

Thesis Abstracts

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Alma Arnaboldi

TL dating applied to Palazzo Raimondi and Palazzo Pallavicino Soldi, Cremona (Italy)

October 2015

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Degree: Masters

Supervisors: Anna Galli, Marco Martini, Ivan Veronese

Since the stratigraphic techniques initially developed for archaeology have been extended to the history of architecture, the relative internal sequence of the various building phases of a monument can be usually precisely determined. Their absolute chronology is however sometimes problematic or controversial. In such cases, the contribution of TL dating could be conclusive. It must be remembered, however, that care has to be taken when associating the TL age of a brick to that of its building structure because the date determined is the last firing of the sample. In case of reuse of materials from pre-existing structures, dates are older than the building; in case of upkeep or mimetic restoration, dates are younger. The main advantages of the TL dating of building are the availability of large quantities of material, the homogeneity of environmental radioactivity and the lesser extent of humidity fluctuation.

A recent conservation campaign at the Palazzo Raimondi and Palazzo Pallavicino Soldi, Cremona (Italy) gave the opportunity to sample bricks and mortars belonging to different structures (basement, ground floor and main floor). The relevance of these sites is due to the very old tradition in Cremona in using earthen mortars, materials and execution techniques very rarely employed and studied. This thesis work was aimed at dating the bricks to support the study of the earthen mortars.

TL dating was performed following the standard fine-grain technique using a home-made system based on the photon counting technique with a photomultiplier tube (EMI 9635QB) coupled to blue filters (Corning BG12). Artificial irradiations were carried out by a 1400 MBq ^{90}Sr - ^{90}Y beta source (Nominal dose-rate: 1.4 Gy/min) and a 37 MBq ^{241}Am alpha source (Nominal dose-rate: 14.8 Gy/min). Internal annual alpha and beta dose-rates were obtained by total alpha counting with ZnS scintillator discs and flame photometry analysis.

For what concerns Palazzo Raimondi, the calculated ages span in a huge range (300-750 years BP), the Bayesian statistical approach has been applied. The experimental results, all obtained from the basement structure, have been combined in a single formal analysis with the historical boundary corresponding to the foundation. In this way it has been possible to ascribe the fabrication of bricks to the second part of the fifteenth century.

For what concerns Palazzo Pallavicino Soldi the samples belong to different historical periods: the Middle Age (12th century), Renaissance (15th century) and the seventeenth century. The older samples are coeval to a pre-existing structure; the younger samples refer to restoration works documented in archives.

Michela Cantù

Characterization and dating of ancient mortars with additives

December 2015

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Degree: Ph.D.

Supervisors: Maria Pia Riccardi, Serena Chiara Tarantino, Emanuela Sibilìa

In the town of Cremona (Northern Italy) it is very common to find masonries both in rural and historic edifices made of bricks and earthen mortars with good physical and mechanical properties. Aim of this thesis was to carry out an archaeometric study of the earthen mortars from five edifices in Cremona (15th - 19th century) to deepen our information on well-established tradition of production and use of earthen mortars and its evolution throughout centuries. A combination of mineralogical, petrographic and geochemical

analyses was used together with the information from historical documents, the latter allowing a precise dating of the analysed samples.

The characterization of the raw materials was carried out by means of optical and electronic microscopy, XRPD and FTIR measurements. The archaeometric investigation allowed the identification of two recipes, differing in the amount of lime (and sometimes sand) added to the basic mixture of earth: the widespread lime-poor earthen mortars and the lime-rich earthen mortars, recognised only in the masonries of Palazzo Magio Grasselli (end of 18th century). Moreover, a two-step technological change in earthen mortars manufacturing that took place between the 17th and 18th century has been recognised: it consisted in the addition of larger amount of lime (from the 17th century) and of organic additives (from the 18th century). This was likely done to improve the mechanical properties of the earthen mortars.

The application of scientific dating methods is of great help in building archaeology, to support and/or validate the information obtained from the historical sources. However, the use of Optically Stimulated Luminescence (OSL) for mortar dating, recently proposed, is not still a routine technique. In the present work OSL was applied to the samples of earthen mortars already well dated on historical ground. To our knowledge, this is the first attempt to extend the applicability of OSL techniques to such materials.

In this case, OSL dating exploits the solar bleaching of quartz grains occurred during the production of mortar. Despite the high quartz content of the analyzed mortars, the solar bleaching efficiency was found not good, probably due to the opacity of the raw materials. In fact, OSL dating multi-grain technique was found unapplicable due to the high amount of poorly bleached grains. The single-grain approach, allowing the selection of the well-bleached grains gave better results and was therefore systematically applied. For data elaboration, several statistical models were used and compared. The simple average and Central Age Model (CAM) significantly overestimated the expected age. The Minimum Age Model (MAM) gave a more accurate evaluation of the equivalent dose. In particular the un-log 3-parameters MAM gave good results on the oldest samples but with large errors. The Internal External Consistency Criterion (IEU) gave generally more precise results, but is less accurate than MAM.

It clearly appeared that another criticality of this application is the recent age of the samples. Further studies are required to improve the efficiency of the low OSL signal detection and to reduce errors associated to the estimated equivalent doses.

Daniela Constantin

On the dating of the last glacial cycle in loess deposits using quartz optically stimulated luminescence

October 2015

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Degree: Ph.D.

Supervisors: Vlad Codrea, Alida Timar-Gabor

Luminescence dating of loess deposits in the Danube Basin and the Chinese Loess Plateau was conducted for the Last Glacial cycle samples using 4-11 μm and 63-90 μm quartz. Ages of older samples are underestimated for all grain sizes. For equivalent doses > 100 Gy (30-40 ka) the fine quartz chronologies underestimate the coarse quartz results.

Single aliquot regenerative dose protocol shows that the laboratory dose response obtained for the two fractions of quartz is different and is fitted with the sum of two exponential functions. The higher saturation in fine grains is not a result of the previous alpha irradiation history and time-resolved (TR-OSL) experiments show that this is not caused by contamination with a different mineral nor it is a consequence of multiple luminescence centres active in the quartz detection window. Laboratory saturation characteristics are not affected by previous irradiation, bleaching, or thermal treatments employed.

For doses > 200 Gy natural and laboratory dose response curves diverge, with natural signals showing earlier saturation. The signals measured on fine quartz after adding thousands of Gy on top of the natural dose are below the saturation level for both Romanian and Chinese quartz samples. Poor dose recovery may, in part, cause age underestimation at high doses. This suggests an initial dose-dependent sensitivity change that is not corrected for by the use of the response to a test dose.

Thus, a dose dependent phenomenon underlies the cause of the age discrepancy. Until the mechanisms of this phenomena are explained both sets of SAR OSL ages are suspected to be inaccurate and question the reported chronologies beyond 30-40 ka.

A summary of the thesis can be downloaded from: <http://doctorat.ubbcluj.ro/ro/sustinerile-publice-ale-tezelor-de-doctorat/>

Robert R. Hendricks

Timing of the Emplacement of an Ancient Coastal Deposit of Georgia Determined by Optically Stimulated Luminescence and Electron Spin Resonance Optical Dating

December 2015

McMaster University, Hamilton, Canada

Degree: Ph.D.

Supervisor: Dr. W. Jack Rink

ESR, OSL and TT-OSL dating methods were applied to samples collected from six of the Ancient Coastal Deposits (ACDs) along the southern Georgia Coastline. Samples were collected from the Princess Anne (the youngest and most seaward ACD), Pamlico, Talbot, Penholoway, Wicomico, and Okefenokee ACDs with the goal of determining the age of formation of these features. Ground Penetrating Radar

(GPR) was used to determine the subsurface morphology and target lithologies for age determination. OSL and TT-OSL dating was attempted on samples collected from the youngest two ACDs, the Pamlico and Princess Anne, at McMaster Universities AGE Lab. ESR samples collected from all of the ACDs studied were measured at Florida State University as well as Osaka University. ESR analysis measured the Al signal, the Ti-Li signal, measured using two different methods, as well as the Ti-H signal. A number of low additive dose points were added to the ESR dose plan to attempt to create a better dose response curve for the low saturating Ti-H signal in attempt to better utilize the signal.

While the geochronological methodology did not prove useful for determining the age of all of the ACDs it did result in depositional age estimates for the Cypresshead Formation at 433-2978 ka and Satilla Formations at 243-417 ka using the Ti-Li ESR signal as a maximum age estimate. The GPR, ESR, and core data all point to the conclusion that the ACDs of the Georgia Coast are geomorphic modifications and not the result of a unique depositional process. Based on the discrepancy between the depositional age of the Cypresshead and Satilla Formations as determined by ESR in this study and the ages of the ACDs published by others from Georgia (Markewich et al., 2013) or other areas of the Atlantic Coast (Wehmiller, 2004; Willis, 2006) it can be concluded that paleo sea-levels modified the Cypresshead and Satilla Formations in to the morphology seen today at some point after their initial deposition.

A PDF of this thesis can be downloaded from: <https://macsphere.mcmaster.ca/> OR by contacting the author at hendricks.robert.r@gmail.com

Daniel N. Livsey

Holocene sea-level, climate, and estuarine stratigraphy of Baffin Bay, Texas: studying past changes in coastal systems to elucidate future coastal response to changing sea-level and climate

September 2015

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Degree: Ph.D.

Supervisor: Dr. Alexander Simms

Recent studies along the northwestern Gulf of Mexico document rapid back-stepping of estuarine environments of up to 20 km and 150 m/yr at ca. 8.2 thousand years ago (ka), 4.8, ka, and 2.6 ka. If such rapid changes in coastal environments occurred today along the urbanized coast of the Gulf of Mexico major economical and ecological loss would occur. Of these three backstepping events, only one can be tied to a known cause the 8.2 ka event related to a rapid increase in the rate of relative sea-level rise. However, the cause of the latter two, the 4.8 and 2.6 ka events are largely unknown. To determine the relative roles of changes in sea level and cli-

mate in these two events, paleo sea-level, climate, and stratigraphic records, are presented from upper Baffin Bay, Texas and neighboring playa Laguna Salada. This abstract has been abbreviated to highlight the paleoclimate reconstruction that utilizes optically stimulated luminescence (OSL) age-dating for a playa chronology, the portion of the dissertation of specific interest to Ancient TL readers.

Paleoclimate records are sparse along the southern Texas coast. A new quantitative drought proxy is derived from a transfer function between X-Ray Fluorescence (XRF) elemental data from a Texas playa core and a tree-ring drought record. The playa core age-model was based upon twenty-two OSL ages obtained from quartz grains isolated from down-core sand lamina. Lamina sampled for OSL age-dating are characterized by well-sorted, sharp-based sand lamina that are interpreted to have been deposited as subaerial aeolian bedforms observed in the field. Radiocarbon ages were not collected given the paucity of organic matter. Using the transfer function, a 954-year tree-ring drought record was extended to ca. 3,000 ka. Ba, Br, and Pb were utilized as predictor variables. Machine learning algorithms, utilized to derive the transfer function, had maximum validation accuracies of 94%. Changes in the extended drought record correspond with the timing of the Roman Climate Optimum, Medieval Warm Period, Little Ice Age, and changes in North Atlantic sea surface temperatures (SST). Increased drought frequency is coeval with nearby dune migration ca. 0.2 ka, 1.9 ka, and 2.6 ka. The highest drought frequency in the record occurs during the Medieval Warm Period ca. 1.0 ka followed by a decrease in drought frequency during the Little Ice Age ca. 0.4 ka. Increased drought frequency accompanies increased North Atlantic SST since 3 ka. This trend of warm North Atlantic SST and dry conditions over the study area follows secular meteorological observations and tree-ring records. These results indicate that lacustrine derived XRF element data can be used as a quantitative tool to reconstruct past drought records, and that North Atlantic SST modulated drought in southern Texas for the last 3,000 years.

Within Baffin Bay, five flooding surfaces occur through a time-period of ever decreasing rates of relative sea-level rise and within error of periods of drying in southern Texas at ca. 1.0 ka, 2.6 ka, 3.4 ka, 4.8 ka, and 5.5 ka. I hypothesize that these flooding surfaces, occurring when sea level in the Gulf of Mexico was rising < 2 mm/yr, and during independently documented drying events, were primarily driven by changes in climate through declines in fluvial sediment supply to the coast.

A PDF of this thesis can be downloaded from: ProQuest Dissertations & Theses. As of 12-10-15 ProQuest is preparing to make the thesis discoverable.

Loïc Martin

Characterization and modeling of archaeological objects for their dating by Paleo-dosimetric methods. Simulation of the dosimetric parameters with Geant4.

December 2015

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Degree: Ph.D.

Supervisors: Norbert Mercier, Sébastien Incerti

The paleodosimetric dating methods allow to obtain the age of some mineral materials which behave as natural dosimeters, the age being derived as the ratio of the dose accumulated from the dated event and the natural dose rate to which the sample was subjected. The determination of the dose rate is usually based on simple models which only allow explicit calculations, but these models reproduce very imperfectly the diversity of situations encountered. Computer modeling allows to represent more complex and more realistic models, which lead to numerically simulate the dose rate. In this work, the toolbox Geant4, allowing to simulate particle-matter interactions by the Monte-Carlo method, was used to create complex models, as well as modeling tools accessible and adaptable to different types of samples and dosimetric situations.

Simulations were first made with basic models in order to study the origin of dose rate variations in sediments, highlighting the limits of the simple models commonly used. This information was used to guide the development of modeling tools based on the Geant4 codes, and also to specify the protocols of samples analysis for gathering the data needed for numerical simulations.

The DosiVox software allows to easily model a wide variety of samples through a voxelised representation of the object and its environment, and to simulate the radioactivity for calculating the spatial distribution of the dose rate. It was developed with a view of accessibility and current use. Comparisons with dosimetric situations previously studied or measured showed the relevance of the modelings, and the possibilities of this computer tool are exposed through a series of examples and applications.

The characterization of the beta dose rate distribution in heterogeneous sediment is one of the most complex problems to be treated. In addition to a study aims at assessing the potential of DosiVox to consider these situations, the DosiSed software was developed specifically for modeling poly-mineral sets of grains. This tool was used to study the heterogeneity of the dose rate in real samples and allowed to include some of the results in a dating problematic.

Thays Desiree Mineli

Evaluation of the high sensitivity Brazilian quartz as personal dosimeter based on optically stimulated luminescence

April 2015

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Degree: Masters

Supervisors: Eduardo Tavares Costa; André Oliveira Sawakuchi

The use of ionizing radiation is becoming increasingly frequent, whether in industry or in the medical field. The harmful effects of ionizing radiation require security measures in its use. One of the most common ways to protect the health of the operator of ionizing radiation equipment is controlling the dose that persons can receive in a given time period. Personal dosimeters are used for this purpose. Protocols to estimate equivalent doses using optically stimulated luminescence (OSL) have been developed for determination of burial ages of sediments, firing of ceramics and personal dosimetry. Studies on dating of sediments in Brazil have found quartz types with very high luminescence sensitivity and excellent dosimetric characteristics. In order to analyze the dosimetric properties of bright Brazilian quartz and evaluate its potential use in personal dosimetry, comparative tests with the most used personal OSL dosimeter ($Al_2O_3 : C$), were made. This work presents the luminescence characteristics of quartz grains commonly found in fluvial (XNG47.2), eolian (TE65B) and coastal (PIN01) Brazilian samples. High sensitivity quartz and $Al_2O_3 : C$ were compared by dose recovery tests using a Single-Aliquot Regenerative dose protocol (SAR) and signal stability tests in samples exposed to beta (given doses: 50.00 mGy, 0.42 Gy, 35.70 Gy and 49.98 Gy) and gamma radiation (given doses: 0.14 mGy, 4.26 mGy, 8.53 mGy and 42.65 mGy). The luminescence measurements were carried out in Risø TL/OSL DA-20 readers using blue stimulation and light detection in the UV band. The studied quartz samples have average OSL sensitivities (beta radiation) ranging from 0.4% to 2.6% of the average sensitivity of the sample $Al_2O_3 : C$. For gamma radiation, quartz sample TE65B showed sensitivity higher (almost twice) than the sensitivity of the $Al_2O_3 : C$ sample. All the analyzed quartz samples showed measurable OSL signal for doses as low as 5 mGy, indicating that they can measure doses in the mGy range. All quartz samples showed OSL signal increasing linearly with dose up to 1 Gy. The studied quartz samples also were able to recover reliable doses using the OSL signal without correction by a test dose, pointing to a stable signal under thermal, optical and irradiation treatments. Equivalent doses calculated using the Central Age Model and without signal correction for a test dose deviated from 1% to 11% of the given dose. Equivalent doses calculated for the quartz samples have a higher accuracy than the equivalent doses calculated for the $Al_2O_3 : C$ sample. The quartz samples have saturation doses ($2D_0 = 114-175$ Gy) higher than the satu-

ration dose for the $Al_2O_3 : C$ sample ($2D_0 = 35$ Gy). The sensitivity of TE65B quartz sample in gamma radiation test was higher than the sensitivity of the $Al_2O_3 : C$ sample. The quartz samples showed insignificant fading in the period of about 1 month (recovered dose with difference less than 10% of given dose). These results indicate that the studied quartz samples are suitable to recover doses from 50 mGy to 50 Gy have potential as dosimeters and encourage further studies to evaluate the use of this type of quartz as sensor element in personal dosimeters.

Ana Luísa Rodrigues dos Santos

Geochemistry, mineralogy and luminescence studies of a pre-historic negative world - from Neolithic to Bronze Age in Alentejo region (Southern Portugal)

May 2015

Centro de Ciências e Tecnologias Nucleares, Instituto Superior Técnico, Universidade de Lisboa Portugal and Universidade de Aveiro, Aveiro, Portugal

Degree: Ph.D.

Supervisors: Fernando Joaquim Fernandes Tavares Rocha, Maria Isabel Marques Dias, Christopher Ian Burbidge

Archaeological research developed in recent years in South Portugal has revealed negative archaeological structures with original architectures and features, posing questions related to their chronology and fill dynamics. These structures (ditches, pits and hypogeum) have been excavated in carbonate-rich rocks and materials derived by weathering of granites, diorites and associated gabbros. They are related with Pre-Historic societies, from the Neolithic through the Bronze Age. In this work, different granulometric fractions of the fill materials of the negative structures and geological contexts have been studied by an innovative approach comprising: (i) chemical composition (neutron activation analysis and X-ray fluorescence), (ii) mineralogical composition (X-ray diffraction), (iii) thermo and optically stimulated luminescence profiling, and (iv) luminescence dating of fill materials.

Results found for granulometry, chemical and mineralogical composition, particularly of the clay fraction, enable differentiation of the paleoenvironments of the fill materials, as well as classification of the carbonate-rich materials: (i) phreatic calcretes, (ii) lacustrine calcretes (young/mature and poor/rich in Mg) and (iii) carbonate detrital formations. Luminescence together with geochemical and mineralogical results (complementing archaeological ones) contributed to the identification of different phases in the stratigraphic sequence, materials provenance and relation with the geological background, with particular emphasis in the role of trace elements, namely rare earth elements. Luminescence dating is often in accordance with archaeological interpretations of stratigraphy. Nevertheless calcite and/or the contribution of geological materials disturb the obtained age. In order to attenuate the effect of calcite, a new methodological approach

is proposed in this work for the dose rate estimation, allowing a re-interpretation of the obtained age.

Thus this work contributes to better establish behaviour of geological materials subjected to natural and anthropogenic weathering effects, in an interdisciplinary point of view, and better understanding the "negative Pre-Historical world" in the Alentejo region.

Kirk F. Townsend

A chronostratigraphic record of arroyo entrenchment and aggradation in Kanab Creek, southern Utah

September 2015

Utah State University, Logan, United States

Degree: Masters

Supervisor: Tammy Rittenour

Arroyos are entrenched channels characterized by near-vertical walls of alluvium and flat channel bottoms. Historic channel entrenchment in the southwest United States during the late AD 1800s and early 1900s has stimulated extensive research on these dynamic fluvial systems. The near-synchronous episodes of arroyo entrenchment and aggradation in Kanab Creek and other drainages in southern Utah during the last ~ 1 ka has led many researchers to argue that hydroclimatic forcings drive arroyo processes. These hypotheses remain largely untested, and there remains considerable uncertainty regarding the timing of these events and the specific mechanisms responsible for arroyo formation.

Previous work established an alluvial chronology for the Kanab Canyon reach of Kanab Creek, but it remained unclear if arroyo events in this reach were continuous with those downstream or synchronous with events in the disconnected arroyo in the upper basin. Using detailed sedimentologic and stratigraphic descriptions coupled with AMS radiocarbon and optically stimulated luminescence (OSL) dating, a new chronostratigraphic record of arroyo entrenchment and aggradation for Kanab Creek is produced in this study. Results suggest at least five periods of fluvial aggradation and episodic arroyo entrenchment during the middle- to late-Holocene, with aggradation occurring from ~ 6.2 to 3.6 ka (Qf1), ~ 3.2 to 2.5 ka (Qf2), ~ 2.2 to 1.5 ka (Qf3), ~ 1.4 to 0.8 ka (Qf4), and ~ 0.75 to 0.14 ka (Qf5). This record is compared to regional alluvial and paleoclimate records to explore potential allogenic and autogenic forcing mechanisms. Rapid transitions from exceptional drought to pluvial periods are quasi-synchronous with regional arroyo entrenchment over the last 1.5 ka, but the lack of clear correlations amongst the regional alluvial records and between paleoclimate records beyond 1.5 ka suggests that internal geomorphic thresholds are important controls on the timing of entrenchment in individual catchments.

Previous research on arroyo dynamics has largely focused on the timing of entrenchment. The few studies that have investigated the processes related to aggradation have used historic observations, and not the stratigraphic record of arroyo

deposits. In this study, the alluvial records from three reaches of Kanab Creek are combined to test models of the processes and geometric patterns of paleoarroyo aggradation. Results indicate that aggradation initially propagates upstream and then transitions to synchronous vertical aggradation along the entire channel profile as arroyos approach complete filling.

A PDF of this thesis can be downloaded from: <http://digitalcommons.usu.edu/etd/4492/>