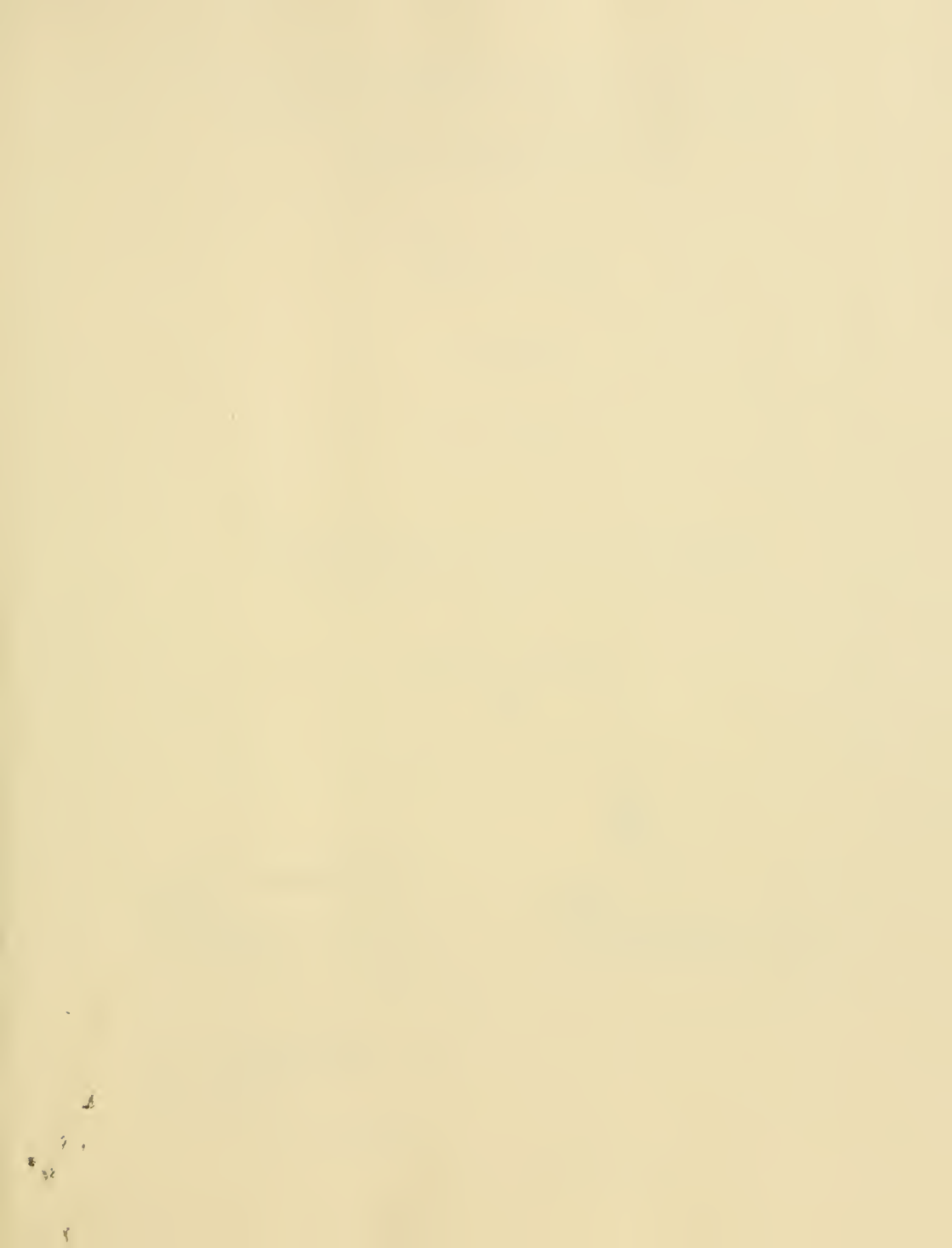AN INVESTIGATION OF SANDSTONE CONSOLIDATION METHOD

FOR THE NORTHERN GOPURA OF THE PHIMAI SANCTUARY,

A KHMER MONUMENT IN THAILAND
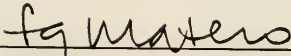
Nuanlak Watsantachad

A THESIS

in

Historic Preservation

Presented to the Faculties of the University of Pennsylvania in
Partial Fulfillment of the Requirements for the Degree of
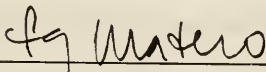
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CHAPTER I

INTRODUCTION

Brief History of Khmer Monument and its Restoration in Thailand

According to the remaining evidence and research by Henry Parmetier and the other French scholars, Khmer architectural activities began around the forth or fifth century AD.[1] Structures built earlier than this period vanished without record due to the perishable building material. From the excavations and study of the ruined elements from the earlier periods, Khmer architecture shows very close resemblances to and strong influence from Indian architecture, especially that of the Pallava and Gupta periods. Khmer and Indian architecture are based on the same religious beliefs— Hinduism and Buddhism. The combination between the assimilated and conventionalized Indian elements and the uniquely Khmer characteristics influenced by the two new local beliefs— the Devaraja and Buddharaja cults— have been found in the architecture since the ninth century. However, the spiritual concept of architecture remained the same as in the earlier period, i.e. to house an image of the deity or the Buddha.

Northeastern Thailand— also called "Isan"— has evidence of Khmer architecture dating to the sixth and seventh century A.D. When the Khmer Empire conquered the Indochina

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1 Ly Kim Long, *An Outline of Cambodian Architecture* (India: Bharatiya Vidya Prakashan, 1967), IV.

1

Penninsula (about 540-1230 A.D.), Thailand— called Siam at that period — was the border of the empire. The country was connected to the Empire's center by imperial roads that ran northwest from Anghor, crossed the Dang Rek Mountains, passed through Burirum Province— where the Phanom Rung and Muang Tum Sanctauries were located— and ended at Nakhon Ratchasima— where the Phimai Sanctuary was located. There were many Khmer monuments, built of sandstone, laterite, and fired brick, constructed along this road and in the surrounding territory. These monuments represented the sanctuary of Hindu gods. However, based on the Devaraja and Buddharaja cults, kings were the representatives of gods, so that the sanctuaries were also the spiritual places for "godlike" kings. According to the size, location, and function of the existing monuments, the Phimai, Phanom Rung, Phanom Wan, and Muang Tam Sanctuaries are the most predominant and important. (See Figure 1.1)

Due to their age, many of these monuments were in a near state of collapse when the survey projects began in the early twentieth century. Only about 60% of the structures were standing while the other 40% had collapsed. Although the condition assessment of the stones—sandstone and laterite—used as building material was quite good, the construction was unstable. (See Figure 1.2)
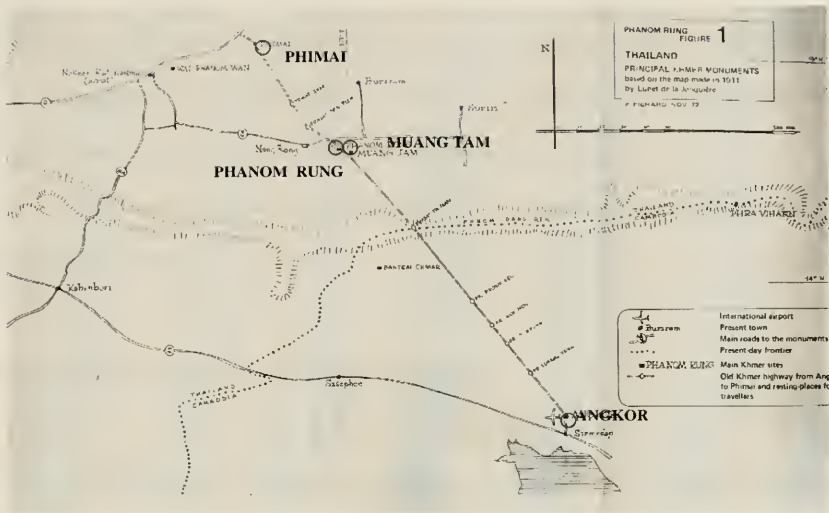
Figure 1.1: Map of Khmer Imperial Route of P.Pichard, 1972,
based on the map of Lunet de la Jonquiere, 1911

Figure 1.2: Collapse of the Viharn Sanctuary located in the border of Thailand and Cambodia.
(July 2000)

3

The restoration of stone monuments in Thailand began systematically in 1962 with the cooperation of UNESCO with the French government. The methodology used to restore such monuments was anastylosis[2], fostering a tradition of disassembly and reconstruction. Since then, the Division of Fine Arts of the Department of the Interior in Thailand has been in charge of supervising all of these historic stone monument preservation projects.

Causes of Structural Failure of Khmer Monuments [3]

Before being excavated and restored, all Khmer monuments were in an advanced state of ruin. Walls were destabilized and some were crumbling down to the base, and the vaulted roofs had almost collapsed. The causes of the structural failure can be attributed to several factors including intrinsic foundation and construction faults, material deterioration, and human interaction. These are discussed below.

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2 Anastylosis is a stone restoration method that re-erects collapsed stone monument using original stone blocks and as little new material as possible. It was first proposed by Nikolaos Bakanos in the First International Congress of Architects and Technicians of Historic Monuments in Athens in 1931. According to chapter 15 in Venice Charter, anastylosis is recommened for restoring the historic monument as quoted "…All reconstruction work should however be ruled out "*a priori.*" Only anastylosis, that is to say, the reassembling of existing but dismembered parts can be permitted. The material used for integration should always be recognizable and its use should be the least that will ensure the conservation of a monument and the reinstatement of its form…"

3 According to the proposal of and article about the Restoration of Khmer Temple in Thailand projects that have been done.

The foundations of Khmer monuments are often defective. They were built of one or more layers of laterite, either in blocks or as rubble, under the first course of sandstone. Due to the heavy loads and the infiltration of rainwater, the laterite foundation became crushed and disaggregated. In addition, the physical and chemical properties of the laterite itself varied for each block. This resulted in uneven load bearing as a function of its origin, orientation, and composition, and resulted in unstable support. The aesthetics of construction also contributed to failure due to unequal support under the walls. The builders placed sandstone blocks to support the heavy loads of the walls only in places where it was necessary; the outside elevation needed the aesthetic appearance, so that the builders placed sandstone blocks as the wall base, while they placed only unstable laterite and sand fill base to support the same wall on the interior elevation. (See Figure 1.3)
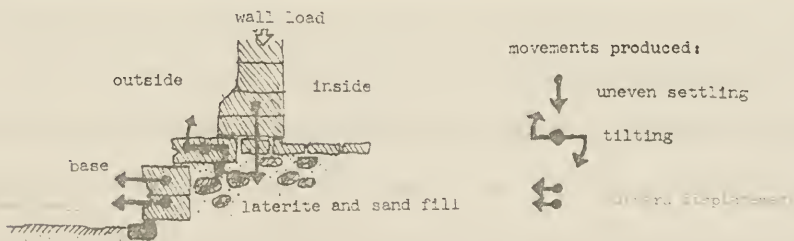
Figure 1.3: Sketch of structural failure due to unequal support of the foundation.

Failure of Construction Techniques

It is necessary to select compatible materials according to their physical and mechanical properties in order to have a more durable and stable construction. In Khmer monuments, builders generally used stone or brick as the main building material. Wood was used as beams, lintels and bracing on the walls, and in some parts of roof construction. Unfortunately, much of the wood was destroyed by rot and termites, resulting in large sections of unsupported masonry, which weakened the walls considerably. Sandstone blocks, sometimes laid in an edge-bedded or face-bedded orientation, could not bear the heavy and different loads, and became tilted, split or broken. That led to the destabilization and collapse of the walls.

Material Deterioration by Water

The foundations were most directly affected by ground and surface water. They are saturated with dampness from the percolation of rainwater between the stones or from the capillary rise from the soil. With the collapse of the roof, the interiors are exposed, so that, after a heavy rainstorm, these unsheltered rooms retain water on the floor for several days before the water evaporates. (See Figure 1.4 and 1.5) Due to the high relative humidity, such water is absorbed into the floor and foundations rather than evaporating. The cycles of wet and dry, moreover, have a physically and chemically destructive effect on the structure of the sandstone as well. Another problem with excessive exposure to

water is that it acts as a vehicle that transports soluble salts to the stone. (The decay mechanism of stone due to water effect will be discussed in more detail in the later chapter.)

Figure 1.4: Interior walls and floors that are exposed due to the collapse of the roof. The Gallery of the Viharn Sanctuary, July 2000.

Figure 1.5: Unsheltered room retains water on the floor for several days before the water evaporates. The Northern Gopura of the Phimai Sanctuary, June 2000

The Khmer monuments in Thailand do not have the same vegetation and biological growth problems as in Angkor. In fact, the sandstone elements that are covered with lichen are in rather good condition[4], while other areas without lichen covering frequently show a dramatic loss of stone due to crumbling and scaling.[5] There is however still no consensus on the effect of vegetation and biological growth on stone. Most researchers agree that biological growth affects the stone, especially the surface, and can cause the stone to eventually deteriorate. This topic will be discussed in detail again in the later chapter.

Human Impact

Although general human activity does not have a direct effect on construction, some specific activities unintentionally damage the construction system. Stealing or removing iron or bronze H clamps and/or some lintels and pediments by chiseling causes holes or gaps within the structure, and, for the large elements, resulting in failure of the

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4 E. Wendler and C. Prasartset. "Lichen growth on Old Khmer Styled Sandstone Monuments in Thailand: Damage Factor or Shelter?" in *Preprints: the 12th Triennial Meeting of the ICOM Committee for Conservation, August 29- September 3, 1999* (Lyon, 1999), 750-754.

5 E. Wendler and C. Prasartset. "Old Khmer Styled Sandstone Monuments in Thailand. Aspects of Weathering and Development of a Conservation Concept," in *Proceeding of the 9th International Congress on Deterioration and Conservation of Stone, Venice, June 19-24, 2000* (Venice: Elsevier, 2000), 765-774.

structural members. The activity has led, in some cases, to scars on the monuments and, in more serious instances, to collapse of the structure.

History of the Phimai Sanctuary and its Restoration

Figure 1.6: Front view of the Phimai Sanctuary in the Nakhon Ratchasima Province of Thailand.
(June 2000)

The Phimai Sanctuary is situated in the Nakhon Ratchasima Province, about 25 miles northeast of Bangkok, the capital of Thailand. It is the largest stone sanctuary in Thailand, and was constructed during the eleventh and twelfth century AD. The Kingdom of Ancient Khmer—King Suryavaram I, and King Jayavarman XI and XII— built the complex for religious purposes as a temple to Mahayana Buddhism and

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Hinduism, not for residential function as a palace. It was built of stone with delicate carved iconography based on the Devaraja cult that believes in the godlike king. (See Figure 1.6)

According to the Restoration of the Phimai Sanctuary Master Plan of 1962 (see Figure 1.7), the sanctuary is composed of 12 monuments built of three main materials: sandstone, laterite and fired brick. The important components are: the principle tower, the main sanctuary located in the inner courtyard; the inner Gopura, the arched gateways and gallery; and the outer Gopura, the arched gateways and boundary wall.

The siliceous sandstone principal tower was successfully restored with anastylosis more than 30 years ago. This was carried out between 1964 and 1969 at the request of the French Ambassador in Thailand, by B.P. Groslier, who was then director of Archaeological Research at the Ecole Francais d'Extreme-Orient and Curator of the Angkor Monuments. The sanctuary was reconstructed on a new reinforced concrete foundation with an inserted bracing structure inside the walls and under the vaults, and the tower was restored to its original height. (See Figure 1.8)

Unlike the tower, the outer Gopuras were built of a litharenite sandstone[6] which is weaker and more susceptible to weathering. (See Figure 1.9) The condition of the

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6 Litharenite or lithic arenite sandstone is a type of sandstone that is composed of mostly quartz, more than 25% rock particles and less than 10% feldspar. It can be called "Lithic Sandstone" meaning that the

Gopuras was very critical when the survey was conducted. (See Figure 1.10) The

building had collapsed, some pieces of stones had fallen down, and the stone itself was

deteriorated in sections to a very high degree, as noted in 1972,

> ...Above all, only the inner sanctuary, built in good quality
> siliceous white sandstone, has been restored. It was not
> possible to carry out any work on the entrance pavilions
> [Gopura], galleries and enclosing walls, which are built in
> a very fine grained red sandstone with a ferruginous clay
> binder, of exceedingly poor quality crumbly, broken and
> eroded due solely to the effects of aging...[7]

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percentage of rock particles exceeds that of feldspar. Information from *Sand and Sandstone* written by
Pettijohn, Potter and Siever.

7 P. Pichard. Restoration of a Khmer Temple in Thailand (Paris: Unesco, 1972), 10.
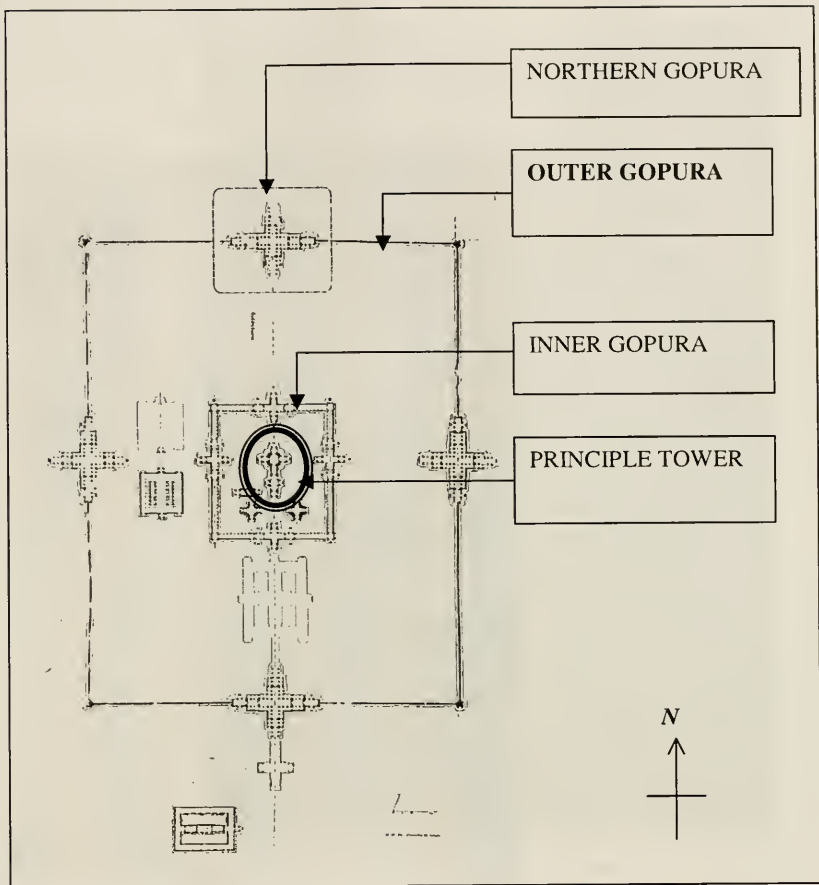
Figure 1.7: Restoration Master Plan of the Phimai Sanctuary, 1962.

Figure 1.8: The Principle Tower after anastylosis restoration, 1964-1969.
(June 2000)

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Figure 1.9: The condition of the Northern Gopura of the Phimai Sanctuary before restoration.
(Fine Arts Division, the Department of Interior, 1998)

Figure 1.10: The condition of the Northern Gopura of the Phimai Sanctuary before restoration.
(Fine Arts Division, the Department of Interior, 1998)

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Current Problems in the Northern Gopura of the Phimai Sanctuary

The Division of Fine Arts of the Department of the Interior in Thailand recently attempted to restore the Northern Gopura by anastylosis. They tried to follow the previous successful reconstruction project carried out on the main sanctuary in 1962. All of the remaining stones were dismantled first and new concrete was poured before replacing the historic stones into their original locations. Unfortunately, because of the extremely disaggregated and delaminated condition of the stone blocks, most of them were damaged during dismantling and were not reusable. (See Figure 1.11) Subsequently, new stone replacements were obtained in order to re-construct the Gopura; the new stone was brought from quarries in Sikhew district in the Nakhon Ratchasima Province, and, although the same type of sandstone, the fresh stone contains more mica than the ancient stone. Hence, the original concept and purpose of the preservation project—to preserve the ancient stones and original construction—had to be changed due to the loss of the original masonry. Consequently, the project might be considered a failure from the point of view of conservation although it was a successful reconstruction. (See Figure 1.12)

The main preservation activity for the Khmer monuments in Thailand focuses only on architectural and structural restoration, or reconstruction without considering the value and importance of the original building materials. This occurred because the conservators lacked knowledge of material properties and the causes of deterioration. That misdirected

activity can be easily seen in the restoration project of the Northern Gopura and others at that site. It resulted in application of inappropriate preservation methods which led to the unfortunate use of new stone blocks in the monuments. Without a clear understanding of the original construction technology—materials and methods, attractions through time, existing conditions and decay mechanisms, and the environmental context-- any consideration of intervention strategies is difficult.

Figure 1.11: Ancient stone block broke during the restoration project.
(Fine Arts Division, the Department of Interior, 1998)

Figure 1.12: New stone replacement during anastolysis restoration project.
(Fine Arts Division, the Department of Interior, 1998)

Purpose of Thesis

The structure of the Phimai Sanctuary's Northern Gopura collapsed not only due to structural failure, but also due to the deterioration of the sandstone leading to a reduction of its mechanical properties. Because of the poor condition of the stones, material treatments, such as consolidation, must first be performed to strengthen them before any further intervention is attempted, especially if disassembly is deemed necessary.

This thesis will investigate the effects of consolidation on a litharenite sandstone of Northeastern Thailand in order to improve its strength and hardness. Although it is too late to offer alternative conservation solutions to the Gopuras of the Phimai Sanctuary, there are still many other Khmer monuments built with the same construction techniques and materials that could benefit from the results of further advanced studies and alternate preservation approaches. Hopefully, the conclusion and the recommendations of this thesis can be used to determine conservation guidelines for future preservation projects on the litharenite sandstone monuments of Thailand. Moreover, the whole methodology of investigation can be adopted to better understanding the performance of traditional porous building materials and to evaluate conservation treatments in any project.

CHAPTER II

TESTING PROGRAM

SELECTION OF APPLIED TESTS

The analysis and characterization of building stone is performed in order to understand the physical, chemical and mechanical properties of the material and its mechanisms of deterioration, as well as to assess of its deteriorated condition. These characterizations help to evaluate the effectiveness of any treatment by noting the changes of such properties before and after treatment, by comparing those changes to the criteria or requirements that the treatment should reach from the recommendation of the standards (e.g. NORMAL 20/85).

In order to select testing protocols, previous research and testing standards will be reviewed and summarized. This summary will provide general information about the important properties that should be tested, and offer several tests that have been successfully applied for stone performance evaluations. Availability of the testing equipment also was taken into account when selecting testing program for this research.

According to the surveyed literature[8], not all the physical, chemical and mechanical characteristics including mineralogical and petrographic properties of the studied stone were identified and tested in every case of sandstone research. Rather the choice of tests depended on the researchers' purposes. Researchers most often tested for stone porosity, water absorption, and mineralogy, as well as compressive and flexural strength. These aspects were chosen because they can indicate the effectiveness of an applied consolidant whether or not it improves the stone's overall quality.

In order to test these aspects, different methods and tools were applied and used. To analyze the mineralogical and chemical composition of the stone, including observing and assessing the degree of deterioration, X-ray diffraction and polarized light microscopy and scanning electron microscopy were used. The identification of salt content in the stone was also the most popular test applied to fresh, untreated weathered or non-weathered stone using X-ray diffraction, Fourier transform infrared microspectroscopy (FTIR) or others tools, such as Atomic absorption spectroscopy (AAS) or Ion chromatography (IC).

Testing water absorption through total immersion and capillary action was the most utilized method to measure the stone's water absorption capacity and porosity. Moisture

content was sometimes measured in the drilled holes of the stone using a moisture sensor. Unfortunately, except for one study that utilized microwaves for this measurement, the rest of the test methods and tools were not mentioned.

In order to evaluate the effectiveness of the consolidation treatment, most physical and mechanical tests were conducted before and after treatment. In addition, the effective depth of penetration and weathering resistance were also measured. SEM analysis, drill resistance and biaxial flexural strength were the most often cited tests for depth of penetration, while artificial aging tests with salt crystallization, freeze-thaw cycle and sulfuric acid were the most frequently applied to assess weathering resistance. Weight variation measurements before and after consolidation were also tested to evaluate the amount of consolidant that could penetrate the stone.

In order to determine the appropriate tests to be applied to the project, not only a summary of the literature, but also other researchers' suggestions and some recommendations from many standard sources, including RILEM, NORMAL and ASTM, were also taken into account. NORMAL 16/84 recommends optical examination, thin section analysis, and scanning electron microscopy to determine mineralogy, petrography, and deterioration morphology. Such methods can also identify previous treatments to the stone, if applicable.

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8 See the table of summary in Appendix IV

NORMAL 20/85 recommends some tests for change in weight and color, water vapor permeability, depth of penetration, hydric dilatation, and ultrasonic and/or sonic velocity for instance. It also specifies some artificial weathering tests that should be able to simulated the environmental conditions to which the stone is exposed, including the cycles of acid rain or mist, wet-dry, freeze-thaw, salt crystallization, thermal, salt spray and ultraviolet radiation. The combination of two or three of those tests is recommended.

The international RILEM 25 P.E.M. Committee proposes structure defining tests: porosity accessible to water, bulk and real densities, vapor permeability, and pore size distribution by suction and mercury porosimetry analysis. Observing thin section with SEM is suggested to evaluate effectiveness of treatments. Some water effect in stone should be analyzed: movement of water including saturation and water vapor conductivity coefficient; water absorption under low pressure and water absorption coefficient; linear strain due to water absorption; and water drop absorption. This standard also recommends the tests for compressive, tensile and bending strength, modulus of elasticity, adhesion strength, surface hardness, and abrasive resistance. The durability of the stone before and after treatment can be tested with artificially or naturally weathered aging. Lastly for this standard, the miscellaneous tests, such as the external aspect of the stone, sampling method, thermal expansion and electrical conductivity, are also recommended.

Summary of Tests for Characterization and Effectiveness Evaluation of Stone

Tested Properties	Tested area	Applied test	Resource/place	Advantage	Disadvantage
Material/Element composition	Stone sample	X-ray diffraction (XRD)	LRMS	- Able to identify the characterization of the stone including particular minerals	- Expensive method - Unable to identify any mineral or compound that have the amount less than 10% in the stone - Take time to prepare the mounting sample, and also to obtain the result
Material composition	Stone sample	Infrared spectrometry	LRMS	- Able to identify and quantify organic and some inorganic matters in the stone - Quick sample preparation	- Expensive - Unable to identify the matters that have the same component or organic matrix - Unable to identify most of mineral oxides and sulfides
Microstructure/ Mineral composition	Stone sample	Optical Microscopy	Microscopy Lab, Architectural Conservation Laboratory	- Able to obtain both information about the stone microstructure and composition and the surface alterations	- Destructive It takes time to obtain the data due to vacuum impregnation of the sample before being prepared into thin sections
Morphology/ Chemical composition	Stone sample	SEM-EDS	LRMS	- Able to simultaneously investigate the morphology and chemical composition of the stone	- Very small observed area
Mineralogical composition	Stone sample	UV-fluorescence	N/A	- Rapid, inexpensive and readily available analytical tool for the stone weathering, minerals and salts identification	- Unable to differentiate colors between the fluorescence and the intensities of reagent grade crystalline powders of some salts.

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Properties	Tested area	Applied test	Resource/place	Advantage	Disadvantage
Roughness	Surface	Touch	Architectural Conservation Laboratory	- Simplest method to compare the roughness	- Unable to obtain the information in detail - Subjective
	Surface	Stereomicroscope	Microscopy Lab, Architectural Conservation Laboratory	- More accuracy than observing by naked eyes	- Confusing if monitor has to be used to show images - Unable to observe the same area - Destructive
	Surface	Roughness measurement using Form Talysurf One track method	Young, 1992/ N/A	- Cheap and fitted to in situ measurement - Calculated easily from the registered roughness profile	- Destructive - Various in the results due to the limitation of times of test in the same track (not able to test the same area before and after treatment) - Inaccurate due to the differences between measurement scale and the scale of real modifications of surface morphology
	Microstructure of stone and other residues being in stone	SEM	LRMS	- Able to view 3 dimension - Able to calculate roughness parameters - Precise	- Unable to work on original stone surface that can not be sampled due to it is a destructive method - More expensive and need specific expensive instruments than the other methods - Unable to apply to in situ works
Color	Surface	Observation based on the Munsell Syetem	Architectural Conservation Laboratory	- Useful to quantify visual impressions - Used as reference values for later assessment	- Subjective

Tested Properties	Tested area	Applied test	Resource/place	Advantage	Disadvantage
Compressive Strength	Stone cube	Compressive strength test	ASTM C-170/ LRMS	- Useful to indicate the differences in compressive strength between various dimension stones and/or between the same type of stone - Able to quantify compressive strength values in different stone conditions	- Expensive - Unable to test in situ - Destructive due to a large amount of tested samples
Flexural Strength	Stone cube	Three point bending test	ASTM C-99/ LRMS	- Useful to indicate the differences in modulus of rupture between various dimension stones and/or between the same type of stone - Able to quantify flexural strength values in different stone conditions	- Expensive - Unable to test in situ - Destructive due to a large amount of required sample - Product's individual variations may cause deviation from the accepted values
Hardness	Scratch hardness of the surface	Measure the width of the scratch by "Marten Sclerometer"	Verges-Belmin, 1996/ N/A	- Able to use in situ or in the lab	- Destructive method
	Surface	Abrasive resistance Test	ASTM C-241, ASTM C-418/ LRSM	- Used to measure the relative abrasive resistance of masonry/ concrete surface Useful for evaluation of treatment effectiveness	- No acceptable reference material for determining bias of the procedure

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Properties	Tested area	Applied test	Resource/place	Advantage	Disadvantage
Water absorption	Stone sample	Water absorption test with Rilem Tube or Water (micro) Drop Absorption	Rilem II.4/ Architectural Conservation Laboratory	- Easy to test either in situ or in the laboratory	- Possible to have multiple errors due to the interruption of absorption process or ineffective elimination of adhering water or unstable environmental conditions
		New method of water absorption test proposed by Martin and Mosquera	Martin, 1996 N/A	- Able to quantify more data sets - More accurate than the standard methods	- Complicated and expensive instruments than the standard ones
	Stone sample	Water absorption by total immersion	NORMAL 7/81 /Architectural Conservation Laboratory	- Useful to analyze stone's water absorption capacity - The result obtained can be useful for calculating stone's porosity - Simple and cheap	- Unable to qualify pore size and distribution
	Stone sample	Water absorption through capillary uptake	Ex.10- Teutonico, NORMAL 11/85/ Architectural Conservation Laboratory	- Useful to analyze stone's ability to draw water into its pores and the highest level to which water can be drawn - Simple and cheap	- No mentioned

Properties	Tested area	Applied test	Resource/place	Advantage	Disadvantage
Porosity	Stone sample	Calculation from Water absorption result	Ex.11- Teutonico, ASTM C-97 / Architectural Conservation Laboratory	- Simple - Useful to quantify the percentage of stone porosity	- Possible to have inaccurate result due to the error of the immersion test
	Stone sample	Mercury Porosimetry	N/A	- Useful to identify and quantify the percentage of stone porosity and pore distribution	- Expensive
Water vapor transmission	Stone sample	Water vapor transmission test	ASTM E-96 / Architectural Conservation Laboratory	- Useful to determine whether or not cleaning process should be applied to the stone	- May have some error data due to unstable environmental conditions
Soluble salts	Stone sample	X-ray diffraction	Verges-Belmin, 1996/ LRSM	- Used to identify the efflorescence of salts over the stone surface	-Unable to identify invisible salts in stone, especially after cleaning and the stone has not been dried yet
	Stone sample	IC [Blauer, Verges-Belmin] ICP [Verges-Belmin] AAS [Blauer, Verges-Belmin] FPM [Blauer] LFP [Blauer]	Blauer, 1996 & Verges-Belmin, 1996/ LRSM	- Used to quantify the number of ions in solution of salt remaining in the stone after cleaning although it has not dried yet.	- Possible to have some error data due to the limitation of each tests
	Ground stone sample	Qulitative salt analysis test	Ex.16- Teutonico/ Architectural Conservation Laboratory	- Simple The rough quantification of salts can farther tested with Salt-Test analytical testing strips	- Unable to indicate accurate amount or concentration of salts

Properties	Tested area	Applied test	Resource/place	Advantage	Disadvantage
Weathering resistance	Stone sample	Salt crystallization test	Ex.15- Teutonico/ Architectural Conservation Laboratory	- Simple and cheap - Useful to indicate salt resistance of stone and to evaluate the effectiveness of consolidant	- Sodium sulfate solution is harmful. Care needs to be taken during the test
	Stone sample	Wet-Dry Cycling	ASTM D-5313/ Architectural Conservation Laboratory	- Able to determine the effects on individual pieces of rock for wet-dry erosion - Able to evaluate the durability of stone and the effectiveness of consolidant	- Unable to provide an absolute value; thus, the test cannot be used as the sole basis for the determination of stone durability
	Stone sample	Accelerated weathering test	ASTM G-53	- Useful to simulate natural weathering by wet and dry cycle - Able to indicate the UV-radiation resistance of stone - Useful for evaluating effectiveness of consolidant	- Expensive - UV-radiation is harmful
Depth of Penetration	Treated stone sample	Iodine vapor staining Test	Kumar &Ginnel /Architectural Conservation Laboratory	- Used for determining the depth of penetration of consolidant - Simple Able to estimate concenration of consolidant	- Unable to test dark color material

Transmitted Polarized Light Microscopy

Polarized light microscopy (PLM) is generally the most useful and economically feasible examination method for conservators.[9] It is used to observe the optical characteristic properties of materials, including size, shape, surface, color, transparency, refractive index, crystal system, and dispersion of the optic axial angle on single particle. It can reveal almost any phase of a sample—whether in thin section, cross section or dispersed— and the images observed with the microscope can be identified by listing of its characteristics, comparing with a reference to a photo-micrographic atlas, or referring to known sample.[10]

To prepare the sample for microscopy, two thin sections of the stone— parallel and perpendicular to the stones bedding plane— are mounted, with blue dye saturation to show the stone's open pores.

In this project, transmitted polarized light microscopy was used to observe the micromorphology and physical characteristics of the stone. This microscope was also used as a tool to evaluate the change of physical and chemical microstructure after

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9 Skip Palenik. "An Introduction to Microchemical Quantitative Analysis". McCrone Research Institute (unpublished manuscript).

consolidation treatment. The untreated and treated stone samples were prepared in thin section and cross section slides. Magnifications of 50x, 100x, and 400x were used.

Scanning Electron Microscopy

Scanning electron microscopy (SEM) is used frequently to analyze the micro-textural and elemental composition of masonry materials. It can reveal some of the physical and chemical properties of the stone, including the size, orientation and arrangement of its composite minerals and pores, and deposition of any new material (e.g. consolidant). This method is also able to detect the decomposition of stone, if applicable. Due to a very small amount of sample required, SEM can be considered as a non-destructive method that helps us to understand decay mechanisms. It can show the alterations of the mineral composition, widening of pores' spaces and loosing of particles' binder that are the indicators of stone weathering. In order to supplement the SEM result, X-ray diffraction analysis is required to give a more accurate result of the mineralogical composition.[11]

For this project, SEM was used to evaluate the effectiveness of the treatment as well. Since it can reveal the stone's microstructure, the alteration and modification of the stone before and after treatment can be compared. Comparing the result with the requirements

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10 Walter C. McCrone. *Polarlized Light Microscopy* (Chicago: McCrone Research Institute, 1984), 1403.
11 Helmut Weber and Klaus Zinsmeister. *Conservation of Natural Stone* (Ehningen, DM: Expert-Verl., 1991), 20.

for consolidation treatment-- which will be discussed in the later chapter-- can indicate whether or not the consolidant and application method used in this project were satisfied. The samples analyzed were the thin-section slides and small pieces of stone surface with biological growth. A scanning electron microscope with 100 magnification was used to observe and photograph the stone's surface and microstructure.

Compressive Strength Test

Compressive strength is the load per unit area under which a block fails by shear or splitting.[12] The results obtained from the compressive strength test can be useful for comparing stone strength in different orientation (e.g. parallel, perpendicular to the bed), or between different types of stone.

In this project, the test followed ASTM C 170 to compare the compressive strength between untreated and treated weathered sandstone. Although the standard recommends the use of least 5 stone cubes in a size not less than 2" in each direction (parallel and perpendicular) and each condition (wet and dry), in this project only 3 stone cubes per direction and condition were tested due to the limited amount of available samples. Before testing for the dry condition, the stone cubes (treated and untreated) were dried in the oven at 60°C ± 2°C for 48 hours or until their weights were stable.[13] Then, the cubes

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12 E.M.Winkler, *Stone: Properties, durability in Man's Environment* (New York: Springer-Verlag, 1973), 39.
13 Following the ASTM standard (C170-87), at the 46th, 47th and 48th hour, each cube will be weighed to ensure that the weight is the same, or until 3 successive hourly weight readings are the same.

were cooled down in a desiccator at room temperature (70F and 18%RH). In contrast, before testing for the wet condition, the rest of the stone cubes were immersed in deionized water (22 ± 2°C or 72±4°F) for 48 hours. Then, they were tested immediately after being removed from the immersion and wiped with wet paper to eliminate the surface water.

The test was performed at the Laboratory for Research on the Structure of Matter (LRSM) at the University of Pennsylvania using an Instron Testing Machine 1125 calibrated at a cross head speed of 0.05 inch/minute, and the chart speed at 2.0 inch/minute. (See Figure 2.1) The preferable rate of loading was not greater than 100 psi (690 kPa)/second.

The calculation of the data was as follow:

$$C = W/A^{14}$$

Where:　　　　C = Compressive strength of the sample (psi or Mpa)

W = Total load on the sample at failure (N)

A = Calculated area of the bearing surface (mm^2)

However, in NORMAL 7/81 (the test for water absorption), in the same procedure of drying sample, it recommends to dry and weigh the sample until two successive weighings carried out after 24 hours in time is not greater than 0.1 % of the sample weight.

14 When the ratio of height to diameter (or lateral dimension) is different from the unity (1:1) less than 25%; otherwise another equation has to be used. (See detail in ASTM C170)
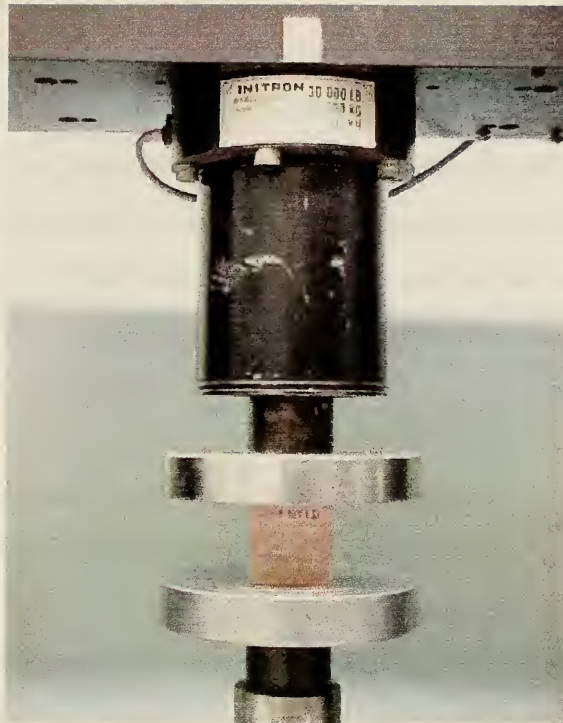
Figure 2.1: Compressive strength test with an Instron Testing Machine 1125. In the photo, a stone cube was tested with the load perpendicular to bedding planes

Flexural Strength Test

Flexural strength or modulus of rupture is the other mechanical property of stone that characterizes the bending or flexural resistance caused by a combination of compressive, tensile and shear load.[15] The test for this strength compares the modulus of rupture between the various orientations of stone within the same type, or indicates differences between various types of stone. It is also one of the most accurate methods for evaluating the effectiveness of strengthening of a consolidant because this test can evaluate and differentiate both the strength and the grain-to grain cohesion of the treated and untreated stones.

Following ASTM C99, 5 or more 4x8x2¼-inch stone samples should be prepared for each direction of stone per each test condition. However, due to the limited availability of the stone, 3 2x4x1-inch samples in each direction per each condition were prepared instead. The testing machine should be as accurate as 1% or lower for the range of 10 to 1000 ft^3 of applied load. The knife edges' length should be at least as long as the width of the samples. In this project, the test was conducted at the Laboratory for Research on the Structure of Matter (LRSM) at the University of Pennsylvania using an Instron Testing Machine 1125 calibrated at a cross speed of 0.05 inch/minute and a chart speed of 5 inch/minute. The gage length was 3 inches. The rate of load applied was not greater

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15 E.M.Winkler, Stone: Properties, durability in Man's Environment, 44.

than 1000 ft³/minute (4450 N/minute). (See Figure 2.2) The calculation of the data obtained from the test was:

$$R = 3Wl/2bd^2$$

Where: R = Modulus of rapture

                     W = Breaking load (ft³ or N)

                     l = Length of span (inch or mm.)

                     b = Width of sample (inch or mm.)

                     d = Thickness of sample (inch or mm.)

Water Absorption Tests

One of the major agents that contributes to the decay of stone is water. It can enter the stone either in the liquid phase, i.e. by suction from wet materials or infiltration of rainwater, or in the vapor phase, i.e. by condensation or adsorption. The amount of water absorbed and its movement is dependent on the physical characteristics of the stone, i.e. its microstructure, porosity, capillarity and permeability. Not only is water itself harmful to the stone, it also acts as a vehicle bringing soluble salt into the stone's pores and causes more deterioration problems. Therefore, the measurement of water absorption is useful to predict a susceptibility to decay, as well as to evaluate consolidation treatments.[16]

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16 Jeanne Marie Teutonico, *A Laboratory Manual for Architectural Conservators* (Rome: ICCROM, 1988), 35.

In this project, two methods of measuring water absorption will be used. One is by total immersion and another is by capillary action. Generally, the capillary rise of water is the most frequent and typical water absorption mechanism that occurs in stone.[17]However, to obtain the maximum amount of absorbed water, and to study the porosity of the stone (in the next step), testing stone saturated through the immersion method has to be conducted as well.

Water absorption by total immersion is to immerse the stones in deionized water at room temperature in order to measure the amount of water that the samples can absorb. The data obtained was reported in terms of percentage of absorbed water to the weight of the dry sample. The water absorption capacity was then calculated and reported as the percentage of maximum quantity of absorbed water to the weight of the sample that had been re-dried after total immersion. The procedure of the test was based on NORMAL 7/81[18]. Another method, the capillary action test, measures the height to which water can penetrate into the stone to indicate the stone's capillarity property. The procedure followed Exercise 10 in the Laboratory Manual for Architectural Conservators. (See Figure 2.3)

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17 Helmut Weber and Klaus Zinsmeister, *Conservation of Natural Stone*, 26.
18 Following the draft translation version translated by Dr. A. E. Charola, Historic Preservation Program, University of Pennsylvania.
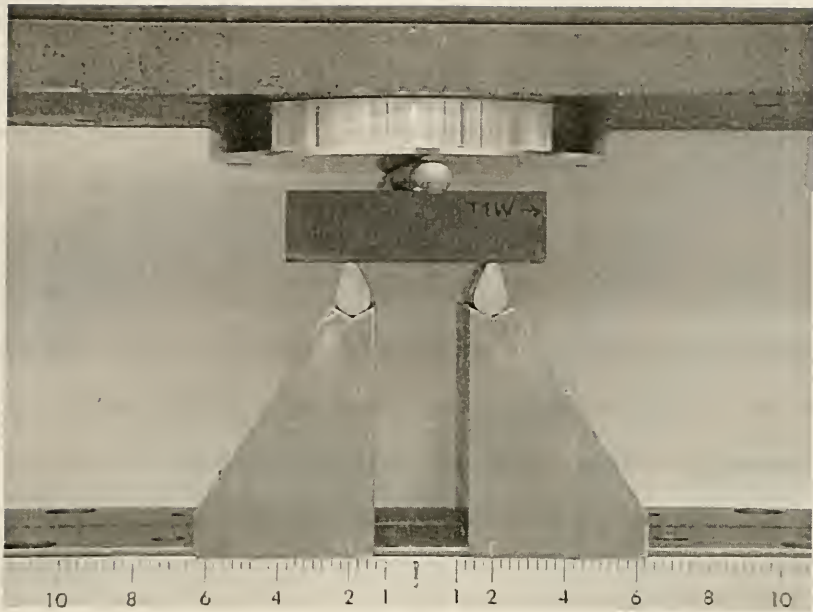
Figure 2.2: Flexural strength test (3-point bending test)
with an Instron Testing Machine 1125 set at 2-inch gage length

Figure 2.3: Water absorption test
Above: Water absorption by total immersion
Right: Water absorption by capillary action

Porosity Calculation[19]

Porosity of the stone is the fraction of the total volume of a solid that is occupied by pores.[20] Generally, the pore structure of natural stones is very complex. Sandstone, a detrital rock, has various dimensions of constituent grains varying between 0.06 and 2 mm. This affects the variety of its pore structure, which is also dependent on the sorting and shape of particles and on the degree of its cement. [21] Usually, the pore structure is oriented, i.e. anisotropic.[22] In some types of sandstone, pores can be entirely filled while in other types, they are only partially filled. Porosity also depends on the presence of clay. If clay is present as clasts that have smaller size than the other grains, such clay material fills the pores in the stone.[23]

The percentage of porosity that can be obtained from calculation will indicate the percentage of pores in the stone. Not only can this result be useful to characterize the stone; it can be useful to evaluate the effectiveness of the consolidant. In this project,

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19 Although most of the previous researches used mercury porosimeter as a tool to study the porosity of stone, the machine is not available in the University of Pennsylvania at the time of this project is being done.

20 Teutonico, A Laboratory Manual for Architectural Conservators, 45.

21 P.Rossi-Doria, "Pore Structure Analysis in the Field of Conservation: State of the Art and Future Developments," in Principle and Applications of Pore Structural Characterization: Proceedings of the RILEM/CNR International Symposium, Milan, Italy, April 26-29, 1983, (Bristol: J.W. Arrowsmith Ltd., 1985), 441-459.

22 P.Christensen and H. Gudmundsson, "Measurement of Porosity in Fresh and Weathered Sandstone by Means of Automatic Image Analysis," in *Principle and Applications of Pore Structural Characterization: Proceedings of the RILEM/CNR International Symposium, Milan, Italy, April 26-29, 1983*, (Bristol: J.W. Arrowsmith Ltd., 1985), 477-481.

23 P.Rossi-Doria, "Pore Structure Analysis in the Field of Conservation: State of the Art and Future Developments," 441-459.

calculating the porosity is the chosen method to measure the porosity of the stone due to its ease when advanced instruments are not available, and can be done following the water absorption by total immersion test. The procedure was based on ASTM C97. The real and apparent density can be calculated when both real and apparent volumes[24] are measured using the Principle of Archimedes method. Then, the porosity can be calculated as follows:

$$\epsilon = 1 - \rho_a / \rho_r$$

Where: ϵ = Porosity

ρ_a = Apparent density

ρ_r = Real density

Water Vapor Transmission Test

A critical property of stone is permeability. This is the ability of the stone to transmit liquid or gas from one place to another. Normally, under conditions of stable temperature and consistent pressure ratios, a balance between the moisture content of the stone and atmospheric moisture will occur. However, whenever one has lower partial water vapor pressure the other, it will absorb the water vapor from the other. Testing simulates this phenomenon, using the stone as a transmitter between a beaker full of water and the atmosphere. The water vapor pressure inside the beaker is higher than that of the air, so

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24 Apparent volume is the volume of stone including its pore space while real volume is the apparent volume minus the volume of its pore space accessible to water.

that the water vapor will be transmitted through the stone disk to the atmosphere. The more porous the stone is, the more water vapor diffuses through the disk.[25]

The water vapor permeability test indicates the time rate of water vapor transmission through a unit area of flat transmitter (i.e. untreated and treated sandstone in this project) of unit thickness induced by a unit vapor pressure difference between two specific surfaces, under specified temperature and humidity conditions.[26] According to one of the recommended requirements for consolidation, such treatments should not change the permeability of the material; this test therefore can be one of the suitable indicators to evaluate an appropriate consolidant.

The test was based on ASTM E96. The stone is cut into cylinders with 3 7/8' diameter and 1" thickness. It is recommended that five samples per type of stone (treated and untreated) should be tested by the same method—two samples being tested with the vapor flow in each direction (perpendicular and parallel to bed planes) and another as a dummy without water inside the beaker. Due to the completed impregnation of the consolidation treatment, both sides of the disk sample were saturated with consolidant. The stone disks and disposal beakers were weighed before being assembled together and sealed with paraffin. Then each assembly—the disposal beaker with stone and 180-ml deionized water—was weighed before being placed in the desiccator. (See Figure 2.4)

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25 Helmut Weber and Klaus Zinsmeister, *Conservation of Natural Stone*, 81.

Figure 2.4: Water vapor transmission test
The assemblies were placed in a desiccator under the average temperature of 69°F
and the average relative humidity of 18.5%

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26 From the definition of terms found in Terminology C168 explained in ASTM E96-95, Standard Test
Methods for Water Vapor Transmission of Materials.

The assemblies were, again, weighed at elapsed times of 5, 15, 30 and 60 minute, and then, at every 24-hour interval for two weeks or until the rate of weight change record was stable. The data from the record was plotted as a weight-time graph to obtain the slope of the graph line, i.e. the rate of water vapor transmission.

The rate of water vapor transmission was be calculated as follows:

$$WVT = G/tA$$

Where: G = Weight change (grams)

            t = time (hours)

            G/t = Slope of the straight line (gram/hour)

Qualitative Analysis of Water-soluble Salts

Stone may naturally contain water-soluble salts as mineral constituents, e.g. halite. However, salts often migrate into stone pores after the stone has been set in place.[27] These salts may come from the ground, through exposure to air pollution or sea spray, or from some construction materials attached to the stone. Salt formation, through dissolution and crystallization in the pores can cause several problems to the stone by producing disruptive pressures while crystallizing, or creating the differential thermal expansion between themselves and their host—the stone. (The decay mechanism by salts will be discussed in details in the Decay Mechanism chapter)

Water-soluble salts are produced by the reaction between stone, water, and polluting compounds in the water or the atmosphere, or sometimes by other components contributed by microorganisms.[28] The main soluble salts that are normally present in stone as deterioration products are sulfates, chlorides, nitrites and nitrates. By measuring and identifying which of these are present, the data obtained can be used as clues to study and predict the decay mechanism and type of deterioration that have occurred in the weathered stone. For quantitative analysis of water-soluble salt content, salt-test analytical testing strips were used to estimate the amount of water-soluble salts in the stone.

Wet-Dry Cycle Test

Change of stone moisture content during wet and dry states causes stone to expand and contract, and consequently, leads to stone friability[29]. Therefore, wet-dry cycle test determines the effects on the stone for erosion control of wetting and drying action, as well as the resistance of the stone to deterioration. This test serves to evaluate the weathering resistance of the stone after treatment compared with that of the untreated stone.

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27 Winkler, Stone: Properties, durability in Man's Environment, 111.
28 Teutonico, A Laboratory Manual for Architectural Conservators, 58.
29 See Appendix I for the definition.

44

The adopted test followed ASTM D-5313. Three untreated and three treated stone cubes (5"x5"x5") were tested. They were first dried in the oven at 60°C for 24 hours. After removed from the oven and cooled in a desiccator, the stones were weighed, and then were immersed in the container full of water for 24 hours (the same procedure as the water absorption by total immersion test). They were weighed again after 24 hours of immersion, and re-dried in the oven at 60°C for 24 hours. The cycle was repeated until evidence of macroscopic deterioration of the stone cubes was observed.

Salt Crystallization Test

As already explained, when stone is contaminated with a solution of salts, crystallization will occur once the water, as the solvent, is evaporated. This process will produce pressures by non-accommodative crystal growth in small pores of stone, and will lead to the disruption.[30] Therefore, the salt crystallization test serves to evaluate the durability of the stone after treatment compared with that of the untreated stone. Salt crystallization is one of the oldest and most aggressive artificial aging tests that can be used to evaluate the effectiveness of the stone consolidation.

The chosen salt solution was a 14% solution of sodium sulfate decahydrate ($Na_2SO_4 . 10 H_2O$) or so called "Mirabite". Three untreated and three treated stone cubes (2"x2"x2") were tested. Before being tested, all samples were dried in the oven at 60°C for 24 hours,

and then cooled in a desiccator. They were weighed before immersion in the salt solution for 24 hours, and then were dried again in the oven for 24 hours. The cycle was repeated until evidence of macroscopic deterioration of the stone cubes was observed.

Depth of Penetration Analysis

Depth of penetration is one of the requirements for effectiveness of consolidants. It can indicate how far the consolidant can penetrate and strengthen the stone—the deeper, the better. Generally, this property of a consolidant depends on its viscosity—the lower, the deeper. It is also, however, dependent on the other factors, such as the pore size and distribution of the treated stone, the application method, and the environmental condition.

In this project, the test followed a new method for determining the depth of penetration of consolidants using iodine vapor developed by Rakesh Kumar and William S.Ginnel.[31] Three 2"x2"x2" stone cubes to which the consolidant had been applied by brushing and which had been cured for 3 weeks were tested. Since the iodine vapor is not physically absorbed by stone, when the stone samples are exposed to the iodine vapor in the closed chamber, iodine will stain only the polymerized consolidant with a faint yellowish brown. The intensity of the stains can indicate the concentration of consolidant—the higher intensity of the stains, the higher concentration of the consolidant. The duration of

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30 Winkler, Stone: Properties, durability in Man's Environment, 119.

exposure depends on the concentration of the consolidant and the original color of the stone compared with that of the stain based on the fact that the longer period can produce the darker stain. In this project, the stones were exposed to the iodine vapor for 3 hours. Generally, the depth of penetration can be measured from the average distance of the appeared stains from the edge to the center of the stone.

Figure 2.5: Depth of penetration analysis by iodine vapor method

31 Rakesh Kumar and William Ginell, "A New Technique for Determining of Penetration of Consolidants into Limestone Using Iodine Vapor," *Journal of the American Institute for Conservation* 36, no.2 (1997): 17-21.

Program of Selected Tests Table

Tested Properties	Applied test	Resource/place	Preparation	Test Equipment & Reagents	Period of test
Material composition	- X-ray diffraction (XRD) - Polarized Microscopy	LRMS Microscopy Lab, Architectural conservation Laboratory	- Grind stone sample Sieve the sample through a #200 sieve - Dry the fine sample in the oven until weight constant - Thin sections	- Sieve # 200 - Oven (110 C) - Balance - Aluminum tray	3 – 4 days of preparation (1 day w/o)* 1-2 days of test (w/o)
Microstructure/ Mineral composition	Polarized Microscopy SEM	Microscopy Lab, Architectural conservation Laboratory	- Prepare mounted thin sections of untreated and treated stone	- Polarized-light microscope - Reflected-light microscope	1-2 days of test (w/o)
Morphology/ Chemical composition	SEM-EDS	LRMS	- Prepare mounted thin sections of untreated and treated stones	- Scanning electron microscope	6 weeks of thin section preparation (w/)* 1-2 days of test (w/o)
Color	Observation by using Munsell Color Book	Architectural conservation Laboratory	- Untreated and treated stone samples	- Munsell Color Book	1 day of test (w/)
Biodeterioration	Stereomicroscope Light microscope SEM	LRSM	- Prepare mounted thin section of weathered stone surface	- Reflected-light microscope - Scanning electron microscope	1 day of test (w/ composition)

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Tested Properties	Applied test	Resource/place	Preparation	Test Equipment & Reagents	Period of test
Compressive Strength	Compressive strength test	ASTM C170/ LRMS	- 3 samples for each condition = 12 non-reusable 5x5x5cm untreated cubes(parallel wet&dry, penpend wet&dry) = 12 non-reusable 5x5x5cm treated cubes(parallel wet&dry, penpend wet&dry)	Instron Testing Machine 1125	1-2 days of test (w/o)
Flexural Strength (Modulus of Rapture)	Three point bending test	ASTM D1635/ ASTM C99/ LRMS	- 3 samples for each condition = 6 non-reusable 2"x4"x1" untreated & 6 non-reusable 2"x4"x1" treated stone pieces(parallel and perpendicular)	Instron Testing Machine 1125	1-2 days of test (w/o)

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Tested Properties	Applied test	Resource/place	Preparation	Test Equipment & Reagents	Period of test
Water absorption	Water Absorption by Total Immersion	Teutonico Lab Manual Ex.8, 12, ASTM C-97/ Architectural conservation Laboratory	- Dry stone in oven (60c) for 24 hours - Cool down the stone = 3 reusable 5x5x5-cm untreated & 3 reusable 5x5x5-cm treated cubes	- Immersion tank Glass beads - Deionized water - Paper towel - Balance - Oven (60 c) - Beaker	2-3 days of preparation (w/) 1-2 weeks of test (w/)
	Capillary Water Absorption	NORMAL 11/85, RILEM II.6, Teutonico Manual Lab Ex.10/ Architectural conservation Laboratory	- Dry stone in oven (60c) for 2 days - Cool down the stone 3 samples for each condition = 6 reusable 5x5x5cm-untreated & 6 reusable 5x5x5-cm treated cubes (parallel & perpendicular)	- Tray - Ruler - Deionized water	2-3 days of preparation (w/ total immersion) 3-4 hours of test (w/)
Water vapor transmission	Water vapor transmission test	ASTM E 96-95, RILEM II.2/ ASTM C355-64/ Architectural conservation Laboratory	- Dry stone in the oven (60c) for 2days - Cool down the stone 3 disks for each condition (2 for tested, 1 for dummy) = 6 reusable untreated & 6 reusable treated disks (parallel & perpendicular)	- 3 stone disks* - Disposal beaker (250 ml) - Balance - Desiccator - Paraffin wax - Heat plate - Disposal pipette - Deionized water - Electrical tape	2-3 days of preparation (w/ total immersion) 2-3 weeks or more for the test (w/)

Tested Properties	Applied test	Resource/place	Preparation	Test Equipment & Reagents	Period of test
Soluble salt contents	XRD	LRMS	- Grind stone sample - Sieve the sample through a #200 sieve - Dry the fine sample in the oven until weight constant	- Sieve # 200 - Oven (110 C) - Balance - Aluminum tray	preparation (0 day w/ composition test) 0 days of test (w/ composition test)
	Soluble salt qualitative analysis	Ex.16-Teutonico/ Architectural Conservation Laboratory	- Grind stone sample	- Deionized water - Test tubes - Funnels - Filter papers - Dilute HCl acid - Diluted nitric acid - Dilute acetic acid - Sulfamic acid - Barium chloride - Silver nitrate solution - Zinc powder - Griess-Ilosvay's reagent	1 day of preparation (w/) 1-2 day of test (w/)

51

Tested Properties	Applied test	Resource/place	Preparation procedure	Test Equipment & Reagents	Period of test
Weathering of stone	Salt crystallization test	Teotonico Lab Manual Ex.15/ Architectural conservation Laboratory	- Dry stone in oven (60c) for 1-2 days - Cool down the stone = 3 non-reusable 5x5x5cm untreated cubes = 3 non-reusable 5x5x5cm treated cubes	- Oven - Beaker - Rod - Tray - Tray cover - Clamps - 14% Sodium sulfate decahydrate - Silica gel - Balance	2-3 days of preparation (w/ total immersion) 1-2 weeks of test (w/)
	Wet-dry cycle	ASTM- D-5313/ Architectural conservation Laboratory	- Dry stone in oven (60c) for 1-2 days - Cool down the stone = 3 non-reusable 5x5x5cm untreated cubes = 3 non-reusable 5x5x5cm treated cubes	- Immersion tank - Glass beads - Deionized water - Paper towel - Balance - Oven (60 c) - Beaker	2-3 days of preparation (w/ total immersion) 1-2 weeks of test (w/)
Depth of penetration	Iodine vapor	Kumar &Ginnel /Archiectural Conservation Laboratory	= 3 non reusable 5x5x5cm-treated cubes (treated by brushing)	- Iodine (solid) - Vacuum container	1 day of preparation 3 hours of test (w/o)

NOTE: (w/) = The period that can be shared with the other tests,

(w/o) = The period that only a specific test can be conduct

CHAPTER III

CHARACTERIZATION AND ANALYSIS OF STONE

Petrographic Examination

The litharenite sandstone in Thailand is mainly composed of quartz (40-50%), with some feldspar (10-25%) and small clasts of chert-arenite, siltstone, hard soil, and volcanic tuff (20-40%).[32] Microscopic analysis revealed the binding cement in this sandstone to be iron oxides, clay, silica, and calcite. Iron oxides and clay cement are easily susceptible to chemical degradation, resulting in disaggregation of grains and exfoliation of stone according to its bed plane splitting.[33] The stone blocks that had been carved and oriented 90° to their natural bed planes display more delamination than those oriented parallel to the bed planes.[34]

From transmitted-light microscopical observation of thin and cross section, the stone appears finely bedded. Two predominant grain textures that differentiate the layers can be observed. (See Figure 3.1) One is composed of large angular grains (0.7-$1x10^{-1}$ mm)

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32 Chiraporn Aranyanark. Raw data. Conservation Sciences Division, Bureau of Archaeology and Museums, Fine Arts Department, Ministry of Education of Thailand.

33 Chiraporn Aranyanark. (Thai) *The Role of Biological Growth on Ancient Monuments*. Conservation Sciences Division, Bureau of Archaeology and Museums, Fine Arts Department, Ministry of Education of Thailand.

of quartz loosely compounded together, the other is of small sub-rounded grains (2.5-$7x10^{-2}$ mm) with very compact packing. Calcite is present mostly in the former coarse beds while it hardly appears in the latter. A numbers of pores also randomly exist between the big grain-size quartz. (The percentage of porosity will be analyzed in the porosity analysis section.) The color of these two layers is also different, i.e. the former is grayish white while the latter is brownish-red, due respectively to the ratio of quartz grain to iron oxide matrix.

Comparison of thin sections of the weathered and freshly cut sandstone reveals the absence of iron oxide cement in the weathered samples. In the freshly cut stone, the grain particles—mainly quartz— are surrounded with a thin brownish-red layer of iron oxides, while in the weathered sample, the grains are loosely compacted together with some calcite binding, and there are more open pores present. Dissaggregating grains from the matrix using concentrated hydrochloric acid confirmed the iron oxide cementing matrix. The presence of calcite was confirmed with the red stain of Aroclor® staining color on the thin sections. (See Figure 3.1) The electron dot map of main constituent elements revealed that silica and oxygen predominated as the quartz grains, while aluminum, iron, and calcium appeared as a network indicating clay and iron oxide cementing materials. (See Figure 3.3) The locations of sodium in the map did not match with that of

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34 Chiraporn Aranyanark, (Thai) "The Deterioration of Sandstone Used in the Monuments in the Northeastern Region of Thailand" (Research Report, Conservation Sciences Division, Bureau of Archaeology and Museums, Fine Arts Department, Ministry of Education of Thailand).

chloride—the only possible salt observed—implying that the sodium might be feldspar.[35] (See Figure 3.2)

Surface depositing on the weathered stone was also examined in reflected light microscopy and scanning electron microscopy. Biological growth could be seen on the surface, especially in the areas that had been carved or deteriorated, as the formation of dark grayish-green layers on the surface. Lichen growth appears as green and gray patches and brown filaments. Under the scanning electron microscope, the depth of the hyphae penetration was about 25 microns. (See Figure 3.5) No evidence of salt crystal deposits or efflorescence was found either in the stone pores or on the surface.

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35 Ernest H. Nickel and Monte C. Nichols, *Mineral Reference Manual*, (New York: Chapman & Hall, 1991), 64.
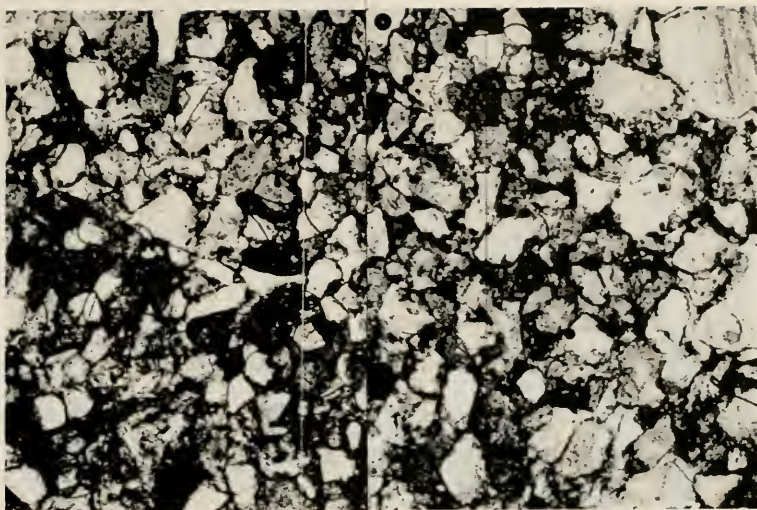
rent grain sizes of each layer could be observed.
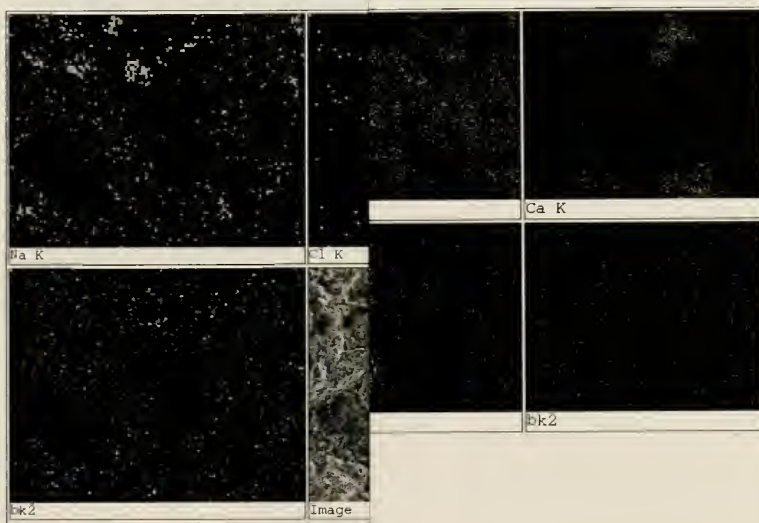
Figure 2: The electron dot maps of the main constituents: silicon, oxygen, calcium aluminum, and iron.
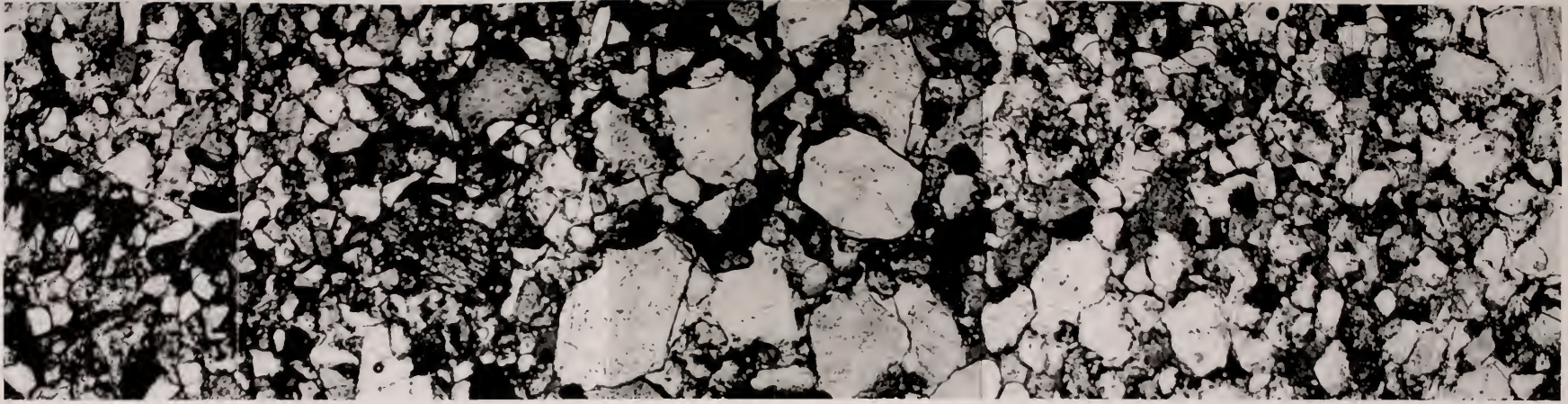
Figure 3.1: Plane-polarized light microscopic image of the stone. (100x magnification). The different grain sizes of each layer could be observed.
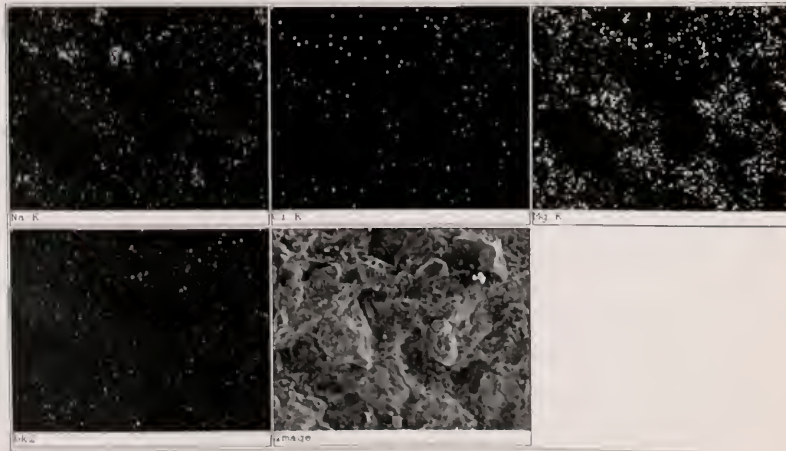
Figure 3.2: The electron dot maps of sodium, chloride, and magnesium.
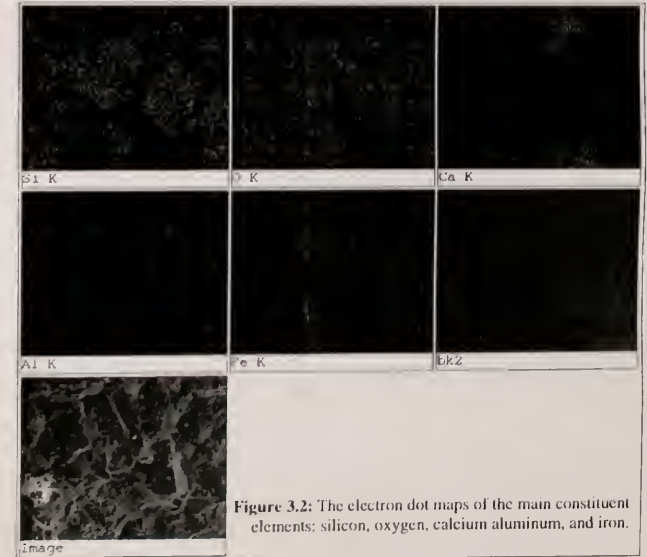
Figure 3.2: The electron dot maps of the main constituent elements: silicon, oxygen, calcium aluminum, and iron.
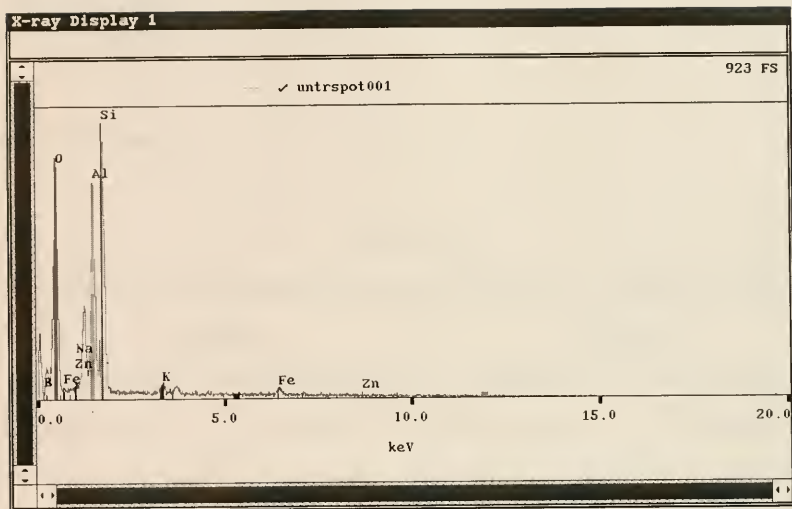
Figure 3.4: X-ray diffraction analysis of untreated weathered sandstone of the Northern Gopura. The main elements are silica, oxygen, and aluminum, with the presence of some sodium and iron.
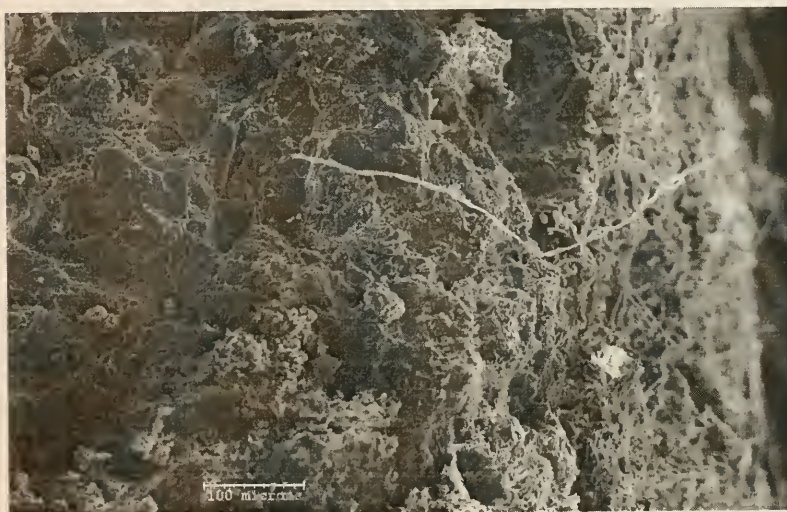
Figure 3.5: SEM image of biological growth depositing on the stone surface. (100x magnification, 15 KV)

Mechanical Testing

Compressive Strength

ASTM C 170			
STANDARD TEST METHOD FOR COMPRESSIVE STRENGTH OF DIMENSION STONE			
Wet Condition		Dry Condition	
Load Direction	Comp.Strength (psi)	Load Direction	Comp.Strength (psi)
Parallel to Bedding	**-2012.50**	Parallel to Bedding	**-10650.00**
Perpendicular to Bedding	**-2300.00**	Perpendicular to Bedding	**-11825.00**

Compressive strength tests revealed the stones were softer when wet, resulting in less load-bearing strength. The wet stones lost more than 400% (422% average) of their compressive strength, when compared to the dry stones. When the load was applied perpendicular to the stone's bed planes, the wet stones could bear 14% more loads than when the load was applied parallel to their bed plane. The dry stones could bear about 11% more load perpendicularly to the natural bed planes than parallel to the bed lines or rifts. At failure, the stones break unevenly parallel to the load direction when the load was perpendicular to the bed planes, and cracked along the bed planes when the load was parallel to the bed planes. Therefore, the dry stones oriented parallel to their bed planes have the highest compressive strength and can bear the greatest load. The wet stone loses its strength because the clay cementing material in the stone probably swells when water is present.

ASTM C 99			
MODULUS OF RUPTURE TEST (3-Point Bending Test)			
Wet Condition		Dry Condition	
Load Direction	*R* (psi)	Load Direction	*R* (psi)
Parallel to Bedding	**-294.42**	Load Parallel to Bedding	**-1252.07**
Perpendicular to Bedding	**-233.47**	Load Perpend. to Bedding	**-1152.89**

The results of this test, like those of compressive strength, show that wet stones are weaker, and bear less load than dry stones. The wet stones lost about 360% average (325% and 394%) of their compressive strength, if compared to the dry stones. In contrast to compressive strength, when the load was applied perpendicular to the stone's bed planes, the wet stones could bear 26% less load than when the load was applied parallel to their bed plane. The dry stones could bear about 9% less load perpendicular to the natural bed planes than those directly to the bed lines or rifts.

Water Absorption by Total Immersion

NORMAL 7/81 WATER ABSORPTION BY TOTAL IMMERSION				
WATER ABSORPTION CAPACITY				
SAMPLE	Original Dry Weight(g)	Saturated Weight(g)	Redry Weight(g)	WAC
1	295.96	311.90	298.26	4.60
2	295.85	311.67	298.08	4.56
3	295.98	311.81	298.13	4.60
Average	**295.93**	**311.79**	**298.16**	**4.59**

The maximum quantity of water that the stone could absorb was 4.57% of the dry weight. The rate of water absorption was high on the first day of immersion, and decreased significantly after one day (less than 0.1% increasing weight between two consecutive weighings every 24 hours) until almost stable after twelve days (less than 0.01% increasing weight).[36]

Porosity of Stone

Stone porosity was calculated as 13.06% by total immersion and 12.78% by hydrostatic weighing.[37]   This value indicates that the stone has relatively low porosity when

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36 See the raw data in Appendix II
37 See Appendix II for the result table of the total immersion test and hydrostatic weighing.

compared with the range of literature values for total porosity, i.e. 0.5-42 %, for sandstone in general.[38]

Water Absorption by Capillary Action

Teutonico's Lab Manual Exercise 10															
WATER ABSORPTION BY CAPILLARY ACTION															
SAMPLE		**HEIGHT(cm)**													
	Min.	1	2	3	4	5	10	15	20	25	30	60	90	120	150	180
Parallel to Bed	1	1.30	1.60	1.75	1.90	1.90	2.10	2.25	2.35	2.40	2.50	2.80	2.85	2.90	2.95	2.95
Parallel to Bed	2	0.80	1.00	1.20	1.30	1.35	1.60	1.60	1.80	1.90	2.00	2.20	2.25	2.25	2.25	2.25
Parallel to Bed	3	0.90	1.10	1.25	1.30	1.40	1.70	1.70	1.80	2.00	2.10	2.30	2.50	2.60	2.60	2.60
Average (cm.)		**1.00**	**1.23**	**1.40**	**1.50**	**1.55**	**1.80**	**1.85**	**1.98**	**2.10**	**2.20**	**2.43**	**2.53**	**2.58**	**2.60**	**2.60**
Perpend to Bed	1	0.8	1.10	1.15	1.20	1.40	1.40	1.50	1.50	1.60	1.60	1.80	1.90	1.90	1.90	1.90
Perpend to Bed	2	0.6	0.90	1.10	1.20	1.20	1.50	1.60	1.65	1.65	1.65	1.80	1.90	1.90	1.95	1.95
Perpend to Bed	3	0.6	0.85	1.15	1.20	1.25	1.50	1.50	1.60	1.70	1.75	1.80	1.85	1.90	1.90	1.90
Average (cm.)		**0.67**	**0.95**	**1.13**	**1.20**	**1.28**	**1.47**	**1.53**	**1.58**	**1.65**	**1.67**	**1.80**	**1.88**	**1.90**	**1.92**	**1.92**

The amount of water that could be absorbed by the stone through capillary action was 2.60 cm parallel to bed plane direction. The amount of water absorbed gradually increased until it reached the maximum point of capillary force after about one hour.

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38 P. Rossi-Doria, "Pore Structural Analysis in the Field of Conservation: State of the Art and Future Developments," in Principle and Applications of Pore Structural Characterization: Proceedings of the RILEM/CNR International Symposium, Milan, Italy, April 26-29, 1983, (Bristol: J.W. Arrowsmith Ltd., 1985), 441-459.

Soluble Salt Content Analysis

The stone sample was ground and mixed with deionized water in a tube in order to dissolve any soluble salts. The solution obtained from this mixture was divided into tubes and tested. From the qualitative analysis of soluble salts in the stone, two types of salts were observed: chlorides and nitrates. With the test reagents, the chemical reactions were as follows:

Chlorides:

Drops of diluted nitric acid (HNO_3 -2N concentration) and silver nitrate solution ($AgNO_3$ –0.1N concentration) were added to the solution in the first tube. Precipitation of silver chloride ($AgCl$) produced a whitish-blue gelatinous precipitate, and indicated the presence of chloride ions in the solution.

$$Cl^- \; + \; AgNO_3 \; \longrightarrow \; AgCl \; + NO_3^-$$
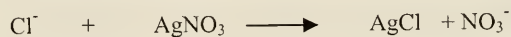
However, the result obtained from a chloride concentration measurement strip indicated that the amount of chloride contaminating in the stone was as little as the lowest concentration, i.e. 0-500 mg/l.

Nitrates:

Drops of dilute acetic acid (CH_3COOH –2N concentration) and Griess-Ilosvay's reagent were added into the soluble salt solution in the second tube. No pink reaction indicated the absence of nitrites. For the nitrate test, a small amount of zinc was added to the same solution in order to convert nitrates to nitrites. The previous procedure was repeated and the pink reaction indicated the presence of nitrates.

A nitrate/nitrite concentration measurement strip indicated that there was 10-25 mg/l of nitrate in the solution, while no nitrite was detected and measured.

It is possible that the chloride in the stone derives from the soil and the original sandstone mineralogy in the northeastern part of Thailand. The "Korat" Terrestrial Sedimentary Rock is composed of several layers of sodium chloride compressed and lifted 30-80 meters from the base, which reaches the underground water level and creates the sub-layer of salt water underground. This salt is transported to the soil by capillary action and diffusion, and then by evaporation. It crystallizes and is deposited in the soil, resulting in the salty soil of the Korat Basin.[39] Nitrates in the stone may derive from the decomposition of nitrogenous material deposited on the stone or soil.[40]

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39 Information from the Department of Mineral Resource, the Ministry of Science and Industry, Thailand, available in Thai language at http://www.dmr.go.th

40 Jeanne Marie Teutonico, A Laboratory Manual for Architectural Conservators, 60.

CHAPTER IV

DECAY MECHANISMS

The decay and deterioration of stone are primarily caused by a combination of weathering processes. This includes weathering agent such as liquid water—either direct precipitation or ground water, general gases and atmospheric pollution, salts—either soluble or insoluble, microflora and vegetation, and animals.[41] Stone deterioration also depends on the specific surface areas exposed to these agents, the susceptibility to weathering of the mineral composition, and the homogeneity of the stone, which affect the degree and rate of alteration.[42] Generally, the decay mechanisms of sandstone are mainly dependent on its physical characteristics and its mineralogical and petrographic composition.[43]

Effects of Water

Water itself can be harmful to any stone once it enters the porous network by absorption or adsorption. Depending on the porosity of stone, water can penetrate in and out from the stone pore network by several ways and in various forms—diffusion and

41 E.M.Winkler, *Stone: Properties, durability in Man's Environment* (New York: Springer-Verlag, 1973), 88.
42 Giovanni G. Amoroso and Vasco Fassina, *Stone Decay and Conservation,* (New York: Elsevier, 1983), 2.
43 P. Malesani et al, "Applications of Mineralogical- Petrographic Techniques to the Study of S. Reparata Basilica (the Old Cathedral of Florence)," in *Proceeding of the 1ˢᵗ International Symposium on the*

condensation of water vapor, transportation of liquid water by capillary action, and evaporation of liquid water. The first phenomenon depends on the relative humidity and temperature of the atmosphere. That means the diffusion of water vapor will occur when a dry stone is placed in a humid atmosphere. The moisture in the air will rapidly diffuse into the stone until it reaches equilibrium. Condensation will occur when the limit of water vapor concentration for a given temperature is reached, and when the vapor arrives on another surface that has the same or lower temperature than the dew point.[44] For the second case, capillary action depends on the stone's pore structure as to whether or not it can be attracted to water due to the oxygen atoms contained in the pore body's compounds. These atoms are negative electrical charges (-) that are attracted to hydrogen atoms (+) in water molecules, and make the pores' wall hydrophilic. The diameter of the pore is also relevant. The smaller the pore, the higher the suction pressure. Lastly, the evaporation of water takes place where a saturated stone is exposed to the flow of air that has a lower relative humidity and a higher temperature. That means the rate of evaporation varies with the increase of temperature, decrease of relative humidity, and especially the increase of airflow velocity. The rate of evaporation can increase about five times when the stone is exposed to a mild breeze, and about three times when it is

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Conservation of Stone, Bologna, June 19-21, 1975, (Bologna: Centro C. Gnudi perla Conservazione delle Sculture all'Aperto, 1976), 81-88.

44 It can be easier explained that when water vapor migrates from a warmer place to a colder one, once it arrives to a place that is colder than its dew point, it condenses.

exposed to the sun.[45] In the case of rising dampness, surface diffusion and water vapor diffusion from the ground seem to be more important than capillary action.[46]

The following deterioration problems have been observed in Thai sandstone due to the effects of water:

Dampness in Stone

This decay mechanism occurs when stone retains water or moisture inside its pores for a prolonged period. Sandstone—like all porous materials, has a pore network of different sizes and shapes.[47] Water can easily evaporate in the pores near the surface; however, water evaporation is more difficult in deeper or very small pores; water is consequently trapped in them. Additionally, in large pores, water molecules can pass into the vapor phase more quickly than in the small pores because in the small pores the attractive forces between the pore wall and water molecule is stronger than the large pores. This can interfere with the evaporation rate.[48]

Water trapped inside the stone can cause several problems. Not only can water dissolve soluble salts and allow them to penetrate the stone causing more problems (in

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45 Giovani and Ippolito Massari, *Damp Buildings Old and News,* (Rome: 1CCROM, 1993), 28.
46 R.Snethlage and E. Wendler, "Moisture Cycles and Sandstone Degradation," in *Saving Our Architectural Heritage,* (New York: John Wiley & Son), 7-24.
47 Robert L.Folk, *Petrology of Sedimentary Rocks,* (Texas: Hemphill's, 1968).
48 Giovanni G. Amoroso and Vasco Fassina, *Stone Decay and Conservation,* 14.

the next topic), but the water itself is also harmful, especially when it changes phases creating pressure inside the pores. The details of this mechanism will be discussed in the following section. When the stone is in a wet state, clay—one of the stone's cementing material—absorbs water, and swells. Consequently, the stone becomes softer and tends to delaminate. In addition, under suitable conditions, water creates the appropriate environment for biological growth on stone, which will also be discussed in a later section.

Wet-Dry Cycling

In tropical climates annual precipitation is extreme. When rain falls, stone will absorb water into its pores by capillary action vertically and horizontally, resulting in wet stone. With solar radiation and wind, the water in wet stone will start evaporating. These fluctuations from day to day and even within a day contribute to wet-dry cycling in stone, causing the stone to dilate and contract due to the change of moisture content in stone – the stone will expand during the wetting state and contract during the drying state. Nevertheless, these phenomena happen only when the water is salt-free. When the water is contaminated with salts, e.g. sodium chloride (NaCl), the stone reacts in opposite ways—contracting during the wetting state producing a smaller grain-to-grain distance, and expanding during the drying state.[49] In summary, the premature deterioration and

49 R.Snethlage and E. Wendler, "Moisture Cycles and Sandstone Degradation," 7-24.

displacement of grains contribute to the loosening of the grain structure, and consequently, the friability[50] of the stone.[51]

The types of damage depend on the zone of the maximum water content. Normally, if salt is present, the greatest salt deposition is the same area as the zone of maximum water effect, which is parallel to the surface. If such a zone is located on the surface, the crumbling occurs. If it is about 1-2 mm beneath the surface, the stone will be damaged with a thin scaling. The deeper the maximum water content zone, the thicker the scaling occurs. Flaking and delamination will take place as a transition between crumbling and scaling zones, and if delamination occurs parallel to the stone's bed plane, it weakens the boundary of each bed plane leading to detachment and loss.[52, 53] (See Figure 4.1 and 4.2)

The climate in Northeastern Thailand contributes to this cycle. The rainy season occupies almost half a year, i.e. from the middle of May to the middle of October, with 1401.2 mm of rainfall. This region also experiences a long period of warm weather with the temperature ranging from a mean maximum of 34.9°C (about 92°F) with the average

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50 See Appendix I for the definition.
51 Ibid.
52 Ibid.
53 See the definition of deterioration problem terms in Appendix I.

at 28.4°C (about 85°F) in summer. Surface wind is influenced by northeast monsoons with some tropical cyclones during the rainy season. [54]

Normally, wet-dry cycling damage simultaneously occurs with freeze-thaw cycling. However, in the Nakhon Ratchasima province, the lowest temperature during a 50-year period (happened in January 12, 1955) was only 4.9°C.[55] Therefore, the freeze-thaw cycling damage has never occurred to the stone at this site or nearby places in the same region.

Weathering of Cementing Material

Since the Lith-arenite sandstone is largely ferruginous, the cementing material is principally clay and iron oxide matrix. Once iron oxide absorbs water from the environment, it will convert into hydrated iron oxides (limonite) or iron hydroxides (goethite). This chemical reaction is associated with the volume change of the compounds leading to exfoliation and granular disaggregation.[56] Moreover, the conversion of iron oxide to limonite and goethite causes the stone to be softer and more friable due to the insufficiency or loss of its cementing material. (See Figure 4.3)

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54 Data from the Meteorological Department. Statistic of Meteorological Elements in Various Parts of
    Thailand During 30 Year Period (1961-1990). See Appendix III
55 Ibid.

Water-Soluble Salts

Water is also the major solvent for transportation of soluble salts into the stone. Salts come from various sources—original components of stone, associated materials, soil, atmosphere, etc. However, it is inevitable that water plays the major role in the deterioration of stone by soluble salts. Salt would not be harmful to stone at all if the water were absent.57 The decay patterns caused by the presence of salts depend on their types and properties, their concentration, the conditions of stone, and the environment that can affect salt solutions.

Crystallization of Soluble salts

As previously discussed, the transportation of soluble salts into stone relies on water. That means water dissolves salts from various sources, such as from the atmosphere and the soil, and then, salt solutions penetrate the stone. Sometimes, only the presence of water can dissolve the salts which already exist in the stone as the stone's natural component or contamination. With the process of water evaporation which occurs after the process of re-migration of the solution to the surface by capillary action, the concentration of the salt solution will be increased. Consequently, crystallization will take place during the state between saturation and supersaturation, but not during the state

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56 Chiraporn Aranyanark, "Major Problems in Conservation of Monuments in Thailand," in Preprinted of the 1st Seminar on Thai-Japanese Cooperation in Conservation of Monuments in Thailand, Bangkok,

of unsaturation.[58] Salts start crystallizing first at the surface of stone when the water has already evaporated and the stone is dry. Efflorescence, therefore, will deposit when the stone is exposed to the low relative humidity with the high rate of water evaporation. The places for salt crystallization, therefore, depend on the solubility of salts and the rate of water evaporation, as well as the moisture supply from the stone interior and the rate of surface ventilation.[59] Cryptoflorescence, on the other hand, can often deposit within the pores, especially the large pores between dry and wet areas, when the water evaporation takes place below the stone surface. This leads to the decay of stone because of salts' hydrostatic crystallization pressure exerting to surrounding stone composition. The surface will be disaggregated, the thin surface layer will crumble, and the bed plane will be blistered or exfoliated[60]. However, there is a contradiction that this kind of pressure is not predominantly harmful to the stone. This is because the solid salts that crystallize by the process of water evaporation from the solution occupy less volume than the original solution, and will not create any harmful pressure to the pores.[61]

Another pressure produced by salt crystallization is hydration pressure. This occurs as a volume increase when salts try to adjust themselves to be in another hydration

March 8-9, 1999.

57 E.M. Winkler, Decay of Stone Conservation of Stone, Vol. I, (New York: IIC, 1970), 1-14.

58 E.M.Winkler, Stone: Properties, durability in Man's Environment, 119.

59 Giovanni G. Amoroso and Vasco Fassina, *Stone Decay and Conservation,* 29.

60 G. Torraca, Porous Building Materials-Materials Science for Architectural Conservation, (Rome: ICCROM, 1981).

61 From A. Elena Charola, "Salt in the Deterioration of Porous Materials: An Overview," *Journal of the American Institute for Conservation* 39, no.3(2000): 327-343. With the review of the article of H. Mortensen (1933).

state.[62] The adjustment will occur when the relative humidity increases resulting in an increase of absorbed water to the stone. This phenomenon, therefore, depends on the change of the atmospheric temperature and humidity. Generally, low temperature and high humidity contribute to the highest pressure.[63] Moreover, the degrees of effect are different in different kinds of salts, and are associated with their hydration pressures and the rate of hydration. Sodium sulfate is one of the most effective deteriorating salts. The hydration of the sodium sulfate thernardite to mirabilite is more rapid than that of the other salts. The hydration-dehydration process of sodium sulfate can repeat several times a day—creating a rapid change that affects the stone more than the amount of pressure the process produces.[64] The rapid change happens because the hydration process of this salt is not dependent on the water absorption by a solid crystal, but on the dissolution of salt crystals and re-precipitation from the solution.[65]

Differential Thermal Expansion of Salts

The differences of thermal expansion of soluble salts compared with that of the substances in stone could also cause the deterioration of the stone. When the temperature increases, the stone, including every substance, will expand but in different ratios. It has been tested that NaCl expands 0.5% while every substance in granite expands less than

62 Giovanni G. Amoroso and Vasco Fassina, *Stone Decay and Conservation*, 45.
63 E.M.Winkler, "Salt Action on Stone in Urban Buildings," in *Application of Science in Examination of Works of Art*, (Boston: Museum of Fine Arts, 1973).
64 E.M.Winkler, Stone: Properties, durability in Man's Environment, 125.

0.2% when the temperature is increased from 0°C to 60°C.[66] Nevertheless, this effect is only a minor cause of the overall salt decay mechanisms.[67]

The decay patterns caused by salts that can commonly be found are flaking and powdering of the stone. Generally, the concentration of salts will be higher in the areas that have more moisture content—about 1-5 cm from the surface depending on the stone's porosity,[68] and these areas are more susceptible to deterioration. However, as previously discussed, if the volume of cryptoflorescence is larger than the pore or the hydration pressure occurs inside the pore producing stress that exceeds the limit of its resistance, the stone will be gradually broken, and become more porous causing it to be more susceptible to further damage caused by water and salts.[69]

According to qualitative salt analysis, there were, however, only two soluble salts found in the stone—chloride and nitrite. Carbonate is the intrinsic constituent which can behave as another salt. There were no salt efflorescence deposits observed. As summarized in these test results, salts do not greatly affect this sandstone within such an environment in the Nakhon Ratchasima province in Thailand. Therefore, this thesis will not focus on this kind of decay mechanism except using sodium sulfate as an agent for the artificial weathering test—salt crystallization.

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65 A. Elena Charola, "Salt in the Deterioration of Porous Materials: An Overview," *Journal of the American Institute for Conservation* 39, no.3(2000): 327-343.
66 C. W.Cooke and I.J. Smalley, "Salt Weathering in Desert," in *Nature* 220 (1968): 1226-1227.
67 A. Elena Charola, "Salt in the Deterioration of Porous Materials: An Overview," 327-343.
68 R. Snethlage and E. Wendler, "Moisture Cycles and Sandstone Degradation," 7-24.

Inevitably, stone in tropical countries suffers from biodeterioration much more than in temperate climates.[70] This is because the hot and humid environment provides the ideal conditions for biological growth. Microorganisms, like fungi, bacteria and algae, grow rapidly and abundantly in countries like India, Sri Lanka, Thailand, Malaysia and Indonesia.[71] Biological growth includes lichens, mosses, and liverwort. They can either physically or chemically affect stone, especially on the surface. Physically, the damage will be the result of the mechanical action of these organisms. The biological growth will burrow beneath the stone surface and disaggregate the mineral grains from their cement. The deposition on the surface of biological growth can clog the stone's pores, lower the evaporation rate, and lead to the longer retaining of moisture inside the stone.[72] Chemically, the biological organisms release some acids and/or complex agents that deteriorate the stone. These agents include the following: organic acids such as formic acid, acetic acid, glycolic acid, citric acid; inorganic acids such as nitric acid and sulfuric

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69 Giovanni G. Amoroso and Vasco Fassina, *Stone Decay and Conservation,* 46.
70 O.P. Agrawal, "An Overview of Problems of Biodeterioration of Cultural Property in Asia," in
    Proceeding of the 3rd International Conference on Biodeterioration of Cultural Property, Bangkok,
    Thailand, July 4-7, 1995, (Bangkok: Thammasat University Press, 1995), 14-34.
71 Ibid.
72 R. Snethlage and E. Wendler, "Moisture Cycles and Sandstone Degradation," 7-24.

acid; and other chemical chelator or ligand radicals that can react with cathode radicals and become several complex salts such as CN⁻, NH₃, etc.[73,74]

Biological growth like lichens, mosses, liverworts, and higher vegetation are visually observed and identified in the field and in the laboratory through visual analysis. In contrast, microorganisms (e.g. bacteria, fungi, and algae) are not easily identified with only optical observation. They need to be studied involving the isolation and characterization of the active microbial agents, the re-creation of the geomicrobial process, and the characterization of active microbial agents.[75]

The effects of biological growth depend on their types. The most effective biological growth that has deteriorated sandstone monuments in Thailand is lichens. They are crustose, foliose and squamulose lichens that often form on the stone surface, especially in the rough areas.[76] In such areas, the algae also grow well. For lichens, the stone beneath the thallus of lichens is burrowed, disaggregated, and loses its cementing material. Where feldspars exist, they can be converted to clay minerals.[77]

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73 Chiraporn Aranyanark. (Thai) *The Role of Biological Growth on Ancient Monuments*. (unpublished, 2000).
74 D. Jones and M.J. Wilson, *Chemical Activity of Lichens on Mineral Surface: A Review*, International Biodeterioration Bulletin 21: 99-105.
75 H.L. Ehrlich, *Geomicrobiology*, (New York: Marcel Deckker).
76 Chiraporn Aranyanark. (Thai) The Role of Biological Growth on Ancient Monuments.
77 Suporn Intasopa and Srisopa Maranet, *Peterographic and Mineralogical Study on Sandstone Affected by Lichens and Algae*, the Department of Mineral Resource, the Ministry of Science and Industry, Thailand. (Unpublished, 2000).

There are some evidences of biological growth depositing on the sandstone of the Northern Gopura of the Phimai Sanctuary. The types of biological growth and their effect, however, need further study in order to provide appropriate treatments to the stone, such as cleaning and protection, which will not be investigated and discussed in this thesis. (See Figure 4.4)
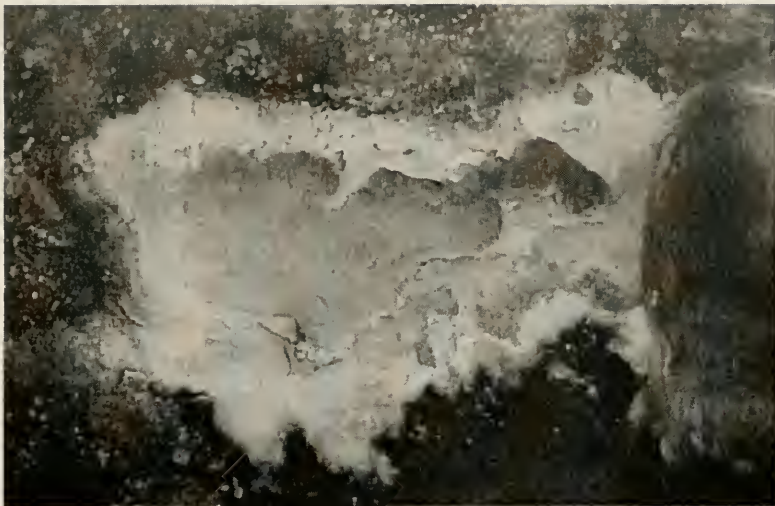
Figure 4.1: Flaking of sandstone surface
The evidence of dampness in stone leading to the flaking of thin layers of the stone surface.
Stone condition under the lintel of the Northern Gopura's Hall(June 2000)

Figure 4.2: Scaling of sandstone surface
The deeper dampness in the stone leads to the flaking and scaling of the stone surface.
The condition of the stone wall of the Northern Gopura. (June 2000)
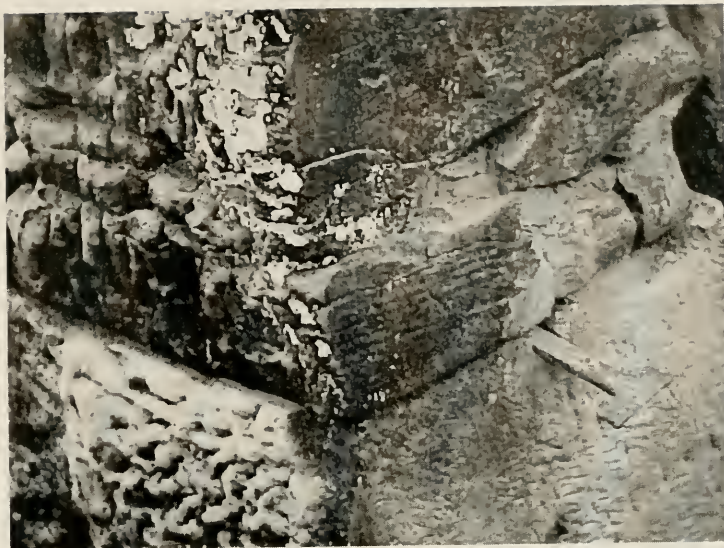
Figure 4.3: Crumbling and dissaggregation of sandstone.
The friability of the sandstone blocks of the Northern Gopura's wall
is due to the loss of cementing material. (June 2000)

Figure 4.4: Lichen on sandstone surface
The biological growth indicates an evidence of dampness in the sandstone
of the Northern Gopura's wall. (June 2000)

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CHAPTER V

CONSOLIDATION METHOD

Sandstone that has lost its integral cementing minerals through chemical weathering shows deterioration patterns of dissaggregation or sugaring, crumbling, flaking, scaling, and delamination.[78]   The internal stone matrix can be restored by introducing a new, durable binding material working as a consolidant within the pore system. This process can improve the mechanical properties of the stone, such as mechanical strength and abrasion resistance.[79] Therefore, the aims of a consolidant are to improve the cohesion of the mineral constituents, to be a good adhesive material between the areas that are deteriorated and that are still in good conditions or well preserved, and to increase or restore the mechanical strengths of the chemically weathered stone. In addition, consolidation should not radically alter the porosimetry of the stone, especially its water vapor permeability, or visually alter its appearance.

This chapter will discuss the methodology and rationale for the choice of consolidant based on the performance criteria for consolidants and a comparative review of consolidation materials focusing on their advantages and disadvantages. The chosen consolidant will be studied in detail following with the application processes.

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78 See Appendix I for the meanings of each deterioration problem technical term.
79 Helmut Weber and Klaus Zinsmeister, *Conservation of Natural Stone,* 53.

To determine the most effective consolidant, the type, quality and degree of deterioration of a weathered stone must first be characterized and evaluated thoroughly in order to find a consolidation material that is compatible with the stone's original constituents as well as with the patterns and degree of deterioration. In addition, there are basic performance requirements that a consolidant must meet. These requirements can provide a framework for selecting an efficient consolidant, and for evaluating its effectiveness. The properties, advantages and disadvantages of each consolidant available in the market have to be compared and evaluated. Selection of a consolidant is ultimately based upon the one that combines the most advantages with the least disadvantages and is the most compatible with the stone, as well as least harmful to both the user and the environment.

Performance Criteria for Consolidation Treatment

NORMAL Recommendation 20/85 specifies four requirements for a consolidant.[80] A consolidant should:

-   Not lead to the formation of deteriorating byproducts.

-   Be uniformly absorbed by the treated stone, reaching all deteriorated areas, and binding the stone to the sound parts.

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[80] A.Elena Charola, "Laboratory Tests and Evaluation of Proposed Masonry Treatment," *APT Bulletin* 16, no.4 (1995): 35-39.

- Have its thermal expansion coefficient as near as that of the treated stone; thus, cracks between the grains and consolidant will not occur when the stone expands or contracts due to the change of temperature.
- Maintain the stone's external appearance. That means it must not alter, discolor, change the texture, or stain the stone substrate.

This thesis considers a more comprehensive list of requirements, including that a consolidant should:

- Deeply penetrate the stone without causing scaling or discontinuities between the treated external zone or surface and the core.[81]
- Cement the grains together in order to increase the mechanical strength.[82]
- Minimally change the original stone's permeability to water vapor.
- Decrease the porosity of the treated stone, if compared with the untreated one. This decrease leads to the reduced liquid water penetration of the stone. With partial reduction in porosity that results in decreasing water penetration, the stone should retain good water vapor permeability.[83]
- Be reversible or allow retreatment. This criterion seems theoretical rather than practical. In practice, it is almost impossible to remove a consolidant that has well penetrated and strongly adhered to the stone without damaging the stone substrate.

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81 M. Laurenzi Tabasso, "Conservation Treatments of Stone," in *The Deterioration and Conservation of Stone*, Unesco, 271-289.
82 Andreas Rohatsch et al, "Physical Preperties of Fine Grained Marble Before and After Conservation," in *Proceedings of the 9th International Congress on Deterioration and Conservation of Stone, Venice, June 19-24, 2000*, (New York: Elsevier), 453-458.

-   Not cause any damage to the treated stone. Absence of deleterious chemical or physical interactions between a consolidant and stone is required.[84]

-   Be minimally hazardous to health and/or safety during handling and application as well as compatible with ecological demands.

-   Be durable against weak acids, alkaline solutions and UV-radiation.

It is practically impossible that a consolidant can be effective and simultaneously meet all of the criteria. Therefore, the evaluation of possible consolidants should concentrate on the necessary and minimum level of stone alteration with the most improvement to the physical properties.

Comparison of Consolidation Materials

Organic Consolidants

-   ***Natural Resin: Linseed Oil and Waxes***

Linseed oil has been applied to strengthen weathered stones since the Middle Ages. Nevertheless, it is not an effective consolidant because it cannot achieve the preferable depth of penetration and long-term durability. Moreover, it is more

83 M. Lauranzi Tabasco, "Conservation Treatments of Stone", 271-289.
84 Helmut Weber and Lklaus Zinsmeister, *Conservation of Natural Stone*, 54.

efficient for low porosity stones rather than for high porosity stones.   Other

disadvantages are its discoloration and irreversibility.[85] In the 1920s, it was criticized

for its harmful effects in consolidation of stone works.[86]

Waxes—either bee or paraffin waxes, have been used for more than 100 years as

consolidants for corroded iron and for decayed stone. For stone conservation, they

are high viscosity agents, making it impossible to achieve deep penetration. They

must be dispersed in varnish by heating the solution, which is then applied hot as a

melted liquid. Although the varnish is later removed from the treated stone, the

waxes still remain due to its irreversibility. The other drawbacks are their strong

discoloration and attraction to dirt during hot weather.

- ***Synthetic Resins***

In the first half of the 20th century, epoxies, acrylic, silanes and silicones were tested

and applied to consolidate weathered stones. It is possible to remove the applied

synthetic resin products from the treated stones, especially from their surface, by

means of solvents.[87] Epoxies, however, cannot be removed because they cross-link,

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85 Giorgio Torraca, "General Philosophy of Stone Consolidation," *The Deterioration and Conservation of Stone*, Unesco, 243-270.
86 A.R. Warnes, *Building Stones*, (London: Benn, 1926).
87 Giorgio Torraca, "General Philosophy of Stone Consolidation," 243-270.

and consequently lose their solubility during their setting period. Synthetic resin consolidants have a low depth of penetration because they are composed of large molecules. To achieve deeper penetration, organic synthetic resins can be directly applied to the stone as monomer molecules. They will polymerize in situ with ultraviolet light or internal catalysts during the curing period. This process, however, can cause some cracks in the treated stone due to stresses produced by the polymerization. Diluting synthetic resin in volatile solvents is another way to improve the good depth of penetration.[88]

Epoxy resins are effective hardeners. They have high strength and gap-filling ability.[89] However, they block water transportation, and discolor (to yellow) when exposed to UV light.[90] The need for their two-part mixture—one is the epoxide group and another is the hardener that reacts with the former and cross-links the molecules to provide the product's strengthen property—complicates the application and risks the mixture accuracy.

Acrylic resins are good in their stability when exposed to oxygen or ultraviolet radiation. They have a good depth of penetration when used in monomer forms. Due to its low viscosity solvent, polymethyl methacrylate (PMMA), polymerized from methyl methacrylate monomer by oxidation under ultraviolet light, performs a good

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[88] Helmut Weber and Lklaus Zinsmeister, *Conservation of Natural Stone*, 60.

depth of penetration in treated stone the process, however, is difficult. Thus, acrylic resin treatments are better in adhesion rather than consolidation. In addition, they cannot be used to consolidate stone structurally.

Polyester resins have been used as stone adhesives and consolidants, especially the former due to their high bonding strength. The drawback is that they are not resistant to ultraviolet radiation and alkalis, while they are also moisture sensitive during curing. Thus, it is not suitable to apply polyester resins to outdoor stone works, especially in tropical climates where sunlight is intense.

Polyurethane resins create urethane bonds produced by reaction between isocyanate and alcohol groups. They tend to form elastic polymers that are not good practical consolidants and are irreversible.

- ***Ethyl Silicate (Tetraethoxysilane, tetraethyl silicate, tetraethylorthosilicate-TEOS)***

Silicic acid esters have been used as a consolidation material for stone for several decades because of the similarity of its polymerized gel to many stone materials.91

89 R.A. Munnikendam, "Preliminary Notes on the Conservation of Porous Building Materials by Impregnation with Monomers," *Studies in Conservation,* 12:158-162.
90 E.M. Winkler, *Stone in Architecture*, (New York: Springer-Verlag Berlin Heidelberg, 1994), 271.
91 Ibid, 159.

For this reason, it is the most frequently used consolidant for sandstone.[92] It will react with moisture, and polymerize through hydrolysis and subsequent condensation process to silica gel (SiO_2) and ethanol (C_2H_5OH) as follows:

$$Si(OC_2H_5)_4 \quad + \quad 4H_2O \quad \longrightarrow \quad SiO_2 + \quad 4\,C_2H_5OH$$

The product from such polymerization in the treated material is an amorphous silica gel. The ethanol, which may be so-called a byproduct, is not harmful to the treated stone.

Ethyl silicate is considered to be an effective consolidant due to its durability with no discoloration. Due to its low viscosity and slow rate of hydrolysis, it deeply penetrates stone pores.[93] Consequently, the deposition of silica gel after the polymerization process will successfully work as a cohesive material not only on the surface, but also in the core of treated stone. Since it does not change the permeability of the treated stone, the water trapped inside the pores before the polymerization can still evaporate from the stone. Nevertheless, treatment with ethyl

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92 E.M. Winkler, *Stone in Architecture*, 270.

93 Peter J. Koblischez, "Polymers in the Renovation of Buildings Constructed of Natural Stone in the Mediterranean Basin," in *Conservation of Monuments in the Mediterranean Basin, Proceeding of the 3rd International Symposium, Venice, June 22-25, 1994*, (Venice: Beni Artistici e Storici, 1994), 849-854.

silicate is irreversible,[94] and care must be taken if the product is dissolved in methanol or methyl ethyl ketone (MEK) which is toxic.

Inorganic Consolidation Materials

- ### *Limewater: Calcium Hydroxide*

Limewater is the oldest and most natural consolidation method for calcareous materials.[95] It has been used for centuries on the consolidation of limestone architectures and sculptures. The technique is to apply a freshly burnt lime paste on the weathered surfaces. However, the very slow rate of calcification and limited water solubility of lime make such a technique ineffective. Additionally, it does not increase the strength of the treated substrates,[96] and is not suitable for consolidation of siliceous materials.

- ### *Barium Hydroxide*

Barium is chemically similar to calcium. Therefore, the barytization process is based on the same chemical principle as the treatment with limewater. It has been used as a

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94 C.V. Horie, *Materials for Conservation*, (Boston, Butterworth, 1987), 159.
95 E.M. Winkler, *Stone in Architecture*, 267.
96 C.A. Price, "The Consolidation of Limestone Using a Lime Poultice and Limewater," in *International Colloquium in Natural Stone Conservation, Munich, May 21-22, 1984*, (Munich: Arbeitsheft 31, 1984), 148-151.

stone consolidant since the 19th century. Once barium hydroxide is applied to stone, it will react with carbon dioxide in the atmosphere, and become barium carbonate. When applied to weathered calcareous stone, the barium carbonate will replace the lost carbonate constituents of the weathered stone, resulting in an improved consolidated stone.

There was an experiment proposed by S.Z. Lewin using a homogeneous solution of barium hydroxide and urea, with immersion application method.[97] He also later proposed the addition of glycerine to the process. Lewin proclaimed that the use of gylcerine could prevent the rapid drying of the solution while the addition of urea provided a good depth of hydroxide penetration. The urea also constituted a source of carbon dioxide for the hydrolysis process. With this new method of application, the barium carbonate—product of barium hydroxide hydrolysis—should deposit more deeply than was originally possible as a stone grain binder in the treated stone.

Despite of the hardness of the treated stone surface, barium hydroxide has some disadvantages in that it has the uneven and slow rate of chemical reaction, produces some discoloration, and is durable for the short-term.[98]

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97 S.Z. Lewin and N.S.Baer, "Rationale of the Barium Hydroxide-Urea Treatment of Decayed Stone," *Studies in Conservation* 19,1 (1974): 24.
98 J.R. Clifton, *Stone Consolidating Materials- A Status Report,* (Washington D.C.: U.S. Government Printing Office, 1980), 19-21.

- ***Waterglass: Sodium or Potassium Silicate***

Sodium and potassium silicate was developed 150 years ago. When a solution of sodium or potassium silicate is applied to stone, it will solidify into Na_2SiO_3 or K_2SiO_3 that can replacce the lost cement, and re-bind the loose grains together. With the combination with carbon dioxide in the atmosphere, the waterglass will gradually be separated into amorphous silicon oxide and potassium or sodium carbonate. The amorphous silicon oxide increases the stone's strength, especially that of quart grain stone rather than calcite grain stone, and can reduce the porosity of the weathered stone by about one-third.[99]

Waterglass does not discolor with ultraviolet light. It is not susceptible to acid rain, but is affected by alkaline solution heated by the sun. In addition, sodium or potassium hydroxide left from the chemical reaction with carbon dioxide tends to deposit on the stone surface as white or gray efflorescence. It has high viscosity and poor depth of penetration (only a few millimeters).[100]

99 E.M. Winkler, *Stone in Architecture*, 269.
100 Helmut Weber and Lklaus Zinsmeister, *Conservation of Natural Stone*, 56.

Summary of Consolidation Materials

Type	Group	Material	Advantage	Disadvantage
Organic	Natural Resins	Linseed Oil	- Strengthening weathered stone - Effectiveness for low porosity stones	- Poor depth of penetration - Ineffectiveness for porous stone - Discoloration of treated stone - Irreversibility
		Wax	- Strengthening corroded iron and decayed stone	- Discoloration of treated stone - Irreversibility - Attraction to dirt
	Synthetic Resins	Epoxy Resins	- High strengthening capacity	- Poor depth of penetration - Alteration of treated stone permeability - Discoloration of treated stone when exposed to UV radiation - Complication of mixture application of epoxide group and hardener
		Acrylic Resins	- Stability when exposed to air or UV radiation - Good depth of penetration, when used in monomer forms - Effectiveness in adhering	- Difficulty of monomer form application - Unsuitability for structural consolidation
		Polyester Resins	- Effectiveness for adhesion and consolidation	- Non-resistance to UV radiation and alkalis - Moisture sensibility - Unsuitability for outdoor material consolidation
		Polyurethane Resins	- Strengthening weathered stone with urethane bonds	- Irreversibility - Difficulty of application

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Type	Group	Material	Advantage	Disadvantage
Organic	Silicic Esters	Ethyl silicate	- Most frequency of using for sandstone consolidation - Durability - No discoloration of treated stone - Good depth of penetration - No change of treated stone permeability	- Irreversibility - Effect of hazardous product's solvent
Inorganic		Calcium hydroxide (Lime Water)	- Suitability for calcareous stone	- Slow rate of calcification - Limited water solubility of lime - No increase of treated stone strength - Unsuitability for siliceous stone
		Barium hydroxide (Baryta Water)	- Replacement of cementing material with barium carbonate	- Effect of noxious salt byproduct formed by calcareous materials react with urea, incorporated in a concentrated barium hydroxide solution - Impossibility to control crystal growth in pore spaces of stone - High toxicity - Uneven and slow rate of chemical reaction - Discoloration of treated stone
		Sodium or Potassium Silicate (Waterglass)	- Extremely cheap cost - Anti-rising dampness property - Ability to replace lost stone cementing agents - Ability to strengthen treated stone with amorphous silica - Suitability for siliceous stone - Resistance to UV radiation and acid rain - No discoloration of treated stone	- Effect of stone surface etching - Effect of alkali carbonate byproduct (partly soluble salt) formed by reactivity of alkali hydroxide with CO_2 - Poor depth of penetration - Rapid precipitation of silica - Effect of glass type incrustation and surface soiling - Reduction of frost-resistance - Susceptibility to alkaline solution

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Selection of Consolidant

According to the literature review[101], 23 articles cited the consolidation materials. The most favored and approved effective consolidants that were applied to sandstone in the researches (17 of 23 applications) were alkoxysilanes with different solvents. Silicic ester; i.e. ethyl silicate or Strengthener OH in its commercial name, was the most frequently used due to its effectiveness.

Based on the characterization of the weathered litharenite sandstone from the Northern Gopura of the Phimai Sanctuary in Thailand, deterioration can be attributed to the loss of its cementing agents—iron oxide, clay, and some calcite. The stone shows the deterioration patterns of disaggregation, flaking and thin delamination. Since the stone is mainly composed of quartz grains (SiO_2), the lost minerals and their cementing should be substituted with a silica-based consolidant in order to be compatible with the original materials. After considering the advantages and disadvantages of each consolidation material, ethyl silicate was chosen as the consolidant to be applied to the stone in this thesis.

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101 See Appendix 1V

Ethyl silicate was first introduced to the conservation field by A.V. Hofman in 1861.[102] It was promoted as the most suitable consolidating material for use on weathered stones.[103] Ethyl silicate, also called tetraethylorthosilicate (TEOS) and tetraethoxysilanes, is a silicon ester that has ethyl groups ($-C_2H_5$) as its alkyl groups[104] (these alkyl groups have already replaced the hydrogen atoms of the original silicic acid molecule.[105]) (See Figure 5.1 and 5.2)
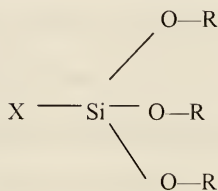
Figure 5.1: Molecular grouping of silicon esters (R= an alkyl group/ X= either an alkyl or alkoxy group).
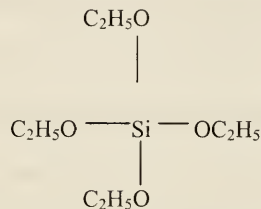
Figure 5.2: Molecular grouping of ethyl silicate. Methyl groups (— C_2H_5) are alkyl groups in a tetraethyl silicate molecular grouping.
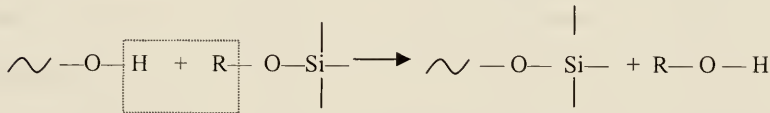
Figure 5. 3: The reaction of alkoxy group (—O—R) with hydroxyl groups (—O—H) derived from either the water molecule or the molecular structure of mineral grains.

102 Anon, "Stone-Preserving Processes, Royal Institute of British Architects," *The Builder*, Vol 19 no. 941, 1861:103-105.
103 A.R. Warnes, Building Stone, Their Properties, Decay and Preservation, (London: Ernest Benn Ltd., 1926), 269.
104 S.Z. Lewin, "The current State of the Art in the Use of Synthetic Materials for Stone Conservation," in *Deterioration and Conservation of Stone, Note from the International Venetian Courses on Stone Restoration*, Studies and Documents on the Cultural Heritage 16, Unesco, 290-302.
105 A.E. Charola, "Brief Introduction to Silanes, Silixanes, Siliconates and Silicate Esters," in *Deterioration and Conservation of Stone, Note from the International Venetian Courses on Stone Restoration*, Studies and Documents on the Cultural Heritage 16, Unesco, 290-302.

When silicon esters are applied to stone, their alkyl groups react with hydroxyl groups—either from the mineral grains or from water molecules. (See Figure 5.3) If the hydroxyl groups come from the mineral grains, the silicon ester will hydrolyze and polymerize with them and become molecular bonds to the surface of the grains. On the other hand, if the hydroxyl groups come from water molecules— not from the mineral grains—the formation of silicic acid, produced through the hydrolysis and polymerization processes, will take place in the pores and replace the lost cementing material physically—without chemically reacting with the grains. Hence, the effectiveness of ethyl silicate as a consolidant depends on types of stone as well. It will yield a good result with the stone that has mineral constituents containing reactive hydroxyl groups or siliceous cementing, such as most types of sandstone.[106] The conditions during application and curing are also relevant. Conditions should be controlled so that the hydrolysis and polymerization processes of silicon ester can occur faster than its evaporation process. Wrapping the treated stone with an impermeable membrane can prevent the silicon ester from evaporating.

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106 S.Z. Lewin, "The current State of the Art in the Use of Synthetic Materials for Stone Conservation," 290-302.

Material

Conservare® OH100[107] was the chosen consolidant in this project. It is mainly composed of tetra ethyl silicate, ethyl alcohol, organic tin compound, and D-n-butyltindilaurate. [108] As already explained, with the internal catalyst and the atmospheric humidity, this silicic ethyl ester-based solution will be converted to a glass-like silicon dioxide gel. This consolidation treatment, therefore, will stabilize the stone by replacing the natural binding material, which has been lost due to the process of weathering (i.e. iron dioxide, clay and calcite), with a new siliceous binder, the amorphous silica gel.

Procedures

The application of this consolidant was carried out in the Architectural Conservation Laboratory of the University of Pennsylvania. Before applying the consolidant, the stone samples were cleaned with deionized water to eliminate any dirt or debris due to natural weathering and the process of cutting. Then they were dried at room temperature for one week or more. It is recommended that the surface temperature during application should

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107 OH is an abbreviation for a German phase *"ohne hydrophobierung"* that means "non-water repellant".
Saray E. Gray, "A Study of Composition in Materials after Treatment" (Master thesis, University of
Pennsylvania, 2000), 105.
108 PROSOCO, Inc. Product Data for OH100 Consolidation Treatment.

be relatively cool to prevent rapid evaporation of the consolidant solvent, and to ensure the proper penetration.[109] It is suggested that the stone surface and air temperature during application should be between 50-68°F with the relative humidity greater than 40%.[110] The stone surface and room temperature in the laboratory fume hood was 64°F and the relative humidity was 32% when applying the consolidant to the stone.

In order to evaluate the effectiveness of the consolidant, complete impregnation with the consolidant was the applied method. The application method was capillary absorption. According to the data obtained from the characterization of the stone, the average height of the water uptake was 2.60 cm parallel to the bed plane direction, and 1.92 cm perpendicular to the bed plane direction. Therefore, the consolidant should be applied to every side of the stone cubes (5x5x5 cm). The cubes were placed over glass beads in a glass container, and then the consolidant was added to the container until the base of the cubes was immersed to a height of 1 cm. The cubes were immersed for an hour on each side or until the heights of absorbed consolidant were constant. Then the stones were turned over to another side, and the application was repeated. During this capillary application, the container was covered with plastic film to prevent the rapid evaporation of the solvent in the consolidant. Immediately after the stones had been saturated, they were rinsed off with methyl ethyl ketone (MEK) in order to eliminate surface crust

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109 Ibid.
110 Ibid.

formation.[111]   Lastly, the stone samples were left in the fume hood with a loose plastic wrap (average temperature of 65.6°F and relative humidity of 33.8%) to dry, and to let the consolidant cure for 3 weeks.[112]

For *in situ* application, brushing would be the easiest and most practical method for applying the consolidant to the stone wall before being dismantled to stabilize the foundation.   Hence, the ethyl silicate consolidant was applied to 3 stone cubes by brushing in order to simulate this *in situ* application.   Hence, the penetration depth of the consolidant—another requirement for consolidant effectiveness—would be evaluated on the stone treated with this application method.   The application was repeated as cycles until the stone samples were saturated.   The number of the cycles was determined by the visible excess material remaining on the stone surface.   When the consolidant remained on the surface for 60 minutes following the last application, it meant the stones had already been saturated. Therefore, ten cycles of application were done with three successive brushings at 5-minute interval each.   The interval time between cycles was 20 minutes.   Like the previous method, immediately after the application was completed the stones were rinsed off with methyl ethyl ketone (MEK), and were dried and cured in the same condition and period as the procedure above.

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111 Recommended by PROSOCO.

CHAPTER VI

EVALUATION OF CONSOLIDATION TREATMENT

Microstructural Examination

The micrographs from the scanning electron microscope reveal ethyl silicate on the stone as a thin deposited film around and over the original stone grains. (See Figure 6.1, 6.2, 6.3, and 6.4) It also filled in most of the pores either naturally caused or later produced by the loss of cementing materials. The x-ray diffraction map displays the locations of silicon and oxygen that are overlapped indicating the presence of silicon dioxide. It could be proclaimed that silicon dioxide in the treated stone act as both the stone grains and cementing networks. (See Figure 6.5) Thus, the chemical reaction between the grains and consolidant was achieved; and therefore, the grains were consolidated by the replacement of the gaps with amorphous silica, which was polymerized from the ethyl silicate.

Color Examination

Ethyl silicate slightly changed the treated stone's color from its original color. After four weeks of curing, the color of both untreated and treated stones were observed visually

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[112] It is recommended that the product needs to cure for 2 or 3 weeks before doing any following treatment.

using the Munsell System as a comparison standard. The color of untreated stone was matched to 5YR 5/3— a reddish brown color-- while that of the treated stone was slightly darker, i.e. 5YR 5/2-2.5—a reddish gray-reddish brown. The degree of color change is minimal, and therefore acceptable. (See Figure 6.5 and 6.11)

According to the manufacturer, Conservare OH® should not discolor or change the color of treated substrate. The minimal discoloration that occurred was possibly caused by insufficient curing time. Since the stone needs about or over one month of curing time to lighten up, the samples will mostly lighten in color over the coming month.

Mechanical Testing

Compressive Strength

ASTM C 170			
STANDARD TEST METHOD FOR COMPRESSIVE STRENGTH OF DIMENSION STONE			
Wet Condition		Dry Condition	
Load Direction	Comp. Strength (psi)	Load Direction	Comp. Strength (psi)
Parallel to Bedding U	-2012.50	Parallel to Bedding U	-10650.00
T	-3912.50	T	-10633.00
Perpend. To Bedding U	-2300.00	Perpend. to Bedding U	-11825.00
T	-3966.67	T	-10650.00

Note: U= Untreated stone/ T= Treated stone

Ethyl silicate used as a consolidant did increase the compressive strength of wet treated stones. There was a 94% compressive strength increase parallel to the stones' bed planes, and a 72% compressive strength increase perpendicular to the bed planes. However, the consolidant insignificantly increased the dry stones' compressive strength (less than 1%).[113]

Although ethyl silicate is not predominantly effective for this type of sandstone when it is dry, it has an outstanding effect on the wet sandstone. Since the main deterioration problem of this stone is that it becomes extremely soft when it is wet, using ethyl silicate as the consolidant can be an effective solution.

113 See Appendix II for the raw data and details of calculations.

ASTM C 99					
MODULUS OF RUPTURE TEST (3-Point Bending Test)					
Wet Condition			**Dry Condition**		
Load Direction		*R* (psi)	**Load Direction**		*R* (psi)
Parallel to Bedding	U	-294.42	Parallel to Bedding	U	-1252.07
	T	-477.27		T	-1555.79
Perpendicular to Bedding	U	-233.47	Perpendicular to Bedding	U	-1152.89
	T	-477.27		T	-1741.74

Note: U= Untreated stone/ T= Treated stone

From the results, ethyl silicate increased the original flexural strength of the wet stones bearing the load parallel to their bed planes by 62%, and by 92% for the load perpendicular to the bed planes. Although the percentage of increase in a dry condition was less than that in a wet condition, the consolidant also increased the flexural strength of the dry stones, i.e. 24% for the bed face side of the stone and 51% for the bed edge side.[114]

It can be concluded that ethyl silicate is undoubtedly effective for increasing the flexural strength of this type of sandstone, especially when the stone is used in the climate of Northeastern Thailand, which is confronted with wet conditions over six months a year.

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114 See Appendix II for the raw data and details of calculations.

Water Absorption by Total Immersion

NORMAL 7/81				
WATER ABSORPTION BY TOTAL IMMERSION				
WATER ABSORPTION CAPACITY				
SAMPLE	Original Dry Weight(g)	Saturated Weight(g)	Redry Weight(g)	WAC
U1	295.96	311.90	298.26	4.60
U2	295.85	311.67	298.08	4.56
U3	295.98	311.81	298.13	4.60
Average	**295.93**	**311.79**	**298.16**	**4.57**
T1	303.12	315.43	304.55	3.57
T2	306.94	318.57	307.45	3.62
T3	302.88	314.94	304.18	3.54
Average	**304.31**	**316.31**	**305.39**	**3.58**

Note: U= Untreated stone/ T= Treated stone

The maximum quantity of water that the stone could absorb decreased from 5.36% of the dry weight of the stone with 4.57% of water absorption capacity (WAC) to 3.94% of the stone's dry weight with 3.58% of WAC when treated with ethyl silicate. The water absorption rates of the treated stones were much lower than that of the untreated one in the first three hours of immersion. However, the treated stone continued absorbing water for a longer period than the untreated stone. The constant weight of treated stones (when the weight increase between two consecutive weighings every 24 hours is less than 0.1%)

was reached after 8 days of immersion while it took only 3 days for the untreated stones to reach their constant weight.[115]

Porosity of Stone

The stone porosity was changed from 13.06% by total immersion and 12.78% by hydrostatic weighing to 9.04% by total immersion and 8.67% by hydrostatic weighing.[116] These values indicate that the porosity of stones is somewhat decreased after being treated with ethyl silicate. According to the microstructural analysis with SEM, this conclusion can confirm the replacement of pores and lost cementing materials of ethyl silicate leading to the reduction of the stone's porosity[117]

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115 See Appendix II for the raw data and details of calculations.
116 See Appendix II for the result table of the total immersion test and hydrostatic weighing.
117 See Appendix II for the raw data and details of calculations.

Water Absorption by Capillary Action

NORMAL 11/85, Teutonico's Lab Manual Exercise 10																
WATER ABSORPTION BY CAPILLARY ACTION																
SAMPLE		HEIGHT(cm)														
	Min.	1	2	3	4	5	10	15	20	25	30	60	90	120	150	180
U Parallel to Bed		1.00	1.23	1.40	1.50	1.55	1.80	1.85	1.98	2.10	2.20	2.43	2.53	2.58	2.60	2.60
T Parallel to bed		0.80	0.90	0.97	0.97	0.97	1.10	1.30	1.37	1.37	1.37	1.30	1.20	1.20	1.20	1.20
U Perpendicular to Bed		0.67	0.95	1.13	1.20	1.28	1.47	1.53	1.58	1.65	1.67	1.80	1.88	1.90	1.92	1.92
T Perpendicular to Bed		0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.02	0.02	0.02	0.02	0.05	0.05	0.05

Note: U= Untreated stone/ T= Treated stone

After treatment with ethyl silicate, the highest level to which water could be absorbed through capillary action parallel to bed plane was reduced from 2.60 cm to 1.37 cm., or almost a 100% decrease. The consolidant significantly affected the capillary action when the bed faces of stones were placed 90° to the capillary direction. The treated stones stopped drawing water after 20-25 minutes while the untreated stone stopped drawing water after two and a half hours.[118]

Ethyl silicate, therefore, can decrease water capillary uptake. This consolidation method can partially solve the problems that the monument now confronts with the rising dampness and moisture on the walls. Nevertheless, further protective treatments, such as

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118 See Appendix II for the raw data and details of calculations.

water vapor barriers, have to be carried out to completely prevent the stone deterioration caused by water effects.

Permeability of Stone

	Perpend to Bedding		Parallel to Bedding	
	U	T	U	T
G (g) (Weight Change)	0.840	0.700	1.300	0.420
G/t (Water Vapor Transmission Rate) (g/h)	0.005	0.004	0.011	0.004
WVT (Water Vapor Transmission) (g/h/sq.m)	0.990	0.820	2.160	0.860

From the results, ethyl silicate slightly changed the capacity and rate of water vapor transmission of the stone in perpendicular to bed plane direction, i.e. a 17% decrease, but greatly changed the stone's capacity and rate of water vapor transmission in parallel to bed plane direction, i.e. a 151% decrease.[119] Such change is unsatisfactory because the consolidation treatment can cause an accumulation of moisture and salts inside the treated stone, resulting in further deterioration problems after treatment.

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119 See Appendix II for raw data and details of calculation

Wet-Dry Cycling Test

From the results, the untreated stone that had a pre-existing crack broke after 2 cycles of the wet and dry testing while the treated stone with the same condition (having a pre-existing crack) resisted the wet-dry cycle until the end of the test. The other untreated stone started cracking during the second round of immersion, and finally broke after 4 wet-dry cycles. The other untreated and treated stones retained their appearances until the end of the test (5 wet-dry cycles). Neither the untreated nor the treated stone had surface crumbling during the test. (See Figure 6.8 and 6.9)

It can be concluded that ethyl silicate increases the wet-dry cycling damage resistance of the treated stone, especially the stone that has already cracked before consolidation. This consolidant can be very useful for consolidating the weathered stone block that has to be reused with anastylosis restoration. Although the weathered untreated stone loses its mechanical strength when wet, it may resist the damage caused by wet-dry cycling if the wet period does not last long and the stone does not have pre-existing damages. In addition, the wet-dry cycle does not cause both untreated and treated stones to crumble.

Salt Crystallization

One untreated stone cracked and broke along bed planes after 4 cycles of salt crystallization test. The other untreated stone had cracks after 6 cycles, and then broke at

the seventh cycle. In contrast, a treated stone started cracking during the fourth cycle, but did not break until the end of the test (10 cycles). The rest of the treated stones retained the same appearance as before the test. Unlike the wet-dry cycling damage, a 0.68% average amount of salt crystals in the stone, i.e. the amount of increased weight of dry stones, could cause the first minor damages, such as crumbling or scaling, to the untreated stone.[120] In addition, the untreated stone became soft and clayish after 3 cycles of immersion in sodium sulfate solution. In the test, efflorescence of sodium sulfate was deposited on the surface of both untreated and treated stones, but appeared first on the untreated stones. The salt tended not to remain in the treated stones in the first 4 cycles due to the decrease of the dry stones' weights during those periods. (See Figure 6.7)

Although ethyl silicate does not prevent the damage caused by the internal stresses produced by salt crystallization, it strengthens the treated stone to resist such stresses longer.

Depth of Penetration Analysis

Since the stone's color was very dark—reddish brown-- testing the consolidant's depth of penetration with iodine vapor/staining did not yield a good result. The yellowish brown stain could be observed only on the surface of the stone that was covered with a film of

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120 See Appendix II for the raw data and details of calculations

ethyl silicate. Depth of penetration, however, could be simply observed when the stones were wet. Although the ethyl silicate slightly discolored the dry treated stones, the color difference was significantly distinguishable when the stones were wet. (See Figure 6.10 and 6.11)

When applied by brushing, Conservare OH® penetrated the stones about 7 mm deep (4 mm, 8 mm and 8.5 mm in the three tested samples respectively). According to several researchers' suggestions, stone consolidant should penetrate to the depth that all deteriorated zones are solidified, and attached to the unweathered sound core of the stone. In this case, the depth obtained from the application is, therefore, unsatisfactory. However, Santamaria argues that depth of penetration of ethyl silicate is less than 5 mm in general.[121] Thus, about a 7-mm depth of penetration is acceptable.

Since the deterioration of this sandstone is caused by water effects, resulting of a function of water penetration, the consolidant should improve cohesion in the range of water absorption through capillarity. From the results obtained from the water absorption by capillary action test, the consolidant significantly reduced the water uptake parallel to bed plane and definitely decreased water absorption through capillarity perpendicular to bed plane. Thus, the consolidant proved its effectiveness. Although the consolidant cannot penetrate the stone core, the obtained depth of penetration is so satisfactory that it is

adequate to improve the stone cohesion, especially in the perpendicular to bed plane direction.

Conclusion

The results of the evaluation tests for the effectiveness of ethyl silicate used as a consolidant for the Northern Gopura's sandstone prove that ethyl silicate is relatively effective. It can reestablish the grains' cohesive strength, and thus strengthen the stone. It can chemically bind the grains together, as well as fill in the pores with amorphous silica. Consequently, the stone compression and flexural strengths are increased, its porosity is decreased, and its susceptibility to weathering is reduced. Although ethyl silicate is not a water repellant, i.e. it does not make the stone hydrophobic, it can decrease the capillary movement of water in the stone. There are, however, some disadvantages of ethyl silicate used in this experiment: decreasing the water vapor transmission and darkening the color. The change of the stone permeability is significant and has to be taken into consideration, while the slight discoloration is unnoticeable, especially in the dry stone.

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121 S.P. Santamaria et al, "Protection of the Stone of "San Bartolome" Church in Logrono, Spain," in *Proceedings of the 7th International Congress on Deterioration and Conservation of Stone, Lisbon, Portugal, June 15-18, 1992*, (Lisbon: Laboratorio Nacional de Engenharia Civil, 1992), 1335-1340.
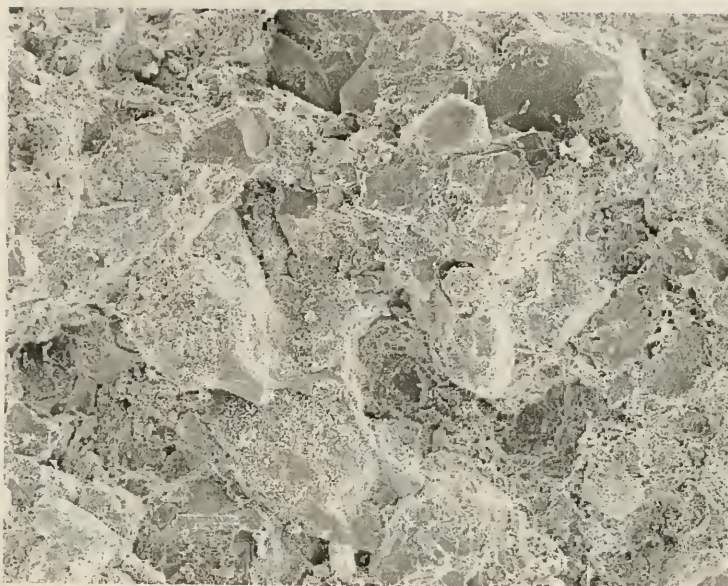
Figure 6.1: SEM image of untreated stone (100x magnification, 15 KV)
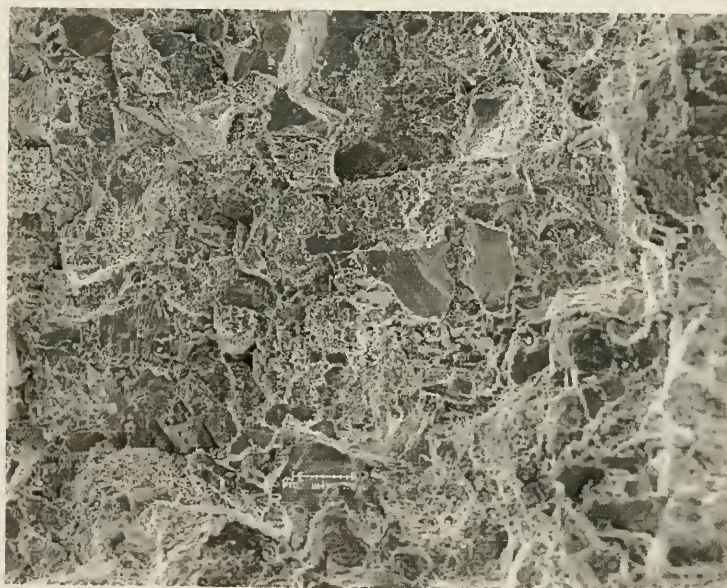
Figure 6.2: SEM image of treated stone (100x magnification, 15 KV)

110
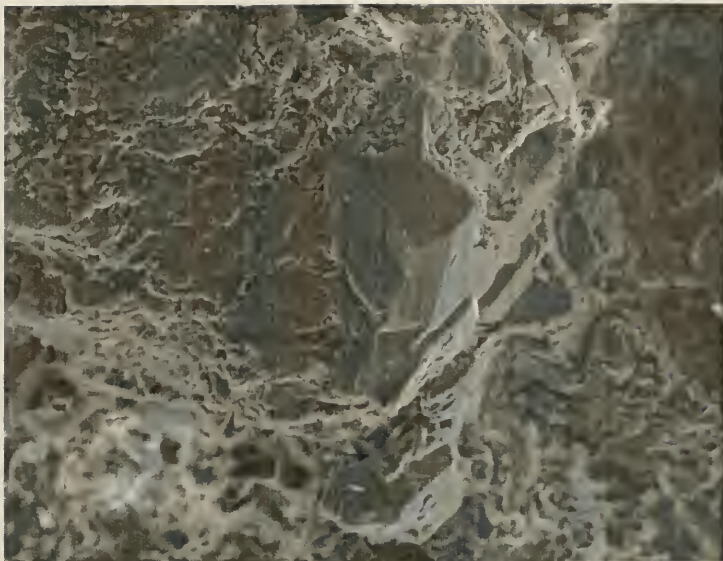
Figure 6.3: SEM image of untreated stone (600x magnification, 15 KV)
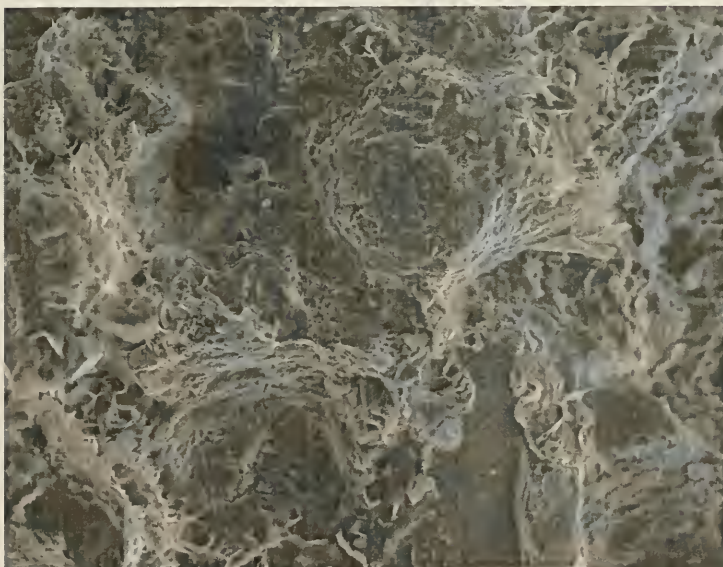
Figure 6.4: SEM image of treated stone (600x magnification, 15 KV)

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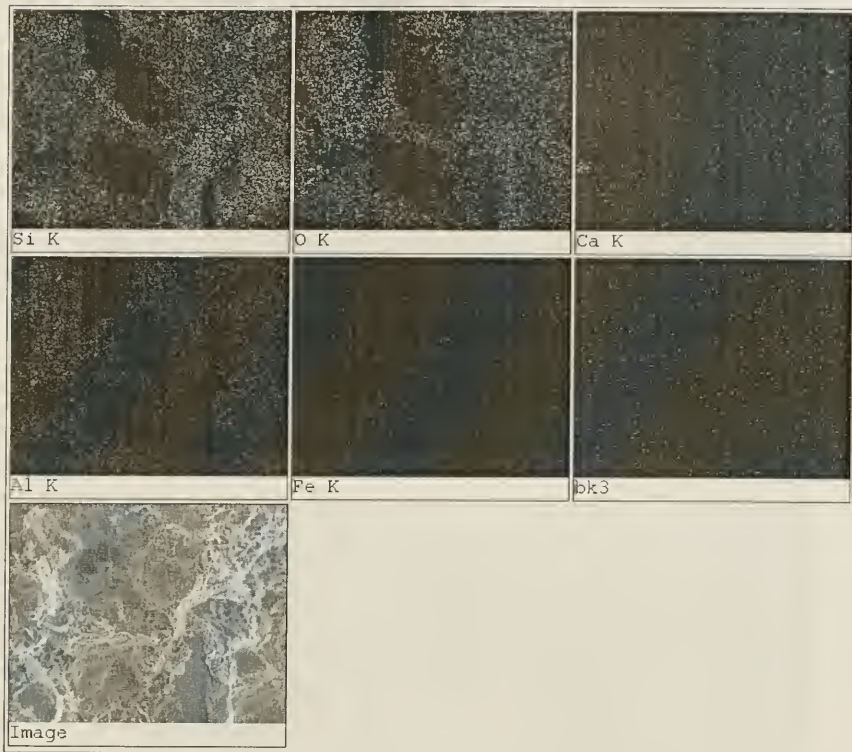
Figure 6.5: X-ray Diffraction Map of the main elements in a spot of treated stone.

The map displays the location of silica, oxygen, calcium, aluminum, and iron. The matched location between silica and oxygen indicates the location of silicon dioxide—either the original quartz grains or the amorphous silica polymerized from ethyl silicate. In the map, silicon dioxide spreads throughout the whole area, and is also seen as a network around the grains, which is visible in the SEM image at the left bottom of the map. This network indicates the replacement of cementing elements—aluminum, iron, and calcite—with silicon dioxide. The black spots in every map, however, can be either other elements if seen in color in the background map or the geometric error of X-ray diffraction analysis if also seen in black in the background map.

Figure 6.6: Color Comparison of untreated and treated stone after 3 weeks of curing time
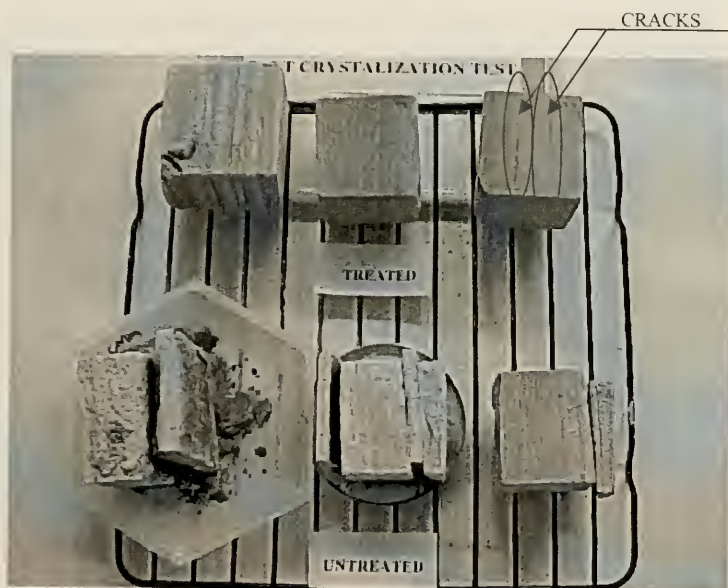
Figure 6.7: Untreated and treated stones after salt crystallization test (10 cycles)
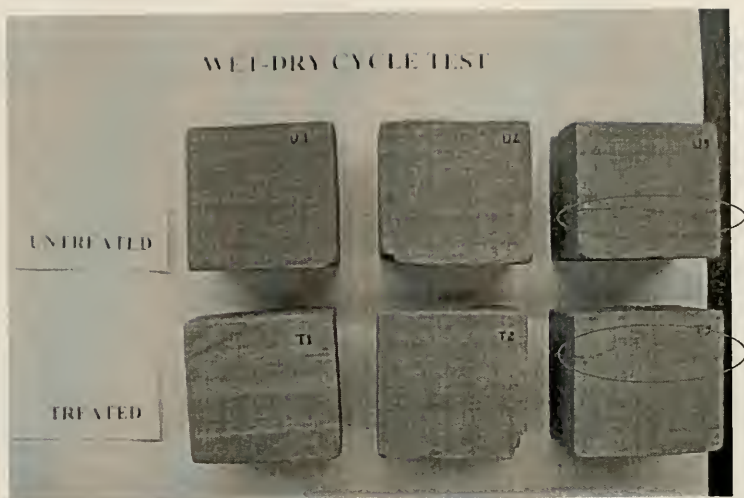
Figure 6.8: Untreated and treated stones before wet-dry cycling test
The untreated stone 3 and treated stone 3 had pre-existing cracks before being tested.

Figure 6.8: Untreated and treated stones after wet-dry cycling test
The untreated stone (#3) that had pre-existing cracks before being tested broke while the treated stone (#3) could resist the damage caused by wet-dry cycling. The untreated stone (#2) broke after the test although the pre-existing damage did not perform.

114
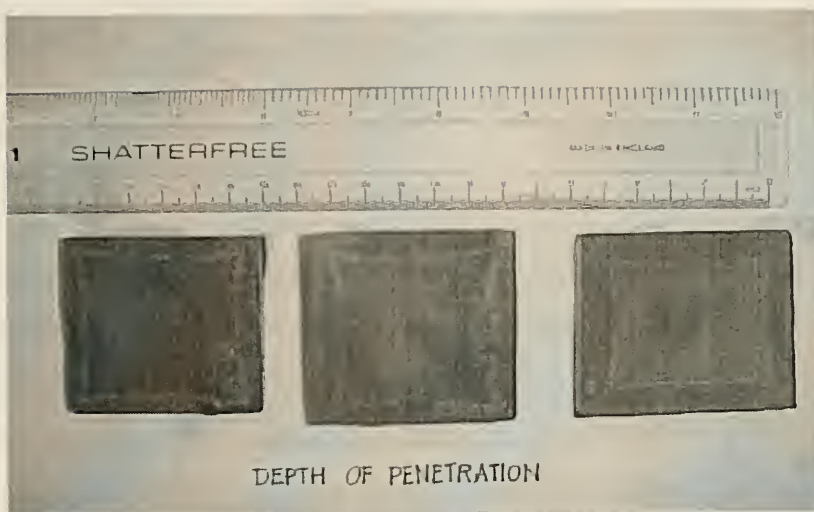
Figure 6.10: Depth of penetration measurement carried out in wet treated stone cubes

Figure 6.11: Slight discoloration of dry treated stone

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CHAPTER VII

CONCLUSION AND RECOMMENDATION

Conclusion

The sandstone from the Northern Gopura of the Phimai Sanctuary in Thailand is mainly composed of quartz (by 50%), feldspar and some small clasts. Due to the amount of feldspar and clast, the stone is geologically characterized as litharenite sandstone, i.e. a type of sandstone that is composed of over 25% clasts and less than 10% feldspar. Its cementing material is silica, iron oxide and clay, as well as, calcite in some concentrated areas of the stone. Since the stone has been used as a construction material for the monument confronted with weathering for more than 900 years, it has had deterioration problems: grain disaggregation or sugaring, crumbling, flaking, scaling, and bed plane delamination. These problems are caused by chemical degradation and dissolution of cementing materials resulting in the loss of the grains' composition and cohesion. Consequently, the weathered stone is no longer able to bear load and support the monument structure, especially when wet. In addition, the stone breaks easily, especially when removed during the restoration project.

The main agent that caused the deterioration problems to the stone is water. Located in Northeastern Thailand with a tropical climate, the monument is confronted with precipitation almost six months a year during the rainy season. Water softens, and

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consequently, weakens the stone. The dampness in the wall directly causes the stone to deteriorate through wet and dry cycles and chemical degradation processes, and indirectly accelerates or supports the other deterioration problems, such as soluble salt crystallization and biological growth. Effects of salt and biological growth, however, do not play predominant roles in deteriorating the stone.

In order to reestablish strengths of the stone, consolidation treatment was carried out. Conservare OH®, a commercial ethyl silicate product, was applied to the stone through capillary rise and brushing and cured for 3 weeks before the evaluation tests for the consolidant effectiveness were done. The comparison of the untreated weathered and treated weathered stones' properties indicated whether or not the consolidant qualified and met with the standard requirements and recommendations.

Ethyl silicate proved that it could strengthen the stone and decreased the porosity and the absorbed moisture content, but caused some negative side effects. The polymerized amorphous silica chemically replaced the lost cementing material and filled the stone's pores resulting in the consolidation of stone grains' cohesion and the reduction of the stone porosity. Substituting the original cementing material—iron oxides, clay, and calcite—with amorphous silica could change the stone to be stronger and more durable; the consolidant changed the ferruginous sandstone into siliceous sandstone which is very durable. The stone compressive and flexural strengths were increased, especially in the case of the wet stone that is very soft and weak. The stone porosity was decreased, and

the amount of absorbed water and depth of water capillary absorption were, consequently, minimized. The stone treated with ethyl silicate also withstand the deterioration of wet-dry cycling and salt crystallization better than the untreated stone. Therefore, if compared with the weathered stone, the treated stone can support more loads, and simultaneously, can resist weathering and deteriorating agents longer. However, there were also some drawbacks to this ethyl silicate used in the consolidation. Ethyl silicate was somewhat unable to penetrate the stone deeply (although the obtained depth was so adequate that the water uptake depth was decreased resulting in the solved rising dampness problem). The other disadvantages were the decrease of the stone's permeability, and the slight discoloration of the stone. These disadvantages should be taken into consideration and need further studies.

Discussion and Recommendation

For the disadvantages that occurred in the experiment, they might be caused by deficient sample preparation and inappropriate consolidation application. With the two methods of application, capillary uptake and brushing, the effectiveness of ethyl silicate was lessened. Brushing did not yield a good depth of penetration[122] while total impregnation through capillary uptake might cause the pores to clog resulting in a significant change of the stone's permeability—although the latter method perfectly decreases the stone's porosity. Stone sample preparation may be the other cause of unsatisfactory results.

Stone sawing may have clogged the stone pores with fine stone dust. In addition, an inadequate curing period of the consolidant might cause an incomplete polymerization leading to darkening of the treated stone.

Change of application method may solve these unacceptable results. Providing an application method that can lengthen the contact time between stone and consolidant is one of the solutions. Professor Marchesini has suggested to use large brushes or cotton wool pads wrapped with plastic film, as well as to use syringes connected to consolidation material's container, to slowly feed the consolidant to stone.[123] Applying consolidant under vacuum pressure is another solution. Through this method, the consolidant will be drawn to the stone through capillary action slowly, but deeply, due to the artificial pressure difference. However, capillary uptake, under either atmospheric or vacuum pressure, is not a practical application method for an object as large as the walls of the monument, especially when the main purpose of the consolidation treatment is to pre-consolidate the stone wall before dismantling, not to apply the consolidant to a single stone block. Therefore, combinations of spraying or brushing with vacuum pressure methods offer options for a good result of depth of penetration, and simultaneously, a practical application *in situ*. Additional consolidation by immersion post-dismantling can certainly be considered as well for seriously deteriorated stone blocks.

122 M. Laurenzi Tabassco, "Conservation Treatments of Stone," 271-289.
123 Ibid.

If the stone permeability reduction beyond the consolidation treatment with ethyl silicate is inevitable, further stone treatments and maintenance should be carried out to prevent or reduce absorbed moisture content in the stone. These have to be done along with applying ethyl silicate, which itself can decrease the amount and depth of absorbed water. Stone water repellency should be investigated, and a protective agent should be applied to the stone in addition to the water drainage system that should be added to the monument. To protect stone from continuing to absorb ground water, cutting the path of water transportation from the ground is suggested, such as providing a waterproof membrane in newly constructed foundations. Sheltering the walls is an intrusive intervention, but yield a good result for rainwater protection. However, if the decrease of water vapor transmission is caused by the clog of pores with fine stone dust due to stone sawing, cleaning the sample with ultrasonic bath can eliminate any remaining debris; thus, yield a more accurate results.[124]

Although discoloration, especially in dry stone, is barely noticeable, it also has to be taken into account. Lengthening the curing period of the consolidant leading to the complete polymerization may yield a better result. In case there are some crust formations or stains of ethyl silicate remaining on the treated stone surface, cleaning the

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[124] Suggested by the thesis advisor, Prof. Frank G. Matero, Historic Preservation Program, University of Pennsylvania

stone with ammonium hydroxide or by pressure water rinsing may eliminate these surface deposits.[125]

Additionally, in order to select freshly cut stone to replace the broken weathered stone, the type of fresh stone must be considered. Using stone from the same quarry as that of the ancient stone can give a compatible appearance and property. The fresh stone, however, will suffer from the same deterioration problems in the future. Hence, pre-consolidating and pre-water repellency as well as providing an appropriate maintenance program may be needed to lengthen the fresh stone durability. In order to obtain the greatest material strengths, the fresh stone should be naturally bedded when used in a wall supporting compression load, and face bedded when used as lintel, sill or any structural member that has to support tensile or shearing load.

Lastly, in order to complete the effectiveness evaluation program and ensure the long-term durability of ethyl silicate consolidation, the evaluation tests need to be conducted again in another ten to fifteen years. In order to ensure or lengthen the effectiveness of consolidation treatment, occasional reapplying the consolidant is also recommended.

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125 Ibid.

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A GLOSSARY OF HISTORIC MASONRY DETERIORATION PROBLEMS[126]

Blistering

This term is used to describe swelling accompanied by rupturing of a thin uniform skin both across and parallel to the bedding plane. Blistering is generally found on a surface close to the ground. It may remain a relatively constant condition scattered over the masonry surface, but it eventually results in greater surface conditions (exfoliation, delamination and spalling).

Cracking

This term is used to describe narrow fissures from 1/16 to ½ inch wide in a block of masonry. Small cracks within a single block of masonry may not be serious, but longer and wider cracks extending over a larger area can lead to a greater damage such as masonry breaking or structural collapse.

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126 Anne E. Grimmer, A Glossary of Historic Masonry Deterioration Problems and Preservation Treatments, (Washington D.C.: U.S. Government Printing Office).

Crumbling

This term is used to describe the condition of brittleness or tendency of masonry to break up or dissolve. It may be caused by an inherent cohesion weakness of the masonry and gradual dissolution of cementing material, or it may be the result of external factors, such as salts or moisture, that can affect the strength or durability of the masonry.

Delamination

This term is used to describe a condition in which the outer surface of the stone splits apart into laminae or thin layers and peels off the face of the stone. Because of their layered composition, this may be a natural condition of sedimentary stones. The presence of clay-rich layers can accelerate the process of delamination. Delamination takes place along the natural bedding planes of the stone.

Delamination differs from spalling in that it is a condition confined to natural stone and it is not a condition that occurs in manufactured products, such as brick.

Exfoliation

This term is used to describe natural stone deterioration: peeling, scaling, or flaking off of the stone surface in thin layers. Exfoliation is caused by the expansion and contraction of

trapped moisture, the chemical action of stone composition, or stone weathering. The process is not limited along natural bedding planes, resulting in an unevenly layered surface.

Flaking

This term is used to describe an early stage of peeling, exfoliation, delamination, or spalling. The appearance of detachment of small, flat, thin pieces of the outer layers of stone from a larger piece of stone can be explained as flaking. The process is usually caused by capillary moisture or freeze-thaw cycles that occur within the stone. It commonly occurs in masonry coatings, the area that has lost the adhesion between the coating and the masonry substrate, or the masonry that has had a water repellant applied resulting in trapped moisture.

Friability

This term is used to describe the tendency for a stone or any masonry material to break up, crumble or powder easily.

Sugaring

This term is used to describe a characteristic of some masonry having gradual surface disintegration problem. The process may be caused by salts dissolved in and transported through the stone by moisture, and consequently results in dissolution of cementing material.

Weathering

This term is used to describe a natural disintegration and erosion of stone caused by wind and rain, resulting in granular and rounded surfaces. Acid rainwater in particular can damage carbonate stone, or increase the natural weathering rates that results in noticeable softening or loss of masonry details.

APPENDIX II

RAW DATA AND CALCULATIONS OF TESTS

COMPRESSIVE STRENGTH TEST

	WET CONDITION				DRY CONDITION		
	Load (lbs.)	A (sq.inch)	Comp.Str(psi)		Load (lbs.)	A (sq.inch)	Comp.Str(psi)
Load Parallel to Bedding				**Load Parallel to Bedding**			
U1	broken *	N/A	N/A	U1	-10700	1	-10700.00
U2	-7300	4	-1825.00	U2	-10600	1	-10600.00
U3	-8800	4	-2200.00	U3	-10550	1	-10550.00
Average	**-8050**	**4**	**-2012.50**	Average	**-10650**	**1**	**-10650.00**
T1	test failed	N/A	N/A	T1	-11000	1	-11000.00
T2	-11300	4	-2825.00	T2	-10900	1	-10900.00
T3	-20000	4	-5000.00	T3	-10100	1	-10100.00
Average	**-15650**	**4**	**-3912.50**	Average	**-10667**	**1**	**-10666.67**
Load Perpend. to Bedding				**Load Perpend. to Bedding**			
U1	-10400	4	-2600.00	U1	-10050	1	-10050.00
U2	-8200	4	-2050.00	U2	-13600	1	-13600.00
U3	-9000	4	-2250.00	U3	-11000	1	-11000.00
Average	**-9200**	**4**	**-2300.00**	Average	**-11825**	**1**	**-11825.00**
T1	-13800	4	-3450.00	T1	-12500	1	-12500.00
T2	-16800	4	-4200.00	T2	-11300	1	-11300.00
T3	-17000	4	-4250.00	T3	-13400	1	-13400.00
Average	**-15867**	**4**	**-3966.67**	Average	**-11900**	**1**	**-11900.00**

* Sample was broken during being immersed

Note: Data are negative due to they are compression loads/ strength values.

PERCENTAGE OF INCREASED COMPRESSIVE STRENGTH

	WET CONDITION			DRY CONDITION		
	C (psi)	Increased C	% Increase	C (psi)	Increased C	% Increase
Load Parallel to Bedding						
Untreated	-2013			-10650		
Treated	-3913	-1900	94.41	-10667	-17	0.16
Load Perpend. to Bedding						
Untreated	-2300			-11825		
Treated	-3967	-1667	72.46	-11900	-75	0.63
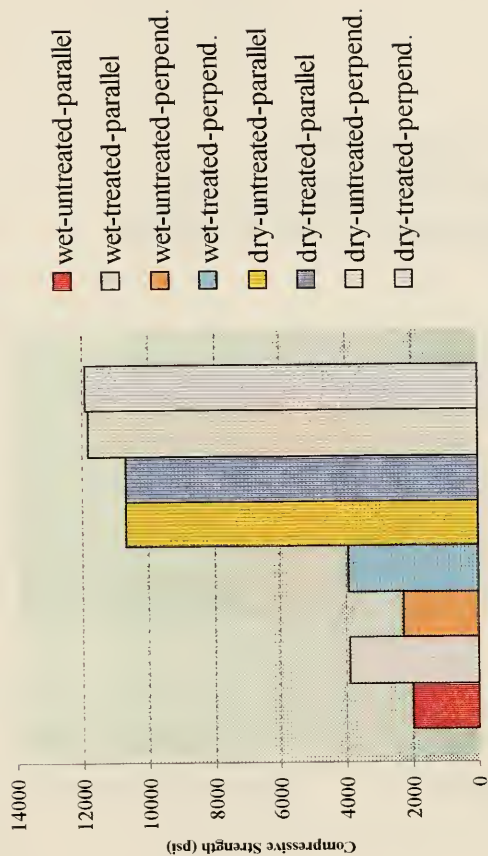
COMPRESSIVE STRENGTH TEST

Legend:
- wet-untreated-parallel
- wet-treated-parallel
- wet-untreated-perpend.
- wet-treated-perpend.
- dry-untreated-parallel
- dry-treated-parallel
- dry-untreated-perpend.
- dry-treated-perpend.

X-axis: Stone Condition
Y-axis: Compressive Strength (psi) — 0, 2000, 4000, 6000, 8000, 10000, 12000, 14000

COMPARISON
OF COMPRESSIVE STRENGTH
OF WET & DRY UNTREATED STONES

Compressive Strength (psi)

14000
12000
10000
8000
6000
4000
2000
0

Condition

- wet-parallel
- dry-parallel
- wet-perpendicular
- dry-perpendicular

COMPARISON
OF COMPRESSIVE STRENGTH
OF WET & DRY TREATED STONES

Compressive Strength (psi) vs Condition

Legend:
- wet-parallel
- wet-perpendicular
- dry-parallel
- dry-perpendicular
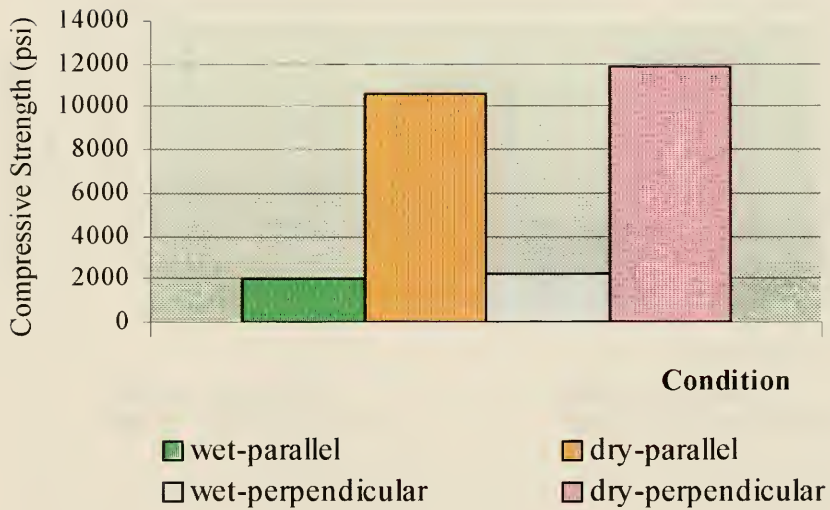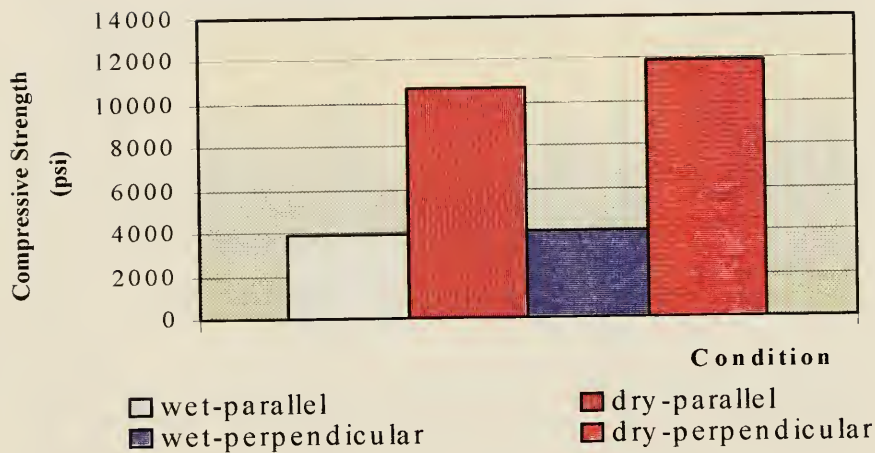
MODULUS OF RUPTURE TEST

	WET CONDITION					DRY CONDITION			
	Load (lbs.)	Span(in.)	2(bxdxd)*	R (psi)		Load (lbs.)	Span(in.)	2(bxdxd)*	R (psi)
Load Parallel to Bedding					*Load Parallel to Bedding*				
U1	-225	2	4.84	-278.93	U1	-1020	2	4.84	-1264.46
U2	-250	2	4.84	-309.92	U2	-1000	2	4.84	-1239.67
U3	-140	3	4.84	-260.33	U3	-1040	2	4.84	-1289.26
Average	**-238**	**2**	4.84	**-294.42**	Average	**-1010**	**2**	4.84	**-1252.07**
T1	-415	2	4.84	-514.46	T1	-1470	2	4.84	-1822.31
T2	-360	2	4.84	-446.28	T2	-1040	2	4.84	-1289.26
T3	-410	2	4.84	-508.26	T3	-1470	2	4.84	-1822.31
Average	**-385**	**2**	4.84	**-477.27**	Average	**-1255**	**2**	4.84	**-1555.79**
Load Perpend. to Bedding					*Load Perpend. to Bedding*				
U1	-140	2	4.84	-173.55	U1	-760	2	4.84	-942.15
U2	-180	2	4.84	-223.14	U2	-1100	2	4.84	-1363.64
U3	-245	2	4.84	-303.72	U3	-930	2	4.84	-1152.89
Average	**-188**	**2**	4.84	**-233.47**	Average	**-930**	**2**	4.84	**-1152.89**
T1	-350	2	4.84	-433.88	T1	-1400	2	4.84	-1735.54
T2	-405	2	4.84	-502.07	T2	-1410	2	4.84	-1747.93
T3	-335	2	4.84	-415.29	T3	-1405	2	4.84	-1741.74
Average	**-363**	**2**	4.84	**-450.41**	Average	**-1405**	**2**	4.84	**-1741.74**

* b=2", d=1.1"

Note: The data are negative because they are compression loads/ strength values.

PERCENTAGE OF INCREASED COMPRESSION STRENGTH

WET CONDITION

	R (psi)	Increased	% Increased R
Load Parallel to Bedding			
Untreated	-294		
Treated	-477	-183	62.11
Load Perpend. to Bedding			
Untreated	-233		
Treated	-450	-217	92.92

DRY CONDITION

	R (psi)	Increased	% Increased R
Load Parallel to Bedding			
Untreated	-1252		
Treated	-1556	-304	24.26
Load Perpend. to Bedding			
Untreated	-1153		
Treated	-1742	-589	51.08

FLEXURAL STRENGTH TEST

Legend:
- wet-untreated-parallel
- wet-treated-parallel
- wet-untreated-perpend.
- wet-treated-perpend.
- dry-untreated-parallel
- dry-treated-parallel
- dry-untreated-perpend.
- dry-treated-perpend.

Y-axis (Flexural Strength (psi)): 0, 200, 400, 600, 800, 1000, 1200, 1400, 1600, 1800, 2000

X-axis: Stone Condition

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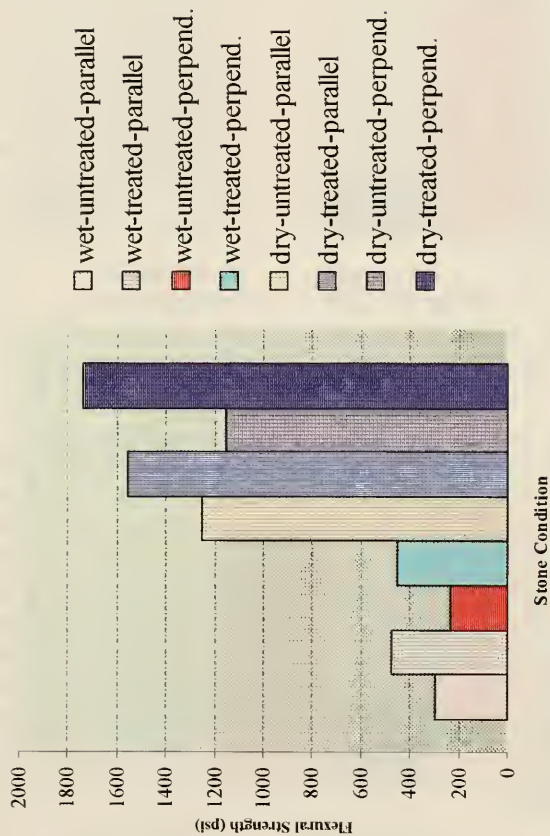
COMPARISON OF FLEXURAL STRENGTH OF WET & DRY UNTREATED STONES

COMPARISON OF FLEXURAL STRENGTH OF WET & DRY TREATED STONES

WATER ABSORPTION BY TOTAL IMMERSION (UNTREATED STONE)

Weight of Sample (grams)

DATA																				
SAMPLE	0	0	1	2	3	8	24	48	72	96	120	144	168	192	216	240	264	288	312	336
U1	295.96	297.95	301.48	303.30	304.41	307.89	310.46	310.68	311.12	311.18	311.26	311.35	311.40	311.50	311.55	311.58	311.72	311.82	311.85	311.90
U2	295.9	297.93	301.54	303.32	304.55	308.10	310.34	310.54	311.00	311.07	311.13	311.18	311.27	311.36	311.38	311.46	311.55	311.60	311.63	311.67
U3	296	298.37	302.44	304.76	305.98	309.04	310.48	310.73	311.25	311.32	311.31	311.35	311.51	311.59	311.62	311.67	311.81	311.81	311.81	311.81
Average	295.93	298.08	301.82	303.79	304.98	308.34	310.43	310.65	311.12	311.19	311.23	311.29	311.39	311.48	311.52	311.57	311.69	311.74	311.76	311.79
Wt Ch		2.15	3.74	1.97	1.19	3.36	2.08	0.22	0.47	0.07	0.04	0.06	0.10	0.09	0.03	0.05	0.12	0.05	0.02	0.03
Rate		25.84	4.08	1.97	1.19	0.67	0.13	0.01	0.02	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.01	0.00	0.00	0.00

Percentage of Water Absorbed

%WA																				
SAMPLE	0	0	1	2	3	8	24	48	72	96	120	144	168	192	216	240	264	288	312	336
U1	0.00	0.67	1.87	2.48	2.86	4.03	4.90	4.97	5.12	5.14	5.17	5.20	5.22	5.25	5.27	5.28	5.33	5.36	5.37	5.39
U2	0.00	0.70	1.92	2.52	2.94	4.14	4.90	4.97	5.12	5.14	5.16	5.18	5.21	5.24	5.25	5.28	5.31	5.32	5.33	5.35
U3	0.00	0.81	2.18	2.97	3.38	4.41	4.90	4.98	5.16	5.18	5.18	5.19	5.25	5.27	5.28	5.30	5.35	5.35	5.35	5.35
Average	0.00	0.73	1.99	2.66	3.06	4.19	4.90	4.97	5.13	5.16	5.17	5.19	5.23	5.26	5.27	5.29	5.33	5.34	5.35	5.36

WATER ABSORPTION CAPACITY

	Org D W	Sat W	Redry W	
SAMPLE	Mo(g)	Mmax(g)	Md(g)	WAC
U1	295.96	311.90	298.26	4.57
U2	295.9	311.67	298.08	4.56
U3	296	311.81	298.13	4.59
Ave.	295.93	311.79	298.16	4.57

POROSITY BY WATER ABSORPTION

	Mass of Pore = Volume of Pore	App V	% Prst
SAMPLE	Mp = Vp	Va(cc)	% voids
U1	15.94	113.00	14.11
U2	15.82	133.00	11.89
U3	15.83	120.00	13.19
Ave.	15.86	122	13.06

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POROSITY BY HYDROSTATIC WEIGHING
(UNTREATED STONE)

SAMPLE	Dry W. M1(g)	Imm W. M2(g)	Air W. M3(g)	Pore V. Vp(cc)	App.V. Va(cc)	Real V. Vr(cc)	Real D. Pr (g/cc3)	App D. Pa(g/cc3)
U1	295.96	191.30	311.10	15.14	119.80	104.66	2827.82	2470.45
U2	295.85	193.50	311.45	15.60	117.95	102.35	2890.57	2508.27
U3	295.98	189.90	311.10	15.12	121.20	106.08	2790.16	2442.08
Ave.	295.93	191.57	311.22	15.29	119.65	104.36	2835.57	2473.30

	Porosity		%Prst
SAMPLE	E	%E	
U1	0.13	12.64	
U2	0.13	13.23	
U3	0.12	12.48	
Ave.	0.13	12.78	

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WATER ABSORPTION BY TOTAL IMMERSION (TREATED STONE)

DATA

SAMPLE	0	0.08	1.00	2.00	3.00	8.00	24.00	48.00	72.00	96.00	120.00	144.00	168.00	192.00	216.00	240.00	264.00	288.00	312.00	336.00
									Weight of Sample (grams)											
T1	303.12	303.56	304.07	304.41	304.52	305.29	307.89	311.70	314.59	314.74	314.78	314.83	314.93	315.08	315.08	315.23	315.40	315.37	315.37	315.43
T2	306.94	307.87	308.32	308.59	308.82	309.77	311.92	313.62	315.73	316.33	316.73	317.03	317.36	317.73	317.81	318.07	318.28	318.38	318.47	318.57
T3	302.88	303.35	303.75	304.02	304.16	304.80	306.34	307.78	309.69	310.31	311.11	311.72	312.64	313.38	313.85	314.33	314.58	314.72	314.82	314.94
Ave	304.31	304.93	305.38	305.67	305.83	306.62	308.72	311.03	313.34	313.79	314.21	314.53	314.98	315.40	315.58	315.88	316.09	316.16	316.22	316.31
Wt. Ch.		0.61	0.45	0.29	0.16	0.79	2.10	2.32	2.30	0.46	0.41	0.32	0.45	0.42	0.18	0.30	0.21	0.07	0.06	0.09
Rate		7.36	0.49	0.29	0.16	0.16	0.13	0.10	0.10	0.02	0.02	0.01	0.02	0.02	0.01	0.01	0.01	0.00	0.00	0.00

%WA — Percentage of Water Absorbed

SAMPLE	0	0.08	1.00	2.00	3.00	8.00	24.00	48.00	72.00	96.00	120.00	144.00	168.00	192.00	216.00	240.00	264.00	288.00	312.00	336.00
T1	0.00	0.15	0.31	0.43	0.46	0.72	1.57	2.83	3.78	3.83	3.85	3.86	3.90	3.95	3.95	4.00	4.05	4.04	4.04	4.06
T2	0.00	0.30	0.45	0.54	0.61	0.92	1.62	2.18	2.86	3.06	3.19	3.29	3.39	3.52	3.54	3.63	3.69	3.73	3.76	3.79
T3	0.00	0.16	0.29	0.38	0.42	0.63	1.14	1.62	2.25	2.45	2.72	2.92	3.22	3.47	3.62	3.78	3.86	3.91	3.94	3.98
Ave	0.00	0.20	0.35	0.45	0.50	0.76	1.45	2.21	2.97	3.12	3.25	3.36	3.50	3.64	3.70	3.80	3.87	3.89	3.91	3.94

WATER ABSORPTION CAPACITY

SAMPLE	Org.D.W M0(g)	Sat. W. Mmax(g)	Redry W. Md(g)	WAC
T1	303.12	315.43	304.55	3.57
T2	306.94	318.57	307.45	3.62
T3	302.88	314.94	304.18	3.54
Average	304.31	316.31	305.39	3.58

POROSITY BY WATER ABSORPTION

SAMPLE	Mass of Pore = Volume of Pore Mp = Vp	App. V. Va (cc)	% Prst % voids
T1	12.31	130.00	9.47
T2	11.63	137.50	8.46
T3	12.06	131.00	9.21
Average	12	132.83	9.04

POROSITY BY HYDROSTATIC WEIGHING
(TREATED STONE)

SAMPLE	Dry W. M1(g)	Imm W. M2(g)	Air W. M3(g)	Pore V. Vp(cc)	App.V. Va(cc)	Real V. Vr(cc)	Real D. Pr (g/cc3)	App D. Pa(g/cc3)
T1	303.12	183.65	314.90	11.78	131.25	119.47	2537.21	2309.49
T2	306.94	185.90	318.10	11.16	132.20	121.04	2535.86	2321.79
T3	302.88	181.00	314.35	11.47	133.35	121.88	2485.07	2271.32
Average	304.31	183.52	315.78	11.47	132.27	120.80	2519.22	2300.76

	Porosity	
SAMPLE	E	%E
T1	0.09	8.98
T2	0.08	8.44
T3	0.09	8.60
Average	0.09	8.67

Water Absorption by Total Immersion

Rate of Water Absorption by Total Immersion

149
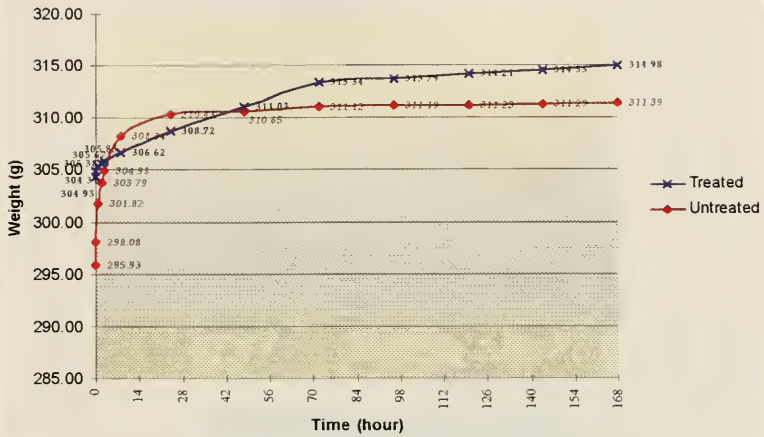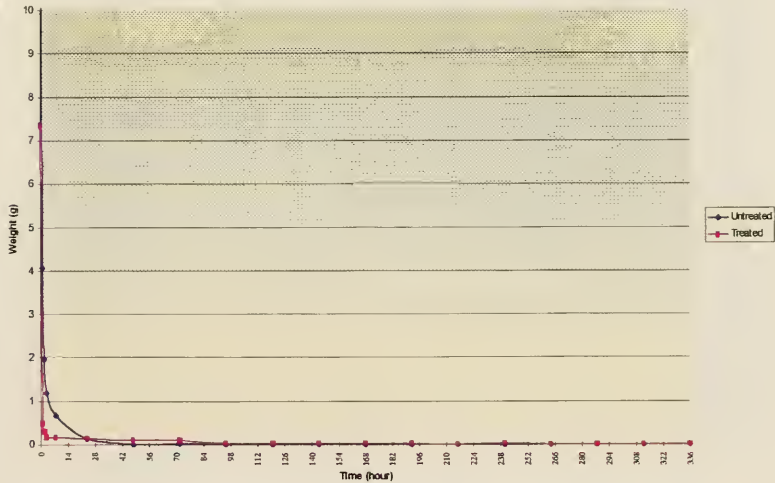
COMPARISON OF % POROSITY

150
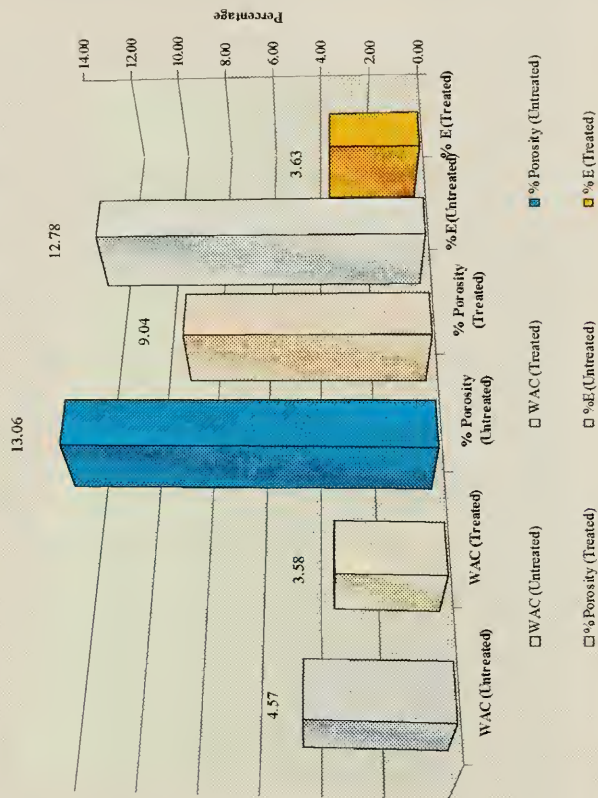
CAPILLARY ACTION (TREATED STONE)

SAMPLE	min										Height (cm)					
		1	2	3	4	5	10	15	20	25	30	60	90	120	150	180
Parallel to Bed	1	1.30	1.60	1.75	1.90	1.90	2.10	2.25	2.35	2.40	2.50	2.80	2.85	2.90	2.95	2.95
Parallel to Bed	2	0.80	1.00	1.20	1.30	1.35	1.60	1.60	1.80	1.90	2.00	2.20	2.25	2.25	2.25	2.25
Parallel to Bed	3	0.90	1.10	1.25	1.30	1.40	1.70	1.70	1.80	2.00	2.10	2.30	2.50	2.60	2.60	2.60
Average (cm.)		1.00	1.23	1.40	1.50	1.55	1.80	1.85	1.98	2.10	2.20	2.43	2.53	2.58	2.60	2.60
Perpend to Bed	1	0.8	1.1	1.15	1.2	1.4	1.4	1.5	1.5	1.6	1.6	1.8	1.9	1.9	1.9	1.9
Perpend to Bed	2	0.6	0.9	1.1	1.2	1.2	1.5	1.6	1.65	1.65	1.65	1.8	1.9	1.9	1.95	1.95
Perpend to Bed	3	0.6	0.85	1.15	1.2	1.25	1.5	1.5	1.6	1.7	1.75	1.8	1.85	1.9	1.9	1.9
Average (cm.)		0.67	0.95	1.13	1.20	1.28	1.47	1.53	1.58	1.65	1.67	1.80	1.88	1.90	1.92	1.92
SAMPLE	$min^{1/2}$	1	1.41	1.73	2.00	2.24	3.16	3.87	4.47	5.00	5.48	7.75	9.49	10.95	12.25	13.42
Parallel to Bed	1	1.3	1.6	1.75	1.9	1.9	2.1	2.25	2.35	2.4	2.5	2.8	2.85	2.9	2.95	2.95
	2	0.8	1	1.2	1.3	1.35	1.6	1.6	1.8	1.9	2	2.2	2.25	2.25	2.25	2.25
	3	0.9	1.1	1.25	1.3	1.4	1.7	1.7	1.8	2	2.1	2.3	2.5	2.6	2.6	2.6
Average (cm.)		1.00	1.23	1.40	1.50	1.55	1.80	1.85	1.98	2.10	2.20	2.43	2.53	2.58	2.60	2.60
Perpend to Bed	1	0.8	1.1	1.15	1.2	1.4	1.4	1.5	1.5	1.6	1.6	1.8	1.9	1.9	1.9	1.9
	2	0.6	0.9	1.1	1.2	1.2	1.5	1.6	1.65	1.65	1.65	1.8	1.9	1.9	1.95	1.95
	3	0.6	0.85	1.15	1.2	1.25	1.5	1.5	1.6	1.7	1.75	1.8	1.85	1.9	1.9	1.9
Average (cm.)		0.67	0.95	1.13	1.20	1.28	1.47	1.53	1.58	1.65	1.67	1.80	1.88	1.90	1.92	1.92

CAPILLARY ACTION (TREATED STONE)

Height (cm)

SAMPLE	min	1	2	3	4	5	10	15	20	25	30	60	90	120	150	180
Parallel to Bed	1	0.80	0.80	0.90	0.90	0.90	1.10	1.20	1.20	1.20	1.20	1.20	1.20	1.20	1.20	1.20
Parallel to Bed	2	0.70	0.80	0.80	0.80	0.80	1.00	1.50	1.50	1.50	1.50	1.50	1.20	1.20	1.20	1.20
Parallel to Bed	3	0.90	1.10	1.20	1.20	1.20	1.20	1.20	1.40	1.40	1.40	1.20	1.20	1.20	1.20	1.20
Average (cm.)		0.80	0.90	0.97	0.97	0.97	1.10	1.30	1.37	1.37	1.37	1.30	1.20	1.20	1.20	1.20
Perpend to Bed	1	0	0	0	0	0	0	0		0.05	0.05	0.05	0.05	0.05	0.05	0.05
Perpend to Bed	2	0	0	0	0	0	0	0	0	0	0	0	0	0.05	0.05	0.05
Perpend to Bed	3	0	0	0	0	0	0	0	0	0	0	0	0	0.05	0.05	0.05
Average (cm.)		0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.02	0.02	0.02	0.02	0.05	0.05	0.05

SAMPLE	min$^{1/2}$	1	1.41	1.73	2.00	2.24	3.16	3.87	4.47	5.00	5.48	7.75	9.49	10.95	12.25	13.42
Parallel to Bed	1	1.3	1.6	1.75	1.9	1.9	2.1	2.25	2.35	2.4	2.5	2.8	2.85	2.9	2.95	2.95
	2	0.8	1	1.2	1.3	1.35	1.6	1.6	1.8	1.9	2	2.2	2.25	2.25	2.25	2.25
	3	0.9	1.1	1.25	1.3	1.4	1.7	1.7	1.8	2	2.1	2.3	2.5	2.6	2.6	2.6
Average (cm.)		1.00	1.23	1.40	1.50	1.55	1.80	1.85	1.98	2.10	2.20	2.43	2.53	2.58	2.60	2.60
Perpend to Bed	1	0.8	1.1	1.15	1.2	1.4	1.4	1.5	1.5	1.6	1.6	1.8	1.9	1.9	1.9	1.9
	2	0.6	0.9	1.1	1.2	1.2	1.5	1.6	1.65	1.65	1.65	1.8	1.9	1.9	1.95	1.95
	3	0.6	0.85	1.15	1.2	1.25	1.5	1.5	1.6	1.7	1.75	1.8	1.85	1.9	1.9	1.9
Average (cm.)		0.67	0.95	1.13	1.20	1.28	1.47	1.53	1.58	1.65	1.67	1.80	1.88	1.90	1.92	1.92

Capillary Action (Untreated Stone)

Capillary Action (Treated Stone)

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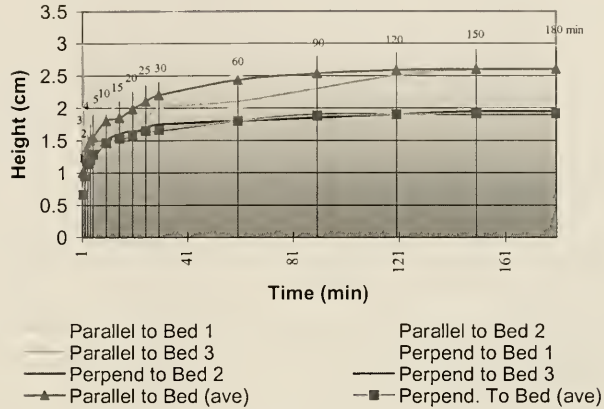
WATER VAPOR TRANSMISSION TEST
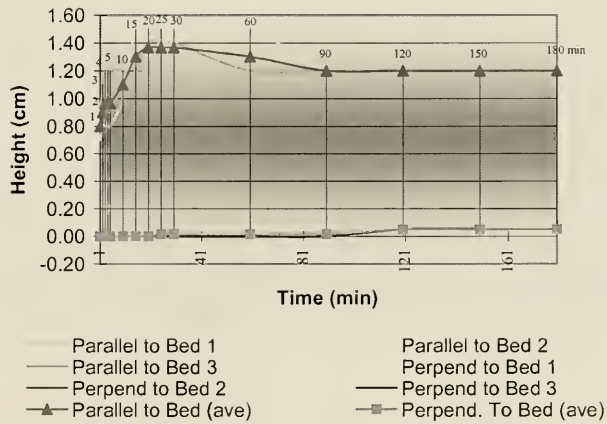
	0	1	2	3	4	5	6	7	8	9	10	11	12	13	14	15	16	17	18
Time (day)	0	1	2	3	4	5	6	7	8	9	10	11	12	13	14	15	16	17	18
Time (hour)		24	48	72	96	120	144	168	192	216	240	264	288	312	336	360	384	408	432
%RH	18	24	24	18	18	18	19	19	19	18	18	18	19	19	18	18	18	18	18
Perpend BP																			
U Dummy	280 3	280 3	280 3	280 4	280 4	280 4	280 5	280 5	280 5	280 5	280 6	280 6	280 6	280 6	280 7	280 7	280 8	280 8	280 8
U1	469 8	469 8	469 8	469 8	469 8	469 9	469 8	469 8	469 7	469 6	469 5	469 4	469 3	469 2	469	468 9	468 8	468 7	468 6
U2	470 5	470 3	470 3	470 4	470 4	470 4	470 4	470 3	470 2	470 1	470	469 9	469 8	469 6	469 5	469 4	469 3	469 2	469 1
U3	467 7	467 7	467 7	467 7	467 7	467 7	467 7	467 6	467 5	467 4	467 3	467 2	467	466 9	466 8	466 6	466 5	466 4	466 3
Average	469 32	469 27	469 27	469 29	469 29	469 30	469 29	469 24	469 12	469 03	468 96	468 80	468 71	468 55	468 43	468 32	468 20	468 09	467 97
WtChng(G)		0 05	-0 01	-0 01	0 00	-0 01	0 01	0 05	0 12	0 09	0 07	0 16	0 09	0 16	0 12	0 11	0 12	0 11	0 12
G/t		0 002	0 000	-0 001	0 000	0 000	0 000	0 002	0 005	0 004	0 003	0 007	0 004	0 007	0 005	0 005	0 005	0 005	0 005
WVT		0 44	-0 08	-0 11	0 00	-0 06	0 06	0 41	1 02	0 75	0 58	1 30	0 77	1 30	0 99	0 91	1 02	0 94	0 99
T Dummy	246 2	246 2	246 2	246 2	246 2	246 2	246 2	246 3	246 3	246 3	246 3	246 3	246 3	246 3	246 3	246 3	246 4	246 4	246 4
T1	468 3	468 3	468 4	468 4	468 4	468 4	468 4	468 4	468 3	468 3	468 3	468 2	468 2	468 1	468 1	468	467 9	467 8	467 7
T2	467	467	467	467	467	467 1	467 1	467	466 9	466 9	466 8	466 6	466 4	466 2	466	465 9	465 7	465 5	465 4
T3	467	467 1	467 1	467 1	467 1	467 1	467 1	467 1	467 1	467 1	467	467	467	466 9	466 9	466 8	466 7	466 7	466 6
Average	467 46	467 48	467 48	467 48	467 48	467 48	467 51	467 50	467 49	467 43	467 36	467 26	467 20	467 08	466 99	466 88	466 77	466 67	466 57
WtChng(G)		-0 02	0 00	0 00	0 00	-0 03	0 01	0 01	0 06	0 02	0 05	0 10	0 06	0 12	0 09	0 11	0 11	0 10	0 10
G/t		-0 001	0 000	0 000	0 000	-0 001	0 000	0 001	0 002	0 001	0 002	0 004	0 002	0 005	0 004	0 004	0 005	0 004	0 004
WVT		-0 17	0 03	0 00	-0 03	-0 22	0 06	0 11	0 47	0 19	0 39	0 83	0 50	1 02	0 75	0 88	0 94	0 86	0 83
Parallel BP																			
U Dummy	292 7	292 8	292 9	292 9	292 9	292 9	293	293	293	293	293 1	293 1	293 1	293 1	293 1	293 2	293 2	293 3	293 3
U1	467 5	467 5	467 5	467 3	467 2	467	466 8	466 6	466 3	466	465 8	465 5	465 4	465 1	464 9	464 7	464 5	464 3	463 9
U2	417	416 9	416 8	416 1	415 8	415 4	415 1	414 7	414 1	413 7	413 5	412 9	412 6	411 9	411 5	411 2	410 9	410 7	
U3	451 9	451 9	451 9	451 9	451 8	451	450 3	450 2	450 1	450	449 9	449 8	449 7	449 6	449 4	449 2	448 9	448 7	448 5
Average	445 48	445 45	445 40	445 10	444 93	444 49	444 09	443 85	443 49	443 25	443 06	442 74	442 55	442 27	442 03	441 78	441 51	441 27	441 01
WtChng(G)		0 027	0 053	0 297	0 167	0 447	0 400	0 233	0 363	0 243	0 187	0 323	0 190	0 277	0 240	0 250	0 267	0 243	0 263
G/t		0 001	0 002	0 012	0 007	0 019	0 017	0 010	0 015	0 010	0 008	0 013	0 008	0 012	0 010	0 010	0 011	0 010	0 011
WVT		0 22	0 43	2 46	1 38	3 70	3 31	1 93	3 01	2 02	1 55	2 68	1 57	2 29	1 99	2 07	2 21	2 02	2 18
T Dummy	304 9	305	305	305	305	305 1	305 1	305 1	305 1	305 1	305 1	305 1	305 1	305 1	305 1	305 1	305 1	305 1	305 1
T1	466 9	466 9	466 9	466 9	466 8	466 8	466 8	466 7	466 5	466 4	466 4	466 2	466 2	466	466	465 9	465 8	465 7	465 6
T2	465 6	465 5	465 6	465 5	465 5	465 4	465 4	465 3	465 1	465	465	464 8	464 7	464 6	464 4	464 3	464 2	464 1	
T3	466 9	466 9	466 9	466 9	466 8	466 7	466 7	466 6	466 4	466 3	466 2	466 1	466	465 9	465 8	465 7	465 5	465 4	465 3
Average	466 44	466 43	466 46	466 42	466 37	466 31	466 25	466 16	466 01	465 92	465 84	465 70	465 63	465 49	465 41	465 30	465 19	465 09	464 99
WtChng(G)		0 007	-0 030	0 040	0 050	0 063	0 060	0 087	0 157	0 087	0 080	0 140	0 073	0 133	0 087	0 107	0 113	0 093	0 103
G/t		0 000	-0 001	0 002	0 002	0 003	0 003	0 004	0 007	0 004	0 003	0 006	0 003	0 006	0 004	0 004	0 005	0 004	0 004
WVT		0 06	-0 25	0 33	0 41	0 52	0 50	0 72	1 30	0 72	0 66	1 16	0 61	1 10	0 72	0 88	0 94	0 77	0 86

	Perpend		Parallel	
	U	T	U	T
G (g)	0.84	0.70	1.26	0.42
G/t (g'h)	0.005	0.004	0.01	0.004
WVT (g/h/s q.m)	0.99	0.82	2.09	0.86

RATE OF WATER VAPOR TRANSMISSION

Legend:
- UT Perpend
- T perpend
- UT parallel
- T parallel

Y-axis: Time (hour)
X-axis: Rate
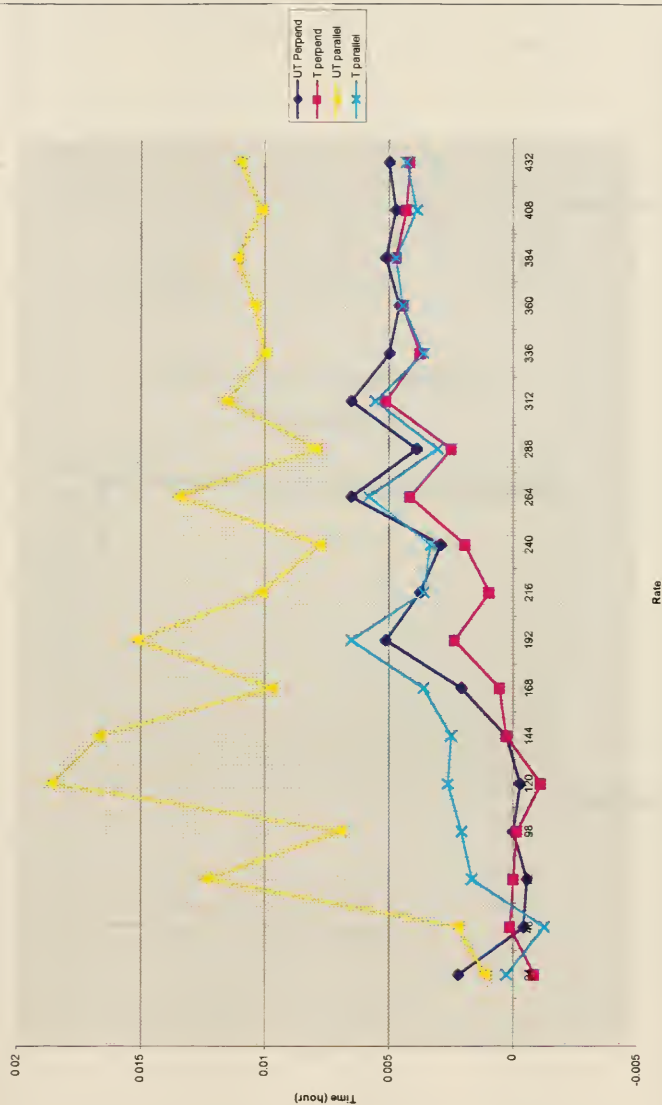
Water Vapor Transmission
(Untreated Stone)

Legend:
Ppd BP-U1 — Ppd BP-U2 — Ppd BP-U3
— Ppd BP-Uave (green square) — Prl BP-U1 — Prl BP-U2
Prl BP-U3 — Prl BP-Uave (triangle)

Water Vapor Transmission
(Treated Stone)

Legend:
PpdBp-T1 — PpdBp-T2 — PpdBp-T3
PpdBp-Tave (green square) — PrtBp-T1 — PrtBp-T2
PrtBp-T3 — PrtBp-Tave (triangle)

SALT CRYSTALLIZATION TEST

Cycle	1		2		3		4		5		6		7		8		9		10		Redry	Salt(g)	% salt(w)
	Dry	Salt	Dry	Salt	Dry	Salt	Dry	Salt	Dry	Salt	Dry	Salt	Dry	Salt	Dry	Salt	Dry	Salt	Dry	Salt			
U1	342.47	360.85	342.93	362.87	343.31	363.90	342.95	367.38	341.07													0.48	0.14
U2	288.25	297.94	288.33	298.81	288.59	299.16	288.69	298.94	289.84	299.48	290.96	300.63	292.93	301.06	292.06							4.68	1.62
U3	304.09	314.34	303.66	314.73	303.84	314.94	303.84	314.59	304.80	313.84	304.88	314.88	306.70	314.89	304.96							2.61	0.86
Ave.	311.60	324.38	311.64	325.47	311.91	326.00	311.83	326.97	311.90	306.66	297.92	307.72	299.82	307.98	298.51							2.59	0.87
T1	381.79	388.05	380.89	396.62	381.61	398.40	381.22	401.44	384.08	402.49	388.03	403.32	385.25	405.05	387.68	407.98	390.04	410.39	392.28	412.77	393.89	12.10	3.17
T2	274.85	277.90	274.24	280.50	274.38	282.74	274.34	282.59	275.39	282.20	276.27	284.35	278.40	284.96	277.87	285.34	278.89	286.23	280.06	288.01	281.93	7.08	2.58
T3	312.99	315.76	312.22	317.84	312.11	320.23	311.99	321.70	312.96	323.13	314.46	325.47	317.13	326.46	315.88	328.18	318.03	329.99	319.87	331.27	321.23	8.24	2.63
Ave.	323.21	327.24	322.45	331.65	322.70	333.79	322.49	335.24	324.14	335.94	326.25	337.71	326.93	338.82	327.14	340.50	328.99	342.20	330.74	344.02	332.35	9.14	2.83

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CLIMATOLOGY OF NOTHEASTERN THAILAND[127]

Geographical Situation

Thailand is located in the tropical area between latitudes 5° 37'N to 20° 27'N and longitudes 97° 22'E to 105° 37'E. The total area is 513,115 square kilometers or around 200,000 square miles. The boundaries of Thailand with adjacent areas are :

- *North* : Myanmar and Laos

- *East* : Laos, Cambodia and the Gulf of Thailand.

- *South* : Malaysia.

- *West* : Myanmar and the Andaman Sea.

Topography

According to the climate pattern and meteorological conditions Thailand may be divided into 5 parts i.e. Northern, Northeastern, Central, Eastern and Southern Parts. The Nakhon Ratchasima Province is in the Northeastern Part. The topography of this part is as follow.

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[127] Information from the Local Climatology Sub-Division of the Climatology Division in the Meteorological Department, August 2000.

Northeastern Part

This region is naturally a high level plain called northeast plateau. Northwest-southeast oriented Phu Phan ridge in the northeastern portion separates this part into two basins. One is a large high level plain in the west. The another is smaller and slope towards the east. This part is divided into 19 provinces (e.g. Nakhon Ratchasima, and Buri Ram).

General Climatic Conditions

The climate of Thailand is under the influence of monsoon winds of seasonal character i.e. southwest monsoon and northeast monsoon. The southwest monsoon which starts in May brings a stream of warm moist air from the Indian Ocean towards Thailand causing abundant rain over the country, especially the windward side of the mountains. Rainfall during this period is not only caused by the southwest monsoon but also by the Inter Tropical Convergence Zone (ITCZ) and tropical cyclones which produce a large amount of rainfall. May is the period of first arrival of the ITCZ to the Southern Part. It moves northwards rapidly and lies across southern China around June to early July that is the reason of dry spell over upper Thailand. The ITCZ then moves southerly direction to lie over the Northern and Northeastern Parts of Thailand in August and later over the Central and Southern Part in September and October, respectively. The northeast monsoon which starts in October brings the cold and dry air from the anticyclone in China mainland over major parts of Thailand, especially the Northern and Northeastern Parts which is higher

latitude areas. In the Southern Part, this monsoon causes mild weather and abundant rain along the eastern coast of the part.

The onset of monsoons varies to some extent. Southwest monsoon usually starts in mid-May and ends in mid-October while northeast monsoon normally starts in mid-October and ends in mid-February.

Season

From the meteorological point of view the climate of Thailand may be divided into three seasons as follows :

Rainy or Southwest Monsoon Season (Mid-May to Mid-October)

The southwest monsoon prevails over Thailand and abundant rain occurs over the country. The wettest period of the year is August to September. The exception is found in the Southern Thailand East Coast where abundant rain remains until the end of the year that is the beginning period of the northeast monsoon and November is the wettest month

Winter or Northeast Monsoon Season (Mid-October to Mid-February)

This is the mild period of the year with quite cold in December and January in upper Thailand but there is a great amount of rainfall in Southern Thailand East Coast, especially during October to November.

Summer or Pre-monsoon Season (Mid-February to Mid-May)

This is the transitional period from the northeast to southwest monsoons. The weather becomes warmer, especially in upper Thailand. April is the hottest month.

Surface Temperature

Upper Thailand i.e. the Northern, Northeastern, Central and Eastern Parts usually experiences a long period of warm weather because of its inland nature and tropical latitude zone. March to May, the hottest period of the year, maximum temperatures usually reach near $40^{o}C$ or more except along coastal areas where sea breezes will moderate afternoon temperatures. The onset of rainy season also significantly reduces the temperatures from mid-May and they are usually lower than $40^{o}C$. In winter the outbreaks of cold air from China occasionally reduce temperatures to fairly low values, especially in the Northern and Northeastern Parts where temperatures may decrease to near or below zero.

Seasonal temperatures (°C) in the Northeastern Part of Thailand

Temperature	Region	Winter	Summer	Rainy
Mean	Northeast	24.0	28.4	27.7
Mean maximum	Northeast	30.1	34.9	32.2
Mean Minimum	Northeast	18.1	22.7	24.1

Extreme maximum temperatures (°C) in Summer

Region	Maximum temperature	Date/Month/Year	Province
Northeast	43.9	28 Apr 1960	Udon Thani
	42.3	31 Mar 1979	Nakhon Rachasima

Extreme minimum temperatures (°C) in winter

Region	Minimum temperature	Date/Month/Year	Province
Northeast	-1.4	2 Jan 1974	Sakon Nakhon
	4.9	12 Jan 1975	Nakhon Ratchasima

Rainfall

Upper Thailand usually experiences dry weather in winter because of the northeast monsoon which is a main factor that controls the climate of this region. Later period, summer, is characterized by gradually increasing rainfall with thunderstorms. The onset of the southwest monsoon leads to intensive rainfall from mid-May until early October. Rainfall peak is in August or September which some areas are probably flooded. However, dry spells are commonly occur for 1 to 2 weeks or more during June to early July due to the northward movement of the ITCZ to southern China.

According to a general annual rainfall pattern, most areas of the country receive 1,200 - 1,600 mm a year.

Seasonal Rainfall (mm) in the Northeastern Parts of Thailand

Location	Winter	Summer	Rainy	Annual rainy days
Northeast Region	72.8	211.1	1,111.9	117

Relative Humidity

Thailand is covered by warm and moist air in most periods of the year except the areas farther in land the relative humidity may significantly reduces in winter and summer. For

example, the extreme minimum relative humidity values shows only 9 % at Suphanburi and Chiang Mai on 25 February 1972 and 24 March 1960, respectively. In the Southern Part which is maritime characteristic the humidity is relatively higher.

Relative Humidity (%) in the Northeastern Parts of Thailand

Region	Winter	Summer	Rainy	Annual
Northeast	69	65	80	72

Cloudiness

Cloud cover is normally less from November to March. Perfectly clear skies are generally found that is a reason why extreme temperatures usually occur. Most clouds in this period are high clouds but cumulus and cumulonimbus may be seen on some occasions. During the southwest monsoon, most clouds in the sky are convective clouds. Clear skies are seldom occur in this period except during June which have a few days.

Thunderstorms

Thunderstorms in upper Thailand often occur in the period from April to October while those in the Southern Part will occur in March to November. The maximum frequency of thunderstorms in upper Thailand is in May. Convection and the confluence of two

different air streams, cold and warm, are the main factor of thunderstorms. The afternoon and evening thunderstorms occur from the convection while the other from the confluence of winds of different airstreams.

Surface Wind

The pattern of surface wind directions is characterized by the monsoon system. The Prevailing winds during the northeast monsoon season are mostly north and northeast in upper Thailand and east or northeast in the Southern Part while they are south, southwest and west over the country during the southwest monsoon. In summer, prevailing wind are mostly south, especially in upper Thailand.

The data below is the monthly mean wind speed (knots) and prevailing wind in the Nakhon Ratchasima Province during the last 5 years.

YEAR		JAN	FEB	MAR	APR	MAY	JUN	JUL	AUG	SEP	OCT	NOV	DEC
1996	spd.	.8	1.5	1.0	1.3	1.1	1.0	1.5	1.5	1.2	1.2	1.6	1.5
	prev.	NE	NE	S	S,NE	SW	SW,S	SW	SW		SW	NE	NE	NE
1997	spd.	.6	1.3	1.5	1.2	1.8	2.0	1.8	1.8	1.4	1.4	1.3	1.2
	prev.	NE	NE	NE	S	S	SW	SW	SW	W	NE	NE	NE
1998	spd.	.9	1.0	1.1	1.2	1.4	1.4	1.3	1.0	1.0	1.4	1.5	1.6
	prev.	NE	NE	S	S	S	SW	S	W	NE	NE	NE	NE
1999	spd.	1.3	1.7	1.1	.8	1.4	1.8	1.9	2.1	1.3	1.5	1.8	3.0
	prev.	NE	NE	S	E	SW	SW	SW	SW	SW	E	NE	NE

YEAR	JAN	FEB	MAR	APR	MAY	JUN	JUL	AUG	SEP	OCT	NOV	DEC
2000 spd.	1.4	1.5	1.3	1.2	1.2	1.5	1.9	1.9	1.4	1.4	2.8	2.4
prev.	NE	NE	NE	S	SW	SW	W	W	W	NE	NE	NE
Mean spd.	1.0	1.4	1.2	1.1	1.4	1.5	1.7	1.7	1.3	1.4	1.8	1.9
Prev. wind	NE	NE	S	S	SW	SW	SW	SW	SW,W	NE	NE	NE

Tropical Cyclones

Tropical cyclone affecting Thailand usually moves from the western North Pacific Ocean or the South China Sea. Considering its strength it may be characterized by wind speed as follows :

- *Tropical Depression* : the maximum sustained winds up to 33 knots (61 kilometers per hour)

- *Tropical storm* : the maximum sustained winds between 34 - 63 knots (62 - 117 kilometers per hour)

- *Tropical Depression* : the maximum sustained winds up to 33 knots (61 kilometers per hour)

- *Tropical storm* : the maximum sustained winds between 34 - 63 knots (62 - 117 kilometers per hour)

- *Typhoon* : the maximum sustained winds 64 knots and above (118 kilometers per hour and above)

Thailand normally receives the effect of tropical depressions because of its location farther in land and some mountain ranges which obstruct and decrease the wind speed before moving into Thailand except the Southern Part has a relatively high risk of tropical storms and typhoon. By considering the annual mean, tropical cyclones usually move across Thailand about 3 - 4 times a year. During January to March, Thailand has never received the effect. According to the historical data, it can be seen that April is the first month which tropical cyclone move across Thailand. The relatively higher frequencies are found from May, particularly September and October. They usually pass through the Northern and Northeastern Parts in early southwest monsoon season and will move across the southern Thailand from October to December.

The frequency of tropical cyclones moving through Thailand during 49 years (1951 - 1999)

Region	Jan	Feb	Mar	Apr	May	Jun	Jul	Aug	Sep	Oct	Nov	Dec	Total
Northeast	-	-	-	-	1	5	4	15	27	22	4	-	78

APPENDIX IV: SUMMARY OF LITER

Ref. No.	Year	Author	Country	Material	Consolidation Material (Number Days of Curing) / Notes
9.1P135	2000	Matovic	Yg	Fresh Sandstone	
9.1P181	2000	Sahlin	Swd	Siliceous Sandstone	CEN TC246/W1014
				Calcereous Limestone	
9.2P3	2000	Baccaro	It	Sandstone	
				Limestone	
				Cement Mortar	
				Efflorescence	
9.2P361	2000	Leroux	Fr.	Sanstone	21
			Ge.	Limestone	
			Pt.	Marble	
			It	Siliceous Chalk	
9.2P395	2000	Maxova	Cze	Sandstone	20 / 30 30 30 / 30 30 30 / 30
				Spongolite	
				Traventine	
9.2P413	2000	O'Corner	Aus	Siliceous Sandstone	NM
9.2P513	2000	Twilley	USA	Micaceous Sandstone	
9.2P561	2000	Messori	It	Stone & Plaster	15
9.2P765	2000	Wendler	Tha	Clay Rich Sandstone	42 / 42
BulkPack	1999	Lotzmann	USA	Sandstone	28
BulkPack	1999	Pleyers	USA	Sandstone	
7P1033	1992	Wheeler	USA	Siliceous Sandstone	14 14 / 14 14 / 14
				Calcitic Limestone	
7P1073	1992	Valdeon	Sp	Dolomite	20
				Limestone	
				Sandstone	
7P1083	1992	Baronio	It	Sandstone	80 / 50
				Soft-mud Brick	
				Lime Putty Mortar	
7P1103	1992	Wendler	Ger	Clay Rich Sandstone	42
7P1113	1992	Witte	Bel	Ferrugineous Sandstone	42
7P1231	1992	Moropoulou	Grc	Calcereous Sandstone	
7P1327	1992	Calatayud	Spn	Calcereous Sandstone	10 / 20
7P1335	1992	Santamaria	Spn	Calcereous Sandstone	15 / 20
7P1343	1992	Rizzi	It	Calcereous Sandstone	
6P86	1988	Theoulakis	Grc	Sandstone	
6P236	1988	Fitzner	Ger	Sandstone	
6P330	1988	Alessandrini	It	Sandstone	6h
				Limestone	
				Marble	
6P356	1988	Heiman	Aus	Sandstone	
6P367	1988	Derkowska	Pol	Sandstone	
				Limestone	
				Mortar	
				Brick	
				Wood	
6P466	1988	Cabalova	Cze	Sandstone	
6P524	1988	Ausset	Fr	Stone	
6P563	1988	Domaslowski	Pol	Sandstone	
				Limestone	
6P712	1988	Hauff	Ger	Sandstone	
BulkPack	1983	Furlan	Swz	Calcereous Sandstone	

Total for each test		
Total for each subject		
% of each test per type		
Total of study type	122	
% of each study type	11	

Ref. No.	Year	Author	Country	Material
9.1P135	2000	Matovic	Yg	Fresh Sandstone
9.1P181	2000	Sahlin	Swd	Siliceous Sandstone
				Calcereous Limestone
9.2P3	2000	Baccaro	It	Sandstone
				Limestone
				Cement Mortar
				Efflorescence
9.2P361	2000	Leroux	Fr.	Sanstone
			Ge.	Limestone
			Pt.	Marble
			It	Siliceous Chalk
9.2P395	2000	Maxova	Cze	Sandstone
				Spongolite
				Traventine
9.2P413	2000	O'Corner	Aus	Siliceous Sandstone
9.2P513	2000	Twilley	USA	Micaceous Sandstone
9.2P561	2000	Messori	It	Stone & Plaster
9.2P765	2000	Wendler	Tha	Clay Rich Sandstone
BultPack	1999	Lotzmann	USA	Sandstone
BultPack	1999	Pleyers	USA	Sandstone
7P1033	1993	Wheeler	USA	Siliceous Sandstone
				Calcitic Limestone
7P1073	1993	Valdeon	Sp.	Dolomite
				Limestone
				Sandstone
7P1083	1993	Baronio	It	Sandstone
				Soft-mud Brick
				Lime Putty Mortar
7P1103	1993	Wendler	Ger	Clay Rich Sandstone
7P1113	1993	Witte	Bel	Ferruginous Sandstone
7P1233	1993	Moropoulou	Grc	Calcereous Sandstone
7P1327	1993	Calatayud	Spn	Calcereous Sandstone
7P1335	1993	Santamaria	Spn	Calcereous Sandstone
7P1343	1993	Rizzi	It	Calcereous Sandstone
6P96	1988	Theoulakis	Grc	Sandstone
6P236	1988	Fitzner	Ger	Sandstone
6P330	1988	Alessandrini	It	Sandstone
				Limestone
				Marble
6P356	1988	Heiman	Aus	Sandstone
6P367	1988	Derkowska	Pol	Sandstone
				Limestone
				Mortar
				Brick
				Wood
6P466	1988	Cabalova	Cze	Sandstone
6P524	1988	Aussel	Fr	Stone
6P563	1988	Domaslowski	Pol	Sandstone
				Limestone
6P712	1988	Hauff	Ger	Sandstone
BultPack	1988	Furlan	Swz	Calcereous Sandstone

	Total for each test
	Total for each subject
	% of each test per type
	Total of study type
	% of each study type

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