MEDISTONE

Preservation of ancient MEDIterranean sites in terms of their ornamental and building STONE

Newsletter n°5
September 2007

Contents.....

Editorial

Welcome to the firth issue of MEDISTONE Newsletter. BRGM as co-ordinator of the MEDISTONE project, launches in the name of the consortium this firth newsletter to inform about the aims and the work under progress of the project since May 2007.

MEDISTONE project (call FP6-2003-INCO-MPC-2; contract n°015245) proposes to contribute to the knowledge and the conservation of three of the most important archaeological sites in North Africa (Volubilis in Morocco, Djemila in Algeria, the Alexandria Lighthouse in Egypt) :

- identifying stones used and determining their origins in terms of geographic areas and, if possible, the former quarry sites
- establishing diagnosis of the state of conservation of the stones, and describing mechanisms of the deterioration to stone for semi-arid continental climate
- providing answers to the main problems regarding stone conservation / restoration that are liable to be met at the selected sites i.e. reassembling fractured and fissured stones.

This newsletter is part of MEDISTONE ongoing dissemination activities. All partners are acknowledged for their contributions.

David Dessandier, co-ordinator, BRGM

Last minute to the attention of MEDISTONE Partners : Extraordinary meeting the 15-16 of November in Marseille (France)

According to the situation in Algeria (problem of security for foreign people linked to terrorism), any on-site mission is cancelled. An extraordinary meeting will take place the 15 and 16 of November in Marseille (France) at BRGM to adapt the objectives and corresponding technical tasks and budget of MEDISTONE and to work on a new version of the "description of work" to submit to the European Commission.

Works under progress

WP1 - IDENTIFICATION OF STONES AND DETERMINATION OF THEIR PROVENANCE:

During the period (May - September 2007), several on-site and in-laboratory works were carried on, mainly:

- Training of the Moroccan doctorate student Mohammed El Rhoddani (partner n°11 / University of Meknès) on in-laboratory stone analyses.
- Technical synthesis works on ornamental and building stones of Volubilis (Morocco).
- Continuation of in-laboratory analysis on stones in BRGM laboratories.

→ 18 April-18 may 2007: Training of the Moroccan doctorate student Mohammed El Rhoddani (partner n°11 / University of Meknès) on in-laboratory stone analyses (place: Scientific and technical Centre of BRGM - Orléans, France):

The training (duration 1 month) permitted to Mohammed to discover and practice several analytical technical, and to apply them to 32 samples of building stones from Volubilis site and its supposed corresponding ancient quarries. He was trained by BRGM specialists from the Monitoring Measurement Analysis Department on the following analytical procedures:

- Samples preparation protocols (drying, grinding).

- Calcite-Dolomite content measurement.
- Total Porosity and bulk density measurement.
- Water absorption measurement.
- Identification of mineral phases (major mineral and clays) by X-Ray diffraction.
- Petrographic observation by optical microscope
- Texture observation and chemical mapping by Scanning electron microscope (SEM).

- Chemical analyses of major elements by X fluorescence and of trace elements by ICPMS.
- Isotopic analyses of C13/O18.

→ 21-25 may 2007 : Technical synthesis works on ornamental and building stones of Volubilis (place: Volubilis site and Moulay Idriss Zerhoun area Morocco) :

A team composed of Dr David Dessandier (partner n°1 / BRGM), Prof. Said Kamel (partner n°11 / University of Meknès), Mr Mohammed El Rhoddani (doctorate student / University of Meknès), Dr Myrsini Varti-Matarangas (partner n°6 / IGME) and Dr Lise Leroux (partner n°2 / CPP-LRMH) worked on the typology of building stones by the mean of field visual controls (on Volubilis site and quarries areas) and microscopic observations of thin sections. A proposal of table of content of the "Atlas of ornamental and building stones (delivrable D1) was also defined.

→ Continuation of in-laboratory analysis on building and ornamental stones from Morocco, Egypt and Algeria (place: Scientific and technical Centre of BRGM - Orléans, France):

The in-laboratory characterisation of stones (limestones, marbles, granites..) sampled on monuments (Volubilis, Djemila, Alexandria lighthouse) was carried on and still in progress. The typical methodology of study of the stones is described below:

- Mineralogical and petrographic characterisation (X-ray diffraction, optical microscopy and Scanning electron microscopy).
- Chemical composition (major and trace elements measurement).
- Porous medium and properties of fluid transfer (total porosity, natural saturation degree, capillarity coefficient measurements).
- Mechanical properties (compressive strength test).
- Isotopic chemistry (C13/O18) applied to carbonated stones.

WP2 - DIAGNOSIS OF THE CONSERVATION STATE OF THE STONES:

Concerning the WP2 and the study of stone degradation in the 3 sites, the period was devoted to:

- Preparation of polished sections and thin sections from the 3 sites and for Egypt on few samples from the Aswan quarries.
- First petrographical examination
- First X rays diffractions on few samples
- 15 days training stage of the Egyptian student Ashraf Nageh

REPORT on the venue of A. Nageh (doctorate student / Suprem Concin of Antiquities / Partner n°14) in CICRP

Ashraf Nageh came for a training stage of 2 weeks from the 16th to the 27th of July 2007. The purpose of this first short stage was to start working together in order to set up a general methodology for the study of the alteration of the stones from the lighthouse of Alexandria. An introducing general visit of the center was done in order to provide to Ashraf Nageh a view of the people, the scientific tools and the scientific activities of our institute.

Ashraf presented to us the work already done on the different stones under optical petrographical microscope in Egypt. With the help of an Egyptian geologist, he had observed the main petrographic features of each type of stone (granite, granodiorite, quartzite, limestone, marble) and we validated together these observations on thin sections.

After this first general overview, we focused the study on the degradation of granite stones for which we have many samples of different origins:
- antique sculptures found during undersea excavations of the lighthouse remains and exposed in different places in Alexandria
- stones considered as original parts of the lighthouse found in the base of Qaitbay fortress
- samples from the quarries (Aswan region)

The degradation of granite was investigated on raw samples and thin sections by the means of petrographical analyses, Raman microscopy, SEM examination and EDS analyses and XRD analyses.

Study of thin sections

Thin sections were made in Egypt and in Cicrp. The thin sections made in Egypt were covered with glass. They were used for observations under the optical polarising microscope. The few uncovered ones made at the surface of the decayed materials at Cicrp were used both for examination and microanalyses.

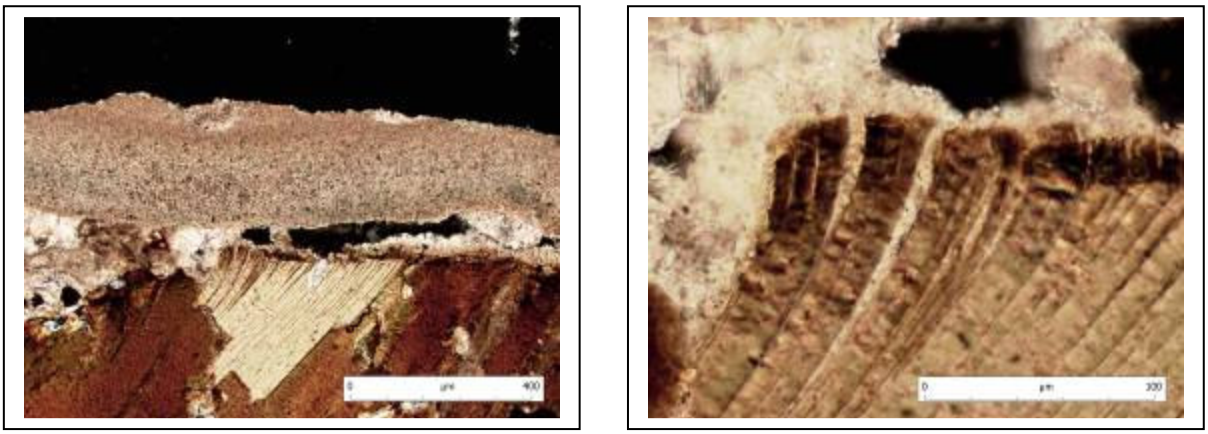


Figure 1: surface of a granite scale covered with a shell concretion covering and exfoliation of biotite with calcite filling underneath (detailed view on the right photography) – thin section, crossed nicols (Photos P. Bromblet).

On thin sections, we put the focus on the alteration features that could be observed under optical microscope. Feldspars and especially plagioclases are partially sericitised. Biotites seem to be partially chloritized. Near the surface, intergranular and intragranular cracking networks were noticed and correlated with the scaling of the stone. Biotites are exfoliated (Figure 1) and iron oxides have precipitated in their boundary. Apart from the biogenic calcite of the shells covering the stone, two generations of calcite have at least crystallised in the porosity. The first one made of large monocrystalline grains can have a hydrothermal origin. The second one, more abundant, is microcrystalline and fills the cracks. Its origin is currently not yet determined. Photographies at different scales were taken with digital camera. Soluble salts were not observed: they had been dissolved by the water used for the realization of the thin sections. Raman microscope was successfully tested to identify in situ some of the secondary minerals (calcite, iron oxides neoformation…). Few areas were selected in order to perform some elementary chemical analyses with a low vacuum electronic microscope (LV-SEM) fitted with an energy dispersive spectrometry (EDS). These analyses were performed during the following week at LRMH (Champs sur Marne) thanks to the collaboration of Lise Leroux (LRMH, partner 2).

Study of raw samples

Soluble salts were studied directly on the raw samples by SEM/EDS. The potentialities of this technique which needs no preparation of the samples/thin sections to observe the soluble salt crystallisations were experimented on a fragment of granite during a preliminary session in the University of Provence before the summer leave of this department. Sodium chloride crystallisations (halite) and various calcite neoformations were observed in the porosity of the granite.

Two samples of granite coming from the Aswan quarries which had been brought by Ashraf Nageh, were dedicated to mineralogical investigations by the means of XRD analyses. XRD analysis were performed on the raw material and also on the clay fraction according to the methodology developed within the framework of another student's work in Cicrp to extract and analyse very small amount of clay minerals in small samples.

Both samples had been observed and analysed. One showed a deposit which had a cream colour (Figure 2). This sample was divided in two parts:

- One was immersed in distilled water in order to put in solution the hydrophilic materials and also to put particles of the deposit into a suspension,
- On the other part of the sample, the deposit was scratched under microscope, in order to analyse the total powder after grinding and the clay fraction

The second sample was fresh granite made. It had been grounded in particles with a size under 100 µm and XRD analysis was performed on it.

Figure 2: Orange cream deposit in a fissure near the surface of a granite sample coming from the Aswan quarries (1 graduation corresponds to 1 millimetre - photo JM. Vallet)

XRD analysis on the scratched sample confirmed the presence of calcite and iron oxides. XRD on the extracted clay fraction showed also the presence of kaolinite.

At last, during his stage, Ashraf Nageh enhanced his bibliography on the studies of granite alteration and collected information that we had or we were able to find with him on the different kinds of alteration that can affect this kind of stone as a geological formation and a building material (hydrothermal alteration, weathering etc).

Conclusions

The venue of Ashraf Nageh allowed very fruitful discussions on the more pertinent methodology for studying the alterations of the lighthouse stones. This preliminary work that we performed together and the discussions that took place have led us to precise several points.

It would not be reasonable to study all the types of stone in detail. We wish to do a general and illustrated description of the alteration of each type of stone with its hypothetical origin and mechanisms and to perform deeper investigations on granite stone which is in fact the main stone material forming the remains of the lighthouse.

Now that the methodology has been validated, Ashraf Nageh will come a second time in CICRP for few weeks to perform XRD, Raman and SEM analyses on chosen area of raw samples and/ or thin section or polished sections. Several tasks have been defined to prepare this second venue in order to optimize the mission:

- New thin section without glass side have to be made on the surface of some samples showing degradation features (scaling, powdering) to allow more microscopic observations of the alterations combined with in situ microanalyses (Raman microscope and EDS analyses).
- Each thin section has to be described in a first step in order to determine the most interesting areas for relevant microanalyses.
- Possibility to scratch samples in order to analyse the deposits which are present on the inner parts of the scales will be investigated
- Parts of each sample have to be grounded into powders for soluble salts analyses that will be performed in France (BRGM).

During this second training, Ashraf Nageh will bring few new samples in Alexandria:

- weathered granite from an antique sculpture (column?) which has never been undersea
- weathered granite from an embankment built during the 19th century

These several samples should help us to distinguish the alteration phenomena occurred undersea after the collapsing of the lighthouse from the weathering of the Aswan granite in the marine atmospheric environment of the town.

The best period to come another time in Marseille would be February or March of the next year after a planned mission in Egypt (measurement of ultrasonic velocity on decayed sculptures that should be restored within the project).

During our work, we have met an almost insurmountable difficulty due to the fact that granite stones are never fresh granite rock but altered and weathered rock materials extracted from outcroppings. A good understanding of the alterations which have taken place in the granite stones and sculptures of Alexandria lighthouse requires a good knowledge of the previous inherited alterations due to the cooling of the geological formation, the circulation of hydrothermal solutions and the weathering that affected the rock material before its extraction in the antique quarries. In addition to the investigations already done in Egypt, Ashraf Nageh should spend some weeks with our colleagues of IUAV (Venice) who are studying within the framework of the WP1 the material from the quarries.

WP3 - DEVELOPMENT OF APPROPRIATE CONSERVATION / RESTORATION TECHNIQUES:

The WP3 work is in progress in accordance with the overall plan of the project. After field missions in the studied sites, the efforts are now centred on the techniques required for reassembling fractured stones and the development of mortar mix-designs based on local materials.

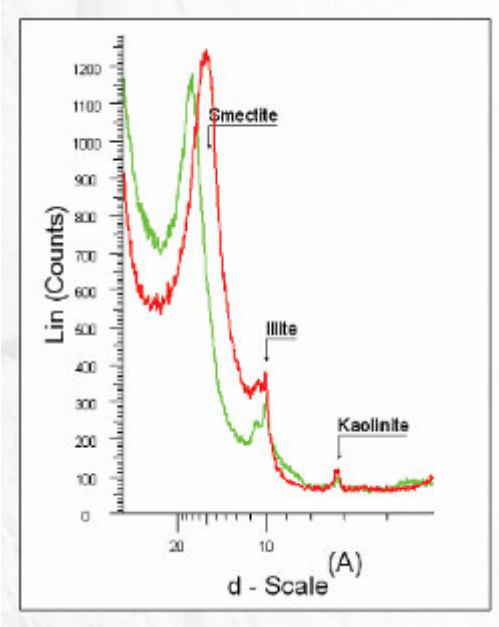
Laboratory works (leader: partner n°2 – CPP-LRMH):

Basically, we now have the samples from the 3 sites, the target is to identify the compatible product with each stone-type through a full characterisation/understanding of stone composition/mineralogy/thermal & mechanical behaviour. There are a number of key laboratory data acquisitions that are currently used by CPP/LRMH:

Dilation behaviour:
Calibration of the purchased dilation equipment is achieved and tests protocols were established according to the stones types (*mainly relative to the occurrence of expansive minerals as clays*). Dilation behaviour under temperature cycling, of the samples coming from Volubilis have been achieved as well as the behaviour of two ready-to-use products.
To fully understand the dilation of stones and to reach the stone compatibility with reassembling products, additional data are under acquisition:

Clays separations:
The nature of expansive minerals and their proportion within a stone can change radically its thermal dilation, indeed in Volubilis a long-time needed to reach dilation equilibrium of some samples, required mineral separation in order to identify the expansive phases involved. Clay minerals as potential expansive phases have been separated through a normalised procedure.

X-ray diffraction:
The X-ray diffraction is used for mineralogical identification of studied whole samples, as well as for the characterisation of the expansive phases when mineral separation was needed/achieved. It was the case for example for the soft yellowish limestone coming from Volubilis.



X-Ray diffraction of fine particles (less than 2 µm) showing the occurrence of expansive clay mineral (Smectite).

Additional methods: To fully understand the thermal dilation behaviour of stones other physical characteristics are needed. The CPP/LRMH is undertaking a pore-size distribution (using mercury porosimeter) of all samples studied and will shortly start the determination of the dynamic modulus of elasticity through pulse velocity of some selected samples.

Considering representative samples:
Large sampling is a recurrent issue when considering monument conservation/restoration. Indeed, precise determination of stone physical parameters requires representative specimen to be tested. There is then a minimal volume-threshold, the Elementary Representative Volume (ERV) for which a given material can be considered as continuous and homogeneous in the considered scale. The ERV is an intrinsic characteristic of a given material for a considered parameter, its reflect a scale effect and should be small enough to take into account the microstructure and in the meantime large enough to allow a description of a global behaviour. More the larger is the sample (in comparison to ERV), less are the measurement uncertainties.
When considering monuments sampling, the determination of the ERV will have a direct impact on the sampling procedure, of course destructive, but the less disturbing as possible for the monument. This problem is faced systematically when sampling building.

Keeping in mind the consideration given to the aspects concerning the reproducibility of tests and the representativeness of samples, the collected blocks from the studied sites were shaped in a well defined geometry. Cylindrical morphology (6cm-high and 2 cm in diameter) was used to decrease side-effects'. But this shape could not be considered for all samples, particularly for soft limestone of Volubilis due mainly to lake of hardness, which were shaped as prism. To ovoid the natural heterogeneity of the rocks, the laboratory tests are undertaken at least on 3 specimens from each facies.

Tests conducted for the choice of a suitable mortar to be used at the sites (leader: partner n°5 – LITHOS):

Empirical tests were conducted of which the results were evaluated only on a visual basis and not on an analytical basis. These samples will be subject to such laboratorial and analytical testing in order to be able to choose the most suitable sample to be used on-site. The reason for these tests was to improve the natural characteristics of a traditional mortar, in that one of our main priorities was to use products available locally.

After a first series of tests, it was decided to use as the base mortar a mix of quick lime, pozzolana and calcareous stone powder that are easily available in Morocco and Algeria (in Morocco lime can be found only as quick lime and in the form of stones, while in Algeria lime is partially slaked and in a powdery form).
In order to make these mortars perform better, we tested a series of additives in order to render them more fluid, in order to help the mix retain water longer and in order to avoid cracking.

Tested products

Base mortar:
1.5 parts quick lime slaked into a paste (slaking the quick lime)
2 parts sifted pozzolana
1 part stone powder

Fluidifyers:
Genium 27 Basf
Genium 51 Basf
Flocable Degussa
Ledan TL2 Tecnoedile Toscana

Bulking agents:
Tylose
Metil-cellulose
Fumed silica

These mortars were compared with two specific products commonly used in conservation for the filling of fissures and fractures in stone material:
Ledan Ta1 Tecnoedile Toscana
PLM M Cts

Formulation of the tests:

PROPORTIONS

	TEST 1	TEST 2	TEST 3	TEST 4	TEST 5	TEST 6
NAME OF PRODUCT	+GLENIUM 27	+GLENIUM 51	+FLOWCABLE	+LEDAN TL2	PLM-M	TA1
POZZOLANA	4 parts	4 parts	4 parts	4 parts		
STONE POWDER	2 parts	2 parts	2 parts	2 parts		
PUTTY LIME	3 parts	3 parts	3 parts	3 parts		
WATER	80% of 1 ½	80% of 1 ½	25 L for 100 Kg of paste	1 ½ parts	70% by weight	
QUANTITY OF PRODUCT	1.2 L for 100 Kg of binder	0.7 L for 100 Kg of binder	6% by weight of the paste	1% by weight	Qb.	Qb

TESTS CONDUCTED

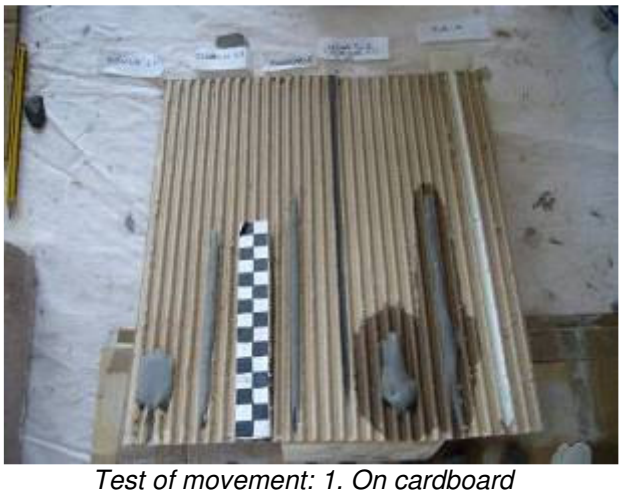
In order to facilitate the execution of the tests (in time and costs) the tests were conducted on materials other than stone but which could represent the performances required by a mortar, like fluidity within small fissures, water retention capacity, etc.

FLUIDITY TEST

This test was conducted in order to verify the mortars characteristics for conditions in which they are injected or poured. The following tests were conducted:

1. Injected the mortars into cardboard and observing which was able to move the farthest distance.
2. Pouring them into open cardboard and observing the distance travelled.
3. Same test on brick.

Tests of injection through the tubes in cardboard



Test of movement: 1. On cardboard

2. On brick

TESTS FOR WATER RETENTION AND ANGLE OF CONTACT

These tests observed the angle of contact and thus the propensity of the mortar to lose water immediately or not. Other tests demonstrated the time and quantity of water loss of the paste. The tests were conducted on cardboard and on brick to observe a pour. And on cardboard and on brick in order to observe the dispersion of the pour.

Test on cardboard

Test on brick

Test on cardboard

Test on brick

CRACKING TESTS

These were tests to observe the shrinkage of the mortars. All of the visual results have been collected and will be controlled with laboratory analysis in order to best choose the most suitable product. A summary of the qualitative results can be found in the table below:

	Flowcable	Glenium 51	Ledan	PLM-M
Quantity of product	+	+++	++	+
Fluidity	+++	++	+	+++
Water Retention	+	++	+++	+
Lack of Cracking	+++	++	++	+++
Hardness	+++	++	++	+++
Price	+++	++		+

Support & training of young researcher from Southern countries:

Besides its research of new reassembling techniques and in-situ application missions, the Cercle des Partenaires du Patrimoine/LRMH places great importance on the long-term applications and dissemination of WP3 results in Southern countries.

The CPP/LRMH support the training of young researchers (*PhD students*) by providing individual supports in two main ways:

- putting the student involved in the research undertaken: involving the student in the laboratory work in France and in the research of local traditional mortar in their countries. The student will then assembles competencies and ensures their continuity;

- giving the students responsibility, allowing them to become independent and used to laboratory techniques.

For example for the PhD student from Egypt, in-service training was provided on: Scanning Electron Microscope, Ionic chromatography, Dilation and petrophysics characterisation. This training was combined with bibliography research using the on-line database of LRMH and the library.
A proposal work of the student on the WP3 relative to the mix-design of local mortar has been submitted to Egyptian partner (Supreme Council of Antiquity).

Support & Training	Figure of 2007
PhD student received at CPP/LRMH	
Ashraf Nageh	from Supreme Council of Antiquities, Egypt
Naïma Rebahi	from University of Boumerdès, Algeria
Support provided	
In-service training	Laboratory techniques
Scientific exchange	Library, and publications in the subject
Period of the stay	
Ashraf Nageh	weeks 28 & 31
Naïma Rebahi	weeks 48 & 49

Result dissemination

As a leader of WP3, one of the objective of CPP/LRMH is to provide the Southern countries with applied scientific results (reassembling techniques), and to increase the visibility and the readability of the on-going work :

- by publication: The already obtained results will be widely disseminated via an oral communication entitled '*Thermal and hygric stone dilation : a key step in the path of reassembling fractured stones of Volubilis archaeological site*' *by Mohamed NASRAOUI & Jean-Didier MERTZ,* to be given in an international meeting hosted by Morocco (Marrakech): 2ème Rencontre Internationale sur le Patrimoine Architectural Méditerranéen 'RIPAM2' October 24-26, 2007. A paper is expected to be published in a Moroccan magazine 'NATURALIA MAROCANA' edited by Natural History Museum.

- by raising media awareness: building contact with the local media as the live interview given in the archeological site of Djamela by Mohamed NASRAOUI to Radio Setif (Algeria).

- by research access for general public: During the days of the Patrimony (15-16 September 2007) the MEDISTONE project has been presented to general public through a double poster and direct communication with the visitors.

Focus on partners

PARTNER N°9 : University M'Bougara of Boumerdès (Algeria)

The university M'Hamed Bougara of Boumerdes (UMBB) is founded in 1998,from groupment of six national institute wish have existed since 1970.The UMBB is a public university, supervised by the ministry of high education and scientific research.

Manpower and faculties:
The manpower of the students for 2007/2008 is with approximately twenty thousand (20 000), the administrative staff, technical and of service is about 630 people; however the teaching framework is with approximately of 900 between professor, master of conferences, master assistant and assistant.
The UMBB comprises four (04) faculties (Faculty of Science, Faculty of Science of the engineer, faculty of hydrocarbon and chemistry, commercial science and Faculty of Law), localized in four sites (south campus, northern, center and west) and a university annex localized in the city of Bouira, gathering 25 departments.

Department of materials engineering:
The department (GM) comprises three options: ceramics and glass, concrete and binder, wood and composite. Several teaching laboratories and a research laboratory of mineral materials and composite (LMMC), this last comprises the following sections: mechanical section (mechanical resistance compression, inflection, traction and tiredness)
section rheology (viscosity, thixotropy, potential zeta etc)
section granulometry (laser particle-measurement instrument, method Study BET)
section mineralogy (diffraction X-ray, fluorescence X, atomic absorption) thermal section (elongation (dilation coefficient); DTA, TGA, TG, T).

International conventions :
University Henri Poincaré Nancy 1 (FRANCE) / University of Technology of Troyes (FRANCE) / Higher institute of Materials and the Mechanical engineering (FRANCE) / University Paul Sabatier Toulouse III (FRANCE) / University of Pau and the Countries of Adour (FRANCE) / University libre of Bruxels (BELGIUM) / University of the Oil and Gas of Moscow (RUSSIA) / Technical University of Saint-Petersburg (RUSSIA) / Electrotechnical University of Saint-Petersburg (RUSSIA). Technical university of Ostrava (CZECH REPUBLIC) / Technical National University of Ukraine "Polytechnic Institute of Kiev" (UKRAINE) / Total Professors Associated (FRANCE) / School of the Mines of Nantes (FRANCE) / University of Versailles Saint-Quentin-en-Yvelines (FRANCE) / University of Technology of Belfort-Montbeliard (FRANCE) / Technical National university of the Oil and Gas of Ivano-Frankivsk (UKRAINE)

Various Informations

Next planned meetings

- 22 March 2008 in Meknès (Morocco) at the Moulay Ismail University – Second MEDISTONE Project Progress Meeting with participation of whole of the partners.
- WP1: Technical synthesis works about Alexandria Lighthouse planned the 8-9 of November in Venice, Italy.
- WP2: from 27 to 31/10/07 / Volubilis (Morocco), partner 3 with partners 10, 11 and 1: ultrasonic velocity measurements on the site, complementary sampling and observations, surface and atmospheric temperature measurements, training of the doctorate student Mohamed El Rhoddani.
- WP2: January 2008 / Alexandria (Egypt), partners 1, 3, 14, 5 days for ultrasonic velocity measurements on selected sculptures and complementary observations and sampling (Aswan granite weathered in the marine atmospheric environment of Alexandria)
- WP2: European partners (n°3 and 7) will meet in January 2008 in Dresden (Germany) in order to synthesize their work on the 3 sites and to prepare the next general Medistone workshop (Meknès, March 2008).

Next training for the doctorate students

- Training of the Moroccan doctorate student Mohammed El Rhoddani (partner n°11 / University of Meknès) and the Algerian doctorate student Naïma Rebahi (partner n°9 / University of Boumerdès) on diagnosis of the stone conservation state and on the stone conservation techniques (November - December 2007 – Place: CICRP Marseille and LRMH Paris, France).

Next issue of Newsletter

N°6 (January 2008).

Next dissemination of results

First MEDISTONE Workshop on the theme "Preservation of ancient Mediterranean sites in terms of their ornamental and building stone: 1. Identification and origin determination of stones" planned the 20-21 March 2008 in Meknès / Morocco

Presentations of POSTER or COMMUNICATION planned at the:

- 2ème Rencontre Internationale sur le Patrimoine Architectural Méditerranéen – RIPAM2 – Marrakech, Morocco – October 24-26 , 2007.
- 11th International Congress on Deterioration and Conservation of Stone – Torun, Poland – September 15-20, 2008.

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