SAHC2014 – 9th International Conference on
Structural Analysis of Historical Constructions
F. Peña & M. Chávez (eds.)
Mexico City, Mexico, 14–17 October 2014

PRELIMINARY RESEARCHES ON LIME-BASED INJECTION GROUTS FOR CONSERVATION OF EARTHEN ARCHITECTURAL HERITAGE AND RUINS

Shibing DAI [1], Xiaoniu FANG[2], Jun HAN[3] and Zhangyong HU[4]

[1] Architecture Conservation Laboratory, CAUP of Tongji University
Room 113 Wenyuan Building, No. 1239, Siping Road, Shanghai CN-200092, PR China
e-mail: ds_build@163.com

[2] Jingangshan University
No. 28, Xueyuan Road, Ji'an, Jiangxi Province, CN-343009, PR China
fangxiaoniu@163.com

[3] Shanghai Geological Engineering Investigation Co. Ltd, No.930 Lingshi Road , Shanghai CN-200072, PR China
13901709830@vip.163.com

[4] Shanghai DS Building Materials Co Ltd.
No.102 Xubei Road, Huaxin Town, Qingpu District, Shanghai CN-201705, PR China
hzy_materials@163.com

Keywords: Earthen Heritage Architecture, Cracking, Detachment, Grouting, Quick lime, Natural Hydraulic Lime, Micro Lime.

Abstract. *The most common defects of earthen architectural heritage and ruins are cracking and detachment. The technical criteria for appropriate grouts are mechanically and chemically compatible with original earthen substrate. Various formulations of earthen materials mixed with air limes, natural hydraulic limes and micro lime have been preliminarily tested. The compatibility of injection grouts are evaluated according to injectability, shrinkage, adhesion to earthen substrates and retreatbility. It can be concluded that for cavity filling the mix of quick lime with earth is most suitable. For the surface reattachment or cracking filling of earthen ruins without pressure injection, the mixture of natural hydraulic lime with fine earth seems to be to be most effective. Alcohol-based micro lime shows a surprised result to reattach delaminated plasters.*

1 INTRODUCTION

Earthen architectural heritage end ruins are the most relevant cultural heritage in China not only because of construction tradition, but also because of miserable history. Most of remarkable earth-timer architecture were burnt down during the wars and only as ruins passed to our generation. Besides earth castles in Fujian, earthen city wall in Pingyao, which are listed as world heritage, but there are still plenty of architecture in historic towns and streets. It is also known that there are mural paintings were found directly on earth surface.

Figure 1: Mural painting on top of lime washed loess earth in Taiyuan

The conservation of earthen architecture and ruins become the one of most challenging tasks in the conservation fields. Among them crack filling to stabilize the structure or back filling to strengthen the delaminated decorative finishing are most relevant.

Until now, there are few publications on the compatible injection grouts. L. Shekede etc. proposed some basic criteria for compatible earth repair materials [1,2]. From 2007 to 2010 injection grouts based on quick lime were tested and applied to small slice of city wall of Pingyao, Shanxi province [3].

This paper summarizes the latest results of national research projects.

2 TYPES AND CAUSES OF CRACKS AND DETACHMENTS

The cracking and detachments vary from dimension and causes.

2.1 Cracking due to shrinkage or wrong intervention

The most common defects of earthen architecture are cracking and detachments of decorative surface. The typical cracks are caused by shrinkage (Fig. 2)

Figure 2: Cracks due to shrinkage
(Left: archaeological ruin in Chengdu Jinsha Museum,Right: Pingyao City wall from compacted earth)

The test on the vertical linal shrinkage (VS) according to China Standard [4] has showed that the soils from Grand Bao En Temple Ruins deformed very differently. The hammed soils or soil with brick debris show low shrinkage, while the soft slightly hammed soil without any debris show high shrinkage up to 37mm/m, while the natural soil ranges between (Figure3).

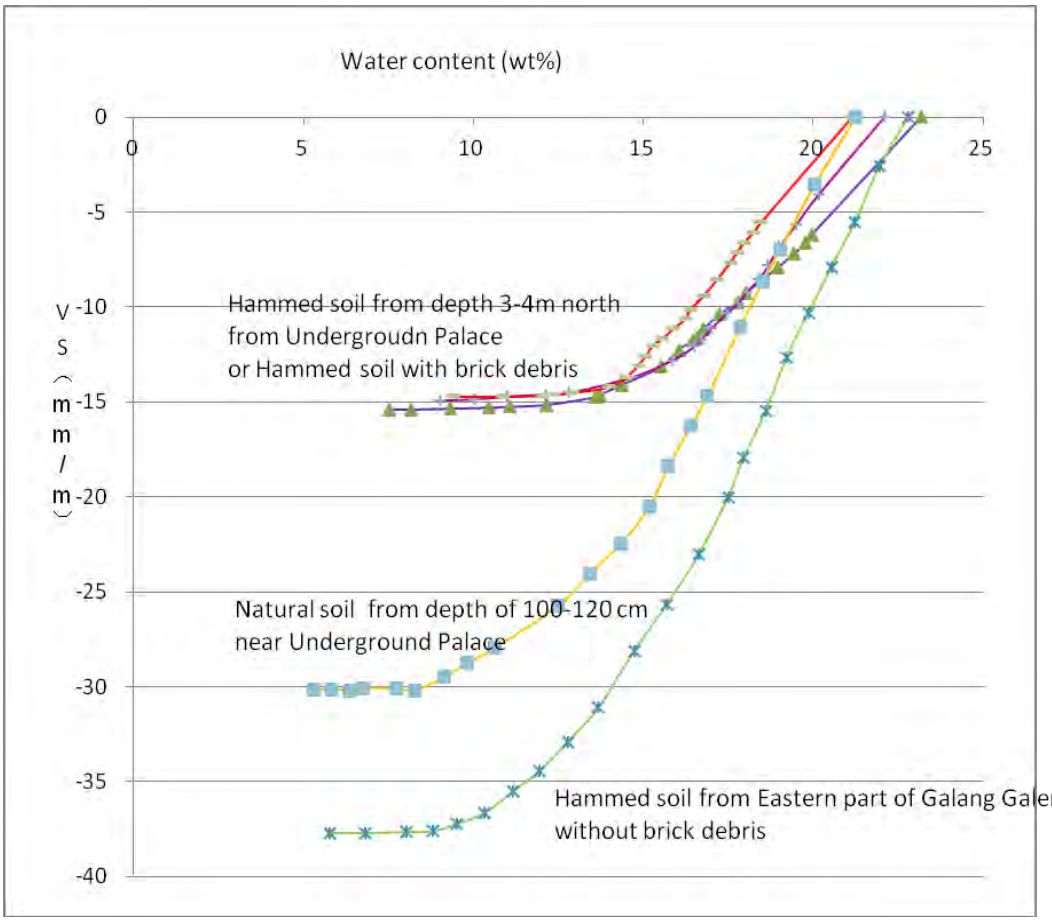

Figure 3: Shrinkage curves of various earths from Grand Bao En Temple, Nanjing

So it can be expected that once the soils in the area of ruin dry out, they will crack in different range and may affect not only the image but also the stability.

The second common cracks are of structural performance. A complicated crack system has been identified within the ancient city wall of Pingyao (Figure4) and most of them become rain water cannel and may cause collapse of entire wall. One of restoration measures is to fill

such cracks and voids with water resistant fillers, which, however shall be both mechanically and chemically compatible with existing old earth materials.

Figure 4: Complicated crack system within the city wall of Pingyao

It is also not uncommon that cracks are caused by intervention during the function changes (Figure 5).

Figure 5: Cracks within hammed earth wall caused by subsequent window opening

2.2 Detachment of decorative finishing on earthen substrate

The traditional architectural decorative finishing with historic values on earthen substrate is lime-based, either lime plasters or lime washes. In China, historic lime plaster of high quality is almost pure lime, with less addition of aggregates.

Mineralogically lime plaster is different from earth (Table 1) , which cause different thermal and hygric reaction between finishing and substrates.

Table1: Technical performances of lime plaster and earth materials

	Lime plaster	Earth	Comments
Mineralogy	Calcite (80-90%), seldom dolomite	Quartz, clay minerals	Almost totally different
Thermal expansion coefficient ($x10^{-6}K$) *	Calcite(parallel to c-axils: 23.3 , vertical to c-axils: -5.2)	Quartz: parallel to c-axils: 7.8 , vertical to c-axils: 14.3)	Different
Thermal conduction coefficient (w/（m·k） *	Calcite(parallel to c-axils: 5.0 , vertical to c-axils: 4.2)	Quartz: parallel to c-axils: 11.3 , vertical to c-axils: 6.5)	Different
Water stability	Form-table under saturation of liquid water	Become plastic when water content exceeds the plastic limits	Plasters will lose from earth substrate if the earth is affected by water (e. g. roof leakage or flooding)

* According to Dreyer,W.,1974

3 THEORETICAL IDEAS AND TECHNICAL CRITERIA

Grouting has long been implemented as measurement to reintegrate cracks and voids or to reattach plasters to their substrate. Although grouting is not a reversible, but it is re-treatable if the materials and application technique are compatible with earth materials.

3.1 Mechanical and chemical compatibility

The technical criteria for appropriate grouts are mechanically and chemically compatible with original earthen substrate. Among them, low strength, high water vapour permeability of the injection grouts are most relevant. Furthermore, they shall be injectable, injectability means the finished mix shall be injected with help of conventional injection machines or pressure-less. The performance of compatible grouts shall also be formulated according to injection technique and dimension of cracks or voids.

The grouts shall adhere to earthen substrate and retreatable in case of new cracking or delamination. Since most of cracks within earthen wall od between architectural finishing and earthen substrate are water transmission channel, so the filling grouts shall be water-resistant, or form-stable under water ingression, so that the grouts based on pure earth without binders are not under consideration in our research programmes.

3.2 Lime as binder

Clay minerals are binder as well, however it is not water resistant, so the grouts based on earth alone with other binders are preferably used in the regions of extreme dry climate.

Lime is most tradition binder to consolidate earth. Both practical observation (Figure 6) and laboratory tests show that air lime, chemically $Ca(OH)_2$ migrates and reacts with clay minerals consolating earth micro structure.

Figure 6: Earth sample taken from City wall top, which restored around 1979 showing migration of lime into earth

According to the latest European standards[5], limes are classified into 4 categories: air lime (CL or DL), hydraulic lime (HL), natural hydraulic lime (NHL) and formulated lime (FL). Natural hydraulic lime (NHL), formulated lime (FL) and hydraulic lime (HL) are limes with hydraulic properties. There are some research publications on the application of NHL for masonry grouting [6, 7 ,8], but very few on earthen grouting.

One of the latest approach is the nano-lime or micro-nano lime, which are produced either by top-down or bottom-up techniques[9, 10, 11]. The particle size of nano-lime produced by bottom-up technique can reach up to 100-200nm while the particle size of lime produce by top-down technique ranges form 1000-2000 nm (Figure 7). The advantage of nano-micro lime is that all those lime is based on alcohol, water –free to avoid efflorences caused by water evaporation. All conventional injection grouts are water-based, high volume water is brought into cracked substrate and may cause severe damages at the evaporation zones.

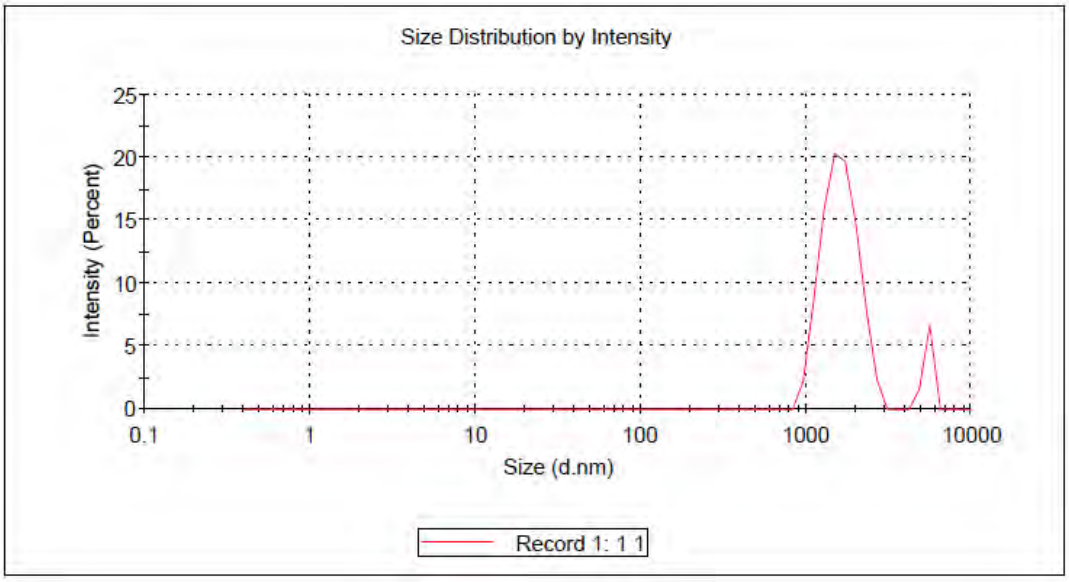

Figure 7: Ca(OH)2 -Size distribution of micro lime produced by top-down technique

4 PRACTICAL APPROACHES

From 2006 various formulations of earthen materials mixed with quicklime, dispersed air lime, dispersed micro lime and nano-lime (alcohol-based) and natural hydraulic limes with and without polymer additives have been tested and some of them have been applied for the conservation of earthen city walls and lime plasters. The compatibility of injection grouts are evaluated according to injectability, shrinkage, adhesion to earthen substrates and retreatbility. Most of those research projects have not been completed, only few results can be reported.

4.1 Injection grouts for void filling

In order to find proper injection and filling materials, 6 types of mixture have been tested (Li, personal information). Those mixtures are composed of

Group A: loess soil with acrylic emulsion;
Group B: loess soil with potassium water glass
Group C: Loess soil with cement-based soil stabilizer
Group D: Loess soil with quick lime
Group E: Loess soil with quick lime and sticky rice soup
Group F: loess soil with sticky rice soup

The injectability, setting time, shrinkage , compressive strength, adhesion between soil and injection grouts of all groups have been tested. The results showed that only the Group D and C can set within reasonable time and injectable. However the mixture of loess soil with quick lime shows less shrinkage than the mixture with cement-based soil stabilizer. The compressive strength loess soil and quick lime ranged from 0.36-0.39 N/mm^2 after app. 35 days, lower than the compacted loess soil, but water resistant. This formulation has been optimized and applied to a pilot trial area in 2007-2008(Figure 8), the results will be evaluated after 1-2 decades.

Figure 8: Voids filling with lime earth mix (photo by Li, Y. , personal information)

4.2 Injection grouts for crack filling

Three formulations are compared （Table 2）. All three grouts are injected with help of syringe into cracks simulated to Grand Bao En Temple ruins. The preliminary results showed that the mixture of modified NHL plus sieved soil adheres well to the earth substrate. The flow property of NHL mixture is also more satisfied that others.

Tablec2: injection filling grouts for earthen substrate

Types of binder	Mix ratio	Flow properties	Performance
NML, Nano-Micro Lime (500 g/L in ethanol)	3L +1.2kg earth（≦ 0.125mm）	Injectable, no separation within 10 min	Based on ethanol, water-free, no extra water will be brought into earth to avoid efflorences; good penetration to earth, but with high shrinkage
Lime putty , app. 50 wt% Ca(OH)2	3kg Lime putty +1.2kg earth（≦ 0.125mm） +1.2kg water	Injectable, no separation within 10 min	Water-based, may cause efflorences; moderate penetration into earth, high shrinkage
NHL2	Modified NHL2+1.2kg earth（≦ 0.125mm） +1.2kg water	Injectable, no separation within 10 min	Water-based, may cause efflorences; low penetration, but low shrinkage

Figure 9: Performance testing of three types grouts into earth cracks simulated to ruins

Figure 10: Cross section and carbonation of injection grouts based on limes

4.3 Injection grouts for reattachment

Historically and even today, bonding agent on the basis of acrylic resin dispersion is still practically used as back-filling to reattach the delaminated mural paintings and architectural finishing. Since acrylic dispersion reduces water vapour transmission, more compatible materials are required.

Within the national research projection for the conservation of the lime plaster in Jin Gang Shan, two mixtures of injection grouts are formulated in the laboratories:

Formulation 1(ML): micro lime with $Ca(OH)_2$- content of approx 50wt % , dispersed in ethanol , 50% dried fine soil (less than 0.25mm) , without any plasticifiers.

Formulation 2 (NHL): 99.5% NHL2 (Hessler-Kalk)+0.5% plasticizer + 50% dried fine soil (less than 0.25mm), all 3 components are premixed with dry mortar mixer for 10min than water are added in ratio of 55 wt% powder +45wt% water.

The prepared grouts were injected to artificially simulated hollow space between earthen substrate and delaminated plaster. The IR scanning showed that the microlime mix flowed much faster than that of NHL-mix. After hardening for 90 days the two samples are cross-cut, carbonation, adhesion between old materials and new grouts are macroscopic and microscopic studied.

The NML-mix was not carbonated, while the NHL-mix was completely carbonated (Figure 11).

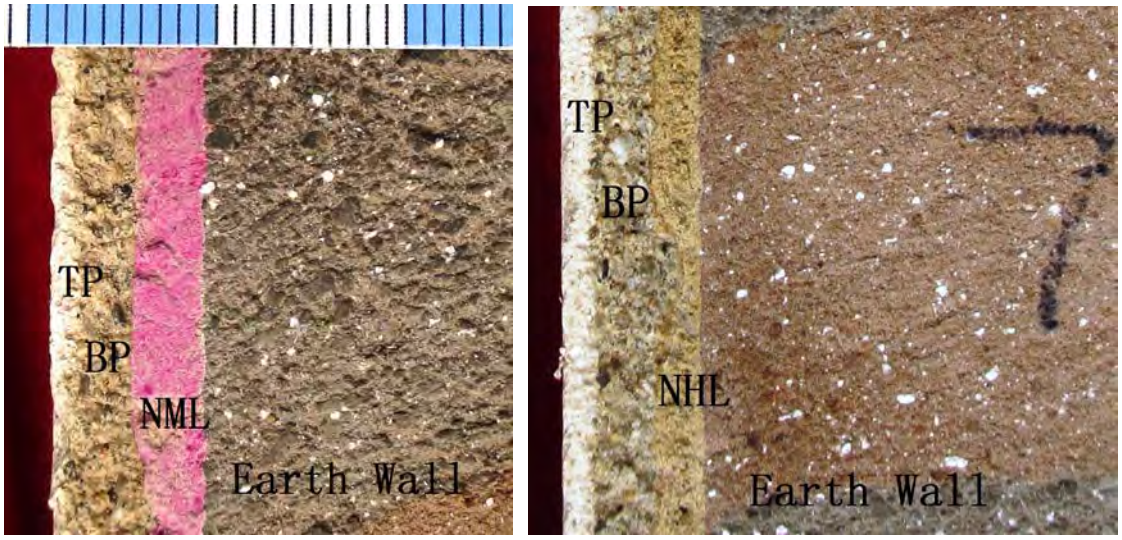

Figure 11: Carbonation of 2 injection grouts to attaché lime plaster
(TP: Old top lime plaster, BP: old base plaster, NHL: Injection grouts based on NHL2; NML: injection grout based on micro lime)

Under microscope it becomes clear that the adhesion between NML-grout and old plaster and earth wall are satisfied, however the adhesion of the new NML- grout between old plasters and earth wall is very well, the entire injection grout is homogeny, no separation from edge to middle or from bottom to top has been identified. There are only micro fissures vertically or to the edge between grout and old materials, but this will not affect the adhesion between old and new materials dramatically.

The NHL-grout adheres well directly to the old materials, but there is obviously separation along the contact zone in the grout, there are continuous fissures in the middle of the grout, which may be caused by shrinkage of the grout during setting.

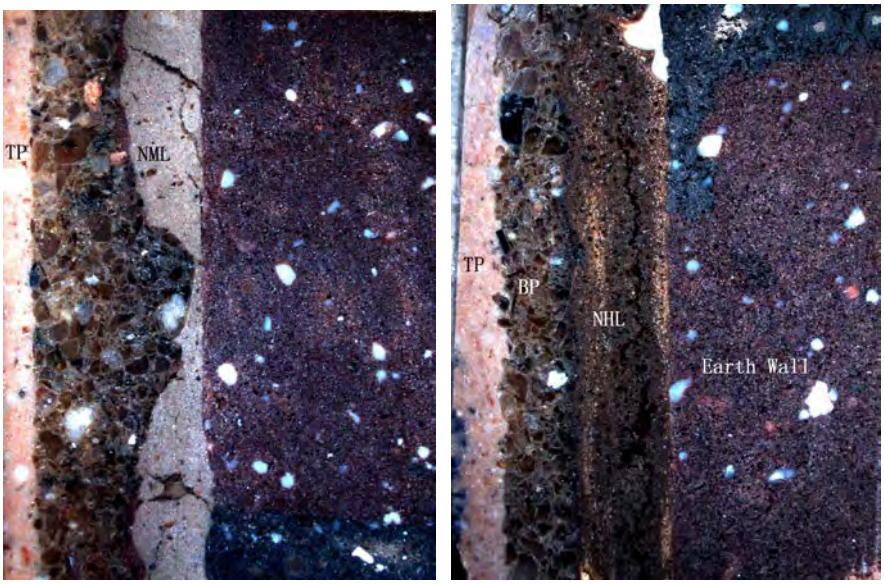

Figure12: Microscopic photos of the injection grouts（TP= old top lime plaster, BP=old base lime plaster, NML=new microlime injection grout, NHL= new natural hydraulic lime grout）

5 CONCLUSION AND DISCUSIONS

The most common defects of earthen architectural heritage and ruins are cracking and detachment. Cracking is either caused by differential shrinking of earth while losing the moisture after construction or excavation, or by structural intervention during the service period. Detachment of architectural surface on earthen substrate is mainly caused by thermal and hygric fluctuations. In the past two decades, injection grouts become the most favourable agent to fill cracks and voids within earthen structure or to reattach the delaminated cultural surfaces. The technical criteria for appropriate grouts are mechanically and chemically compatible with original earthen substrate. Furthermore, they shall be injectable, well adhere to earthen substrate and retreatable. The grouts shall be water-resistant, or form-stable under water ingression. The formulation of compatible grouts shall also be formulated according to injection technique. Various formulations of earthen materials mixed with quicklime, dispersed air lime, micro lime and natural hydraulic limes with and without polymer additives have been preliminarily tested and some of them have been applied for the conservation of earthen city walls and lime plasters. The compatibility of injection grouts are evaluated according to injectability, shrinkage, adhesion to earthen substrates and retreatbility. on the not completed research results, it can be concluded that for void filling of massive city wall or adobe masonry, the mix of quick lime with earth, which is one of the traditional lime technology in China is most suitable, which, however, shall be injection with low pressure. For the cracking filling of ruins, the mixture of NHL and soil seems to be a appropriate mixture. The more or less surprised result for re-attachment of delaminated lime plaster on earth substrate is the micro lime, which is dispersed in alcohol with particle size within 1-2 micrometer. The reasons are not clarified. The further research works will concentrate on natural hydraulic quick lime for crack filling. Both micro air lime and micro natural hydraulic lime will be interesting binder for reattachment of lime plaster or mural paintings.

6 ACCKNOLEGMENT

This article is completed under the financial support of China National “Twelfth Five Years” Key Research and Development Project granted No. 2012BAC11B01-2, the Architec-

ture Conservation Laboratory (part of the of the Key Laboratory of Ecology and Energy-saving Study of Dense Habitat, Tongji University, Ministry of Education, PR China) and other national research founds . The authors thank Yaxin Tang, Shangyou Zhu, Yuee Zhou, Lin Chen, Lei Li for assisting laboratory testing and mock-ups in collaboration with Shanghai DS Building Materials Co. Ltd. The authors express deep appreciation to other members of research team from Jinggangshan University and staffs of Jinggangshan Museum for sharing data and achievement in different fields. The authors also acknowledge Mr Li Hongsong, Mr Li Yuan for good cooperation during the research program to consolidate the Ancient City Wall of Pingyao, Shanghai Wenbao Construction Engineering Ltd. for completion of the pilot conservation test at the Liu's Ancestral Hall. Thanks Ms Zhou Yuee for correction of the manuscripts and improvement of the layout.

REFERENCES

[1] L. Shekede, S. Rickerby, Theory into practice: establishing compatibility in earth-based repair materials, L. Rainer and A. Bass Rivera eds. *Proceedings from the International Colloquium org. by the Getty Conservation Institute and the National Park Service,* Mesa Verde National Park, Colorado, USA, September **22-25**, 2004.

[2] Daniela Geyer, *Vergleiche Erprobung von Injektionsmassen auf Lehmbasis zur Konservieurng historischer Lehmbauten,* Diplomarbeit in der Stuienrichtung Konservierung und Restaurierung von Wandmalerei und Architekturfassung, Fachhochschule Potsdam, Germany , 2007.

[3] Hongsong Li, *Scientific researches for the strengthening design* of Ancient City Wall of Pingyao, Shanxi Province, un-published report, 2013.

[4] GB/T50123-1999 *Standard for Soil Test Methods,* Beijing , China,1999.

[5] BS EN 459-1: *Building lime. Definitions, specifications and conformity criteria.* UK: British-Adopted European Standard; 2011.

[6] S.B. Dai, J. H. Wang, Lime-based Materials and Practices for Surface Refitting of Cultural Heritage *in "Structural Analysis of Historic Constructions" ed. By Xianglin Gu & Xiaobin Song* Trans Tech Publications Inc. Switzerland. ISBN-13 978 -0-87849-239-8 Ad. Materials Research Vols 133-134 at http://www.scientific.net ,,2010.

[7] Shi-bing Dai, Building limes for cultural heritage conservation in China. *Heritage Science*, 1:25 at http://www.heritagesciencejournal.com/content/1/1/25, 2013.

[8] Bass, Angelyn. *Design and evaluation of hydraulic lime grouts for in situ reattachment of lime plaster to earthen walls*. Master's Thesis, University of Pennsylvania, 41, 1998.

[9] G. Ziegenbalg Colloidal calcium hydroxide – a new material for consolidation and conservation of carbonatic stones. In: Lukaszewicz E, Niemcewicz P (ed) *Proceedings of the 11th international congress on deterioration and conservation of stone,* 15-20 September 2008. Torun, Uniwersitet Mikolaja Kopernika, Torun, Poland, pp 1109–1115.

[10] CaloSil Injection Grout W, from: IBZ Salzchemie., Freiberg/Germany. Seewww.ibz-freiberg.de, 2013.

[11] A. Daehne and Ch. Herm, Calcium hydroxide nanosols for the consolidationof porous building materials - results from EU-STONECORE, *Heritage Science*, http://www.heritagesciencejournal.com/content/1/1/11, 2013.