : it is based on the volume increase during recrystallisation from Na_2SO_4 to $Na_2SO_4 \cdot 10\ H_2O$ in water solution at a temperature of 32.7°C.

3.- APPLICATION

Crystallisation test by total immersion is a laboratory test preferable for stone samples treated by impregnation.

This is a accelerated weathering test to assess the resistance to weathering agents, especially to damage caused by salts, of impregnated stones in comparison with the same kind of stone without impregnation. The test is also useful for showing the behaviour during weathering of the contact zone between the impregnated surface layer and the mass of the stone not reached by the impregnation.

4.- TEST METHOD

4.1 - Sample preparation

4.1.1. Number and dimensions of samples

A representative number of stone specimens for every type of impregnation under test and of reference specimens without impregnation of the same kind of stone have to be prepared. Cube with 50 mm sides should be used as standard size. If samples of other (larger) dimensions (for example : when the impregnation zone is very deep) are used, this has to be recorded in the test report.

4.1.2. Method of impregnation

For cases that the impregnation material under test is applied to the stone specimens in the laboratory the following method of impregnation should be used : the dry test cubes are impregnated on their four vertical planes for example by brushing. In cases of stones with bedding, the bedding has to be orientated vertically, i.e. two of the impregnated cube planes are parallel and two are across the bedding. The impregnation of the cubes on only four sides, leaving the bottom and top free of impregnation, guarantees the same degree of salt soaking for all test specimens, independent of the hydrophobic or highly pre-filling properties of the impregnation under test. It also imitates the conditions in impregnated facades, where soluble salts are present in the walls, attacking the impregnated surface from behind.

The test procedure should not be started sooner than four weeks after impregnation.

4.1.3. Drying of samples

The samples are dried at 60° ± 5°Ctill constant mass. The constant mass is reached when the difference between two successive weighings (at a time interval of 24 h) is not more than 0.1 % of the mass of the sample, determined with an accuracy of 0.01% of this mass. The samples are cooled at room temperature.

The drying temperature of 60° C ± 5° C is chosen instead of a higher one in order to avoid deterioration of the organic materials used to treat the stone.

4.2 - Test Procedure

During the test procedure cycles of soaking the samples in salt solution and drying them are repeated. A test cycle consists of :

- immersion in 10 % Na_2SO_4 solution for 2 hours
- drying on a preheated oven at 60° C for 19 hours*
- cooling to 20°C within 3 hours
- control weighing after every second cycle

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* in the case of very fine grained stones, a longer drying period may be necessary.

The test cycle is repeated 15 times or until a nearly complete destruction of the samples is achieved. After the final cycle, the salt is removed from the samples by 7 day immersion in often renewed tap water. The samples then are dried at 60° C to constant weight for determination of the final weight at 20°C.

5.- EXPRESSION AND INTERPRETATION OF RESULTS

The relative resistance of the samples is expressed as their loss of weight after 15 test cycles or as the number of cycles required for their complete destruction. The loss of weight of the samples is calculated as the difference between their initial weight and their final weight as a percentage of their initial weight.

More informative in particular is the process of the sample destruction during the test. It can be recorded in diagrams; the loss of weight of the specimens after every weight - controlled cycle expressed as a percentage of their initial weight is plotted against the respective number of the test cycles. Results obtained in separate test series can only be compared if the sample dimensions and the drying and soaking temperature have been the same. An additionnal visual and photographic recording of the kind of damages of the samples during the test, especially of damages in the impregnation zones and in the interface to the unimpregnated part of the stone (for example the flaking off of the impregnation zone) can be used as important hints to assess the behaviour of the different impregnations applied to stone materials under test.

III. ASTM C279-88

Designation: C 279 – 88 (Reapproved 2007)

Standard Specification for Chemical-Resistant Masonry Units[1]

This standard is issued under the fixed designation C 279; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ε) indicates an editorial change since the last revision or reapproval.

1. Scope

1.1 This specification covers solid, kiln fired brick and tile made from clay, shale, or mixtures thereof, suitable for indoor and outdoor use in masonry construction subjected to chemical environments.

1.2 The physical and chemical properties of brick and tile differ from supplier to supplier, mainly because their composition is determined by the source of raw materials. Regardless of the differences, brick and tile are considered to be of three types as follows:

1.2.1 *Type I*—For use where low absorption and high acid resistance are not major factors.

1.2.2 *Type II*—For use where lower absorption and higher acid resistance are required.

1.2.3 *Type III*—For use where minimum absorption and maximum acid resistance are required.

NOTE 1—*Types I, II,* and *III* may not differ significantly in thermal shock resistance. The suitability of a given brick, for a particular application should be determined at the time of purchase by agreement between the purchaser and the supplier.

NOTE 2—*Types I* and *III* were formerly designated Type "H" and "L" respectively.

1.3 The values stated in inch-pound units are to be regarded as the standard. The values given in parentheses are for information only.

1.4 *This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

2. Referenced Documents

2.1 *ASTM Standards:* [2]

C 20 Test Methods for Apparent Porosity, Water Absorption, Apparent Specific Gravity, and Bulk Density of Burned Refractory Brick and Shapes by Boiling Water

C 67 Test Methods for Sampling and Testing Brick and Structural Clay Tile

C 397 Practice for Use of Chemically Setting Chemical-Resistant Silicate and Silica Mortars

C 723 Practice for Chemical-Resistant Resin Grouts for Brick or Tile

E 11 Specification for Wire Cloth and Sieves for Testing Purposes

3. Physical Properties

3.1 *Strength*—The brick and tile when tested in accordance with Test Methods C 67 shall conform to the requirements for modulus of rupture (flexural strength) for the type specified, as prescribed in Table 1.

3.2 *Water Absorption*—The brick and tile when tested in accordance with Test Methods C 20 shall conform to the requirements for water absorption (based on the 2 h boil) for the type specified, as prescribed in Table 1.

3.3 *Sizes*—The sizes of the brick and tile shall be as specified by the purchaser. The length, width, and depth measurements of the brick or tile shall be within ±3 % of the specified dimensions when tested in accordance with Test Methods C 67.

3.4 *Warpage*—The brick and tile when tested in accordance with Test Methods C 67 shall conform to the requirements as shown in Table 2. (**Warning**—The above tolerances may not be consistent with the recommended mortar joint sizes contained in Practices C 397 and C 723. If brick or tile with tighter tolerances than those described in 3.3 or 3.4 are required, the purchaser shall negotiate such requirements with the manufacturer.)

3.5 *Surface Textures*—Brick or tile surfaces should be textured in order to promote better bonding. Texturing may be accomplished by scoring, wire cutting, matting, or other means consistent with a manufacturer's process. If texturing is done, the protrusion or indentation shall not exceed ⅛ in. (3 mm) in depth.

[1] This specification is under the jurisdiction of ASTM Committee C15 on Manufactured Masonry Units and is the direct responsibility of Subcommittee C15.02 on Brick and Structural Clay Tile.

Current edition approved June 1, 2007. Published July 2007. Originally approved in 1951. Last previous edition approved in 2001 as C 279 – 88 (2001).

[2] For referenced ASTM standards, visit the ASTM website, www.astm.org, or contact ASTM Customer Service at service@astm.org. For *Annual Book of ASTM Standards* volume information, refer to the standard's Document Summary page on the ASTM website.

TABLE 1 Physical and Chemical Requirements for Brick and Tile

Designation	Modulus of Rupture (Brick or Tile Flatwise) min. psi (MPa)	Water Absorption Maximum % by 2 h Boiling Test		H_2SO_4 Solubility Maximum % Weight Loss
	Average of 5 Brick or Tile Low Individual	Average of 5 Brick or Tile High Individual		Average of 5 Brick or Tile
Type I	1250 (8.6) 1000 (6.9)	6.0	7.0	20
Type II	1250 (8.6) 1000 (6.9)	4.0	5.0	12
Type III	1250 (8.6) 1000 (6.9)	1.0	1.5	8

TABLE 2 Tolerances on Warpage

Minimum Face Dimensions, inches (mm)	Maximum Permissible Warpage, inches (mm)
8 and under (203) and under	3/32 (2.4)
over 8–12 (203 to 305), incl	1/8 (3.2)
over 12–16 (305 to 406), incl	5/32 (4.0)

4. Significance and Use

4.1 The brick and tile covered herein are intended essentially for use in chemical environments where resistance to thermal shock may or may not be a consideration. The brick and tile are normally used with chemical-resistant mortars.

5. Precision and Bias

5.1 A statement on precision and bias will be added at a later date.

6. Visual Inspection

6.1 The brick and tile shall be free of open surface laminations or cracks which would impair the performance of the construction.

NOTE 3—Open laminations or cracks within the brick or tile observed in the brick or tile cut or broken during testing, should be noted with their size and number indicated as part of the test report. If internal open laminations or cracks, or both, are reported, the purchaser shall determine the suitability of such brick or tile for his application.

6.2 *Black Heart*—Brick or tile when broken may have a dark area that has a steely appearance and is sharply delineated from the surrounding normal color of the brick. It is known as *black heart* or *black core*. Black heart is generally the result of the reduction of iron minerals during the firing process. Its presence, regardless of size, in brick or tile which otherwise meet the physical and chemical requirements of this specification, shall not be cause for rejection.

7. Sulfuric Acid Solubility Test

7.1 *Apparatus*:

7.1.1 *Crusher*, jaw-type.

7.1.2 *Sieves*, ¼-in. (6.3-mm) and No. 4 (4.75-mm) sieves (equivalent to 3-mesh and 4-mesh sieves, respectively, in the Tyler series), conforming to Specification E 11.

7.1.3 *Mechanical Shaking Device*, producing a lateral and vertical motion of the sieve, accompanied by a jarring action so as to keep the sample moving continuously over the surface of the sieve.

7.1.4 *Drying Oven.*

7.1.5 *Analytical Balance and Weights*, 0.01-g sensitivity.

7.1.6 *Desiccator.*

7.1.7 *Erlenmeyer Flask*, 750-mL, of heat-resistant and chemically resistant glass.

7.1.8 *Water-Cooled Condenser.*

7.1.9 *Hot Plate.*

7.1.10 *Fritted-Glass Funnel*, fine porosity.

7.1.11 *Suction Pump.*

7.2 *Preparation of Sample*—Prepare the sample from at least five masonry units selected in accordance with Test Methods C 67. Remove and discard the skin surface from a quarter of each unit selected and crush the remaining pieces in a jaw-type crusher, with the jaws set so that the grain size of the product ranges from material retained on a ¼-in. (6.3-mm) sieve to material passing a No. 4 (4.75-mm) sieve. Reduce this material either by mixing and quartering or by a mechanical splitter to approximately a 1000-g sample, and screen in a mechanical shaking device for 15 min, using the No. 3 [6.75-mm] and No. 4 [4.75-mm] sieves. Thoroughly mix the portion of the material passing the No. 3 [6.75-mm] sieve and remaining on the No. 4 [4.75-mm] sieve (Note 4), and then quarter down to obtain two 50-g samples. Dry these samples in a drying oven at 240°F (120°C) for at least 16 h, and then cool in a desiccator.

NOTE 4—Although it is recognized that some types of material tend to break down in a manner yielding various-shaped particles, no attempt shall be made at hand selection.

7.3 *Procedure*—Transfer each of the 50-g samples, weighed to the nearest 0.01 g, and 250 mL of sulfuric acid (sp gr 1.706, or 78 weight % 60° Baumé) to 750-mL Erlenmeyer flasks. Insert water-cooled condensers and boil on hot plates for 48 h (Note 5). Cool the flasks and contents sufficiently to permit handling, and decant the solutions through fritted-glass funnels with the aid of suction, retaining the samples in the flasks. Add about 250 mL of water to the flasks, boil for 10 min, and decant with the aid of suction through the same funnels as used previously. Repeat this washing procedure three times. On the fourth decantation of wash water, transfer the samples to the funnels, using hot water to aid in the transfer. Dry the funnels and contents in an oven at 240°F (120°C) for at least 16 h, and cool in a desiccator. Remove material from the funnels, brushing out the fines if necessary, and weigh to the nearest 0.01 g.

NOTE 5—Regulate the temperature of the hot plate so as to maintain a gentle boiling solution avoiding any considerable agitation of the sample. The use of a variable transformer in series with the hot plate is suggested.

7.4 *Calculation and Report*—Calculate the loss in weight as a percentage of the original weight. Make duplicate determinations and report an average of the two results to the nearest 0.1 %.

8. Keywords

8.1 absorption; acid resistance; acid solubility; ceramic; chemical-resistant; masonry; physical properties; solid brick

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