

STUDY OF A NATURAL RESIN USED AS JOINT FOR THE BRICK MASONRY OF HINDU TEMPLES IN MY SƠN (VIETNAM).

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SUMMARY

The authors were involved with the archaeologists of the Fondazioni Lerici, Politecnico of Milan, in the preservation of some Hindu temples in Mỹ Sơn, Vietnam. The characterisation of the brick- masonry materials was carried out at the Politecnico of Milan. Particularly interesting also for future developments was the successful study of the natural resin used to bond the bricks in the masonry and the research of new compatible resins to be used for the repair and restoration. The results of the research are presented together with some proposal for future research.

INTRODUCTION

A team of the DIS- Politecnico of Milano was asked in 2000 by the archaeologists of the Lerici Foundation (Politecnico of Milan) and by the Institute for Conservation of Monuments in Hanoi to visit the Mỹ Sơn (Vietnam) archaeological site. The Hindu temples built by the Cham people between the 6th and the 14th cent AD need preservation and repair. A decision was taken by the Italian team to work out a pilot project for the preservation of the so called group G.

The attention of the brick masonry of the team was particularly attracted by peculiar technique of construction; in fact the horizontal and vertical bonding between bricks was so tight that they did show a sort of continuity in the wall. Moreover, this bond was still preserved after centuries and no (or very small) biological decay was noticed on the surface of the wall and the decay did not penetrate inside the wall. The binder used between bricks was recognised later by experimental investigation as a natural resin.

The most modern techniques of Vibrational Spectroscopy (Infrared and Raman) were used to identify the nature of the material at the molecular level. Strong indications were observed in the spectra that one of the original components of the resin (*dipterocarpol*) may have suffered along the years by self-oxidation in the presence of atmospheric oxygen. During the various visits it was also realized that a supposedly similar vegetal resin was presently available on the market and used for caulking boats. The analysis was also extended to a glue extracted from some local trees (Dầu Rái).

From the infrared spectrum it turned out that the examined resins (viscous liquid at room temperature) consist mostly of two main components: A (collected after evaporation under vacuum for 24 hrs at room T) is identified as alloaromadendrene (or its demethylated derivative); B (solid residue after evaporation) contains a large fraction of *dipterocarpol*.

The original resin is very similar (but not fully identical) to the natural resin available now. This resin (Daù Rài) was used during the preservation intervention between 2004 and 2005 to joint the bricks together on some buildings of the group G of temples,. The operations since then seem to be successful. The research made on masonry materials will be here reported. While the restoration work will proceed in the next year, the idea of realising thin joints for new masonry to be used in humid areas will be developed.

DESCRIPTION OF THE SITE AND OF THE CONSTRUCTION TECHNIQUE

The archaeological area of Mỹ Sơn is situated in Central Vietnam, 30 km South-West from Da Nang; it is located in a valley surrounded by low mountains dominated by the Rang Meo mountain, and it is crossed by the Thu- Bôn river. Mỹ Sơn area (Figures 1 and 2) is 15ha wide and composed by several groups of buildings made with brick masonry, each of them organized around a main temple (*Kalan*). Mỹ Sơn is the most important holy place of the Champa kingdom; the Cham people built here more than seventy buildings, from the 6th to the 14th cent AD, but nowadays only thirty with at least 1m in elevation are still recognizable.

The site was burnt and plundered in the past centuries during the Chinese and Kmer occupations until it was finally neglected and covered by the jungle. It was rediscovered after centuries of abandonment in 1898, and studied at the beginning of the 20th C. by a French architect, Henry Parmentier from the École Française d'Extrême Orient (EFO) (Parmentier 1918); a Vietnamese-Polish expedition (lead from 1982 until 1986 by Kasimierz Kwiatkowski and Hoang Dao Kihn) carried out restoration works on some group of buildings damaged during the war at the end of the sixties (Kwiatkowski 1990).



Figure 1. The archaeological area of Mỹ Sơn.



Figure 2. Mỹ Sơn: view of the groups C and D of temples.

The buildings in Mỹ Sơn were made by fired bricks thinly joined by natural resin. The stone was used as building material only for pillars, lintels and some decoration. The wall section is made of two leaves with small connections or three leaves, externally in bricks and with brick rubble in the middle (Figure 3). The most peculiar characteristic of the brick masonry was the special construction technique which realised the bond between bricks so tight that they did

not practically show real joints. The special building technique protects the walls from the affection of the vegetation: where the thin joint is not damaged, there are only very low biological attacks (Figure 4) (Condoleo 2007).

In order to realize a more tight physical bond the technique of *rub-joining* was used during the wall construction before applying the resin. Scratches can be seen on the horizontal and vertical surface of the bricks in contact with other bricks. The scratch can be clearly observed by a magnifier but even by naked eyes (Figure 5) (Bo Xay 2004).

Following some hypotheses that organic natural materials could have been used as binder between the bricks, as in historic buildings in other parts of Vietnam and South East Asia, a careful study was carried out on materials sampled from the Mỹ Sơn masonry walls.



Figure 3. Section of the wall.



Figure 4: The thin mortar joints in prospect.



Figure 5. Scratches due to the rub-joining procedure.

TESTS ON SAMPLED MATERIALS

In order to carry out a laboratory research on the masonry materials, samples were taken during several visits to the site, starting from the first one in 2000. The samples were collected from the groups A, D, E, G, being the last one the group of interest for the Italian pilot project. At DIS, Politecnico of Milan, the authors carried out several tests on the sampled materials in order to detect the properties of: bricks, brick assemblages and joint material.

Material sampling

Due to the difficulty of sampling without damaging the walls, the number of samples was rather small to be statistically representative of the materials used. Nevertheless the experimental research gave rather reliable results as it will be shown in the next Sections. All the brick samples were taken from the material available on the soil (buildings A1, A13, B9, D4, G1) or on the ruins (E4, E5, E7) in order to avoid spoiling the standing parts of the buildings. When possible, couplets of bricks stuck together by the original joint were collected (Figure 6). Following the hypothesis that the bricks were bond together using an organic binder coming from local trees, samples of vegetal materials were also collected for laboratory research (Figure 7).

In 2001 also a special glue from vegetal origin used for caulking of boats was bought at the local market in Hoi An (RES1); in 2004 another resin coming from local trees (called Daù

Rai) and sold as glue was bought close to Mỹ Sơn site (RES2) (Figure 8). During the excavation on G group some fragment of natural resin were found and also taken to Milano for analyses.



Figure 6. Couplets of bricks stuck together.



Figure 7. Vegetals collected in Mỹ Sơn in 2000



Figure 8. The “Dau Rai” tree and the resin extracted from it (2004)

Chemical analyses on bricks, joints and filling

These analyses concerned the bricks and the binder in the external joints and in the inner leaf of the walls. Chemical analyses according to ASTM C 114 (1996) were carried out on the sampled materials (Ballio et al. 2001).

The results showed that the composition of the brick is the same, despite the apparent visual difference, but also that the composition of the bricks and of the so called joint is the same. The presence of a very low content in CaO in the joint (from 2.33 to 4.48%) showed that no lime is present in it. Chemical analyses were also carried out on the material sampled from the internal leaf of the walls. They show that this material does not differ from the one of the bricks.

Mechanical and physical tests on bricks

Physical and mechanical tests (mono-axial compression and indirect tensile tests) were carried out on the brick samples. Dimensional variations in water were measured; nevertheless they have not to be taken into account, being very small.

Physical tests were performed on small 4 to 6 brick cubes (400mm side) cut from the bricks; the tests were carried out according to the European standard (UNI EN 2001). The results show a certain in-homogeneity; nevertheless some orientation values can be given as an average: (i) bulk density = $1,630 \text{ kg/m}^3$, (ii) I.R.S from 0.41 to $1.92 \text{ kg/m}^2/\text{min}$, (iii) water absorption coefficient = $160.09 \text{ g/cm}^2 \times \text{s}^{0.5}$, (iv) water absorption by total immersion between 18.18 and 23.99 %. Some XRD tests carried out on a specimen from A1 show that the bricks were fired at a temperature below 900°C . Specimens from A1 were fired at 900°C in order to control the previous results. The higher value of the bulk density ($1,608.7$ against $1,558.8 \text{ kg/m}^3$) and the lower water absorption (21.94 against 23.99 %) respect to the other A1 specimens seems to confirm the fact that the bricks were fired at a temperature below 900°C . Compression tests were carried out on cubes ($40 \times 40 \times 40 \text{ mm}$). Once again all the values were very much scattered, between 8 and 14 N/mm^2 ; nevertheless the cubes from A1 fired at 900°C gave the maximum strength; confirming that the original bricks were fired at a lower

temperature than 900°C. The modulus of elasticity E and the Poisson coefficient were also calculated and the values are typical of a rather soft material (Binda et al. 2006).

ANALYSES OF THE BINDER IN THE JOINTS AND OF THE NEW NATURAL RESINS

The presence of organic material between the bricks was firstly determined according to a test suggested in (Road Research 1955). Analyses were carried out on the joint material, which has been separated from the bricks, the core of the brick and its external part. The results were that a predominant content of organic material was found in the joint and a minor content in the bricks.

Spectrochemical analysis.

The “Giulio Natta” Department of Chemistry, Materials and Industrial Chemistry of the Politecnico of Milano (G. Zerbi) and the Institute of Biology of the Faculty of Science of the University of Milan (F. Tomé) have performed the chemical characterization of the resin found in the joint.

The identification of the main components of the organic materials has been carried out mostly by means of infrared spectroscopy with FT-interferometers. Since each sample necessarily consists of a mixture of a variety of chemical compounds the spectroscopic analysis focussed at the identification of the main components which could justify the chemical and physical behaviour of the materials. To help in the analysis a few procedures of separation of the components have been followed such as evaporation *in vacuo* and extraction with suitable solvents.

The idea behind these analyses is that the materials used in the building of the Mý Son temples be fully related to what nature offers in the area and what people can manufacture locally. In particular the organic materials used may originate from the resins of the trees which grow in the surrounding area. Trees of the species of the *dispterocarpaceae*.

The results of the analysis are as follows:

- i) Identification of the chemical nature of sample RES1 (a special resin, liquid at room temperature, used for caulking boats) and RES2 extracted (as viscous fluid from a local tree called Daù Rai).

The infrared spectrum of RES1 (Figure 9) shows a complex pattern which becomes simpler by keeping the sample under vacuum for 24 hrs.

A dry materials has been obtained as residue after evaporation and its infrared spectrum (Figure 10) is certainly simplified if its interpretation has to be carried out on the basis of a “group frequency” analysis. Dammarenediol seems to be the substance with the highest relative concentration with respect to many other possible substances in much lower concentration.

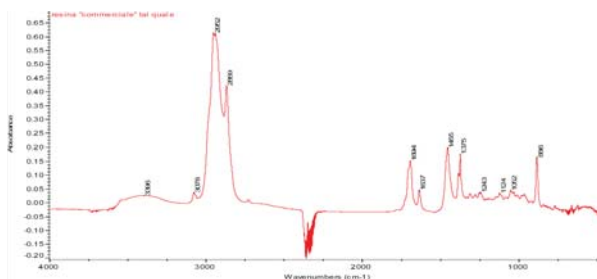


Figure 9. FT-IR spectrum of RES1 (fluid sample)

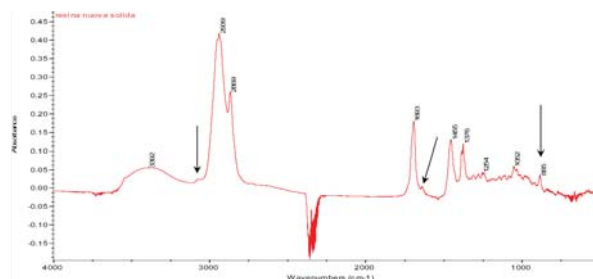


Figure 10. FT-IR spectrum of the solid residue after evaporation of RES1.

The disappearance of the bands near 3078, 1637 and 886 cm^{-1} strongly suggests that the substance which has evaporated contains in its structure C=C bonds. Difference spectroscopy (spectrum of the liquid-spectrum of the solid residue) provides the infrared spectrum of the volatile component of RES 1 (Figure 11) which can be identified as alloaromadendrene.

Next step is the analysis of RES2 which was analogously evaporated on a SeZn glass. Comparison of the spectrum of RES2 Figure 12 with that of Figure 10 shows that the two materials are practically identical, thus indicating that the local people obtained the glue from local trees.

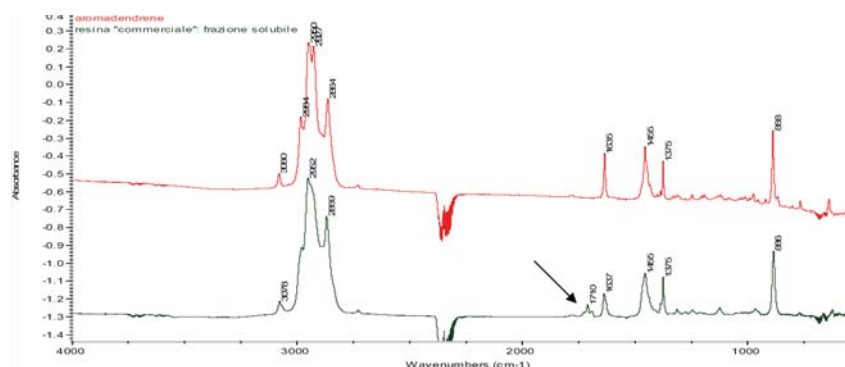


Figure 11. Comparison of the FT-IR spectra of the volatile component of RES1 (black) and the spectrum of aromadendrene (red).

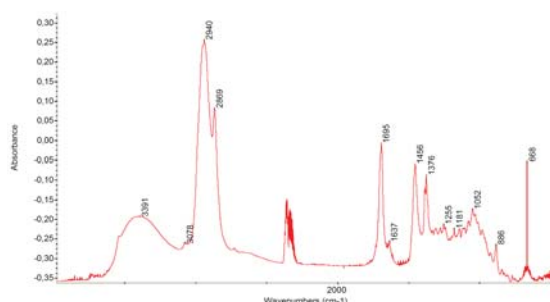


Figure 12. FT-IR spectrum of the residue of RES2 after evaporation *in vacuo*

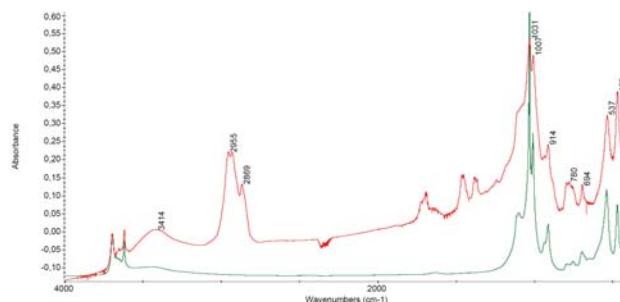


Figure 13. FT-IR spectrum of RES1c (red) showing the existence of kaolin (green).

ii) Next our attention was focused on samples bought on the market at Hanoi which was presented either as a solid block (RES1b) and a powder (RES1c). From the spectrum the material which makes up the solid block (RES1b) is substantially identical to the solid residue from RES1 obtained after evaporation (Figure 10). However the spectrum of the powder RES1c shows additional absorption bands which allowed to identify the existence of kaolin, thus indicating that the powder is not only the ground resin but it also consists of

a mixture of additional materials in which kaolin is the main additional component (Figure 13).

- iii) Analysis of the material scratched away from the joint (JRES1). Figure 14 shows the infrared spectrum of JRES1 as collected on the site. No absorption from the organic component can be observed since it is too weak with respect to the overwhelming infrared absorption by the components which make up the brick.

The absorption was removed by the inorganic material from the brick by extracting the organic fraction with carbon tetrachloride and recording the spectrum of the solute after evaporation of the solvent. Figure 15 shows the great similarity of the organic component extracted from JRES1 with the solid residue of RES1. The spectrum shows, however, that some chemical modifications have occurred from RES1 to JRES1 mostly associated to the C=O functional groups in the molecules (band at 1739 cm^{-1} from the brick and 1693 cm^{-1} in the solid residue of RES1). It is well likely that the resin taken from the brick, in the early days could have been just the resin from the trees; self-oxidation processes which possibly occurred along the many years could justify the spectral changes observed.

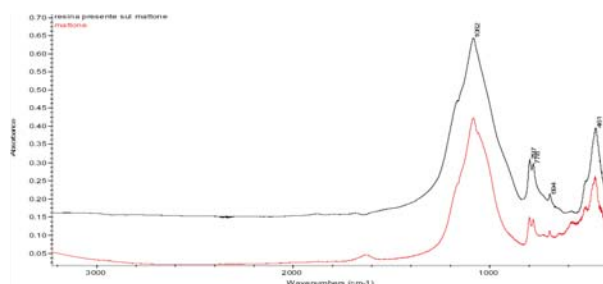


Figure 14. FT-IR spectra of JRES1; black sample with resin, red spectrum of a pure brick.

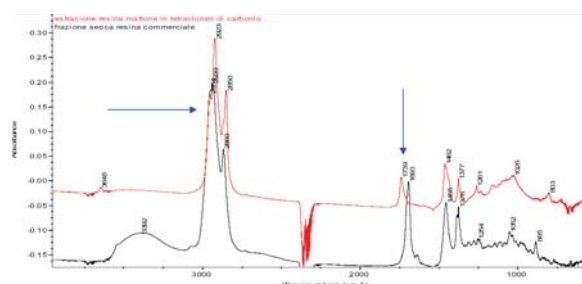


Figure 15. Comparison of the FT-IR spectra of a) material extracted with carbon tetrachloride from the brick (red) and b) the FT-IR spectrum of the solid residue of RES1.

- iv) Analysis of sample ARCHES (small fragments of resin found among the excavated bricks). The infrared spectrum of ARCHES shows again the spectrum of the solid fraction of the resin, but additional substances occur in the sample. Their identification requires more extended studies.

Mechanical tests on couplets

The compression test carried out on a couplet of bricks with the joint has been compared with a test carried out on a couplet of bricks without joint. The strength values found for the couplet without joint are lower than the ones obtained with the jointed couplet showing once again the role of the thin joint when well bonded to the bricks (Table 1). It can be observed by comparing the results that the strength of the couplet is very similar to the one of the bricks varying between $8\text{ and }14\text{ N/mm}^2$ (see previous section) and this is due to the fact that the joint is very thin and no mortar is between bricks. It is well known that mortar joints give regular stress distribution but lower the strength and the stiffness of the masonry.

Figures 16a and b shows the different behavior in the two situation as revealed by the ESPI (Electronic Speckle Pattern Interferometry) diagrams. Continuity in the horizontal displacement was the characteristic of the couplet with the joint.

Table 1. Compression tests on couplets.

Specimen	σ_c [N/mm ²]	ε_v [μm/mm]	E (30-60%)			$\frac{\Delta \varepsilon_l}{\Delta \varepsilon_v}$ (30-60%)
			E _{joint} [N/mm ²]	E _{prism} [N/mm ²]	E _{brick} [N/mm ²]	
G.4a without joint	11.48	11.55	1352	1623	3186	0.12
G.4b with joint	12.58	14.41	1470	1439	2147	0.09

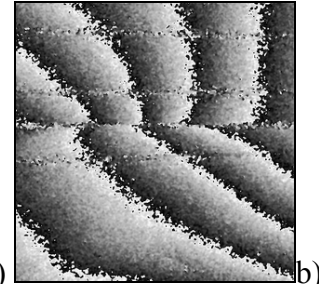
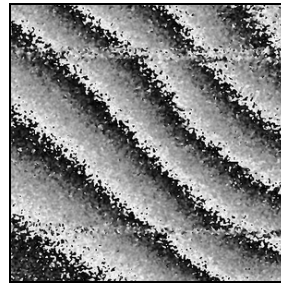
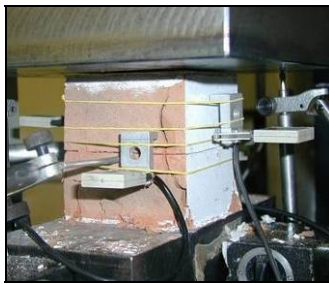


Figure 16. ESPI images during the compression test on the two couplet: a) specimen with joint; b) specimen without joint.

NEW RESINS USED FOR THE RESTORATION

The effectiveness of the second resin coming from Daù Rai tree was tested at DIS Laboratory in Milan, where it was used natural without heating or modifying it. Fourteen specimens made with two portions of bricks from Mý Sơn (80x40x50 mm dimension) (Figure 17) and glued together with the chosen natural resin were prepared and cured under different conditions in order to simulate the local climate in Mý Sơn. Some of the joints were very thin, other thicker in order to test the influence of the thickness variation which can occur on site. After gluing they were left under low compression load for two days. Then they were submitted to different temperature and R.H. in a climatic chamber or also to cycles as listed in Table 2. At the end of the treatments they were submitted to direct tensile tests.

Table 2. Tensile tests on couplets



Specimen	Curing condition	σ_t (N/mm ²)
C.1	30°C (4 days)	0.25
C.2	30°C (4 days)	0.24
C _{thin.1}	8h 20°C, 90% R.H. 16h 70°C, 50% R.H. (60 days)	0.11
C _{thin.2}	8h 20°C, 90% R.H. 16h 70°C, 50% R.H. (60 days)	0.17
C _{thick.1}	8h 20°C, 90% R.H. 16h 70°C, 50% R.H. (60 days)	0.16
C _{thick.2}	20°C, 90% R.H. (60 days)	0.11
C _{thick.3}	20°C, 90% R.H. (60 days)	0.04
C _{thick.4}	20°C, 90% R.H. (60 days)	0.04
C _{thin.3}	20°C, 90% R.H. (60 days)	0.16
C _{thin.4}	20°C, 90% R.H. (60 days)	0.10
C _{thin.5}	20°C, 90% R.H. (60 days)	0.07
C _{thick.5}	in water (60 days)	0.06
C _{thin.7}	in water (60 days)	0.07

Figure 17. Specimen made with two portions of brick and glued by resin

The testing machine was a servo-controlled hydraulic press of 0.5 t working under displacement control. The test results are also reported in Table 2. The lowest values were reached by specimens with thick joints cured at 20°C and 90% R.H for 60 days and by the specimens cured into water for 60 days, while the highest values were reached by the specimens cured at 30°C and with thin joints. These results were taken into account when applying the resin on site. It can also be observed that the direct tensile strength measured are some specimens was higher than strength measured by the authors on some old masonry with joints based on lime.

POSSIBLE FUTURE APPLICATION TO NEW THIN JOINT MASONRY

New thin joint mortars have been studied and produced during the last decade in Europe as a mean to join Aircrete and other lightweight blocks and particularly in UK, where the potential benefits of extending the use of thin joints to brickwork have been the subject of recent research at Kingston University (Fried et al. 2005). The experimental research was also applied to dense concrete block masonry for which a tentative to reduce the joint thickness of 3mm by using a glue mortar failed due to the fact that the thin joint could not accommodate variation in the block height (Figure 18). The appealing possibility of using thin glue mortar joints for brickwork was also mentioned even if the possibility of the mortar squeezing into the brick face represents a limit for the application.

The type of joint found in Mý Son would be so thin if applied to new brick masonry, provided that the brick dimensional variations would be carefully reduced, that no squeezing would occur. In Figure 19 an example of new masonry used as support in the restoration of Mý Son is given. So a future development of the presented research could be the application of resins as a joint to new brickwork. This technique could be applied in humid climates where the natural resin is available or an artificial resin could be synthesized for the purpose.



Figure 18. New thin joint masonry made with dense concrete blocks and glue mortar.



Figure 19. New masonry in Mý Son used to stabilize the ruins of group G.

CONCLUSIONS

In conclusion the experimental research carried out on the sampled materials from Mý Son temples allowed to state that: (i) the bricks were fired at a temperature below 900°C, (ii) no lime was used for the mortars and the tight bonding between bricks, (iii) the joint between bricks is so thin that no moisture seems to penetrate from the outside, (iv) the contact between the bricks was realized by the presence of a natural resin, (v) the thin joint also seem to help

increasing the strength of the masonry even if the bricks are rather soft and with low strength, (vi) the biological growth affects in a much lower degree the decay of the original walls with tight joints than it does for the repaired walls where cement mortar was used, (vii) the natural resin can be easily substituted by the present resin coming from the Daù Rai trees.

A further step of the research will be to check the possibility of using natural or synthetic resins for thin joint concrete and brick masonry to obtain more durable masonry and with good direct tensile strength.

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