



2<sup>nd</sup> International Congress

# CHEMISTRY FOR CULTURAL HERITAGE

9-12 July 2012, Istanbul / TURKEY



TURKISH  
CHEMICAL SOCIETY

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EUROPEAN ASSOCIATION FOR  
CHEMICAL AND MOLECULAR SCIENCES





**Organized by** CHEMISTRY FOR CULTURAL HERITAGE  
2<sup>nd</sup> International Congress  
July 9-12, 2012 Istanbul / TURKEY

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## SCIENTIFIC PROGRAMME

### July 9th (Monday)

<b>08.00 – 10.00</b>	Registration
<b>10.00 – 10.30</b>	<b>Welcome Address</b>
<b>10.30 – 11.30</b>	<b>Plenary Lecture</b> Yenikapı shipwreck and Their Contribution to the History of Ship Construction Technology <i>Cemal Pulak (Texas A&amp;M University)</i>
<b>11.30 – 12.00</b>	<b>Coffee Break</b>
<b>12.00 – 13.00</b>	<b>Oral Session 1: Application of instrumental techniques in issues of cultural heritage</b> <i>Chair person: Gregory Smith (Indianapolis Museum of Art)</i>
12.00 - 12.15	<b>OP.01.01 - The effect of lead white in the stability of oil paint films</b> <i>Mette R. Sørensen, Demian R. Sanpedro, Dolores J. Yusá Marco, Laura Fuster López, Kim P. Simonsen, Mikkel Scharff, Marion F. Mecklenburg</i>
12.15 - 12.30	<b>OP.01.02 - Usability of r-FTIR-, r-UV/Vis- and XRF-spectroscopy for non-destructive characterization of materials on watercolor paintings, graphic objects and oil paintings</b> <i>Wilfried Vetter, Manfred Schreiner</i>
12:30 - 12.45	<b>OP.01.03 - Evaluation of the effect of different paint cross section preparation methods on the performances of fourier transformed infrared microscopy in total reflection mode</b> <i>Silvia Prati, Francesca Rosi, Giorgia Sciotto, Emilio Catelli, Costanza Miliiani, Brunetto Brunetti, Rocco Mazzeo</i>
12.45 - 13.00	<b>OP.01.04 - An advanced multivariate approach for processing XRF spectral and hyperspectral data from non-invasive in situ analyses on painted surfaces</b> <i>Giorgia Sciotto, Paolo Oliveri, Silvia Prati, Marta Quaranta, Silvia Bersani, Rocco Mazzeo</i>
<b>13.00 – 14.00</b>	<b>LUNCH</b>
<b>14.00 – 15.30</b>	<b>Oral Session 2: Application of instrumental techniques in issues of cultural heritage</b> <i>Chair person: Kim Simonsen (Royal Academy of Fine Arts School of Conservation, Copenhagen)</i>
14.00 - 14.15	<b>OP.02.01 - Study of the alteration processes of Prussian blue in laboratory-prepared and genuine paint layers</b> <i>Louise Samain, Jana Sanyova, David Strivay</i>

## July 9th (Monday)

14.15 - 14.30	<b>OP.02.02 - Analysis of tyrian purple in samples from the eastern mediterranean basin: an HPLC study for the assessment of the purple biological source</b> <i>Ioannis Karapanagiotis, Dimitrios Mantzouris</i>
14:30 - 14.45	<b>OP.02.03 - Characterization Of The Objects In Historical Textiles In Favour Of Restoration By Multi-Analytical Techniques</b> <i>Nilgun Kahraman, Emine Torgan, R.gurcan Oraltay, Recep Karadağ</i>
14.45 - 15.00	<b>OP.02.04 - In-situ, non-invasive, unilateral NMR investigations of lead carboxylate formation in traditional oil paints</b> <i>Eleonora Del Federico, Silvia Centeno, Cindie Kehlet, Amelia Catalano, Boris Itin, Alexej Jerschow</i>
15.00 - 15.15	<b>OP.02.05 - Vibrational study of bone black pigments</b> <i>Alessia Daveri, Francesca Rosi, Riccardo Vivani, Brunetto Giovanni Brunetti, Costanza Miliani</i>
15.15 - 15.30	<b>OP.02.06 - Trends in nanotechnology-based analytical methods in cultural heritage</b> <i>Ludmila Otilia Cinteza, Ana Emandi</i>
<b>15.30 – 15.45</b>	<b>Coffee Break</b>
<b>15.45 – 17.30</b>	<b>Oral Session3: Application of instrumental techniques in issues of cultural heritage</b> <b>Chair person: Manfred Schreiner (Akademie der bildenden Künste, Wien)</b>
15:45 - 16.00	<b>OP.03.01 - Surface characterization of 19th century and modern daguerreotypes using high-resolution field emission scanning electron microscopy (HR FE SEM)</b> <i>Patrick Ravines, Anne West, John Minter, Robledo O Gutierrez, Rob Mcelroy, Peter Bush</i>
16.00 - 16.15	<b>OP. 03.02 - EBSD &amp; EDS to characterize the surface of 19th c and modern daguerreotypes</b> <i>Patrick Ravines, Lisa H Chan, Matt Nowell, Robledo Mcelroy</i>
16.15 - 16.30	<b>OP.03.03 - Deterioration of the Daguerreotype Image by Environmental Pollutants</b> <i>Robyn E Hodgkins, Silvia A Centeno, Alejandro G Schrott</i>
16.30 - 16.45	<b>OP.03.04 - Identifying varnish compositions of historic tintypes: recipes and reality</b> <i>Corina Rogge</i>
16.45- 17.00	<b>OP.03.05 - Analyses of paint layers on tinned and leaded steel. Investigation of coat of arms of the Order of the Elephant from the Royal Collection Knights' Shields in Denmark</b> <i>Clara Bratt Lauridsen, Kim Pilkjær Simonsen, Jana Sanyova</i>

## July 9th (Monday)

17.00 - 17.15	<b>OP.03.06 - Characterization of iron slag and iron ore used in the ancient iron smelting work in Manipur, India by XRF, XRD, EDX, TL and FTIR</b> <i>Md Raheijuddin Sheikh</i>
17.15 - 17.30	<b>OP.03.07 - Plant gums used as binding media: analytical problems, databases, occurrence and physicochemical evolution</b> <i>Anna Lluveras Tenorio, Celia Duce, Maria Rosaria Tinè, Maria Perla Colombini, Ilaria Bonaduce</i>
<b>17.30 – 19.00</b>	<b>Poster Session: Application of instrumental techniques in issues of cultural heritage</b>
PS1-01	<b>Criteria and indicator risk assessment: A European overview</b> <i>R. Mazzeo, M. Quaranta, S. Prati</i>
PS1-02	<b>Synthesis and Spectroscopic Characterization of an Old Pigment</b> <i>Pınar Çakar, Ayşegül Belet</i>
PS1-03	<b>Identification of inks used in 15th century Ottoman manuscript by non-destructive techniques</b> <i>Ayşegül Belet, Pınar Çakar</i>
PS1-04	<b>The contribution of micro-Raman and FTIR spectroscopy in art history study of wall painting from the 16th century church in Republic of Macedonia</b> <i>Lidija Robeva Cukovska, Jehona Spahiu, Biljana Minceva Sukarova</i>
PS1-05	<b>The characterisation of ochres used in Australian Aboriginal paintings</b> <i>Barbara Stuart, Paul Thomas, Alexandria Hunt</i>
PS1-06	<b>A study on the polychromy and technique on some Armenian illuminated manuscripts by Raman microscopy</b> <i>Yeghis Keheyan, Pietro Baraldi, Gayane Eliazian, Yeghis Keheyan</i>
PS1-07	<b>A Study of Chrysographic Lines in a few Surviving Folios of the Quran</b> <i>Faranak Bahrololoumi, Roya Bahadori, Nader Motalebi Kashani</i>
PS1-08	<b>An in-situ, non-invasive XRF and Reflectance FTIR investigation of the wall paintings and mosaics at the house of Neptune and Amphitrite, Herculeaneum</b> <i>Katelin Fallon, Eleonora Del Federico, Dimitri Hazzikostas, Cindie Kehlet, Hiba Schahbaz, Amelia Catalano, Katerina Posch</i>
PS1-09	<b>An HPLC-DAD method for dyestuff identification. Application on textiles excavated from Faiyoum, Egypt</b> <i>Ioannis Karapanagiotis, Paraskevi Kamaterou, Omar Abdel Kareem</i>
PS1-10	<b>Identification of Materials and Pigments Used in a Mural Painting in a Historical House in Yazd</b> <i>Roya Bahadori, Mona Montazeri Hadesi, Somaye Jahangiri</i>
PS1-11	<b>Scientific Examination of Two Iranian Historical Paintings on Wood</b> <i>Roya Bahadori, Seyed Hamid Ahmadi</i>
PS1-12	<b>Raman spectroscopy and Py-GC/MS applied for the identification and characterization of synthetic organic pigments</b> <i>Marta Anghelone, Dubravka Jembrih Simbürger, Valentina Pintus, Manfred Schreiner</i>
PS1-13	<b>HPLC-DAD Natural Dye Analysis on Carpets from National Palaces Museum (Yıldız Palace-Chalet Kiosk) Istanbul-Turkey</b> <i>Hülya Keser, Türkan Yurdun</i>

## July 9th (Monday)

PS1-14	<b>Identification of Natural Dyes in Historical Writings by High-Performance Liquid Chromatography with Diode-Array Detection</b> <i>Türkan Yurdun</i>
PS1-15	<b>The painting technique of Macedonian painters: Py/GC/MS and GC/MS based techniques for the analysis of organic materials used by Dicho Zograph workshop</b> <i>Anna Lluveras Tenorio, Lidija Robeva Chukovska, Alessia Andretti, Maria Perla Colombini, Biljana Minceva Sukarova</i>
PS1-16	<b>An Application of FTIR to Aging Process Assessment of the Beeswax-Treated Paper Heritage in Korea</b> <i>Choi Kyoung Hwa, Kang Yeong Seok, Cho Jung Hye, Yang Eun Jung, Jeong Hye Young</i>
PS1-17	<b>Characterization of archaeological glass beads in Thailand: Hor-Ek prehistorical site</b> <i>Krit Won In, Sanitpong Khunsong, Teerasak Kamwanna, Sawet Intarasiri, Pisutti Dararutana</i>
PS1-18	<b>Characterization of 19th century Daguerreotypes by nondestructive analytical techniques</b> <i>Marília Peres, Luís Dias, José Mirão, Anabela Gomes, Estela Jardim, Fernanda Costa, Teresa Ferreira</i>
PS1-19	<b>Material study on a Liturgical Cope from the 16th century belonging to D. Teotónio of Braganza, a Portuguese Archbishop</b> <i>H. Moreiras, A. Manhita, C. Frade, E. Lopes, P. Tomas, J. Mirao, C. Dias, P. S. Rodrigues, A. T. Caldeira, T. Ferreira, P. S. Rodrigues</i>
PS1-20	<b>XRF-analysis of Glagolitic manuscripts</b> <i>Gunn Pöllnitz, Manfred Schreiner, Heinz Miklas, Melanie Gau</i>
PS1-21	<b>The Évora Inquisition Banner – Historical and Material Characterization</b> <i>L. Tobias, L. Dias, H. Mestre, A. Conde, P. Monteiro, A. Candeias, C. Dias, A. Caldeira, T. Ferreira</i>
PS1-22	<b>Study of natural organic colourants in the Andean textile collection of the British Museum</b> <i>Catherine Higgitt, Thibaut Devière, Colin Mc Ewan, Helen Wolfe, Ana Roquero, Jenny Figari</i>
PS1-23	<b>The influence of the SERS substrate on the sensitive identification of fresh and old varnishes using Raman spectroscopy</b> <i>Ioana Catalina Vacaresteanu, Ana Emandi, Sorana Ionescu, Ludmila Otilia Cinteza</i>
PS1-24	<b>Microanalysis of Persian coin found in Thailand</b> <i>Krit Won In, Wanwisa Dhanmanonda, Teerasak Kamwanna, Saweat Intarasiri, Pisutti Dararutana</i>
PS1-25	<b>Statistical methodology in numismatic studies - the case of Dacian gold and silver coins</b> <i>Catalina Chiojdeanu, Bogdan Constantinescu, Ernest Oberländer Tarnoveanu</i>
PS1-26	<b>Chemical Characterization of Bronze Samples using Elements determined by Inductively Coupled Plasma-mass Spectrometry</b> <i>Chunyan Zhao</i>
PS1-27	<b>XRF Analysis of Nine Renaissance Nuremberg Trombones</b> <i>Bernadette Frühmann, Hannes Vereecke, Manfred Schreiner, Wilfried Kausel</i>
PS1-28	<b>Interdisciplinary Studies for Investigations of the Historical Objects</b> <i>Ugur Genc, Şinasi Ekinci, Asiye Başsarı, Mehmet Görgülü, Fatih Tepgeç, Recep Karadağ, Emine Torgan, Koray Dağlı</i>
<b>19.00</b>	<b>Welcome Reception</b>

## July 10th (Tuesday)

<b>08.00 – 09.00</b>	Registration
<b>09.00 – 10.00</b>	<b>Plenary Lecture</b> The Latest Link in the Long Tradition of Maritime Archaeology in Turkey: The Yenikapı Shipwrecks <i>Ufuk Kocabaş (Istanbul University)</i>
<b>10.00 – 11.30</b>	<b>Oral Session 4: Transformation on heritage surface due to environmental factors</b> <i>Chair person: Emine Caner (Middle East Technical University)</i>
10.00 - 10.15	<b>OP.04.01 - Dolomitic lime mortars: carbonation complications and susceptibility to acidic sulfates</b> <i>Heather Hartshorn, Norman Weiss, George Wheeler</i>
10.15 - 10.30	<b>OP. 04.02 - Non-destructive diagnostic and monitoring studies on cultural heritage – in-situ examination of state of deterioration</b> <i>Ayşe Tavukcuoğlu, Emine Nevin Caner Saltık</i>
10:30 - 10.45	<b>OP. 04.03 - Examination Of Deterioration In Nemrut Dağ Limestones And Development Of Their Conservation Treatments With Nanodispersive Ca(OH)<sub>2</sub> Solutions</b> <i>Evin Caner, Emine Caner Saltık</i>
10.45 - 11.00	<b>OP.04.04 - The relevance of spectroscopic imaging techniques on test drillings to assess the physicochemical damage of building materials</b> <i>Nagore Prieto Taboada, Olivia Gómez Laserna, Alfredo Sarmiento, Irantzu Martínez Arkarazo, María Ángeles Olazabal, Juan Manuel Madariaga</i>
11.00 - 11.15	<b>OP.04.05 - Positive and Negative Results of the Chemicals Used in the Restoration of the Fatih Mosque and Sultan I. Mahmut Library between 2007-2012</b> <i>Gönül Cantay, Tuğba Keleş, Gülşah Altun</i>
11.15 - 11.30	<b>OP.04.06- Origin and development of blue-and-white porcelain in ancient china</b> <i>Weidong Li, Hongjie Luo, Xiaoke Lu, Lanhua Liu, Xinmin Sun</i>
<b>11.30 – 12.00</b>	<b>Coffee Break</b>
<b>12.00 – 13.00</b>	<b>Oral Session 5: Transformation on heritage surface due to environmental factors</b> <i>Chair person: Annemie Adriaens (University of Gent, Belgium)</i>
12.00 - 12.15	<b>OP.05.01 - Voltammetry of Microparticles for the characterization of copper and bronze corrosion products on Roman artefacts</b> <i>Alice Elia, Annemie Adriaens, Mark Dowsett</i>
12.15 - 12.30	<b>OP.05.02 - Analysis of silver content and trace metals in medieval coins from Sweden, using a <math>\mu</math>-spot X-ray fluorescence instrument (<math>\mu</math>XRF), and comparing results to other techniques</b> <i>Kaj Fredrik Thuresson, Cecilia Von Heijne</i>

## July 10th (Tuesday)

12:30 - 12.45	<b>OP.05.03 - Micro X-ray fluorescence analysis (<math>\mu</math>XRF) of silver denarii from medieval Poland</b> <i>Julio M Del Hoyo Meléndez, Lukasz Bratasz, Marta Wlodarczak, Anna Klisinska Kopacz</i>
12.45 - 13.00	<b>OP.05.04 - Towards standard practices in outdoor bronze coating testing: characterisation of bronze patina growth in coastal environment</b> <i>Paola Letardi, Giorgia Ghiara, Simona Scrivano, Stavroula Golfomitsou</i>
<b>13.00 – 14.00</b>	<b>LUNCH</b>
<b>14.00 – 19.00</b>	<b>City Tour Visit to Yenikapı Excavation</b>
<b>19.00</b>	<b>Social Dinner</b>

## July 11th (Wednesday)

<b>08.00 – 09.00</b>	Registration
<b>09.00 – 10.00</b>	<b>Plenary Lecture:</b> The coordinated use of synchrotron spectroelectrochemistry for corrosion studies on heritage metals <i>Annemie Adriaens (University of Gent, Belgium)</i>
<b>10.00 – 11.30</b>	<b>Oral Session 6: Application of instrumental techniques in issues of cultural heritage</b> <i>Chair person: Rocco Mazzeo (University of Bologna)</i>
10.00 - 10.15	<b>OP.06.01 - Laboratory Induced Biodeterioration of Stained Glass by Fungi</b> <i>Alexandra Rodrigues, Márcia Vilarigues, Maria Filomena Macedo</i>
10.15 - 10.30	<b>OP.06.02 - Nature's first green is gold: analysis of a lost Frank Lloyd Wright wisteria mosaic</b> <i>Corina Rogge, Patrick Ravines, Jonathan Thornton, Peter Bush</i>
10:30 - 10.45	<b>OP.06.03 - Preliminary Archaeometrical Studies of Glass Finds From Istanbul Yenikapi Marmaray and Metro Excavation</b> <i>Ali Akin Akyol, Yusuf Kagan Kadioglu</i>
10.45 - 11.00	<b>OP.06.04 - Study of 18th century glass mosaics: The reds and carnagionni of Alessio Mattioli</b> <i>Sara Canaveira, Vânia S. F. Muralha, Susana Coentro, Carlo Stefano Salerno, Teresa Morna, António Candeias, Luis Mirão</i>
11.00 - 11.15	<b>OP.06.05 - <math>\mu</math>-Raman analysis of Hispano-Moresque historical tiles</b> <i>Susana Coentro, Luís Cerqueira, Rui Silva, Solange Muralha</i>
11.15 - 11.30	<b>OP.06.06 - Luminescent ionic liquids for glass cleaning: restoring Stained Glass transparency</b> <i>Joana Delgado, Márcia Vilarigues, César A.t. Laia, Luís Branco</i>
<b>11.30 – 12.00</b>	<b>Coffee Break</b>
<b>12.00 – 13.00</b>	<b>Oral Session 7: Application of instrumental techniques in issues of cultural heritage</b> <i>Chair person: Ufuk Kocabaş (Istanbul University)</i>
12.00 - 12.15	<b>OP.07.01 - Analysis of Wax Paint and Fatty Acid Soaps by Pyrolysis Gas Chromatography</b> <i>Joy Mazurek, Michael Schilling</i>
12.15 - 12.30	<b>OP.07.02 - Identification and Pathology of Lapis Lazuli Pigment in Illustrated Books by PIXE</b> <i>Masoud Bagherzadeh Kasiri, Maryam Sharafi</i>
12:30 - 12.45	<b>OP.07.03 - Opal: Australia's National Gem Stone - Materials Chemistry and Conservation</b> <i>Paul Stephen Thomas</i>
12.45 - 13.00	<b>OP.07.04 - Chemical Characterisation of Waterlogged Woods in Yenikapi 12 by FT-IR Analyzes</b> <i>Öznur Özden, Namık Kılıç, H. Işıl Özsait-Kocabaş, Aslı Gökçe Kılıç</i>

## July 11th (Wednesday)

<b>13.00 – 14.00</b>	<b>LUNCH</b>
<b>14.00 – 15.30</b>	<b>Oral Session 8: Development and evaluation of cleaning and conservation of materials</b> <i>Chair person: Elizabeth E. Peacock (University of Gothenburg, Sweden)</i>
14.00 - 14.15	<b>OP.08.01 - Thermal property dependence of paraloid B-72 cast films on solvent choice</b> <i>Gregory Dale Smith, Daniella Ash, Anne Getts</i>
14.15 - 14.30	<b>OP.08.02 - The use of nitroxides in the control of biofilm formation on cultural materials</b> <i>Stefanie Ann Alexander, Robyn Sloggett, Carl H. Schiesser</i>
14:30 - 14.45	<b>OP.08.03 - Complementary Analysis of Proteins in the 14th-century Double-Sided Processional Banner, Saint Mary Magdalen Holding a Crucifix; (reverse) The Flagellation, by Spinello Aretino</b> <i>Hae Young Lee, Sarah Kleiner, Julie Arslanoglu</i>
14.45 - 15.00	<b>OP.08.04 - Identification of animal adhesives using DNA amplification</b> <i>Anne Marie Eriksen, Hans Viborg Kristensen, Peder Bøllingtoft, Knud Bo Botfeldt, Arne Redsted Rasmussen</i>
15.00 - 15.15	<b>OP.08.05 - Silane based products for tile protection</b> <i>Teresa Pinto Santos, Amir Zomorodian, Maria Fátima Montemor, Maria Fátima Vaz, Ana Paula Carvalho</i>
15.15 - 15.30	<b>OP.08.06 - In situ investigation of metal protective coating films with mid-infrared spectroscopy</b> <i>Stamatis C Boyatzis, Vassilike Argyropoulos, Antonios M Douvas, Galateia Kriezis, Amalia Siatou</i>
<b>15.30 – 16.00</b>	<b>Coffee Break</b>
<b>16.00 – 18.00</b>	<b>Oral Session 9: Development and evaluation of cleaning and conservation of materials</b> <i>Chair person: Hadi Özbal (Boğaziçi University)</i>
16.00 - 16.15	<b>OP.09.01 - Deposition of a lead dodecanoate coating on a lead or lead alloy metal surface</b> <i>Michel De Keersmaecker, Karolien De Wael, Annemie Adriaens</i>
16.15 - 16.30	<b>OP.09.02 - Modern repairs of Chinese bronzes: study of materials, restoration techniques and state of conservation</b> <i>Johanna Muller, Kwang Tzuu Chen</i>
16:30 - 16.45	<b>OP.09.03 - Preparation of Pozzolanic Lime Mortars for Repairing Limestones of Nemrut Dağ Monument</b> <i>Bilge Alp Güney, Emine Nevin Caner Saltık</i>
16.45 - 17.00	<b>OP.09.04 - The development of organic consolidants for heritage Sydney sandstones</b> <i>Tamae Fukumoto, Paul Thomas, Barbara Stuart</i>

## July 11th (Wednesday)

17.00 - 17.15	<b>OP.09.05 - Microstructural and mechanical comparison of different nanostructured lime-based consolidant products applied on renders</b> <i>Giovanni Borsoi, Maria Do Rosário Veiga, António Santos Silva</i>
17.15 - 17.30	<b>OP.09.06 - Study of the superficial cohesion and chromatic changes of marbles treated with polymers and exposed to artificial ageing</b> <i>Mara Camaiti, Halit Canol</i>
<b>17.30 – 19.00</b>	<b>Poster Session: Transformation on heritage surface due to environmental factors</b>
PS2-01	<b>Optical sensor for the formic acid detection prepared through the immobilization of colorimetric dyes in a polymeric matrix assembled by the layer-by-layer method</b> <i>Inês Coutinho, Márcia Ventura, Márcia Vilarigues, A. Jorge Parola</i>
PS2-02	<b>The review of Historical and Technical studies of ancient bronzes</b> <i>Soodabeh Yousefnejad</i>
PS2-03	<b>Infrared reflection absorption (IRRAS) and Raman spectroscopy to study early stages of atmospheric silver corrosion</b> <i>Rita Wiesinger, Irene Martina, Manfred Schreiner</i>
PS2-04	<b>Chemical simulation of a decaying process observed in weathering steel sculptures exposed to an urban atmosphere</b> <i>Julene Aramendia, Leticia Gomez Nubla, Kepa Castro, Maite Maguregui, Juan Manuel Madariaga</i>
PS2-05	<b>Microbiodeterioration study of Anselmo Pineda collection saved by National Library of Colombia: Evaluation and Selection of Chemical Control Products</b> <i>Maria Camila Patiño, Aydee Camila Vargas, Ada Luz Manrique, María Ximena Rodríguez, Sandra Angulo, Jose Fernando Mikan, Luz Stella Villalba</i>
PS2-06	<b>Investigation on some antioxidants for ink damaged paper</b> <i>Olga Darcanova, Aldona Beganskiene, Aivaras Kareiva</i>
PS2-07	<b>Effects of atmospheric pollutants and meteorological factors on a column from a 19th century building (Palma, Spain)</b> <i>Catalina Genestar, Carmen Pons, Rafael Forteza</i>
PS2-08	<b>Stone weathering studies on the Portal des Mirador from the Cathedral of Palma</b> <i>Catalina Genestar, Francisca Alba, Victor Cerdà</i>
PS2-09	<b>Portable non-destructive analytical techniques to diagnose the impacts of modern urban atmospheres and other environmental factors on archaeological sites: The case of Insula IX, 3 (Pompeii, Italy)</b> <i>Maite Maguregui, Anastasia Giakoumaki, Ulla Knuutinen, Kepa Castro, Irantzu Martínez Arkarazo, Juan Manuel Madariaga</i>
PS2-10	<b>Combined use of SEM-EDS, micro-XRF and Raman spectroscopies to evaluate the composition of black and salt crusts on building stone surfaces formed by natural and anthropogenic sources</b> <i>Estefanía Calparsoro, Anastasia Giakoumaki, Maite Maguregui, Nagore Prieto Taboada, Juan Manuel Madariaga</i>
PS2-11	<b>Manufacturing techniques of Porcelain Bowls from Xing Kiln in 6th-7th centuries A.D.in China</b> <i>Jiming Xu, Weidong Li</i>

## July 11th (Wednesday)

PS2-12	<p><b>Meybod Ziluo Museum: Preventive Conservation plan, Microclimate and Indoor Climate Control</b>  <i>Faeze Sadat Asadi Firuoazabadi, Mohammad Taghi Ashoori, Farnak Bahrololumi, Atefeh Sadat Asadi Firuoazabadi, Parichehr Moradi</i></p>
PS2-13	<p><b>Exhibition Method And Environment Condition Proposals For Yenikapı Shipwrecks</b>  <i>Aslı Gökçe Gökçay</i></p>
<p><b>Poster Session: Development and evaluation of cleaning and conservation of materials</b></p>	
PS2-14	<p><b>Evaluation of the effectiveness of laser removal of graffiti on granite by means of ftir</b>  <i>Jose Santiago Pozo Antonio, Teresa Rivas Brea, Ana Jesús López Diaz, Alberto Ramil</i></p>
PS2-15	<p><b>Novel material for stone protection with water-repellent and antibacterial properties</b>  <i>Georgian Nedelcu, Teodora Miclaus, Maria Marinescu, Mariana Pruna, Ludmila Otilia Cinteza</i></p>
PS2-16	<p><b>Study of new pigment products for filling the red marble in the conservation works</b>  <i>Halit Canol, Ozan Deveoglu</i></p>
PS2-17	<p><b>A multi-analytical investigation on samples from the Macedonian Tomb "Makridi", Thessaloniki, Greece</b>  <i>Svetlana Vivdenko, Fani Athanasiou, Ioannis Karapanagiotis, Panagiotis Manoudis, Dimitrios Lampakis, Haralambos Tsagalides</i></p>
PS2-18	<p><b>Novel Fully Conjugated 2H- and Metal- Phthalocyanine Network Polymers: Synthesis, Characterization and Dielectric Spectra Analysis</b>  <i>Hamada Abdel Razik, Khaled Mahmoud</i></p>
PS2-19	<p><b>Improving paper mechanical properties and printing quality by using carboxymethyl cellulose as a strength agent</b>  <i>Asmaa Mohamed El Shafey, George Nubar Simonian, Fouad Taha Abd El Halim, Fathey Fahim Abd El Latif, Abeer Mohamed Adel</i></p>
PS2-20	<p><b>Disinfection of cultural property made of cellulose materials by herbs: Old materials, new technologies</b>  <i>Narges Pedram, Shahmira Rahimi</i></p>
PS2-21	<p><b>Evaluating the Application of Natural Pesticides on Korean Traditional Paper</b>  <i>Young Hee Kim, Chang Wook Jo, Jin Young Hong, Soo Ji Kim, Jeung Min Lee, Jung Eun Choi</i></p>
PS2-22	<p><b>Sol-gel Method for the Conservation of Copper</b>  <i>Erika Borovikovaite, Janina Lukseniene, Algirdas Selskis, Jurate Senvaitiene, Rimantas Ramanauskas, Aivaras Kareiva</i></p>
PS2-23	<p><b>Physico-chemical characterizations of nickel substituted calcium hydroxyapatites</b>  <i>Badraoui Bechir, Sendi Nesrine, Elaloui Elimame</i></p>
PS2-24	<p><b>Soltanieh dome tiles: scientific investigation and diagnostic for conservation</b>  <i>Parichehr Moradi, Ali Reza Razeghi, Farnak Bahrololumi, Faeze Alsadat Asadi</i></p>
PS2-25	<p><b>Two common adhesives in restoration of potteries (Researching and comparing)</b>  <i>Roxana Minoueipour</i></p>

## July 11th (Wednesday)

PS2-26	<b>Characterization of morphological and chemical changes at micro- and nano-scale in contemporary paintings treated with biocides</b> <i>Annette Suleika Ortiz Miranda, Maria Teresa Doménech Carbó, Antonio Doménech</i>
PS2-27	<b>Application of surface analysis methods in historical artifacts</b> <i>Soodabeh Yousefnejad</i>
PS2-28	<b>Occupational Safety Of Chemicals Used In Restoration Of Cultural Heritage Materials</b> <i>Hazal Özlem Ersan</i>
<b>19.00</b>	<b>Gala Dinner (Bosphorus Tour by Boat)</b>

## July 12th (Thursday)

<b>08.00 – 09.00</b>	Registration
<b>09.00 – 10.00</b>	<b>Plenary Lecture:</b> TScience-based Doctoral Degrees in Conservation: Room for Conservators and Science Graduates Alike <i>Elizabeth E Peacock (University of Gothenburg, Sweden)</i>
<b>10.00 – 11.30</b>	<b>Oral Session 10: Application of instrumental techniques in issues of cultural heritage</b> <b>Chair person:</b> <i>Fusun Okyar (TeneArt, İstanbul)</i>
10.00 - 10.15	<b>OP.10.01 - MOVIDA: A new tool for on-the-spot data documentation and analysis for Cultural Heritage investigations</b> <i>Anna Amat, Costanza Miliani, Brunetto Giovanni Brunetti</i>
10.15 - 10.30	<b>OP.10.02 - The Books of Hours in the collection of the National Palace of Mafra: Study of materials and Conservation</b> <i>Ana Rita Araújo, Maria João Melo, Vânia S. F. Muralha, Conceição Casanova, Ana Lemos, Marcello Picollo</i>
10:30 - 10.45	<b>OP.10.03 - Spectroscopic characterization of natural dyes for their non-invasive identification on Mesoamerican Codices</b> <i>David Buti, Costanza Miliani, Antonio Sgamellotti, Brunetto Giovanni Brunetti, C. Lofrumento</i>
10.45 - 11.00	<b>OP.10.04 - Enhancement of Manuscript Paper Materials: Restoration and Physico-Chemical Study</b> <i>Latifa Hajji, Abdellatif Boukir, Ghizlane Serhrouchni Idrissi, Jamal Assouik, Hamid Lakhari, Abdelali Kerbal</i>
11.00 - 11.15	<b>OP.10.05 - Water Adsorption Isotherms and Dimensional Stability for Ancient Ivory</b> <i>Gordon Turner Walker</i>
11.15 - 11.30	<b>OP.10.06 - A restoration methodology of Antique Maps</b> <i>Fatma Banu Çakan, Ayşe Betül Oral</i>
<b>11.30 – 12.00</b>	<b>Coffee Break</b>
<b>12.00 – 13.00</b>	<b>Oral Session 11: Application of instrumental techniques in issues of cultural heritage</b> <b>Chair person:</b> <i>Hadi Özbal (Boğaziçi University)</i>
12.00 - 12.15	<b>OP.11.01 - Morphological Study of Paper Surface by SEM-EDS of the Degraded and Restored Paper Manuscript from Library Al-quarawiyine</b> <i>Latifa Hajji, Jamal Assouik, Nouredine Idrissi Kandri, Abdellatif Boukir, Rachid Benslimane</i>
12.15 - 12.30	<b>OP.11.02 - Structural Analysis by Spectroscopy FTIR and XRD of Manuscript Paper Materials dating from 150, 200 and 800 years</b> <i>Latifa Hajji, Abdellatif Boukir, Ahmed El Ghazouali, Abdelaziz Zerouale</i>
12:30 - 12.45	<b>OP.11.03 - Fungal deterioration of artificially aged cotton</b> <i>Katja Kavkler, Nina Gunde Cimerman, Polona Zalar, Andrej Demšar</i>
12.45 - 13.00	<b>OP.11.04 - Transformations of heritage surfaces due to environmental factors</b> <i>Shobhakar Adhikari</i>
<b>13.00 – 14.00</b>	<b>LUNCH</b>
<b>14.00 – 15.30</b>	<b>Closing Ceremony</b>



2<sup>nd</sup> International Congress  
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July 9-12, 2012 Istanbul / TURKEY

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## PLENARY LECTURERS

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PLENARY LECTURES

## **Yenikapı shipwreck and Their Contribution to the History of Ship Construction Technology**

**Cemal Pulak**

*Assoc. Prof. Texas A&M University*

Since 2004, a major construction program to expand the city's public transportation system in Istanbul, Turkey, has led to the discovery of the silted Byzantine Theodosian harbor at Yenikapı, which served ancient Constantinople as a major point of access, mainly from the 4th to the 11th centuries AD. The 36 well-preserved shipwrecks revealed in these excavations represent the largest and the best preserved assemblage of early medieval vessels ever found in the Mediterranean. During the period to which these vessels date, ship construction underwent a gradual but highly significant shift from shell-based, plank-first construction, with the ships' frames added only afterwards for reinforcing the planking, to the modern, skeleton-based method with pre-erected frames determining the vessels' shape. As the Yenikapı vessels represent a wide range of ship types from small fishing boats to light galleys, their materials and designs promise to dramatically increase our understanding of maritime trade, technology, and naval warfare in this period, as well as our knowledge of the economic life and Constantinople's seaborne local and long-distance contacts. These water-logged vessels require extensive post-excavation documentation and study, after which the ships will undergo a multi-year conservation process before being permanently exhibited at a new, purpose-built museum.

## PLENARY LECTURES

## The Latest Link in the Long Tradition of Maritime Archaeology in Turkey: The Yenikapı Shipwrecks

**Prof. Dr. Ufuk Kocabaş**

*Istanbul University, Turkey*

During the construction of the Marmaray railway and metro stations in Istanbul-Yenikapı between 2004 and 2012, no fewer than thirty-six shipwrecks, dating from the Middle Byzantine period to about the fifth to tenth centuries AD, were revealed. The on going archaeological excavations have confirmed that Constantinople's main harbour, Portus Theodosiacus, was once situated in this former natural bay, now silted by the ancient Lykos (Bayrampaşa) river and lying about 300 m from today's shoreline. The harbour is known to have been built in the late fourth century during the reign of Theodosius I (AD 376– 395), in response to the demands of the growing economy and population of the capital city of the Byzantine Empire. According to historical records, the breakwater of the Theodosian harbour stretched from the Davutpasa pier on the west, first eastward and then northeast, thus protecting the bay from the prevailing southwesternly winds. Excavations by the Istanbul Archaeological Museums revealed the presence of a breakwater made of limestone blocks exposed at the west end of the construction site. The artefact inventory of the site is also quite rich. Different types of amphorae and table wares, coins, candles, figurines, leather sandals, ornaments, a large number of nautical artefacts – including rigging equipment such as pulleys, ropes, toggles, and also stone and iron anchors – and also well-preserved shipwrecks have been unearthed by the museum's on going archaeological salvage project. The ships from the Theodosian harbour display a moment frozen in time and have made tremendous contributions to information on shipbuilding technology and development during Antiquity and the Middle Ages. The Istanbul Archaeological Museums turned to the Istanbul University's Department of Conservation of Marine Archaeological Objects to deal with most of the shipwrecks. Department President and project director Professor Ufuk Kocabaş and a hard-working team of Department assistants, full-time specialists, and Istanbul University graduate students have been working for over 5 years in the active construction site in tent-covered pits to document and carefully recover the shipwrecks. Undoubtedly, the shipwrecks constitute the most remarkable artefact group, especially for nautical archaeologists. The thirty-six ships can be divided into three groups: long warships (galleys); sea-going traders; and small, local trading vessels.



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**ORAL PRESENTATION**

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## The effect of lead white in the stability of oil paint films

**Mette R. Sørensen<sup>1</sup>, Demian R. Sanpedro<sup>2</sup>, Dolores J. Yusá Marco<sup>2</sup>, Laura Fuster López<sup>2</sup>, Kim P. Simonsen<sup>1</sup>, Mikkel Scharff<sup>1</sup>, Marion F. Mecklenburg<sup>3</sup>**

*<sup>1</sup>School of conservation, copenhagen*

*<sup>2</sup>Instituto Universitario de restauracion del patrimonio, universidad politécnica de valencia*

*<sup>3</sup>Museum conservation institute, smithsonian institutio*

The cleaning of paintings has been a subject of much discussion for a long time. Organic solvents are still widely used by conservators both in surface cleaning treatments as well as varnish removal. In the last years there has been an increasing interest in understanding the eventual changes in the mechanical behavior of painted structures after being subjected to the effects of solvents.

In the spring 2011 the School of Conservation in Denmark had the opportunity to be part of a larger research project hosted by the Universidad Politécnica in Valencia and the Smithsonian Institution in Washington, D.C. The research project is concentrating on evaluating the changes in the mechanical properties of oil paints and the analysis of their vulnerability to cleaning treatments with organic solvents. More specifically, this early stage of the project is focusing on some paints containing metals ions and the study of their influence in film formation thus conditioning the paint's response to solvents.

For this purpose FT-IR, SEM-EDX -analysis as well as tensile tests have been carried out. This study presents the data corresponding to several natural aged oil paint films containing Zinc White, Titanium White and Lead White. Tests were carried out before and after the films were exposed to selected organic solvents. In addition to this, a discussion of the effect of Lead White on oil paint films containing 'weak' pigments such as Yellow Ochre and Terre Verte will also be shown.

**Keywords:** oil paint films, solvents, lead white, zinc white, titanium white, FTIR, SEM

## Usability of r-FTIR-, r-UV/Vis- and XRF-spectroscopy for non-destructive characterization of materials on watercolor paintings, graphic objects and oil paintings

**Wilfried Vetter, Manfred Schreiner**

*Academy of Fine Arts Vienna, Institute of Science and Technology in Art*

Depending on the applied paint technique the composition of art objects may differ strongly regarding pigments, binding media and supports. Thus, differing analytical questions may rise when particular objects are investigated. In the work presented we assessed the usability of non-destructive compound specific reflection-FTIR and reflection-UV/Vis/NIR as well as element specific XRF for the identification of painting materials on 19th century watercolor paintings, contemporary graphic objects and oil paintings. Due to the unique character of artifacts sampling often is not possible and consequently non-destructive methods were applied for the analyses utilizing self built measuring systems in case of reflection-UV/Vis/NIR and XRF.

The investigations demonstrated a high potential of the complementary methods, as many organic and inorganic pigments and both traditional and synthetic binding media could be characterized even on watercolor paintings, where the paint layers are mainly very thin and a high sensitivity is required. In contrary, only few information about multilayer structures could be obtained. Other limitations particularly concerning earth and lake pigments originated in the lack of suitable reference materials, lack of characteristic interactions with the radiation used and ambiguity of measuring signals. Nevertheless, the results extend the body of knowledge in art history, document the actual condition of the objects analyzed and may further provide important information for the selection of suitable conservation or restoration treatments in many cases, as will be shown by some examples.

**Keywords:** painting materials, non-destructive, reflection-FTIR, reflection-UV/Vis, XRF



### *Fiber optic measuring head*

*Measuring head of the self-built fiber optic UV/Vis/NIR-system for the analysis of paintings and graphic objects. The main features are a 0°/45° measuring geometry, a spot size of 1,5 mm and laser assisted positioning.*

## Evaluation of the effect of different paint cross section preparation methods on the performances of fourier transformed infrared microscopy in total reflection mode

**Silvia Prati<sup>1</sup>, Francesca Rosi<sup>2</sup>, Giorgia Sciotto<sup>1</sup>, Emilio Catelli<sup>1</sup>, Costanza Miliani<sup>2</sup>, Brunetto Brunetti<sup>2</sup>, Rocco Mazzeo<sup>1</sup>**

<sup>1</sup>University of Bologna - Ravenna Campus, Microchemistry and Microscopy Art Diagnostic Laboratory, via Guacimanni 42, 48100 Ravenna, Italy

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The aim of this presentation is to compare the performances of FTIR microscopy in total reflection mode for the analyses of paint cross sections prepared with two recently developed methods aimed at overcoming the contamination from the embedding medium.

Indeed it is known that when flat surfaces are analysed the signal intensity is maximized together with the specular component, even if distorted spectra are obtained. On the other hand rough surfaces can increase the diffuse reflection leading to spectra less intense, but quite similar to transmission ones and allowing to detect diagnostic combination bands in the region 6000- 2000 cm<sup>-1</sup>.

The first method consisted in the use of IR inactive KBr as embedding medium followed by dry controlled polishing in order to obtain flat and homogenous cross sections in which the specular component was maximized. The second method foresaw a treatment of the sample with cyclododecane before embedding it in resin. Then the obtained block was cut to prevent the spreading of the synthetic polymer, obtaining a rough surface capable to increase the diffuse component.

As expected spectra registered both on real samples and on paint reconstructions prepared following the first method were more intense. In particular OH bands were more defined and less noisy. On the other hand, even if spectra registered on samples embedded with the second method were less intense, diagnostic combination bands could be identified which were hard to be distinguished in the spectra collected from samples prepared with the KBr method.

**Keywords:** paint cross sections, FTIR microscopy, total reflection, KBr, Cyclododecane

## **An advanced multivariate approach for processing XRF spectral and hyperspectral data from non-invasive in situ analyses on painted surfaces**

**Giorgia Sciutto<sup>1</sup>, Paolo Oliveri<sup>2</sup>, Silvia Prati<sup>1</sup>, Marta Quaranta<sup>1</sup>, Silvia Bersani<sup>1</sup>,  
Rocco Mazzeo<sup>1</sup>**

*<sup>1</sup>Microchemistry and Microscopy Art Diagnostic Laboratory (M2ADL), University of Bologna, Ravenna, Italy*

*<sup>2</sup>Department of Pharmacy, University of Genoa, Genoa, Italy*

In the last decades, in situ non-invasive analytical techniques have been widely developed for preliminary screening of painted surfaces and for identifying suitable sub-areas to be sampled and submitted to deeper laboratory investigations. Among the analytical techniques commonly used for in situ investigations, X-ray fluorescence (XRF) spectroscopy represents one of the most applied.

The present research work was aimed at developing an appropriate strategy for multivariate treatment of XRF spectral and hyperspectral data sets. In fact, even if multivariate methods are able to account for inter-correlation among variables, which is of high importance for elemental analyses, their application in this field have not been enough investigated. For this reason, an innovative methodological approach is presented, including several chemometric tools – such as peak alignment, spectra normalisation and exploratory analysis – aimed at improving the interpretation of the results. Moreover, spectral profiles were analysed directly, without computation of derived parameters, by means of principal component analysis (PCA). This allows a fast interpretation of the results, which can be accomplished in situ, providing real-time information helpful to define the subsequent sampling strategy. A particular attention was addressed to the selection of proper spectral pre-treatments to be applied on data. In addition, the research was focused on the application of multivariate exploratory analysis on XRF hyperspectral maps by means multivariate chemical maps, which were drawn and studied using brushing procedures. The application of the approach on the famous Renaissance painting “The Ideal City” (Palazzo Ducale, Urbino, Italy) is discussed as a case study.

**Keywords:** XRF, Principal component analysis, Signal processing, Multivariate maps, Painting

## Study of the alteration processes of Prussian blue in laboratory-prepared and genuine paint layers

**Louise Samain<sup>1</sup>, Jana Sanyova<sup>2</sup>, David Strivay<sup>1</sup>**

<sup>1</sup>*Centre Européen d'Archéométrie, Université de Liège, Liège, Belgium*

<sup>2</sup>*Institut Royal du Patrimoine Artistique, Bruxelles, Belgium*

The necessity of understanding degradation and alteration processes in a painting's materials is well established for preservation and art history issues. The task is complex because of the highly heterogeneous character of an ancient paint layer. In this context we focus on a particular pigment, Prussian blue. Prussian blue is a hydrated ferric ferrocyanide complex, first synthesized in 1704 in Berlin. It has been widely used by artists until the 1970s. However, the permanence of Prussian blue had already been questioned by the mid-eighteenth century, because it exhibits a tendency to fade in light and to turn green. To date, little attention has been devoted to the understanding of the degradation processes of Prussian blue in paint layers.

We induced discoloration upon light exposure in commercial and laboratory-synthesized Prussian blue watercolor and oil paint layers by accelerated ageing. Pure Prussian blue painted in a dark shade appears to be extremely light fast but fades when either painted in a lighter shade or mixed with white pigments.

We analyzed the paint layers by various techniques, i.e., UV-visible, Fourier transform infrared, Raman, Mössbauer and X-ray absorption spectroscopy. We attributed the fading of Prussian blue to a reduction of the iron(III) ions at the surface of the paint layers. We also observed a partial oxidation of Prussian in the entire paint layer. Finally we confirmed these results by analyzing works of art containing Prussian blue, i.e., a polychrome sculpture, wallpapers and a mural decoration sample.

**Keywords:** Prussian blue, modern pigment, fading

## **Analysis of tyrian purple in samples from the eastern mediterranean basin: an HPLC study for the assessment of the purple biological source**

**Ioannis Karapanagiotis<sup>1</sup>, Dimitrios Mantzouris<sup>2</sup>**

*<sup>1</sup>University ecclesiastical academy of Thessaloniki, department of management and conservation of ecclesiastical cultural heritage objects*

*<sup>2</sup>Ormylia foundation, art diagnosis center*

Tyrian purple, known also as Royal or true purple, is obtained from the hypobranchial glands of molluscs (Muricidae family). In the Mediterranean basin three mollusc species are found which can provide the purple dye: *Hexaplex trunculus* L., *Bolinus brandaris* L. and *Stramonita haemastoma*.

The relative dye compositions of these molluscs, measured by HPLC, have been used in the past to identify the (specific mollusc) Tyrian purple biological source applied on a historical/archaeological sample. These studies, however, were based on a limited number of data and furthermore they did not include a statistical analysis to take into account the several variables which can affect the HPLC dye compositions of the molluscs.

In the present study HPLC analyses of new mollusc samples, collected from various areas of the Mediterranean, are carried out. These new results, previously published and unpublished analytical data are investigated through an integrated approach: principal components analysis (PCA) is applied, for the first time, to investigate its effectiveness on the separation of the three Mediterranean mollusc species. Prior to this work a systematic investigation to optimize the extraction process (prior to HPLC) of the purple dye is carried out. The extraction efficacies of DMSO, DMF and pyridine, which were previously suggested and used to treat samples containing Tyrian purple, are evaluated. Furthermore, the effects of treatment time and temperature are evaluated.

Finally, the results of the aforementioned studies are used is to discuss the possible biological origin of Tyrian purple detected in historical and archaeological samples.

**Keywords:** HPLC, dye, Tyrian purple

## Characterization Of The Objects In Historical Textiles In Favour Of Restoration By Multi-Analytical Techniques

**Nilgun Kahraman<sup>1</sup>, Emine Torgan<sup>2</sup>, R.gurcan Oraltay<sup>3</sup>, Recep Karadag<sup>4</sup>**

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Characterization of metal threads and identification of natural dyes in historical textiles are important for preservation of cultural heritage. Chemical analysis of originally applied materials is very important part of characterization, since this enables understanding the nature of chemical and physical degradation and determines the cleaning methods. The analysis of objects of historical and cultural interest usually requires the use of various analytical methods and techniques. In this study, HPLC with diode array detection (HPLC-DAD) method was utilized for the identification of dyes in the historical objects. Scanning electron microscopy-energy dispersive spectrometry (SEM-EDS) has been applied for characterization of the metal threads in the objects. Colour measurements are determined by CIL\*a\*b\*.

In this study, the objects which are silk textiles are provided from Topkapı Palace Museum, Istanbul. Dyestuffs were detected using high-performance liquid chromatography with a diode array detector. As for red colour of dyes carminic acid, kermesic acid, alizarin purpurin, ellagic acid; as for yellow colour of dyes luteolin, apigenin; as for blue colour of dyes indigotin, indirubin were found. Four metals threads were analyzed via SEM-EDS. Two of them were Ag threads; the others were gold gilded Ag wires. S and Cl have been found as major contaminants. Au content showed difference from sample to sample. Furthermore, for the same sample, Au content changed depending on the degree of wear of the metal, in some regions outer Au layer was eroded, exposing inner Ag core at the surface.

**Keywords:** HPLC-DAD, SEM-EDS, dyestuff

**SEM results expressed in weight percentages**

Inventory no	Ag% Range	Average Ag (%)	Au% Range	Average Au (%)
13-21	89.1-93.4	90.6	-----	-----
13-512	88.3-91.1	89.7	-----	-----
13-1160	69.4-68.9	69.2	11.6-19.0	15.3
13-1747	63-92.8	78.8	0-18.1	6.7

## In-situ, non-invasive, unilateral NMR investigations of lead carboxylate formation in traditional oil paints

**Eleonora Del Federico<sup>1</sup>, Silvia Centeno<sup>2</sup>, Cindie Kehlet<sup>1</sup>, Amelia Catalano<sup>1</sup>, Boris Itin<sup>4</sup>, Alexej Jerschow<sup>3</sup>**

<sup>1</sup>*Department of Mathematics and Science, Pratt Institute, Brooklyn, NY, USA.*

<sup>2</sup>*Department of Scientific Research, The Metropolitan Museum of Art, New York, NY, USA.*

<sup>3</sup>*Department of Chemistry, New York University, New York, NY, USA.*

<sup>4</sup>*New York, Structural Biology Center, New York, NY, USA*

The reaction of lead-containing pigments and fatty acids in oil binders plays a critical role in the degradation of traditional oil paintings due to the formation of lead carboxylates which can form protrusions and/or increase the transparency of the paint, often causing a visible deterioration of the artworks [1-4]. In this study we apply unilateral NMR to explore the nature of the pigment-binder interactions that lead to carboxylate formation, and to assess the potential of the technique to evaluate the emergence of carboxylates in-situ and non-invasively. Carr, Purcell, Meiboom, and Gill (CPMG) followed by ATR/FTIR, to monitor a characteristic carboxylate peak in samples aged in different conditions, reveal that the addition of lead white to linseed oil increases the T2eff relaxation of the paint film if metal carboxylates are present. Comparison of the CPMG curves of samples prepared with red lead, massicot, lead tin yellow type I or vermilion in linseed oil display three different T2eff behaviors which can be qualitatively correlated to the carboxylate peak height observed by ATR/FTIR at ca. 1530 cm<sup>-1</sup>. These results suggest that T2eff of the paint films depends on the concentration of lead soaps and that single-sided NMR is therefore a suitable technique for monitoring their emergence in-situ and non-invasively.

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3.Mazzeo, R., et al., Analytical and Bioanalytical Chemistry, 2008. 392(1): p. 65-76.

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**Keywords:** Unilateral NMR, traditional oil paintings, T2 relaxation, ATR/FTIR, lead pigments, lead soaps, non-destructive analysis

## Vibrational study of bone black pigments

**Alessia Daveri<sup>1</sup>, Francesca Rosi<sup>2</sup>, Riccardo Vivani<sup>3</sup>, Brunetto Giovanni Brunetti<sup>4</sup>,  
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<sup>4</sup>Centro di Eccellenza Smart, Dipartimento di Chimica, Università di Perugia, Via Elce di Sotto, 8, 06123 Perugia-Italy

Several studies have been devoted to the molecular characterization of carbon-based black pigments by means of different micro destructive techniques [1-3]. One of the main interest on this regard is to find reliable spectral features to discriminate among different carbon-based black pigments according to their source (plants, animals and minerals) and to the manufacturing process (flame carbons, chars and cokes).

In particular the mid infrared spectra of bone black pigments generally show a small sharp band at 2018 cm<sup>-1</sup> that could be used to identify the animal origin of the pigment. This band characteristic of isocyanate, tyocyanate and/or isocyanate groups has been tentatively associated to a degradation product of bone [4,5].

In this study we present a spectroscopic investigation by mid-FTIR and Raman spectroscopy supported by X-ray diffraction, of both commercial and synthesized in laboratory bone black pigments, that allowed to clarify the assignment and the origin of this absorption band.

The band at 2018 cm<sup>-1</sup>, clearly detectable also in reflection mid-FTIR mode, allowed us to assess the opportunity of a non-invasive pigment identification in real artworks. On this regard, in order to evaluate the sensitivity of infrared spectroscopy in reflection mode, respect to transmission mode, model paints made of bone black diluted with lithopone and titanium dioxide in an acrylic binder have been prepared and analyzed.

Finally, to demonstrate the effectiveness of the method, several reflection mid-FTIR spectra from ancient to modern art including canvas, wall and easel paintings, have been considered.

**Keywords:** bone black pigments, mid-FTIR spectroscopy, non-invasive technique

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## Trends in nanotechnology-based analytical methods in cultural heritage

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The scientists can greatly contribute to the preservation of Cultural Heritage due to the tools they can provide for reliable predictions of the degradation of monuments and fabrication of new materials developed for cleaning or consolidation.

This work presents modern trends in developing sensitive, reliable microanalytic techniques for the characterization of the materials in art and archaeology, based on nanoparticle use.

Nanoparticulate SERS active substrates improve Raman spectroscopy employed for the identification and quantification of dye, pigments, varnishes, and represent one of the major contributions of nanotechnology in the conservation science. Several results in the development of SERS method for the characterization of old varnishes, by using active surfaces based on anisotropic silver and gold nanoparticles are detailed.

The functionalization of the cyclic voltammetry electrodes with nanostructured films is another application of nanoscience that produces a spectacular lowering of the detection level in the electrochemical methods.

Examples of silica or hybrid polymer-CNT films were presented, providing an easy-to-use voltammetry method to analyze patina of bronze objects.

The possibility to use other nanoparticle-enhanced methods, currently developed for biomedical or material science, for the analysis of specific materials and degradation processes in art objects will also be discussed.

**Keywords:** nanotechnology, nanomaterials, analytical methods

## Surface characterization of 19th century and modern daguerreotypes using high-resolution field emission scanning electron microscopy (HR FE SEM)

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The tarnish colors observed on historic daguerreotypes vary considerably, ranging from white to blue films, and/or light brown-yellows to browns to dark blue-black films. There is a need to better understand the chemical and physical nature of the daguerreotype surface and hence the composition of tarnish. This paper presents an electron microscopic study of 19th century and modern daguerreotypes using FE HR SEM with magnifications ranging from 50,000 to 250,000X, using low beam voltages, close working distances and imaging in both SE and BSE modes, to corroborate earlier studies and to demonstrate the nano-texture characteristics of daguerreotype surfaces. High-resolution images of both gilded and ungilded modern daguerreotype plates investigated show nano-textured surfaces. The ungilded plates show silver grains with embedded iron oxide rouge polishing material used in the preparation of the daguerreotypes. The gilded plates show smaller gold surface grains with accompanying grain boundaries covering silver and iron. The 19th c plates, which have been gilded, show similar gold grains as seen in the modern gilded plates, and nucleation of tarnish/corrosion nano-crystallites at the grain boundaries. In this scenario, reactive silver at the gold inter-grain boundaries is exposed to air and its atmospheric contaminants thereby allowing tarnish to develop.

Image particles of modern ungilded plates reveal sharp crystalline features when compared with gilded plates. The gilded particles show a finer nano-texture of gold grains than that observed on the background. Tarnish crystallites on image particles on 19th c. plates are also observed.

**Keywords:** daguerreotype, high resolution field emission scanning electron microscopy, surface characterization, tarnish

## **EBSD & EDS to characterize the surface of 19th c and modern daguerreotypes**

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The daguerreotype is the first viable imaging process that gave birth to photography and the imaging revolution. Unlike other silver-based black and white photographic processes, the daguerreotype is an image that rests on the surface of a highly polished silvered copper plate. The final stage of the daguerreotype process is to affix the silver-mercury amalgam image particles to the plate by passive electrochemical coating with a thin gold film. Even though gold is a noble metal, tarnishing readily occurs on all of the components of the daguerreotype, namely the image particles and the background surface. This work presents a study of 19th century and modern contemporary daguerreotypes using the two scanning electron microscopic techniques of energy dispersive X-ray spectroscopy (EDS) and, for the first time, electron backscattered diffraction (EBSD). EBSD is a microstructural-crystallographic technique used to determine the crystallographic orientation of the daguerreotype's metallic surface to elucidate the texture and preferred orientation of the silver and gold grains that are tens of nanometers in size on the background surface. EDS was used to determine the daguerreotype's surface chemical composition and/or elemental analysis. The microstructural, elemental, and crystallographic information observed by EBSD and EDS potentially explains the occurrence of tarnish as corrosion in the inter-granular boundaries of gold grains on gilded daguerreotype surfaces.

**Keywords:** daguerreotype, EBSD, EDS, surface characterization

## Deterioration of the Daguerreotype Image by Environmental Pollutants

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Daguerreotypes are unique images that consist of mercury-silver or mercury-silver-gold amalgam nanoparticles on silvered copper substrates [1]. The sensitivity of the daguerreotype image to light and environmental pollutants, such as hydrogen sulfide and chlorine-containing compounds, as well as to damage by mechanical and chemical cleaning has been previously discussed [1-3]. To further characterize the formation of deterioration products frequently found in historic and artistic daguerreotypes, test samples prepared following 19th century daguerreotype recipes were exposed to either chlorine or sulfur-containing environments, or both. Original daguerreotypes and the exposed samples were analyzed using Raman spectroscopy and SEM-EDS. Deterioration products, such as silver chloride, silver sulfide, silver oxide, and copper sulfide, were identified in the test samples and in the original daguerreotypes by their characteristic Raman bands complemented by ultra high resolution SEM-EDS analysis. To assess the photoreactivity of the alteration products, diffuse reflectance curves were measured using a micro-fading tester for up to two hours. Areas exposed to light at different stages during these experiments were analyzed by Raman spectroscopy and SEM-EDS. Additionally, to improve storage conditions, new silver corrosion sensors have been developed and used to help better assess future daguerreotype housing materials using a modified Oddy test.

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**Keywords:** daguerreotype, environmental pollutants, oddy test, diffuse reflectance, raman spectroscopy, SEM-EDS

## Identifying varnish compositions of historic tintypes: recipes and reality

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The tintype was the most popular photographic technique in the U.S.A. between 1856 and 1900, when millions of these objects were created by studios, itinerant artists, and even amateurs working from published 'how-to' manuals. To correlate the identity of varnishes used with recipes from the historical literature, a collection of more than 200 tintypes was analyzed using a crescendo approach: first, non-contact analysis of UVA-induced visible fluorescence, followed by contact-requiring ATR-FTIR, and finally micro-destructive pyrolysis GC-MS. This approach definitively identifies the varnish and also enables an assessment of the accuracy of the non-destructive analytical techniques. Py-GC-MS revealed that approximately 50% of the tintypes were varnished with shellac (a low cost and commonly available wood varnish), 8% with dammar, and 5% with sandarac. The rest were varnished with mixtures such as shellac with camphor/lavender oil, shellac with colophony, shellac with sandarac, or sandarac with camphor/lavender oil. Not all of these formulations correspond to published recipes, suggesting that tintypists either developed their own preferred mixtures or used what was at hand. CIEL\*a\*b\* values for the lightest-lights and darkest-darks of photographs of tintype UVA-induced visible fluorescence yielded no significant correlation with varnish identity, indicating that this common technique unfortunately has little analytical value for these samples. ATR-FTIR was able to accurately identify shellac and dammar varnishes but was less reliable with varnish mixtures and degraded samples. This study represents the first in-depth technical analysis of tintype materials and suggests that just as tintypes were bourgeois, so were the materials used as varnishes.

**Keywords:** tintype, photography, FT-IR, pyrolysis, GC-MS, fluorescence, historical, ATR, varnish

## **Analyses of paint layers on tinned and leaded steel. Investigation of coat of arms of the Order of the Elephant from the Royal Collection Knights' Shields in Denmark**

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The Order of the Elephant, with roots back to the 15th century, is one of the oldest Orders in Europe that is still practiced. Being the highest Order of Denmark, the Knights of the Order of the Elephant are first of all members of the royalty and heads of state, and the Order includes members from throughout the world. The coat of arms of each member of the Order is painted on a metal shield, which is hung and displayed in the Chapel of the Orders in Frederiksborg Palace church.

Whilst the oldest shields generally are in a good condition, some of the recent ones from the mid 1960s to the 90s show serious delamination of the paint layer. This has led to a concern for the modern part of the collection, which is the object of the present study. Several samples from shields dating from the last part of the 20th century have been analyzed. The results give information about the materials and techniques used in recent times, and lay a foundation for understanding the problems observed. Among the analytical methods are micro-Raman, micro-FTIR, py-GCMS and SEM/EDS.

The project is a collaboration between the School of Conservation in Copenhagen, the Chapter of the Royal Orders of Chivalry in Copenhagen and the Royal Institute for Cultural Heritage in Brussels

**Keywords:** paint on metal, modern paint, raman microspectroscopy, FTIR microspectroscopy, py-GCMS

## Characterization of iron slag and iron ore used in the ancient iron smelting work in Manipur, India by XRF, XRD, EDX, TL and FTIR

**Raheijuddin Sheikh**

*D m college of science, imphal*

Man evolved with materials. To a physicist or chemist, cultural heritage is the aggregation of material evidence of civilization. In this context, physics and chemistry can play an important role in studies of cultural heritage materials of a nation. Ancient people of Manipur smelted iron in local traditional styles and techniques which contributed to its rich cultural heritage. Recently, the author has discovered an ancient iron smelting site at Tumu Hills, Manipur, India. This paper presents the results of the TL dating of iron slag obtained from the place which is found to be of about  $1600 \pm 80$  years, characterization by XRD/XRF, FTIR, EDX spectroscopy. Iron ore samples collected from Lamdeng, Manipur which has been used in the iron-smelting work have also been characterized by the same techniques and the results of which are presented in the paper. The characterization reveals that iron of good quality has been extracted by the ancient people of Manipur in local traditional styles and techniques.

**Keywords:** Fourrier transform infrared, X-ray diffraction, Energy Dispersive X-ray, Scanning electron microscopy

## Plant gums used as binding media: analytical problems, databases, occurrence and physicochemical evolution

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Since the 3rd millennium B.C., polysaccharide materials, such as plant gums, sugar, flour or honey, have been used as binding media and sizing agents in paintings, illuminated manuscripts and polychrome objects. Saccharide materials are considered in ancient artistic recipes and they have also been widely employed for restoration and consolidation processes. Despite their widespread documented use, polysaccharide materials have been identified in very few works-of-art such as mural paintings and polychromies, tempera paintings and watercolors and, besides, their identification is often doubtful, and rarely discussed.

In this research, supported by a Marie Curie Intra European Fellowship within the 7th European Community Framework Programme (SYNOPYE project), we investigate the possibility of reliably identifying plant gums in paintings. In this study the widening of the available databases, the improvement of GC/MS procedures for their analysis as well as the study of how their composition changes over time has been taken into consideration. For this purpose, model systems were studied using thermoanalytical techniques, namely TGA and DSC to investigate the interaction of saccharide binders with proteins and inorganic materials, and to understand how these evolve with time. The same model systems were also investigated by GC-MS to evaluate the modification of the sugar profile of saccharide binders occurring as a result of aging, or the presence of inorganic and proteinaceous materials. The results allowed to develop a new model for the reliable identification of saccharide binders in paintings, based on the evaluation of markers that are stable to ageing and unaffected by pigments.

**Keywords:** saccharide paint binders, GC-MS of polysaccharides, pigments interference, ageing processes, thermoanalytical techniques.

## **Dolomitic lime mortars: carbonation complications and susceptibility to acidic sulfates**

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*Columbia University*

Dolomitic lime mortars, widely used in historic construction and as repair mortars today, are distinguished by their high magnesium content as well as their high plasticity, water retention, and workability. However, the dolomitic lime creates a more complicated chemistry in the resulting mortar in which the magnesium and calcium compounds carbonate at different rates. Although portlandite readily carbonates to calcite, the carbonation of brucite is delayed and could result in a variety of magnesium compounds in the cured mortar. The reaction of these magnesium compounds with acidic sulfates in the environment could lead to the formation of magnesium sulfate salts, which have the potential to deteriorate the mortar itself and surrounding materials. Mortar deterioration could also occur due to the dissolution of the magnesium compounds in the mortar and subsequent material losses.

A series of experiments have been conducted that examine the carbonation of dolomitic lime mortars as well as the extent to which these mortars interact with acidic sulfates. First the production of dolomitic lime was simulated in a laboratory setting, using XRD and SEM to determine the chemical and physical changes that occur during the calcining process. The same analytical techniques were used to examine the level of carbonation in dolomitic lime model mortars. The fundamental reactivity of acidic sulfate solutions with certain compounds in dolomitic mortars was explored using AA. Acidic sulfate attack via acid rain was also simulated on model mortars, and the effects of that exposure analyzed by AA and XRD.

**Keywords:** Dolomitic Lime, Acidic Sulfate Attack, Carbonation, Mortar

## Non-destructive diagnostic and monitoring studies on cultural heritage – in-situ examination of state of deterioration

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The use of quantitative infrared thermography (QIRT) and ultrasonic testing are some of the non-destructive investigation methods that allow in-situ evaluation and monitoring of cultural heritage. Here, some recent studies of METU Materials Conservation Laboratory (MCL) were presented. The state of deterioration of timber and stone materials were examined by the combined use of QIRT and ultrasonic testing together with supportive laboratory tests. The studies have shown that the joint interpretation of QIRT and ultrasonic testing were promising for the:-

-soundness assessment of structural timber elements used in historic stone masonry structures (Aslanhane Camisi, 13th century, Ankara) and traditional timber framed houses (Ayaş & Istiklal Districts, 19th century, Ankara);

-assessment of cracks in an historic stone masonry in terms of their activeness, depth and pattern (Cenabi Ahmet Paşa Camisi, 16th century, Ankara);

-determination of the emergency areas on stone statues and steles in open air conditions that needed conservation treatments, such as the cracks to be treated for the prevention of water penetration and losses by fragmental disintegration or by scales, as well as their monitoring before and after treatment (Nemrut Dağ Stone Monuments, 62 BC, Kahta-Adiyaman).

A good correlation was achieved between the state of deterioration of materials and their thermal inertia characteristics such as rates of warming up and cooling down. Together with ultrasonic velocity measurements, those rates allow in situ soundness assessment of materials and their monitoring on site. Advances in non destructive analyses require multi-disciplinary approach that brings together knowledge on materials science and building science.

**Keywords:** Non-destructive testing (NDT), quantitative infrared thermography, ultrasonic testing, state of deterioration, stone conservation, timber conservation

## Examination Of Deterioration In Nemrut Dağ Limestones And Development Of Their Conservation Treatments With Nanodispersive Ca(OH)<sub>2</sub> Solutions

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Limestone statues of Nemrut Dağ Monument were studied to discover their major deterioration mechanisms and develop their conservation treatments in atmospheric conditions using nanodispersive Ca(OH)<sub>2</sub> solutions. Deterioration was examined at exposed surfaces, internal crack surfaces and compared with relatively undeteriorated parts of limestone. Similar limestones from the geological formations were artificially deteriorated by salt crystallization and were used to serve as reference samples. Standard physical and physicomechanical tests, mineralogical and petrographical analysis by optical microscopy, XRD, SEM-EDX and FTIR were used during examinations.

The decay forms that required urgent conservation treatments were the material loss and detachments formed by fragmental disintegration in those karstic limestones. Some karstic zones were found to be preferred sites of decay by dissolution, recrystallization and crack formation. Swelling nature of clay minerals, iron oxides that move through those karstic zones, as well as biological activity were found to be closely related to that decay phenomenon by fragmental disintegration.

Preparation of high concentrations of nanodispersive Ca(OH)<sub>2</sub> solutions in ethyl alcohol was achieved for conservation treatments of limestone showing fragmental disintegration.

Treatments targeted to the decay zones were tested for their ability to control swelling action of clays, carbonation of nanodispersive solution in the cracks of stone and improvement in the physicomechanical properties of treated limestone. The success of the treatments were evaluated by non-destructive analyses at the site and microstructural analyses that were carried out in the laboratory. The long term behavior of those treatments are needed to be monitored and expressed by measurable parameters.

**Keywords:** Limestone deterioration, clay minerals, iron oxides, Nemrut Dağ Monument, nanodispersive Ca(OH)<sub>2</sub> treatment

## The relevance of spectroscopic imaging techniques on test drillings to assess the physicochemical damage of building materials

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The evaluation of the use of test drillings for the characterization of the physicochemical damage suffered by building materials was carried out by the combination of spectroscopic imaging techniques based on Raman spectroscopy,  $\mu$ -XRF and SEM-EDX.

The cross-section analysis of the drillings reveals:

- i) It is possible to differentiate superficial depositions (crusts and patinas) from penetrating pollution and to determine the thickness affected. For instance,  $\text{Ca}(\text{NO}_3)_2 \cdot 4\text{H}_2\text{O}$  internal deposits were found but it was not identified in the surface due to its solubility and migration capacity. That is, when salts are dissolved by action of rain water, affection can be erroneously assessed.
- ii) The elemental and molecular distributions allow to determine the origin of the compounds/elements found. The identification of some heavy metals as chrome or barium, are usually related to atmospheric particulate matter. However, a homogeneous distribution of these elements in the sample would indicate that they belong to the original composition. Moreover, the depth reached, which is related with the material porosity, could even suggest the physicochemical form of the pollutant.
- iii) SEM-EDX images allowed to detect superficial and internal cracks as well as to identify the responsible for this physical stress. Thus, salts as NaCl,  $\text{CaCO}_3$  and  $\text{CaSO}_4 \cdot 2\text{H}_2\text{O}$  were present inside the cracks, whereas they were absent on the surface.

In conclusion, the cross-section analysis on test drillings which is commonly used in other fields but not in built Heritage, is not only useful but is essential to avoid incorrect and/or incomplete damage assessment of building materials.

**Keywords:** cross-section, drillings, spectroscopic imaging, built Heritage

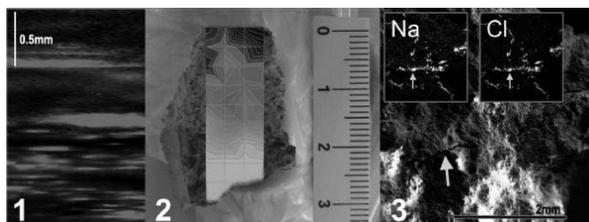


Figure 1

Figure 1.- (1) Monitoring of the internal carbonation (2mm) of a crusted brick by molecular Raman image of calcite. (2) The depth reached by lead, as shown in the  $\mu$ -XRF image, suggests a very penetrating physicochemical form of the pollutant, probably due to fuel combustions. (3) SEM-EDX image of the mortar reveals material cracks due to the physical stress produced by NaCl deposits probably originated by the flood that affected to the building.

## Positive and Negative Results of the Chemicals Used in the Restoration of the Fatih Mosque and Sultan I. Mahmut Library between 2007-2012

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The Restoration of the Fatih Mosque and Sultan Mahmut 1st Library began in the end of the 2007.

In the research process, it has been seen that, the previous restorations, rain and snow waters and also the planting has given the damages to the structures. Therefore to get rid of these damages, the mechanical and chemical operations were used.

The Subject of this paper is based on the positive and negative results of these 5 yeared operations.

**Keywords:** restoration, conservation, mosque, library, historical monument

## Origin and development of blue-and-white porcelain in ancient china

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<sup>2</sup>Chinese academy of cultural heritage

<sup>3</sup>Cultural relics and archaeology institute of henan province

A large amount of evidence has shown that blue-and-white porcelain was successfully produced as early as the Tang Dynasty (618-907 A.D.) in China. Though only very small quantity of Tang blue-and-white porcelain shards were excavated from the Tang stratum Gongyi kiln site which covers Baihe kiln site and Huangye kiln site, they are precious, especially in validating the producing area and time of Tang blue-and-white porcelain according to archaeological stratigraphy.

In the late Tang dynasty, the blue and white porcelain came into being on the basis of white porcelain production. The body and glaze compositions of blue-and-white shards from Gongyi kiln site are close to the white porcelain of late Tang, the type of pigment used is similar to the pigment used for the upper-glaze blue decoration on the white-glazed pottery. The mature white porcelain making technology lays foundation of body, glaze and high temperature firing for the birth of blue-and-white porcelain. The material basis and technology development regularity has been investigated concerning the "Tricolor pottery-white glazed pottery with blue decoration-blue and white porcelain" evolutionary process.

**Keywords:** origin, blue-and-white porcelain, Gongyi kiln

### *Tang blue-and-white shard*



*Tang blue-and-white shard excavated from Gongyi kiln site*

### *Tang blue-and-white shard*



*Tang blue-and-white shard excavated from Gongyi kiln site*

## **Voltammetry of Microparticles for the characterization of copper and bronze corrosion products on Roman artefacts**

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Since long time electrochemistry has been applied to the scientific examination of cultural heritage, especially for the study of metals, alloys and their corrosion problems.

In the last decades the application of voltammetric techniques to archaeometry and conservation issues has undergone a large development.

Voltammetry of Microparticles (VMP), developed by Scholz et al. [1], is a non-invasive and sensitive technique for identifying metals and alloys and it appears promising for the characterization of corrosion products [2]. The paraffin impregnated graphite electrode (PIGE) allows the use of a very small amount of sample microparticles, mechanically transferred simply by rubbing the electrode surface on the sample that needs to be analyzed.

The required amount of sample is in the range of ng- $\mu$ g [2], but PIGE can be also used for in-situ measurements.

This work, done in collaboration with the Flemish Heritage Institute (Vlaams Instituut voor het Onroerend Erfgoed - VIOE) presents the characterization of the corrosion layers presents on archaeological objects from excavation in Flanders.

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**Keywords:** VMP, PIGE, copper corrosion, cultural heritage

## Analysis of silver content and trace metals in medieval coins from Sweden, using a $\mu$ -spot X-ray fluorescence instrument ( $\mu$ XRF), and comparing results to other techniques

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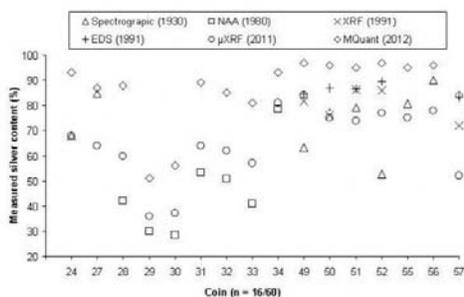
Analysis of metal content in coins is of importance within the field of numismatic research, in order to classify and understand the origin and history of these artefacts.

There are difficulties when analysing the metal content in archaeological coins. Excavated coins are often covered in dirt and may be corroded. Corrosion makes it difficult to estimate archetypal metal content and even after conservation, the desired patina left on the coin may disturb analysis. There may also be inhomogeneity within the metal itself, which makes it hard to calculate representative results.

In this study  $\mu$ XRF was evaluated as a technique for quantifying the silver content and trace metals in medieval coins from Sweden. Recently new software was developed that enables a calibrated  $\mu$ XRF-instrument to quantitatively measure the composition of a sample, without using external standards. This method has been compared to the traditional quantification against reference standards. Analyses were mostly performed on two sided coins minted during the reign of the Swedish king Magnus Eriksson (1319-1364). A total of 60 silver coins were analysed with the  $\mu$ XRF-technique. About 30% of the coins had previously been investigated with a range of other quantification methods, such as XRF, SEM/EDS, NAA and destructive spectrographic analysis, this provided an opportunity for further comparison and a discussion of these quantification techniques.

The results from this study will be used in the ongoing research work of understanding the very complex structure of medieval monetary systems and ethics in Sweden at that time.

**Keywords:** analysis,  $\mu$ XRF, XRF, silver content, coin, bracheates



### Comparison of different methods: Analysing silver content in medieval coins from Sweden.

Silver content was analysed using six different methods: Destructive spectrographic analysis, neutron activation analysis, XRF, EDS and  $\mu$ XRF with and without the application of MQuant (a quantification software).

Older analysis was performed as early as the 1930th; others are from the 1980-90th; compared with analysis from 2011 and 12.

## Micro X-ray fluorescence analysis ( $\mu$ XRF) of silver denarii from medieval Poland

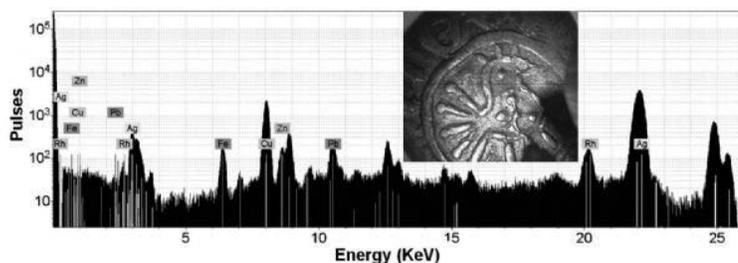
**Julio M Del Hoyo Meléndez, Lukasz Bratasz, Marta Włodarczak, Anna Klisinska Kopacz**

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X-ray fluorescence (XRF) analyses have become a standard method in cultural heritage science due to their non-invasive and non-destructive nature. Chemical analyses of metal alloy collections provide information about the manufacturing process, the provenance of raw materials, and the geographical distribution of ancient mints. 46 silver denarii from the early Piasts (10-11th centuries AD) belonging to the collection of the National Museum in Krakow have been characterized using micro-X-ray fluorescence spectrometry and the results have been evaluated in the context of literature data. The research has focused on evaluating the use of this technique as a screening tool for elemental surface characterization of the alloys. All denarii are made of Ag and Cu, with minor amounts of Pb, Fe, Au, and Zn. Preliminary semi-quantitative analyses have revealed silver contents in the 92-97 % range for several of the surveyed coins. In spite of the problems associated with the use of different instruments and various experimental conditions, the results are in agreement with previous data obtained for similar coins from the same time period and geographical location. In addition, the advantages and limitations of the technique when examining numismatic collections are explained. A known drawback of this method is silver surface enrichment, which is a common process observed in archaeological silver-copper alloys sometimes leading to incorrect interpretation of the data. This study has the goal of providing elemental information, which will serve to enhance the current knowledge about geographical and chronological diversification of Polish numismatic collections.

**Keywords:** X-ray fluorescence, numismatic collections, non-destructive analysis, Polish denarii

**Figure 1**



*Micro-XRF spectrum acquired from the surface of a Boleslaw Chrobry denar. Inset: photography of the object showing the measurement point.*

## **Towards standard practices in outdoor bronze coating testing: characterisation of bronze patina growth in coastal environment**

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Experimental testing to evaluate coating performance in outdoor exposure is a well established practice for technological and industrial applications, with a number of available standards. In the field of cultural heritage, the usefulness of well established procedures has been recognised at European level and standards related to stone, mortars, and waterlogged wood have already been drafted. However, in the field of metallic cultural heritage this is yet to be established taking into account the key parameters affecting the corrosion process of alloys.

One of the widely addressed topics in the field of metal conservation is the testing of protective treatments for outdoor bronze monuments: in the last 15 years many studies have clearly enlightened the complex behaviour of copper and its bronze alloys and the key role of patinas in the effectiveness of inhibiting and protective treatments. In order to evaluate a procedure for bronze treatments, bronze coupons have been exposed in a standard weathering station inside the Genoa Harbour and the patina growth have been monitored using several analytical techniques such as X-ray diffraction (XRD), colorimetry and Eddy current to monitor the patina thickness development. The results are discussed in comparison to the present knowledge of patinas of outdoor bronze monuments and their effective use in coating testing. Statistical and chemometric tools have also been used to evaluate the results and examine the accuracy of the techniques used.

**Keywords:** bronze, atmospheric corrosion, patina, methods

## Laboratory Induced Biodeterioration of Stained Glass by Fungi

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Over the past decades, several studies revealed that microorganisms can act as biodeteriogens of glass surfaces. Since historic glasses and stained glasses are very rich in modifying elements, such as potassium and calcium, they are particularly vulnerable to environmental aggressions (namely, water corrosion), and also to microbial attack that will promote glass biodeterioration.

In this work, glass microbially induced corrosion is being studied by a laboratory induced fungi contamination of a few glass samples. These are reproductions made under laboratory conditions in order to simulate three distinct colors (colorless, purple and brown) found in two stained glass panels from D. Fernando II's collection (dating from 15th and 17th century), and therefore were prepared using recipes deduced from the  $\mu$ -EDXRF analysis results.

The presence of fungi had been previously detected on these stained glass windows by molecular biology analysis. The results showed that the two predominant genera were *Penicillium* sp. and *Cladosporium* sp., so these two were selected to be inoculated over the glass samples. The fungi biodeterioration of the glass surfaces and their chemical alteration is being studied by means of  $\mu$ -EDXRF,  $\mu$ -Raman and  $\mu$ -FTIR spectroscopy, optical microscopy, SEM-EDX and ICP-AES analysis. The consequences of the surface attack made by these two fungi species will be discussed comparing two initial glass surface states: non corroded and corroded glass surfaces.

**Keywords:** Biodeterioration, stained-glass, glass reproductions, fungi.

## Nature's first green is gold: analysis of a lost Frank Lloyd Wright wisteria mosaic

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Frank Lloyd Wright (1867-1959), one of America's premier architects, designed and built the Darwin Martin House Complex, an exemplar of prairie style architecture, in Buffalo, NY (1903-1905). The focal point of the main house was a glass mosaic depicting wisteria vines that surrounded the central fireplace. The mosaic was a collaborative effort: made by the Giannini & Hilgart Studio, a Chicago art glass firm and perhaps designed by Wright or Blanche Ostertag, an illustrator known to have designed another Wright and Giannini fireplace. During the years the house sat vacant the mosaic was destroyed and the tesserae dispersed. Luckily, fragments were discovered in the fireplace grate at the outset of a renovation, and we analyzed these by XRF, SEM-EDX, FT-IR, confocal microscopy, and pyrolysis GC-MS analysis to determine how the mosaic had been manufactured. Design glass of four compositions, all containing gold, was adhered to a clear backing glass with mortar and grout that was also gilded, in keeping with the gold theme used throughout the structure. Also in keeping with this theme the blossoms were gilded glass, not purple or white as published reports suggest. The unique crackled gold surface of the green glass leaves, a hallmark of Giannini's work, was made by application of pure gold to the surface of the pre-cut glass, which was then heated to induce traction crackling. Our findings have guided efforts to reproduce this crackled gold surface and will allow an ongoing restoration to recreate this 'hardest hue to hold'.

**Keywords:** glass, gold, mosaic, Frank Lloyd Wright, SEM, FT-IR, pyrolysis, GC-MS, confocal, analysis

## Preliminary Archaeometrical Studies of Glass Finds From Istanbul Yenikapi Marmaray and Metro Excavation

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Istanbul Yenikapi region so called ancient Theodosius Harbour at historical peninsula has been excavated by the Directorate of Istanbul Archaeology Museums since 2004. In the Yenikapi Excavations, about 25000 artefacts were found by the year 2008. Glass finds are probably belong to Early Byzantine and Ottoman Period.

The aim of the researches on glasses is to characterise to find their historical origins. During the excavation period 2011, a glass set including mouth, body, chunks and bottom pieces was prepared for this purpose. The glass samples were primarily visually analysed under microscope to get their production techniques, then their colour values were determined by chromometric analysis and the samples were photographically documented and catalogged. Most of the pieces were turquoise, light and dark olive green, bluish, green, light blue in colours and their thickness varied between 0.10 and 11.20 mm.

The element compositions of the samples were analysed by the spectroscopic methods of X-Ray Fluorescence Spectroscopy. Complementary results showed that the glass samples are the typical soda-lime glasses. but the Si content of the samples were a bit lower than the expected values. That might be the reason of decomposition of the glasses when the time being. It was understood that the origin of the raw material as sand was the sea sand and soda was the only alkaline source of the glasses. Plant ash should not be added for the sample. Co, Cu is the colour related elements for the coloured glasses.

**Keywords:** Archaeometry, Glass, Glass Analyses, Istanbul Yenikapi Excavation Glasses

## Study of 18th century glass mosaics: The reds and carnagionni of Alessio Mattioli

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The São João Baptista chapel, in the São Roque church (Lisbon), has four mosaic panels that were made in Rome in the 18th century (Fig.1). The glass tesserae were produced by the most famous Italian glass-maker of that time, Alessio Mattioli, who became very well-known for developing many shades of glass colours, especially the reds and skin tones ("canagioni"). These colours are associated with a very thin black layer in the tesserae, which is called "scorzette".

The objective of this study was to chemically and morphologically characterize the glass material, particularly the tesserae associated with the scorzette layer. The aim is to understand the composition of the scorzette and how it is formed. Also, this range of colours show more susceptibility to degradation, resulting in darkening of the exposed surface of the tesserae, and it is important to understand if this fact is related to their specific chemical composition.

The study employed a multi-analytical approach, including energy-dispersive X-ray fluorescence ( $\mu$ -EDXRF), Raman microscopy, scanning electron microscopy with X-ray microanalysis (SEM-EDS), and observation techniques such as SEM and optical microscopy.

Preliminary results show that reds and carnations are lead-rich glasses with copper as their main colorant, in the form of cuprite. Orange is a mixture of red glass and lead antimonite (Naples Yellow). The scorzette shows a particular copper-rich composition and it is associated, in orange tesserae, with an antimony-rich yellow layer immediately before the glass body (Fig.2). The relationship between the chemical composition of the tesserae and degradation will be discussed.

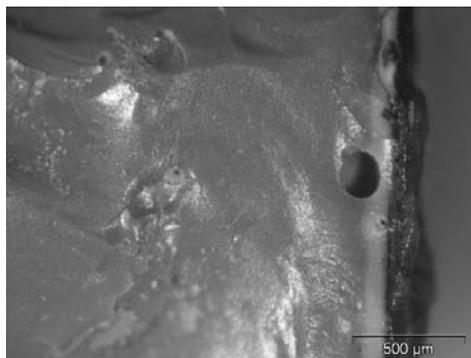
**Keywords:** Mosaics, Glass tesserae, Mattioli, Scorzette, Raman, SEM-EDS

*Micromosaic panel. "Anunciación"*



*Image of the mosaic panel ("Anunciación") in São João Baptista chapel, in the São Roque church (Lisbon).  
Made in Rome in the 18th century*

*Image of an orange tessera, showing the scorzette layer*



*Orange mosaic tessera, where a yellow layer is visible between the glass body and the scorzette (black layer).*

## **$\mu$ -Raman analysis of Hispano-Moresque historical tiles**

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A collection of Hispano-Moresque architectural tiles was recently excavated in the archaeological site of the Monastery of Santa Clara-a-Velha, in Coimbra, Portugal. This is a remarkable collection, comprising tiles produced by “alcatado”, “cuerda-seca”, “arista” and relief techniques, dated between the end of the 15th century and the beginning of the 16th century.

Non-destructive techniques were chosen for this study, with emphasis on  $\mu$ -Raman spectroscopy. This technique is able to detect the pictorial elements on a glaze and other materials, and the glaze as a glass material can be characterised as well. A typical Raman spectrum of a glass or glaze material shows two main broad bands, corresponding to the Si-O bending vibration (about 500 cm<sup>-1</sup>) and the Si-O stretching vibration (about 1000 cm<sup>-1</sup>). The shape, intensity and deconvolution of these bands are characteristic of a certain glass composition, and can infer on processing temperatures of manufacture, substitution patterns and discriminating between different groups. The results from the glaze characterisation will later be compared to analysis of other Hispano-Moresque tile collections.

As complementary techniques,  $\mu$ -EDXRF and  $\mu$ -PIXE were used. A quantitative analysis of the chemical composition of different coloured glazes was performed. The results are compared with the ones obtained by  $\mu$ -Raman spectroscopy in order to assess a possible association between the  $\mu$ -Raman spectra and the chemical composition of the glazes.

**Keywords:** Raman, glazed tile, azulejo, Hispano-Moresque

## Luminescent ionic liquids for glass cleaning: restoring Stained Glass transparency

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Medieval stained glass often present encrustations, deposits and dirt that require important cleaning operations, rendering these artistic objects more accessible from an aesthetic point of view and for the preservation of cultural heritage. These crusts are difficult to remove without damaging the glass surfaces. Present cleaning methods require chemicals and/or procedures that can damage the glass surface and painting. Alternative task-specific ionic liquids (ILs), designed for controlled and efficient glass cleaning, were synthesized and tested in order to remove the crusts. Besides removing the crusts, and in order to assure the complete removal of both impurities and solvent (IL) after the treatment, ILs will be functionalized using light emitting markers that will inform the user if the glass surface is completely clean.

The final goal is to produce a luminescent IL capable of solubilising the insoluble carbonates, sulphates and oxalates. Glass samples with a composition similar to medieval glass were produced and part of it had artificial corrosion induced, in order to study glass corrosion and have samples for testing the ILs produced.

Coumarin-3-carboxilate (Coumarin) anion was combined with Aliquat<sup>TM</sup> (an quaternary amine cation) to produce an intrinsic luminescent IL, [Aliquat][Coumarin]. Solubility tests showed that with only 10% of [Aliquat][Coumarin] added to [Aliquat][AOT] (AOT is a Docusate anion) it is possible to increase the amount of Ca dissolved by 2 to 4 times when compared with the pure [Aliquat][AOT] IL. After the characterization of the synthesized ILs, their performance will be evaluated using different spectroscopy and microscopy techniques.

**Keywords:** Stained glass, ionic liquids, glass corrosion, luminescent ionic liquids

## Analysis of Wax Paint and Fatty Acid Soaps by Pyrolysis Gas Chromatography

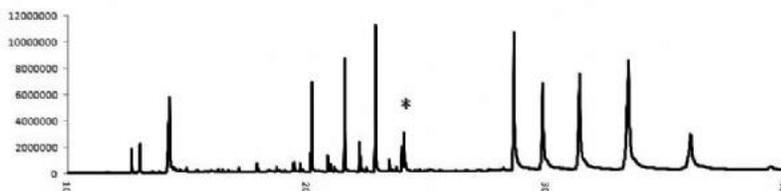
**Joy Mazurek, Michael Schilling**

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Beeswax has been used by artists as a paint medium since antiquity. The ancient Egyptians used it for mummification, and for creating the accompanying images of the deceased, or Fayum portraits. In the encaustic technique, heated tools melt, apply and shape colored waxes on the support. Beeswax can also be formulated into a cold paintable-wax or a water-based emulsion. Punic wax is a well-known example of a wax emulsion described by Pliny, and is thought to be made by saponification of the waxy esters or neutralization of the free fatty acids to soaps, which then emulsify the beeswax in water. Pigments further complicate interpretation of the analytical data because fatty acids are susceptible to form pigment soaps. A solvent-based, brushable wax medium can also be formulated with drying oils and natural resins, and these are common commercial products. Preparation of wax paint replicas from commercial sources and from recipes are analyzed and discussed. The paints were analyzed using a double shot pyrolysis gas chromatograph/mass spectrometer technique for characterizing soaps in wax-based paint media. In the chromatogram below, a lead palmitate soap is shown in ancient Egyptian beeswax by the identification of a palmitone. Although identification of the type or source of wax (plant, mineral, petroleum or insect) is usually straightforward, waxes can be quite difficult to fully characterize in small samples of paint because they are complex mixtures of many classes of organic materials.

**Keywords:** beeswax, fatty acid soaps, pyrolysis, gas chromatography, binding media

### *Pyrolysis of Beeswax from the Herakleides mummy at the Getty Villa*



*Beeswax and Lead Red from Egyptian mummy. A palmitone(\*) is produced during pyrolysis and subsequent degradation of a palmitic acid lead soap.*

## Identification and Pathology of Lapis Lazuli Pigment in Illustrated Books by PIXE

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Lazurite was the main source of blue color in Iranian painting in the past. It is an aluminosilicate mineral belonging to the sodalite group, associated with some other minerals (diposide, quartz, calcite, pyrite to form the lapis lazuli rock). Most of the blue color of the Iranian middle-age and Islamic world paintings decorated with lazurite and lapis lazuli as the blue pigments. In this work, the ancient methods for preparing of lazurite and lapis lazuli, as well as the main reasons of their destruction have been investigated. Four pictures of Yūsof-o-Zalīkhā book related to Safavid period (Iran, 16th century) were analyzed by proton-induced X-ray emission (PIXE) analytical method to find out more about the nature of the blue colors used and the pathology of the damages. For the first time, the extender was introduced as the main cause of lapis lazuli disease. This study demonstrated the potential of PIXE analytical method in the understanding of the ancient techniques of pigments preparing and the pathology of damages.

**Keywords:** Lazurite, Extender, Iranian Painting, PIXE, Lapis Lazuli Disease

## Opal: Australia's National Gem Stone - Materials Chemistry and Conservation

**Paul Stephen Thomas**

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Precious opal was declared as Australia's national gemstone in 1994 based on its importance to the Australian Outback economy (Australian opal fields provide 90% of world opal production) and to the cultural heritage of Outback Australia (opal mining helped the development of outback Australia and provides much of outback folklore). Opal is also important as a part of the fossil record as many plants, invertebrates and vertebrates of the cretaceous age (ca. 110 million years ago) have been preserved through the process of opalisation.

Opal is a hydrous amorphous silica ( $\text{SiO}_2 \cdot n\text{H}_2\text{O}$ ) and is found in Australia predominantly in sedimentary rocks circa 10 to 30 m below the surface. Once an opal specimen has been removed from its environment, however, there is the potential for a small percentage of opals to dehydrate and crack. The phenomenon is a form of stress corrosion cracking. The cracking of specimens destroys the value of precious gem stones and fossil specimens and may result in the loss of historic specimens and, hence, a loss of specimens important to the national heritage. Currently there are no methods available to predict which specimens are susceptible to cracking. To understand the process of cracking, the materials chemistry must first be understood. This paper will present work being carried out at UTS on the materials characterisation of opal from both Australian and international sources with an aim to understand the materials chemistry so that an understanding of the crazing phenomenon observed in some specimens of opal can be developed.

**Keywords:** Australian sedimentary opal, cracking, heritage, materials chemistry

### **Examples of Opalised Fossils - Opalised bivalves**



*Opalised bivalves sourced from Coober Pedy. Opalisation is the infilling of voids in the host rock. Infilling takes on the surface topography of the voids thus retaining the imprint left behind. Specimens are 65 to 110 million years old.*

### **Polished opals exhibiting play-of-colour which have undergone cracking.**



*Samples of polished gem stones which have undergone cracking after polishing. Cracking is not common, but does result in loss of value particularly in the case of historic specimens.*

## Chemical Characterisation of Waterlogged Woods in Yenikapı 12 by FT-IR Analyzes

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Structure of the degradation of the wooden material which had remained under water for long year must be known for conservation. This study includes chemical analyzes of the samples during the excavations in Yenikapı shipwreck. Because of this, FT-IR analyzes made samples from various points in the ship and fresh woods. Obtained values by the FT-IR Analysis was informed about lignin degradation of Yenikapı waterlogged woods.

Polymer degradation is observed due to decrease im OH groups at 3410 cm<sup>-1</sup> band in all degraded samples if compared with the intact samples. Besidex, remarkable decreases are detected in lignin at 1508 cm<sup>-1</sup> band, lignin and polysaccharides (hemicellulose and cellulose) at 1421, 1372, 1265, 1158, 1054 ve 1030 cm<sup>-1</sup> bands, cellulose at 897 cm<sup>-1</sup> band.

**Keywords:** FT-IR analyzes, Yenikapı Waterlogged wood, lignin, shipwreck, cellulose

## Thermal property dependence of paraloid B-72 cast films on solvent choice

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<sup>2</sup>*Winterthur/University of Delaware Art Conservation Program*

Paraloid B-72 is perhaps the most commonly used conservation polymer, and its working properties, performance characteristics, and appearance are known to depend heavily on the resin's physical properties. The glass transition temperature ( $T_g$ ) is critical in the role of this polymer as an adhesive, a consolidant, and a varnish. Modulated differential scanning calorimetry (MDSC) has been used to study the relationship between the  $T_g$  of B-72 films and the solvent used to prepare the casting solution. A Teas chart for B-72 based on approximately 50 solvents has been prepared, and for films cast from the workable solutions, the  $T_g$  was measured at several points in the drying process. Trends between solvent properties and film hardness as indicated by the  $T_g$  are exposed. In general, the "thermodynamic suitability" of the solvent correlates strongly with the glass transition temperature, revealing another useful means of manipulating film hardness and strength for this ubiquitous conservation polymer.

**Keywords:** Paraloid B-72, glass transition temperature, physical properties, calorimetry, conservation polymer, thermoplastic resin, Teas chart



## Complementary Analysis of Proteins in the 14th-century Double-Sided Processional Banner, Saint Mary Magdalen Holding a Crucifix; (reverse) The Flagellation, by Spinello Aretino

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Scientific analysis of materials found in cultural heritage is strengthened when several techniques are collectively employed for the identification and characterization of the materials and techniques used to create a work of art. One of the materials present in artworks is proteins, which are used widely as paint binders and adhesives but also as coatings or isolation layers. The most common proteinaceous binders found in paintings are animal glues, casein, and egg. Analysis and identification of these proteins can be challenging as in many cases the amount of material present is limited and the proteins are degraded. The recent application of immunological and proteomics techniques to works of cultural heritage are emerging as powerful tools, assisting scientists and conservators in the identification and localization of proteins. This current work will discuss analytical methods employed in combination to identify and characterize proteins found in a 14th-century double-sided processional banner, Saint Mary Magdalen Holding a Crucifix; (reverse) The Flagellation, by Spinello Aretino (Italian, born 1345–52, died 1410). The immunological-based techniques ELISA and ImmunoSERS as well as the more established techniques of GC/MS and ATR-FTIR were used to identify and localize egg protein in micro-samples from the banner. In addition, MALDI-MFP analysis was used to provide a more in-depth understanding of the identified proteins. The diversified analytical approach to studying proteins provided an invaluable opportunity to learn more about this rare work of art.

**Keywords:** protein identification, protein localization, ELISA, MALDI-MFP, ImmunoSERS, casein, animal glues, egg, GC/MS, ATR-FTIR

## Identification of animal adhesives using DNA amplification

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Adhesives based on collagenous tissues such as bones, cartilage, connective tissue or skins have been used in the manufacture of art and archaeological objects for thousands of years. The adhesive is mainly manufactured from products based on fish or mammalian species, depending on the original material and the specific purpose of the conservation treatment. Identification of collagenous adhesives are not an easy task, as the adhesive has no obvious macro or micro structure which can identify it down to species level. In the past 20 years new species identification tools has evolved for plant and animal material. Using DNA sampling (e.g. barcoding) the study will address the problematic of identification to species level in collagen based binding medias in artwork and historical objects. This is done by producing different collagen binding media based on old original recipes to see whether the DNA is still available for species identification after the production of the adhesive. Our first studies on bladders from sturgeon gives positive species identification from old original recipes used during the manufacturing of the glue. The results of this study will be of great use within museum studies of objects' origin including manufacture techniques as well as historic restorations and recommendations for future conservation.

**Keywords:** animal glue, DNA, species identification, adhesive

## Silane based products for tile protection

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Inorganic-organic hybrids, like organic modified silanes, are very interesting materials for coatings as they can grant flexibility, robustness and durability characteristic of organic polymers, allied to the hardness, chemical resistance, weatherability and UV-resistance of inorganic silicate polymers.

Organic modified silanes (Ormosil®) like coatings via sol-gel are versatile in coating formulation. Glycidoxypropyltrimethoxysilane (GPTMS) is one of the most important precursor used to prepare inorganic-organic hybrids via sol-gel. Having a combination of two different components, glycidoxy (organic) and silicon alkoxy groups (inorganic), GPTMS can undergo different reactions during the preparation of hybrids by sol-gel route. Hydrolysis of methoxy groups gives silanol groups that condense to form the silicate network. Using a low temperature epoxy ring opening with amine curing agents, an organic network can be formed [1]. In the present work diethylenetriamine (DETA) was used as curing agent.

Hydrolyzed GPTMS and GPTMS/DETA ormosils were applied by immersion on ceramic glazed tiles. Results show significant improvement in mechanical properties and in water absorption properties. Tile samples exhibit an increase in flexural strength, from 12.5 MPa, for untreated samples, to about 19 MPa, upon treatment. Water absorption essays revealed a decrease in the values of initial water absorption rate up to two orders of magnitude, and a decrease in open porosity from about 30% to 14%, upon treatment. The maximum water content expressed in percentage of dry sample weight decreased from 17% (untreated) to near 8% (treated).

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**Keywords:** silane, tile protection, water absorption, flexural strength

## In situ investigation of metal protective coating films with mid-infrared spectroscopy

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Mid-infrared spectroscopy in transfection mode was applied in the study of spin-coated films on bronze and iron substrates of copolymer resins based on acrylate esters (Paraloid family and Inralac from various distributors) and acrylic acid (BASF Poligen® ES91009). Spin-coated films of these materials on bronze and iron substrates were investigated both in their fresh and artificially aged states. Specifically for Paraloid B72 and Poligen® ES91009, their behaviour was compared to that of immersion-applied thick films on pre-corroded metal coupons. For the latter, (an ethylene-acrylic acid copolymer existing in mixed carboxylic acid and ionic carboxylate states in the commercially available water-borne dispersion) the working state has been established as one having purely acidic functionality gradually evolving after application. Therefore, consideration of the thermal history and the post-application (or working) state of the material is imperative for routine analysis. Furthermore, assessing its condition as protective coating involved the detection of interactions of the sensitive acidic carbonyl functionality with its reactive environment (metal surface and corrosion products, moisture, pH, etc). Thus, a band attributed to a carboxylate coordination complex has been detected in the case of films on bronze coupons.

Additional investigation using microscopy-FTIR in transfection mode on a number of immersion-applied and spin-coated films allowed detection of chemical changes of the above coatings in the local vicinity of specific corrosion grains. Scanning areas at the micrometer range can significantly improve detection levels and provide valuable site-specific information in a complementary manner to typical transfection infrared spectroscopy which offers space-average information.

**Keywords:** metal coating, copolymer, infrared spectroscopy, transfection, microscopy-FTIR

## Deposition of a lead dodecanoate coating on a lead or lead alloy metal surface

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In this project, we focus on the protection of the metallic cultural heritage and more specifically on lead or other lead alloy objects. Research activities can proceed in two directions: (1) try to reduce the corrosive substances in the environment and/or apply (2) a surface protection treatment. An aqueous sodium dodecanoate solution has been used for the formation of a protective coating for lead metal surfaces [1,2]. Three deposition methods have been compared: immobilization using cyclic voltammetry [3], immersion and amperometry. Apart from this, we tested a reduction pretreatment of the lead surface (-1.5 V during 600 seconds) in order to obtain a more reproducible coating, resulting in a better corrosion protection behavior. The corrosion inhibition properties were examined using potentiodynamic polarization curves and electrochemical impedance measurements in a standard corrosive environment [4].

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**Keywords:** sodium dodecanoate; lead conservation; corrosion inhibition; deposition

## Modern repairs of Chinese bronzes: study of materials, restoration techniques and state of conservation

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This study focuses on the Institute of History and Philology (IHP) collection of Chinese bronze artefacts which were reassembled in 2000 according to the traditional restoration techniques of Beijing Masters to create an accurate profile for the display. The thirty-three artefacts date either from the Shang Dynasty, Anyang Period, (1200-1046 B.C.) or from the Zhou Dynasty, Eastern Zhou Period (770-221 B.C.). The previous restorations consist in original fragments that are soft-soldered to new replacement pieces made from copper plates. Missing decorative elements are re-created in resin-based putty and calcium-based fillers. The mends and repairs are concealed with an imitation patina, mixture of shellac and pigments, to match the natural surface. The scientific methods employed to identify the modern restoration materials on the one hand, and the brief curators written records on the other hand, give rise to the sequence, description of each step of the interventive procedure, of the modern restoration process.

In a second part, this contribution presents the initial results of a survey to determine these artefacts condition after twelve years exposition in a temperature and humidity controlled environment. The degree and the cause of the artefacts damages are discussed. Finally a risk estimation of this way of joining artefacts is proposed to better predict the collection needs and confidently identify effective strategies.

**Keywords:** Chinese bronzes, restoration technique, condition assessment, chemical identification

## Preparation of Pozzolanic Lime Mortars for Repairing Limestones of Nemrut Dağ Monument

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This study aimed at preparing pozzolanic lime mortars for the repairs of deteriorated limestone statues of Nemrut Dağ Monument in Adiyaman Turkey. Historic mortar samples from limestone rubble masonry of Kahta Castle, a medieval structure in the vicinity of Nemrut Dağ Monument, were analyzed to serve as a model for the preparation of durable and compatible repair mortars. Physical and physicomechanical tests, optical microscopy, chemical tests, SEM-EDX and XRD analyses were used to assess the properties of the historic mortars and repair mortars.

The use of fat lime, aggregates up to 1.5 cm size and pozzolanic fine aggregates were observed in historic mortars. Laboratory grade Ca(OH)<sub>2</sub>, river sand (Cendere), standard sand and pozzolanic additives of metakaolin, fly ash, brick powder were used in repair mortar mixtures.

In repair mortars, setting was found to be predominantly due to carbonation along with pozzolanic reactions. However, abundant presence of stratlingite in mortars with added metakaolin indicated that pozzolanic reactions preceded carbonation in those mortars.

Use of pozzolanic materials increased the uniaxial compressive strength and modulus of elasticity of mortars compared with control samples. Using the same binder:pozzolan:aggregate ratio, highest increase was observed on mortars prepared with added fly ash at the end of 90 days. Durability parameters of repair mortars defined as wet to dry strength ratio were in the very good to excellent range. By using fly ash, design of lime mortars with high water impermeability and high water vapour permeability characteristics was accomplished which was important for the stone repairs.

**Keywords:** pozzolanic lime mortars, repair mortars, limestone, monument

## The development of organic consolidants for heritage Sydney sandstones

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A number of the nineteenth century heritage buildings located in Sydney, Australia, are built from locally quarried sandstone. After more than a century of natural weathering, a number of the sandstone buildings are showing signs of deterioration. The Sydney yellow block sandstones typically contain relatively large amounts of sand bound together by a clay matrix, with small quantities of iron-rich minerals. Previous studies have demonstrated that the degradation can be associated with changes occurring to the clay component at a microstructural level. A variety of consolidants have been investigated for use in the protection of different building materials and polymeric materials have been widely used to minimise the rate of decay. Polymeric composites based on organic polymers and clay may provide improved consolidations properties compared to the polymer alone. A series of polymer composites have been synthesised, characterised and tested for their ability to act as an effective consolidant for Sydney sandstones. The structure of the potential consolidants have been characterised using a range of experimental techniques, including scanning electron microscopy, x-ray diffraction, infrared spectroscopy and thermal analysis.

**Keywords:** sandstone, consolidation, polymer-clay composite, FTIR spectroscopy, X-ray diffraction, Scanning electron microscopy, Thermal analysis

## Microstructural and mechanical comparison of different nanostructured lime-based consolidant products applied on renders

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An important operation for the conservation of historical renders is the restitution of cohesion to the binder-aggregate system, based on the use of materials with aggregating properties. Inorganic consolidants are usually preferred to organic ones due to better compatibility and durability.

The aim of this work is the experimental characterization of several consolidant treatments, namely three commercial nanolime products and two combined applications of a nanolime and a limewater solution of ethyl silicate.

Nanostructured lime-based products present interesting potentialities due to their high penetration depth, homogeneous distribution and high stability.

Nanolimes were moreover applied in combined applications with limewater, a traditional compatible product, and ethyl silicate, a well-known product for consolidation intervention. The combined application could guarantee some benefits and improve the performance of these products.

Consolidant products were applied on weak lime mortar samples (prismatic samples and single mortar layer applied on bricks); these mortars were optimized by studying different binder-aggregate ratios, to simulate old lime mortar with cohesion loss.

The evolution of consolidation effects was accompanied and periodically evaluated. Physical-mechanical characterization was performed on mortar samples after and before the consolidant treatments, analysing superficial hardness and compression and flexural strength, thus the treatments efficacy. Microstructural observations and microanalyses of the consolidation products and of the consolidated mortar samples are also reported.

This work is part of a research project between several leading Portuguese institutions to establish a conservation methodology for historical renders, defining decision criteria and developing the study of specific materials for the main problems detected.

**Keywords:** Consolidation products, Compatibility, Nano-products, Historical renders

## Study of the superficial cohesion and chromatic changes of marbles treated with polymers and exposed to artificial ageing

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Taking in mind the environmental conditions to which numerous marble items, coming from the archaeological site of lasos (Milas, Muğla – Turkey) and exhibited since 1995 in the Balık Pazar, are exposed, a study of the durability of some protective treatments has been realized. Considering that these marbles, exposed outdoor in sheltered position, suffer of superficial decohesion and foliation, and that the main causes of their degradation may be ascribed to abrupt daily changes of the stone temperature which may give repeated and frequent phenomena of water condensation/evaporation, the effect of accelerated ageing simulating the real conditions has been studied.

Marble samples coming from two different quarries (Carrara – Italy, and Yatağan-Muğla - Turkey) have been used as substrate for the treatments. Two acrylic polymers (Paraloid B72 and Paraloid B67), three perfluorinated elastomers (NH, N935 and N215) and a natural polymer (animal glue) have been tested as protective agents. The specimens have been artificially aged, both in a climatic chamber, where temperature and relative humidity changes have been performed for simulating the natural decay, and under UV irradiation for simulating solar radiation. The efficacy and durability of the treatment have been evaluated by testing the water capillary absorption, the peeling resistance and the chromatic behaviour of the treated samples.

Various polymeric materials, mainly of synthetic production but also of natural source, are frequently used for consolidation or protective treatments of marbles and other stone artefacts.

**Keywords:** aggregating agents, fluoroelastomers, marble

## MOVIDA: A new tool for on-the-spot data documentation and analysis for Cultural Heritage investigations

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We present MOVIDA a new tool for the data management and analysis of scientific investigations in the Cultural Heritage field. The software has been developed due to the lack of an adequate tool able to manage all the heterogeneous data generated in a multi-technique investigation of an artwork. MOVIDA can be used as an analytical tool as the investigation is being carried out and allows for the digital preservation of all the information acquired with the different instruments and techniques.

MOVIDA is a java-based software distributed free of charge[1] that runs in the most common OS (linux, mac, windows). The software can be used to record, elaborate and analyze all the data generated in a spectroscopic investigation on the spot. All the information can be this way managed with the same application and easily consulted, compared and related to the corresponding areas of the artwork. MOVIDA has been already tested with very satisfactory results in several MOLAB[2] non-invasive in-situ investigations. It is self-comprehensive and user-friendly and can be used by all the professionals involved in the investigation and preservation of Cultural Heritage whatever their background and computer skills are.

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[2] Acc. Chem. Res 43 (2010) 728-738.

**Keywords:** Cultural Heritage, it, software, investigation, data management



**Movida**

*Movida's screenshot. Investigation by the Molab team of The Taking of the Christ, Caravaggio. National Gallery of Ireland.*

*'Project supported by Laboratorio di Diagnostica di Spoleto (Italy)'*

## The Books of Hours in the collection of the National Palace of Maфра: Study of materials and Conservation

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Three Books of Hours, from the early fifteenth century and belonging to the collection of the Maфра National Palace, were studied by an interdisciplinary team.

In this work we will present the results from the study of the materials and techniques. A multi-analytical approach disclosed the full molecular palette used to create colour and gathered new insights in the production of these lavishly decorated codices. The palette is in agreement with the dating of the manuscripts; and the profusion with which of lapis-lazuli and brazil lakes were applied with a French provenance, possibly from a Paris workshop, as proposed by the Art Historians in the team. Also, concordantly to a previous publication on greens from 15th-16th century manuscripts, we found the use of a copper sulphate type green, which was used both to create dark and light greens. Gold and silver were also lavishly applied; the silver backgrounds display an extensive degradation and darkening.

Molecular analysis was carried out by microEDXRF, Raman microscopy, microFTIR, microspectrofluorimetry and FORS. Together with the spectroscopic investigation, paints were also analyzed by optical microscopy which allows us to understand how the final colour is built up (layers, mixtures) and to better understand degradation phenomena such as the one observed in the silver backgrounds.

Finally, a full conservation condition survey of one of the most deteriorated codex was carried out and its conservation-restoration design planned, on the basis of minimal intervention concept. The interdisciplinary knowledge required to treat these artworks will be discussed.

**Keywords:** Books of Hours; Illumination; Medieval Pigments; microEDXRF; Raman microscopy; microFTIR; microspectrofluorimetry; FORS

***Books of Hours***



## Spectroscopic characterization of natural dyes for their non-invasive identification on Mesoamerican Codices

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Ancient Mesoamerican painted books represent the religious and historical legacy of the Pre-Columbian people. They were produced to record by means of hieroglyphs and ideographs the historical and religious matters. Before the Spanish conquest of Mexico, there must have been a large amount of such manuscripts, but due to the colonial persecution and destruction, only about fifteen have survived. Nowadays, thanks to the development of a variety of non-invasive and portable spectroscopic techniques, the scientific study of Mesoamerican books can be carried out, revealing information on the constituting materials and painting technique, while fully respecting their physical integrity [1]. This work is focused on the characterization of natural Mesoamerican dyes, in particular yellow dyes, that starting from the in situ experience on Pre-Columbian codices are the most difficult materials to be identified through non-invasive portable techniques [1]. Following various historical sources, four species of plants have been selected to extract yellow dyestuffs. By adsorbing dyes on clay substrates hybrid organic-inorganic pigments have been produced by analogy with the Maya blue. The dyes and pigments obtained have been studied both as powder and mock-up painting samples. UV-Vis spectroscopy both in absorption and in emission have been exploited for the characterization of electronic properties to achieve a first classification of organic dyes and pigments. For obtaining a more specific molecular speciation Raman spectroscopy has been attempted. Finally, reflection infrared spectroscopy and X-ray fluorescence have been employed to gain information on the inorganic substrates of the dyes in the hybrid pigment formulations.

**Keywords:** Pre-Columbian Mesoamerican codices, Yellow dyes, Hybrid organic-inorganic pigments, Non-invasive analysis

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## Enhancement of Manuscript Paper Materials: Restoration and Physico-Chemical Study

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Manuscripts are parts of the most valuable cultural heritage. To preserve them we must apply highly sophisticated and modern restoration techniques that are closely appropriate to maintain the integrity of their paper. The conservation-restoration treatments applied at the libraries are very useful in the protection of such heritage and in the safeguarding of any nation cultural and historical memories.

Our research project aims at protecting Moroccan writing heritage, especially manuscript paper materials. To achieve this goal, we studied the process of restoration applied in the laboratory of AL-QUARAWIYINE library of Fez, which allowed us to restore three pieces of manuscripts paper from the same library. The investigation by physico-chemical analyzes allowed us to validate the restoration process by studying the state of celluloses' fibbers of the old manuscript paper and their adhesion with those of Japanese paper used in restoration.

**Keywords:** Conservation-Restoration, Cultural Heritage, Manuscripts, Paper, Physico-chemical analysis.

## Water Adsorption Isotherms and Dimensional Stability for Ancient Ivory

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Historic ivories have a reputation for being extremely sensitive to fluctuations in relative humidity (RH) and although storage and display recommendations have been published in the conservation literature there is little theoretical basis for them. Affected ivories may exhibit warping, cracking and delamination if the RH varies too rapidly or strays beyond acceptable limits. Today the trade in elephant ivories is effectively prohibited under the 1975 CITES convention. However, carving of ivory continues to be practiced in South East Asia, but using fossil mammoth tusks excavated from permafrost deposits in Siberia as a substitute. Mammoth ivory suffers similar problems as historic ivories. Water adsorption isotherms for mammoth ivory have been developed using standard dilutions of glycerol to create different RHs. Dimensional changes in radial, tangential and longitudinal directions have been recorded. Results indicate that dimensional changes in the radial direction are most important with an increase of 6% between 60% and 90% RH. Changes in the tangential direction are 3% over the same range and this fall effectively to zero in the longitudinal direction. These dimensional changes are shown to be due entirely to adsorption of water molecules within the collagen fibrils and resulting swelling. Volumetric and gravimetric measurements of mammoth and historic ivories show that ivory has a total porosity of 19vol%. Hydrazinolysis shows that ivory has a collagen content of 29%. These measurements provide a powerful predictive tool for selecting appropriate storage conditions for historic ivories and in the controlled drying of waterlogged archaeological specimens.

**Keywords:** Mammoth, Ivory, Adsorption Isotherm, Collagen, Porosity, Dimensional Stability, RH

## A restoration methodology of Antique Maps

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Mechanical deterioration process in antique maps may be initiated by improper usage and/or storage conditions. Throughout this process, the structural characteristics of a map gradually changes and it falls out of use. Removal of factors that excite mechanical deterioration processes and consolidation of maps with new materials which bear the characteristics of original materials is a preferred approach. This article focuses on the consolidation methodology of an antique map whose support material lost its integrity due to improper usage and storage history.

**Keywords:** antique map, consolidation, conservation and restoration

## **Morphological Study of Paper Surface by SEM-EDS of the Degraded and Restored Paper Manuscript from Library Al-quarawiyine**

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The aim of our research project is to contribute to the enhancement of the cultural heritage by studying paper material of some Arabic manuscripts available at the library AL-QUARAWIYINE of Fez. In achieving this goal, we will be using the scanning electron microscopy in order to assess the state of conservation of cellulose fibres of paper. Upon completion of this experimentation, we will compare the results obtained since their degraded state until restored process taking into consideration those of Japanese paper used in restoration today. The study of the morphology of paper's surface will help us to observe the fibrous structure of the different samples. In addition, This research by means of EDS elemental analysis will certainly assist us in identifying the additives introduced into the pulp.

**Keywords:** Degraded and Restored State, Scanning Electron Microscopy (SEM), Paper manuscripts, Additives, EDS Elemental Analysis.

## Structural Analysis by Spectroscopy FTIR and XRD of Manuscript Paper Materials dating from 150, 200 and 800 years

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For many centuries, paper was the main material for recording the cultural achievements and memory of the mankind. It is entirely made up of cellulose with small amounts of organic and inorganic additives, which can identify its nature and characteristics. However, they can easily cause its gradual degradation. The development of specific analytical techniques improves the procedures for the study of heritage objects made of cellulose-based materials, especially manuscripts. FTIR spectroscopy and X-ray diffraction methods are among the most important and most effective in the characterization of paper.

The aim of our study is the chemical characterization of paper materials of three samples of Arabic manuscripts dating from 150, 200 and 800 years. The ATR-FTIR spectroscopy helped us to characterize cellulose fibres and chemical additives used during the preparation of the pulp from each sample, while the X-ray diffraction allowed us to characterize the crystalline fraction of cellulose and mineral fillers introduced in the paper.

**Keywords:** Materials paper manuscripts, ATR-FTIR spectroscopy and XRD analysis, Cellulose, Paper dating from 150, 200 and 800 years, Mineral fillers.

## Fungal deterioration of artificially aged cotton

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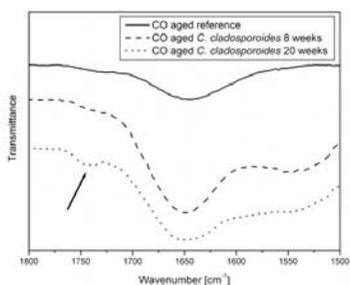
Deterioration of materials is caused by different internal as well as external factors. Biodeterioration by fungi is especially severe. Historical textiles, which have already changed by different external factors (light, relative humidity, temperature, impurities in the air) had already degraded to certain extent and are easier for enzymes to digest.

In the present investigation we analysed the changes caused by six different fungal species on cotton fibres. We used artificially aged cotton fabric, incubated with single fungal species for 8 or 20 weeks. After incubation we analysed the structure and mechanical properties of the inoculated fabrics by spectroscopic methods (Raman and Fourier transform infrared spectroscopy (FTIR)), scanning electron microscope (SEM) and tensile tests.

We compared structural changes caused by six different fungal species. We compared their influence on artificially aged cotton, to see the type, range and intensity of structural changes and mechanical properties of the inoculated fabrics. Structural changes in artificially aged specimens were initially slow, but intensified after 20 weeks. Fungi influenced the supramolecular structure and crystallinity, as observed by Raman spectroscopy. Due to depolymerisation of cellulose macromolecules, a carbonyl band occurred in FTIR spectra (Fig. 1). Structural changes influenced properties of the fibres and fabrics. Breaks and cracks as well as fibrillation of fibres were observed by SEM. Tensile properties of single threads changed, and tensile elongation in the tensile tests curves decreased.

The analyses showed intensive deterioration in inoculated aged specimens. However, different fungal species influenced fibres with different intensities.

**Keywords:** cotton, fungi, biodeterioration, fibre structure, mechanical properties



### carbonyl band

Carbonyl band appeared in the longer incubated specimens (see arrow).

## Transformations of heritage surfaces due to environmental factors

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The surfaces of cultural heritage assets can be both physically damaged and chemically changed by factors such as humidity, temperature, and pollution. The degree of stress to which they are subjected depends upon geographical position, pollution levels, type of materials used, and the success or failure of conservation attempts. This paper looks at the influence of these factors on iron, bronze and lead artefacts excavated from the site of St Mary Magdalen leper hospital, near Winchester, UK (c.1000-1150). The paper discusses preliminary results from an interdisciplinary project, which was started at the University of Winchester last year. The method involves soil samples taken from the archaeological site, in close proximity to the metal objects found. Each soil sample has been geologically classified and a number of chemical analyses have been undertaken. Carbon content, sulphate, phosphate and chlorides have been identified. The paper also includes a discussion of the corrosion products of iron, hydroxide and akageneite; the causes of 'bronze disease' and its corrosion products, malachite, brochantite and atacamite; and corrosion of lead objects. During the soil analysis process the pH value of the soil samples, and the chloride, phosphate and sulphate ion content were analysed. The conclusions so far are only tentative but it is thought that a concentration of chlorides, phosphates and sulphates in the soil may have accelerated the corrosion of all three types of metal objects resulting in the destruction of their surfaces by cracking, flaking, chipping and bursting.

**Keywords:** Archaeology, artefacts, Corrosion, Iron, bronze, lead, bronze disease, humidity, temperature, and pollution, conservation, sulphate, phosphate and chlorides, akageneite, malachite, brochantite and atacamite, cracking, flaking, chipping and bursting



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**POSTER**

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## Criteria And Indicators For Risk Assessment: A European Overview

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The present contribution is based on the work carried out within the European project Eu-CHIC, which aims at developing a Cultural Heritage Identity Card. In doing so, one relevant point is the identification and analysis of existing directives for risk assessment.

It has emerged that the most complete risk assessment methodologies have been developed in those countries where the risks are highly relevant, reflecting single country situations. Thus, it seems that there is a need to develop guidelines to be globally accepted and shared within a pan-European perspective.

The European Spatial Planning Observation Network (ESPON) has developed several maps addressing specific risks and hazards and their graphical distribution. In addition several research projects (Noah's Arc, Climate for Culture, PERPETUATE, SMooSH, FIRESENSE, etc..) have been funded in order to provide a better understanding of the impact of climate change on cultural heritage, to determine risk priorities at a country and regional level and to develop more reliable tools for historical building monitoring.

As far as risk assessment methodologies are concerned, it is relevant to mention the Risk Map approach developed by the Italian ISCR (Istituto Superiore per la Conservazione ed il Restauro dei Beni Culturali). According to this system, the risk analysis should be based on the definition of hazards, identifying the most relevant according to the geographic location of items, and the careful analysis of the vulnerability (state of conservation) of each single item.

## Synthesis and Spectroscopic Characterization of an Old Pigment

**Pınar Çakar, Ayşegül Belet**

*Restoration and Research Centre, Suleymaniye Manuscript Library, 34112 Istanbul, Turkey*

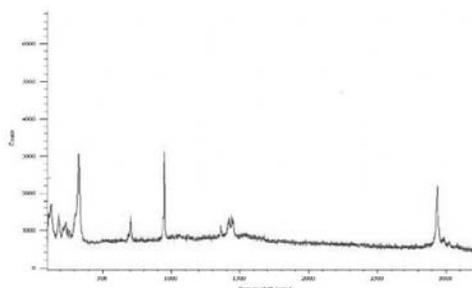
Pigment, dye and ink characterization is a crucial study in the field of manuscript conservation/restoration as well as those of other cultural heritage objects. In this study a pigment, called 'jengar', was synthesized according to an old recipe that tells the usage of it at borders framing the articles in manuscripts. The pigment product was traditionally applied on a paper from 16th century and was subjected to accelerated aging tests. Pigment was chemically characterized before and after aging by means of Raman spectroscopy. A complementary study was done with FTIR spectroscopy and optical changes within the pigment were measured with color spectrophotometer. All the analyses were performed non-destructively.

**Keywords:** jengar, ancient pigment, manuscript, pigment characterization, non-destructive, accelerated aging

### *Jengar pigment*



### *Raman spectrum of the synthesized pigment*



## Identification of inks used in 15th century Ottoman manuscript by non-destructive techniques

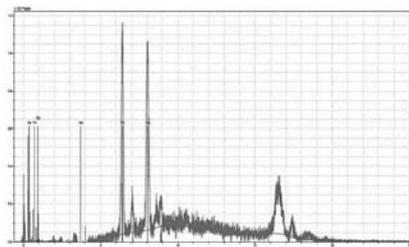
**Ayşegül Belet, Pınar Cakar**

*Restoration and Research Centre, Suleymaniye Manuscript Library, 34112 Istanbul, Turkey*

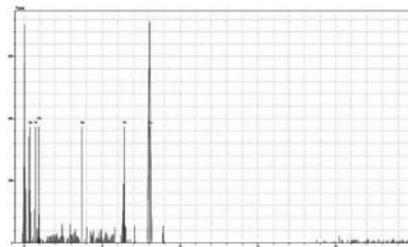
Different types of inks with different composition that varies depends of the time period or the area in which the manuscripts, books and the other written material were written were used for centuries. Iron gall ink and carbon based ink were the most commonly used inks through the western Europe to Central Asia. In this study the inks used in two different part of the manuscript dated to 15th century has been analyzed by non-destructive techniques to compare the disparity and reveal the authenticity of manuscript. The spectra recorded by Raman Microspectroscopy have been compared with both iron gall ink and carbon ink's referans spectra. XRF measurements also have been performed by micro-XRF to identify the metal content of the inks.

**Keywords:** Raman spectroscopy, XRF, iron gall ink, manuscript

*spectrum 1*



*spectrum 2*



*XRF measurement of manuscript*



## The contribution of micro-Raman and FTIR spectroscopy in art history study of wall painting from the 16th century church in Republic of Macedonia

**Lidija Robeva Cukovska<sup>1</sup>, Jehona Spahiu<sup>1</sup>, Biljana Minceva Sukarova<sup>2</sup>**

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<sup>2</sup>Institute of Chemistry, Faculty of Natural Science and Mathematics, University "Ss Cyril & Methodius", Arhimedova 5, 1000 Skopje, R. Macedonia

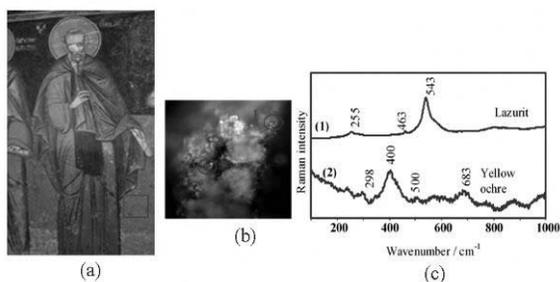
The main objective of this study is the characterization of the traditional painting technique and materials (inorganic and organic) used in the church "St. Nikola"- Monastery of Toplica, Republic of Macedonia. According to the art historians the largest part of the paintings in this church has been created during the first half of 16th century, but there is also an assumption that particular area could date from the end of 14th or beginning of 15th century. Numerous samples of pictorial layers (38 in total) from different murals were collected and analyzed by use of micro-Raman and FTIR spectroscopy.

The aim of the analyses was identifying the artistic pellet (pigments) and obtaining information about the binding medium used in the wall painting. The results of the analysis were implemented in the study of the artwork history, author's attribution and problems related to the conservation restoration issues.

Optical microscopy, as the first employed technique in this study, was applied to the previously prepared cross sectioned samples. The stratigraphy analyses revealed single pictorial surfaces, except in some samples where two pictorial layers were detected indicating re-painting.

Micro-Raman spectroscopy demonstrated invaluable contribution to the unambiguous characterization of the pigments (Table 1), allowing examination of powdered form of the paint specimens (Fig. 1), but also analyzing layer by layer of the cross sections of pictorial sequences (with the thickness of few microns). All measurements were supported by use of FTIR spectroscopy.

**Keywords:** Wall painting, micro-Raman spectroscopy, FTIR



**Figure 1**

Fig. 1. (a) Wall painting in the North chapel; marked area is a sampling position; (b) Raman microscopy image of green paint; (c) Raman spectra of paint mixture: Blue 1; Yellow -2

## The characterisation of ochres used in Australian Aboriginal paintings

**Barbara Stuart, Paul Thomas, Alexandria Hunt**

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Ochre is an important component of paint used in traditional, as well as modern, Australian indigenous art. This mineral-based material, observed in a variety of colours from pale yellow to dark reddish-brown, is mined from particular sites and is coloured by iron oxide. The source material was extensively traded across Australia in the past. The pigment has been traditionally used on a range of surfaces including rock and bark.

An understanding of the chemistry of these pigments can be used to link the source of the pigments to the geographical location of the mine used in production, thus providing archaeologists with valuable insight into ancient social practices. This study involves the examination of ochre paint specimens collected from indigenous bark and rock paintings originating in the Northern Territory in Australia. The project is being carried out in collaboration with archaeologists and conservators from the Australian Museum, Monash University and the University of Bordeaux. A variety of analytical techniques are being employed to investigate the chemical and structural properties of the ochre pigments, including infrared spectroscopy, Raman spectroscopy, x-ray diffraction, thermal analysis, scanning electron microscopy, x-ray fluorescence and inductively coupled plasma – mass spectrometry.

**Keywords:** ochre, pigment, aboriginal, Australia, rock, bark

## A study on the polychromy and technique on some Armenian illuminated manuscripts by Raman microscopy

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<sup>2</sup>Restoration Department of Matenadaran Museum of Yerevan, Armenia, Yerevan-0009, Mashtotsi Ave., 53

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A research on some micro fragments from some Armenian codices has been started with non destructive methods in order to obtain most information on the same samples. Raman microscopy was employed with a red laser. The wide gamut of colors used is expressed through the materials used in different centuries. A wide use of gilding on a preparation (assisa) made with Armenian bole together with a proper binder. Among the most frequently found pigments there are: carbon, white lead, gypsum, calcite, orpiment (with realgar), lazurite, indigo, mercury sulfide, goethite, litharge, massicot, probably mosaic gold (purpurine), azurite, minium, indigo. A green pigment probably is a basic copper sulfate with a well defined Raman spectrum. A frequent green mixture is vergaut, suitable for foliage. The techniques and materials ascertained seem to follow the indications in medieval recipe manuscripts, such as Theophilus, Heraclius, Cennini. The ink used in these codices appear always to be iron gall ink.

A wider number of manuscripts would be desirable in order to deepen our knowledge of illuminated Armenian manuscripts and to ascertain the nature of the compounds not clearly investigated.

**Keywords:** raman microscopy, illuminated manuscripts

## A Study of Chrysographic Lines in a few Surviving Folios of the Quran

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*<sup>1</sup>Codicological Laboratory Nameh Baharestan*

*<sup>2</sup>Iranian Cultural Heritage, Handicrafts and Tourism Organization*

The use of Chrysography for the purpose of book decoration in Iran and surrounding countries is probably traceable to pre-Islamic times. During the Islamic period, some of the oldest specimens of chrysography are those that are found on the folios of fragments of the Quran that have been dated to approximately the beginning of the 2nd century hijri (or 1st half of the 8th century A.D.).

In treatises that concern book decoration, not only techniques of preparing dissolved gold, but also way of making paints and inks that seem like gold, have been listed. However, no study of on the compounds used in such paints and inks had ever been undertaken in Iran.

The present study is an investigation of 24 samples of gold colors used in 12 Quran fragments that date from the Safavid and the Qajar periods in order to identify their constituting compounds. The samples were subjected to the two techniques of Scanning Electron Microscopy in Combination with Energy Dispersive X-ray (SEM-EDX)-in order to identify the elements-and the Attenuated Total Reflectance-Fourier Transform Infrared Spectroscopy (ATR-FTIR) in order to determine the organic compounds used in them. According to the results of these tests, in 12 samples gold and silver were combined in order to achieve the golden color. In 10 other samples some other metal alloy was employed. The metals that were used for this purpose were: copper, zinc, and tin. In two the samples the golden color was achieved by combining saffron and tin.

**Keywords:** Chrysography, Quran Manuscripts, Decoration, SEM-EDX, ATR-FTIR.

## **An in-situ, non-invasive XRF and Reflectance FTIR investigation of the wall paintings and mosaics at the house of Neptune and Amphitrite, Herculaneum**

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Hiba Schahbaz<sup>2</sup>, Amelia Catalano<sup>1</sup>, Katerina Posch<sup>1</sup>**

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A non-invasive, in-situ investigation of the wall paintings and mosaics at the house of Neptune and Amphitrite at Herculaneum, was carried out with the combined use of portable X-ray Fluorescence (XRF) and Reflectance Fourier Transformed (FTIR) Infra-Red spectroscopies with the objective of investigating the presence and nature of undocumented consolidants, as well as to examine the technology of the wall paintings and mosaics. The FTIR measurements of all surfaces revealed the presence of an acrylic coating allowing to date the undocumented consolidation treatment to after 1950. FTIR also revealed the use of solid Egyptian Blue tesserae both in the mosaic and in the nymphaeum.

In-situ XRF identified the nature of the chromophores and opacifiers used in the preparation of the vitreous tesserae, which are in agreement with previous studies [1]. XRF investigations of the wall paintings revealed a rich palette of red pigments: red ochre, red lead, cinnabar and realgar, specifically on the walls of the tablinum. The use of the last two pigments along with the presence of the colored glass tesserae mosaic and nympehnum within a middle class household, suggests its dwellers' uprising social standing [2].

This work has taken place in the context of the Herculaneum Conservation Project's collaborative program for scientific research and site trials that address the conservation challenges at the archaeological site of Herculaneum, Italy.

### References

[1] Inez van der Werf, et al. *Journal of Archaeological Science* 36 (2009) 2625–2634

[2] Andrew Wallace-Hadrill, "Houses and Society in Pompeii and Herculaneum". Princeton University Press, 1994.

**Keywords:** Non-invasive analysis, XRF, FTIR, Reflectance, Mosaics, Wall Paintings, Roman,

## An HPLC-DAD method for dyestuff identification. Application on textiles excavated from Faiyom, Egypt

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<sup>2</sup>Conservation Department, Faculty of Archaeology, Cairo University, Egypt

A method for the identification of alizarin, purpurin and indigotin was developed using high performance liquid chromatography with UV-Vis diode array detection (HPLC-DAD). These dyestuff components were expected to be detected in Egyptian textiles, as described next, and therefore method evaluation for these compounds was important, considering that a newly purchased HPLC system was used. The linear response of the instrument was evaluated at 254 and 288 nm for the two anthraquinones and indigotin, respectively. Good linear correlations between peak areas and concentrations were obtained; the mean value of the correlation coefficient for any of the three standards was  $R^2 > 0.999$ . The limits of detection for the three investigated compounds ranged from 0.05 to 0.08  $\mu\text{g/ml}$ .

The method was then used to analyze several microsamples, removed from significant textile fragments (1st – 3rd c. AC) which were excavated from the area of Faiyom, Egypt. The following compounds were detected in the extracts of the archaeological textiles: alizarin, apigenin, chrysoeriol, ellagic acid, indigotin, indirubin, luteolin, purpurin and rubiadin. Therefore, the following dyes were identified: madder, weld (*Reseda luteola* L.) and an indigoid dyestuff source either indigo (*Indigofera* species or other) or woad (*Isatis tinctoria* L.). Furthermore, the detection of ellagic acid suggests the use of a tannin material. The madder source is probably *Rubia tinctorum* L., because according to the HPLC chromatograms alizarin was found in quantities comparable to purpurin and furthermore, rubiadin was detected in trace.

**Keywords:** HPLC, dye, textile, Faiyom

## Identification of Materials and Pigments Used in a Mural Painting in a Historical House in Yazd

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<sup>2</sup> *Islamic Azad University Central Tehran Branch, Iran*

Malk-o-Tojar house is a historical building in Yazd, a central city of Iran. This house was built at 150 years ago, in Qajar era. The painting of this house was done by a Shirazian painter and this painting is similar to painting in houses of Shiraz. Several samples were taken from one of the niche that wasn't restored. The colors which were applied in this painting are light and dark red, green, blue, golden, yellow and pink.

Chemical tests, light microscopy (LM), X-ray diffraction (XRD), scanning electron microscopy in combination with energy dispersive X-ray microanalysis (SEM-EDX) and Fourier transform infrared spectroscopy (FTIR) were used for the characterization of the structure of pigments, binders and ground layers. Cross sections were studied by a metallurgical microscope.

According to the obtained results, vermilion, ultramarine, mixture of two metals, copper and zinc, lead oxide and verdigris were used as the pigments in this painting. Ground layer was huntite, support layer was mixture of gypsum and quartz, binder and varnish were animal glue and oil respectively.

**Keywords:** Pigment, mural painting, XRD, FTIR, SEM-EDX

## Scientific Examination of Two Iranian Historical Paintings on Wood

**Roya Bahadori<sup>1</sup>, Seyed Hamid Ahmadi<sup>2</sup>**

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*<sup>2</sup>Chemistry & Chemical Engineering Research Center of Iran,*

Painting on the wood is a public decoration of historic aristocratic houses in Iran. In this study two paintings on wood of historical houses in Shiraz and Brojerd for Qajar era (19 century) were studied. The first painting on wood was in the ceiling of Shafiee Ardakani's house in Shiraz in south of Iran. Blue, pink-red, dark and light green, yellow and white were used in this painting. The second painting on wood was the painting of Birjandi's house in Brojerd in center of Iran. This painting was done on small pieces of wood and then, they were fixed on the wall. Black, red, gold, light and dark green, white and blue colors were used In this painting.

Instrumental techniques were used for indentificating of pigments and different layers of both paintings, due to trace amounts of the samples. X-ray diffraction (XRD), Fourier transform infrared spectroscopy (FTIR) and scanning electron microscopy in combination with energy dispersive X-ray microanalysis (SEM-EDX) were used for the characterization of each specimen taken from painting.

According to the obtained results, in the first painting, red lead, basic copper acetate and gypsum were characterized. Also, existence of gypsum, animal glue and oil has been proved in ground, binding and varnish layer of painting respectively. In the second, red lead, copper and zinc alloy, Verdigris, Prussian blue and basic lead carbonate were used. White lead and gypsum were used as ground layer and oil was used as binder and varnish.

**Keywords:** Painting on wood, pigment, XRD, FTIR, SEM-EDX.

## Raman spectroscopy and Py-GC/MS applied for the identification and characterization of synthetic organic pigments

**Marta Anghelone<sup>1</sup>, Dubravka Jembrih Simbürger<sup>1</sup>, Valentina Pintus<sup>2</sup>  
 Manfred Schreiner<sup>1</sup>**

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<sup>2</sup>*Institute of Chemical Technologies and Analytics, Analytical Chemistry Division, Vienna University of Technology, Getreidemarkt 9/161, A-1060 Vienna, Austria*

The Institute of Science and Technology in Arts, Academy of Fine Arts of Vienna, owns a rich collection of paint materials from the 19th and 20th centuries, including inorganic and organic pigments. In the past, the pigments were subjected to extended researches, employing different techniques (i. e. XRF, XRD and FTIR), in order to compile a satisfactory reference database. Recently, the database was extended by including spectra obtained by Raman spectroscopy applied to synthetic organic pigments, focusing on phthalocyanine blue.

For optimizing the quality of the spectra, several measuring parameters were selected and compared. Three different lasers, 532nm, 632.8 nm and 785 nm, with different laser powers at the sample were used. Thus, differentiation between copper and metal-free phthalocyanines, in the form of pigment and lake, was possible. Furthermore, the potential process of photodegradation of these pigments was studied by calculating peaks intensities at 1530 cm<sup>-1</sup> in correlation with the measuring time.

The measuring parameters selected were finally used to perform  $\mu$ -Raman spectroscopy on real samples and their cross-sections. Subsequently modern mural paintings from San Sperate (Sardinia), dated from the 1960s, renowned for their artistic, cultural and political value, were sampled and cross-sections were prepared. After observing the stratigraphy of the paint layers by optical microscopy in visible and UV light,  $\mu$ -Raman measurements were carried out for the pigment identification. Furthermore, analyses with Py-GC/MS revealed that as binding media copolymers of PVAc/VeoVa, 2-EHA/MMA and Styrene-nBA were used by the artists.

**Keywords:** Raman spectroscopy, phthalocyanine blue, Py-GC/MS, synthetic organic pigments

## HPLC-DAD Natural Dye Analysis on Carpets from National Palaces Museum (Yıldız Palace-Chalet Kiosk) Istanbul-Turkey

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<sup>2</sup>Faculty of Pharmacy, Department of Pharmaceutical Toxicology, Marmara University, 34668 Haydarpasa-Istanbul, Turkey.

Carpets in museums are primary evidence for applications, area and time of productions, methods of dyeing, chemical compositions of the natural dyestuffs, dating, trade, etc. The present study aims to identify the colouring compounds contained in microsamples removed from selected carpets. The samples were provided by the National Palaces Museum (Yıldız Palace-Chalet Kiosk, Istanbul, Turkey). The wool microsamples were treated with a solution mixture of H<sub>2</sub>O:MeOH:37%HCl (1:1:2 v/v/v), for a 8 min. at 100 °C. Analyses were performed using high-performance liquid chromatography (HPLC) with diode-array detection (DAD) at 255, 268, 276, 350 and 491 nm. Separation was carried out using a Nova-Pak C18 analytical column with dimensions 150 mm x3.9 mm, 4 µm. Gradient elution was performed using two solvents consisting of A: 0.1% TFA in water (v/v) and B: 0.1% TFA in acetonitrile (v/v), as described previously. Natural dyes of plant origin-alizarin, purpurin (components of *Rubia tinctorum* L.), ellagic acid and gallic acid (components of *Quercus infectoria* Olivier or *Quercus ithaburensis* Decaisne) and of insect origin-carminic acid (component of *Dactylopius coccus* Costa) were identified.

**Keywords:** Carpet, natural dyes, HPLC

## Identification of Natural Dyes in Historical Writings by High-Performance Liquid Chromatography with Diode-Array Detection

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Dyestuffs used in dyeing of textiles, inks, icons, historic maps, printed documents and paintings are made from natural sources such as plants, insects and mineral sources. These dyes include mainly flavones, anthraquinones, indigoids dye and relative compounds. The identification of organic colorants used in writings is an important information source for the restoration and conservation of the historical documents. In this study, organic colorants contained in tiny ink samples (1-2 mg) taken from the damaged areas of manuscripts were investigated using High Performance Liquid Chromatography coupled to a Diode-Array Detector (HPLC-DAD) [1]. Extraction from samples was carried out with HCl solution containing methanol. As the main individual chemical component of the natural dye including ellagic acid was found.

Scanning Electron Microscopy -Energy Dispersive Spectrometry (SEM-EDS) was used to examine coloured ink samples. SEM-EDS results indicated elemental composition of the samples: the presence of mercury sulfide (HgS), iron salts (containing traces of copper) as the components of the ink.

Reference:

[1]. Yurdun T, Karadag R, Dolen E, Mubarak M.S. (2011). Identification of natural yellow, blue, green and black dyes in 15th-17th centuries Ottoman silk and wool textiles by HPLC with diode array detection. *Reviews in Analytical Chemistry*, 30(3-4): 153-164, DOI: 10.1515/REVAC.2011.101

**Keywords:** Ink, paper, natural dyes, HPLC

## The painting technique of Macedonian painters: Py/GC/MS and GC/MS based techniques for the analysis of organic materials used by Dicho Zograph workshop

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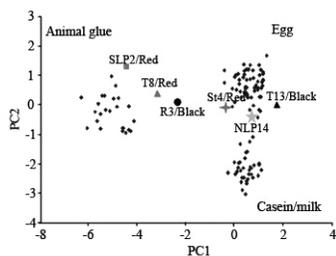
The present work comprises characterization of the 19th century paint materials used by the iconographer Dicho Zograph in the wall painting from four sacral monuments in the Western part of Republic of Macedonia. First indication for the presence of oily and proteinaceous materials was obtained by using solubility tests and histochemical staining test. In order to unequivocally determine the organic materials used as binding media, Pyrolysis Gas Chromatography-Mass Spectrometry (Py/GC/MS) and Chromatography-Mass Spectrometry (GC/MS) were carried on 11 selected samples.

A combined GC/MS analytical procedure was used for the simultaneous identification of glycerolipids, proteinaceous materials, plant and animal resins, as well as natural waxes in seven samples. The procedure is based on a sample multi step chemical pre-treatment (solvent extractions and microwave-assisted chemolysis) that is able to separate the various organic components into two different fractions: amino acid and lipid-resinous fractions.

Binding media analysis showed a rich use of organic material in the painting. Results show that Dicho's Hermeneia painting technique was based mainly on the use of proteins though seldom a drying oil was also utilised. Interestingly, results also evidenced the restoration processes undergone by the churches and the materials used for restoration purposes. Micro Raman spectroscopy data, highlighting the use of a rich palette of pigments, will be also provided in order to complement the data on the painting technique of Dicho Zograph.

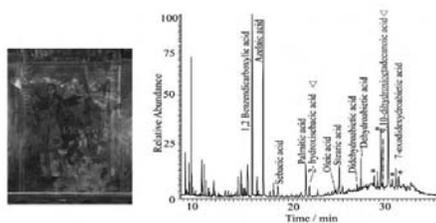
**Keywords:** wall painting, binders, pigments, Py/GC/MS, GC/MS, micro Raman spectroscopy

Figure 1



PCA score plot of the proteinaceous fraction of samples analyzed using GC/MS.

Figure 2



Total ion chromatogram of the lipid-resinous fraction of sample St4/Red. Fatty acids and pine resin markers are indicated. (\*) unknown peaks corresponding to pine resin related compounds; (∇) peaks corresponding to long chain hydroxyl acids.

## An Application of FTIR to Aging Process Assessment of the Beeswax-Treated Paper Heritage in Korea

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Jeong Hye Young**

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The Annals of Joseon Dynasty is one of important paper cultural heritage in Korea registered as "The Memories of the World" by the UNESCO. Among the 1187 volumes of the Annals, 475 volumes were beeswax-treated for long-term preservation but have contradictorily been more damaged than the untreated ones. In this study Fourier transform infrared spectroscopy (FTIR) analyses are carried out for the dry heating-aged beeswax-treated volumes after duplicating to understand differences in various aging processes in different partitions of the original beeswax-treated volumes, and a reliable Oxidation Index (OI) defined as a ratio of peak area of each carbonyl region to that of methylene region is also suggested and validated. FTIR analyses reveal that various carbonyl compounds including a conjugated carbonyl, ketone, aldehyde, carboxyl groups are produced in the aging process. It is observed that OI by conjugated carbonyl compound on the center portion of middle pages in the beeswax-treated volumes is higher than that on top pages; and that the reversed patterns are found in cases of OIs by ketone, aldehyde, and carboxyl at initial aging stage, but at the later aging stages, OIs by ketone, aldehyde, and carboxyl higher on top pages. These results indicate that at the early stage, the aging by oxidation was more active on the outer portion of the beeswax-treated volume, while as the aging proceeds with time, the inside portion is more vulnerable to serious damages by oxidation.

### Acknowledgement

This study was supported by the National Research Institute of Cultural Heritage, Korea.

**Keywords:** FTIR, Oxidation Index (OI), Beeswax-Treated volume, Aging process

## Characterization of archaeological glass beads in Thailand: Hor-Ek prehistorical site

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**Pisutti Dararutana<sup>5</sup>**

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In ancient time, glasses have been used in ornaments and decorations in Thailand for several hundred years as seen by archaeological evidences such as glass beads and decorative glasses. In this work, the blue and red colored glass bead samples from Hor-Ek prehistorical site (Nakhon Pathom Province, central Thailand) were collected to characterize their compositions using SEM-EDS and PIXE. Cu K-edge XANES of the selected samples was also carried out in fluorescent mode in order to determine the oxidation state. It was found that they were LMHK-based glass. The blue and red tonalities were affected from copper with different oxidation states. These glass beads have shape and composition as similar as those from other sites both in Thailand and foreign. It was demonstrated for the long distance trade or exchange network of the ancient time.

**Keywords:** glass bead, Hor-Ek, SEM-EDS, PIXE, XANES

## Characterization of 19th century Daguerreotypes by nondestructive analytical techniques

Marília Peres<sup>1</sup>, Luís Dias<sup>2</sup>, José Mirão<sup>3</sup>, Anabela Gomes<sup>1</sup>, Estela Jardim<sup>4</sup>  
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The Daguerreotype is considered by most of the historians of photography the first invented technique to produce a permanent image using a photochemical reaction. It was developed in France in the decade 1820-1830 by Louis Jacques Mandé Daguerre (1787-1851) and Joseph-Nicéphore Niépce (1765 – 1833). Daguerre presented this new photographic process to the Academie des Sciences de Paris in the session of 7th January 1839. In summary, the process involves the polishing of a silver-coated copper plate and its sensitization with chloride or iodine, exposure to sunlight followed by the image development with heated mercury. The image was then fixed with sodium thiosulfate and a positive photographic image was obtained.

In order to study the morphology and the degradation status of some daguerreotypes, Optical Microscopy (OM) and Variable Pressure Scanning Electron Microscopy (VP-SEM) techniques were used. Local chemical composition was evaluated by in-situ nondestructive spectroscopic techniques such as X-Ray Fluorescence Spectroscopy (XRF) and Energy Dispersive X-Ray Spectrometry (VP-SEM/EDS). This multianalytical approach includes important tools for the characterization of the daguerreotype process as well as for issues related to the restoration and conservation of historical photographic plates.

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**Keywords:** Daguerreotype, X-Ray Fluorescence Spectroscopy (XRF); Variable Pressure Scanning Electron Microscopy (VP-SEM); Energy Dispersive X-Ray Spectroscopy (EDS)

## Material study on a Liturgical Cope from the 16th century belonging to D. Teotónio of Braganza, a Portuguese Archbishop

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Teotónio of Braganza (1530-1602) was the fifth son of Duke Jaime I of Braganza and Joana de Mendonza. The Braganza's were the most important of the Portuguese noble families, which became the last dynasty of the kingdom. Teotónio received at his father's house an exquisite education and later he travelled through Europe. Member of the Society of Jesus (1549), he held several ecclesiastical positions, being the most important of all, Archbishop of Évora. He founded in his archdiocese several monasteries, convents and hospitals. Under his governance, the archbishop's palace and the cathedral suffered renovation and restoration works to where he ordered new sets of ecclesiastical vestments and liturgical objects.

In this work, a liturgical cope dating from the end of the 16th century and belonging to the Évora Museum (ME 172/1) was studied. The cope was cut in a semicircular shape, presenting a flat, stiffened shield-like hood, attached to the shoulders below a wide orphrey, hanging far down the wearer's back. The carmine and yellow silk brocade with silver wire presents natural motifs that follow the vegetable, animal and human forms. The velvet orphrey, rich in silver and golden silver threads, was also decorated in a vegetable pattern, with tulips, artichokes and pine cones.

Dyes identification was done by LC-DAD-MS. Optical microscopy and SEM were used for fibres morphological characterization and evaluation of metal threads degradation products. Mordant analysis and metal threads composition were studied by SEM/EDS. With the purpose of restoration works, evaluation of biological colonization was also done.

**Keywords:** liturgical cope, metal threads, material study

## XRF-analysis of Glagolitic manuscripts

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A current research project (The Enigma of the Sinaitic Glagolitic Tradition) aims to the edition of hitherto inedited manuscripts of Glagolitic manuscripts but also to the comparative analysis of several manuscripts in Glagolitic script from the 10th-11th/12th centuries. In order to classify analogous objects from various collections, used materials were identified to find hints on differing recipes for inks and parchment-manufacturing as well as to evaluate storage-conditions influencing the stability of the objects. As the manuscripts are very sensitive due to their age as well as due to the fact that they were in intense use, it was highly aimed that collecting data of used writing inks and parchments must involve non-destructive techniques. This means that methods with the ability to measure in-situ are needed. XRF covers the demands mentioned and can be applied under specific conditions also as air-path systems. A handheld XRF-device of Spectro Analytical Instruments, type xSORT, designed in the first place for commercial application, was used for non-destructive in-situ analysis, as it offers several advantages: its lightweight and the fast measuring procedure as well as the detection of light elements with Z even below 20, such as Mg, Al, Si and P.

The poster presents preliminary results of the measurements done within the current project including also the evaluation of the qualitative results from different XRF devices used in former and current projects in order to achieve further adaption of the hand-held instrument for the analysis of objects of art and archaeology.

**Keywords:** X-ray fluorescence analysysi (XRF), analysis of manuscripts, handheld XRF

### *handheld\_XRF*



*XRF measurement of a manuscript in the St. St. Cyril and Methodius National Library (Bulgaria) using the handheld device with a tripod and the museum's adapter, which enables to adjust measuring position according to demands of the object.*

## The Évora Inquisition Banner – Historical and Material Characterization

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The Inquisition Banner, belonging to the Évora Museum (ME 171), is an important item from the Portuguese cultural and religious heritage. It was used from the seventeenth until the nineteenth century, but nowadays it is in a very poor conservation status. The severe damage of the red damask is probably due mainly to mechanical degradation. However, microbial and fungal degradation have also contributed for the overall poor conservation status of the Banner.

The Banner has a two-faced central medallion surrounded by a baroque frame, with a golden strip at the top and the inferior part split into two triangular flaps. The more detailed needlework in the frame can be distinguished by contrast with less exquisite embroidery of the medallion. The obverse medallion face depicts the Inquisition coat of arms with a cross at the centre surrounded by a sword on the left and an olive tree on the right. The reverse face depicts the image of Pedro Arbués, who was the provincial inquisitor of Aragon, murdered in 1485, carrying a martyrrium palm.

Samples were collected from different points of the embroidery work, representing the different metal thread typologies and coloured silk yarns. Sterile cotton swabs were used to recover the microbiological contamination. Optical microscopy and SEM were used to study morphological details of the metal and silk threads and biological colonization. SEM/EDS were used to determine the chemical composition of the metal threads. Natural dyes used in the silk embroidery were identified by LC-DAD-MS after chromophore extraction with EDTA/DMF.

**Keywords:** Inquisition Banner, metal threads, material characterization

## Study of natural organic colourants in the Andean textile collection of the British Museum

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The British Museum houses one of the largest collections of Andean textiles outside Peru comprising pieces ranging from the Early Horizon through the Colonial Period. A detailed study of the natural organic colourants and dyeing technologies is currently being undertaken to complement the “traditional” study of iconography and weaving techniques.

Investigation of the organic colourants at molecular level is essential to fully characterise the diverse range of biological sources and manufacturing technologies employed. The analytical approach developed in this research is based on HPLC with “soft” extraction techniques in order to preserve the most sensitive classes of colourants. The method has also been optimised to ensure extraction of all colourants from a single sample and for the small sample sizes typically available from museum objects. This optimised analytical protocol has been tested on a large range of fibre samples dyed with plant-derived colourants recently collected in South America, providing a comparative dataset for the study of the archaeological textiles.

This paper will present the results of a literature review of Andean natural organic colourant sources and the creation of the reference collection of colourants. The results obtained for a number of archaeological samples will also be presented. While addressing key questions about Andean textile production, this paper will demonstrate the wider potential of colourant analysis to explore art historical, archaeological, anthropological and cultural questions. This research will additionally provide new analytical methodologies, better understanding of deterioration processes and improve conservation and display strategies.

**Keywords:** Organic colourants, Andean textiles, dyeing technologies, HPLC, British Museum

## The influence of the SERS substrate on the sensitive identification of fresh and old varnishes using Raman spectroscopy

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The use of Surface-enhanced Raman scattering (SERS) as advanced analytical method developed for the characterization of the materials used in works of art and cultural heritage is a hot topic that attracted increasing attention of scientists and museum researchers.

The influence of the size and morphology of the substrate used in SERS analysis was investigated, in order to ensure a rational design of the nanoparticulate surface for applications in the analysis of fresh and old varnishes.

Several metallic nanoparticles (gold and silver) were used as SERS substrates and analytical procedures were developed and evaluated with two frequently used varnishes.

The SERS active substrates were prepared from nanoparticles with various sizes and shapes (spherical, prism, rods, flower-like nanostructures). Hybrid nanoparticles with polymeric core and metallic shell deposited on the top were used to prepare more efficient and inexpensive SERS surfaces. The nanoparticles and the nanostructured films were characterized by using DLS, SEM and TEM.

The Raman spectra of two commercial varnishes were recorded on various substrates, and the influence of the characteristics of the nanostructured substrate on the intensity of the signals is evidenced. The effectiveness of surface-enhanced Raman spectroscopy (SERS) using hybrid polymeric-metallic nanoparticles (based both silver or gold) has been successfully demonstrated for the identification of varnishes as very low concentrations.

**Keywords:** SERS, nanoparticles, varnish

## Microanalysis of Persian coin found in Thailand

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Elemental analysis of silver coins provides valuable information. In this work, the silver coin belonging to ancient Persian coin dated around the 5th Century A.D. which has been found at archaeological site in Pattani, southern province of Thailand was characterized non-destructively for the first time by using multi-element XRF techniques such as EDS and PIXE. It was found that silver was the major element. Chromium, iron, copper and zinc were found to be the trace elements. These revealed the commercial interaction between Thailand and Persia was more than 1,500 years ago.

**Keywords:** silver coin, Pattani, SEM-EDS, PIXE

## Statistical methodology in numismatic studies - the case of Dacian gold and silver coins

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This paper is a first attempt of statistical analysis of Geto – Dacian gold and silver coins based on their elemental composition, determined by XRF. The study discusses results obtained for a hoard of 143 gold Koson type coins (37 with monogram and 106 without monogram) recuperated by the Romanian Authorities in the autumn of the year 2009 and one of 202 silver coins: Kosons and Macedonia Prima tetradrachms.

Two statistical methods, Cluster Analysis (CA) and Principal Component Analysis (PCA), were employed in the study. Cluster analysis was used for grouping the coins and for comparison with other contemporary emissions. The results revealed differences and analogies between the emissions based on their alloy composition.

From the correlations obtained using the PCA we can formulate several hypotheses regarding the source of the material employed: e.g in the case of the gold Kosons without monogram, the Ag-Cu correlation can point to the proximity of the primary source of the alluvial gold used for manufacturing. PCA correlations can also be used to expose manufacturing aspects: e.g a positive correlation between Cu and Pb found in silver coins indicate the deliberate addition of copper for debasement and the addition of lead for decreasing the melting point of the alloy.

**Keywords:** coins, elemental analysis, cluster analysis, Principal Component Analysis

## **Chemical Characterization of Bronze Samples using Elements determined by Inductively Coupled Plasma-mass Spectrometry**

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In this study, More than 200 bronze objects recovered from the Bronze Age Yinxu site have been analyzed using inductively coupled plasma-mass spectrometry (ICP-MS). The Yinxu site is located in Anyang City, Henan Province, China. This site is large settlement site of the Shang dynasty, and it is dated to 1250~1050B.C. The site is currently known with the earliest Oracle, the largest palace building, the group of ritual bronzes, and bronze smelting workshops, and is so far to have confirmed as the site for the largest capital of kingdom. Bronze objects found in Yinxu site have been analyzed for their elemental composition, In order to answer the archaeological question of a local copper and lead supply, a database for elemental composition of copper artifacts has been established. This paper presents analytical results of the more types of Yinxu bronzes objects. The archaeological context of Yinxu bronzes and their significance for the archaeometallurgy research of the Late Bronze Age are discussed as well.

Keywords: Yinxu site, Bronze, Late Bronze Age, ICP-MS, elemental composition, China

## XRF Analysis of Nine Renaissance Nuremberg Trombones

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Nuremberg was from the beginning of the 16th century till the end of the 18th century the centre of brass wind instrument making par excellence. The high-quality standards achieved by the Nuremberg renaissance brass wind instruments makers are testified by a group of ten preserved trombones made between 1551 and 1598. One of the issues on this group of instruments is related to the material they were made of. Scholarly references stay brief on historical brass alloys as they apply to these pre-1600 brass wind instruments; consequently many questions arise as to the material one should use in making reproductions of such instruments. X-ray fluorescence (XRF) analysis has proven to be a useful method for the determination of the chemical composition of a brass alloy and has been used by several scholars for this purpose.

This poster presents the results of a systematic X-ray fluorescence analysis of a group of nine of the earliest surviving Nuremberg trombones. The measurements had been carried out by a handheld XRF Spectrometer [1], which is equipped with a special museum's adapter to guarantee a secure and repeatable setup. With the included software it was possible to quantify these measurements and therefore classify the different pieces of the samples.

With the help of these measurements a characteristic alloy has been identified which differs considerably from the material used in modern reproductions of Renaissance instruments. Additionally it was possible to identify the replaced pieces in the samples.

[1] Spectro Analytical Instruments GmbH., Kleve, Germany. [www.spectro.com](http://www.spectro.com)

**Keywords:** X-ray fluorescence analysis (XRF), Renaissance trombones



**Image 1**

*The handheld spectrometer on the museum's adapter during analysis of a Renaissance trombone*

## Interdisciplinary Studies in Examining Historical Artifacts

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Istanbul Restoration and Conservation Central Laboratory Directorate, that serves under Ministry of Culture and Tourism - General Directorate of Cultural Heritage and Museums, carries out restoration and conservation works of many concrete cultural heritage remains that are consisted of organic and inorganic substances. Our institution carries out the advanced analysis and examinations it needs for choosing the right method and appropriate materials during the condition diagnostics phase or protection/maintenance practices for historical artifacts with various laboratories that support the studies. As a result of the detailed documentation practices for historical artifacts that are handled with and interdisciplinary laboratory analysis that are within the field of archeometry, historical events that are related to these artifacts may also be enlightened. For instance, 4 linked metal pieces which belongs to the Chain of Golden Horn and is transferred to our laboratory in 2010 has been filmed using X-Ray and Gamma ray in TAEK Çekmece Nuclear Research and Training Center NDT Laboratory and its link forging techniques has been revealed. Different samples from the chain are also analyzed in ICP-MS device in the same center and identities between them are studied.

Inner mechanism and corrosion monitoring procedure that is widely required in conservation works has given successful results in our examinations. A deposit covered bronze coin and metal powder flask, that are among the artifacts sent from Directorate of Erzurum Museum in 2011, has been film with radiographic inspections. Elemental structure of the bronze coin has been learned using non-destructive WDXRF device. Powder flask is also analyzed with non-destructive PTXRF device. As a result of the inspection bronze coin is understood to belong Byzantine period and carry an "M" symbol, the object defined as powder flask is understood to include a mechanism. Sample taken from the liquid oil found in this object is inspected with NMR method in Yıldız Technical University Chemistry Laboratory.

Another object that is studied in our laboratory is a cut hand and textile finding that covers the hand. The piece that belongs to the hand has been examined with experts from Medical and Forensic Anthropology Foundation (MEDADER) and Computerized Tomography and DNA (mitochondrial) analysis have been carried out in this context. It has been understood that the hand in question belonged to an adult male between 30-35 years old, member of K Haplogroup and was separated from the body as a result of being cut.

Unidentified textile finding that was transferred to the laboratory in brunt condition and a banner that is registered to Museum of Shrines Directorate inventory have been examined by being analyzed in Turkish Cultural Foundation Natural Dyes Research and Development Laboratory in 2012. In the inspection carried out, it has been found that the burnt textile finding is plain weave (B 1/1), its warp and weft thread density is 32 cm/fiber. Inspection provided the information that warp thread winding direction is "S" and weft thread winding direction is "Z". A colorant inspection has been carried out with HPLC (High Performance Liquid Chromatography) that is used in the same center and the textile in question is learned to be raw cotton and do not include any organic dyes according to this inspection. When the sample taken from the banner tassel is inspected, it is learned that material was produced from silk and it took its red color from cochineal as carminic acid was used in it.

In poster notice; interdisciplinary laboratory inspections that made in parallel with protection and maintenance works that are carried out on our historical artifacts which are captured as a result of the digs or protected in museums, are included with some sample studies.

**Keywords:** X-Ray, WDXRF, PTXRF, ICP-MS, Tomography, DNA, Textile Analysis

## Optical sensor for the formic acid detection prepared through the immobilization of colorimetric dyes in a polymeric matrix assembled by the layer-by-layer method

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Organic pollutants such as formic acid, acetic acid and formaldehyde, released by some materials (mainly wood) used for storage and display, can play an important role on the alteration of glass structure due to the alkali leaching process. It has been determined that formic acid when present inside storage or display cabinets is the one that affects most glass integrity, accelerating and deepening the alkali leaching from the silica matrix. As this situation can affect many museums on their glass collections, monitoring this compound would be of great importance for indoor preservation.

A new optical sensor based on layer-by-layer (LbL) electrostatic self-assembly process is under development to identify indoor formic acid based on the immobilisation of chemo-responsive dyes in polymeric structures. The sensors are based on an optical response resulting from the reaction between immobilised dyes and the referred organic pollutant. As far as we know a simple and low-cost device for detecting the presence of formic acid in situ are still required to help in the preservation of cultural heritage objects.

Several pH indicator dyes were incorporated in between the polymeric layers of LBL assembled films. The UV-Vis absorption spectra of the dyes in polymeric matrix and in aqueous solutions are similar although the pKa values of the dyes are shifted relative to their values in aqueous solution. Studies on reversibility and reproducibility of the sensors were performed and detection limits were determined through experiments simulating the acidity environment by measuring concentrations of acid formic vapours using passive samplers.

**Keywords:** Layer-by-layer, Optical sensor, Formic acid, colorimetric dyes

## The review of Historical and Technical studies of ancient bronzes

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The use of copper back to the Neolithic period, ten thousand years ago. in the millennia that have followed, the alloying of copper to make bronze and the employment of copper compounds as pigments have been central aspects of the human use of metallic materials. the first undisputed evidence for human exploration of native copper deposits comes from the aceramic Neolithic site of Caynu Tepesi in southern Turkey, where beads of malachite and native copper were found that date back 8150 to 9250 years before the present day, in addition worked copper dating from the eight to the seventh millennium has been found in both Anatolia and Mesopotamia. In Iran Plato the first evidence of using copper refers to Tape Alikosh nine to seven millenniums, with finding needles and other objects.

This article is a review of studies and examination a bout ancient copper and bronzes. the condition of corrosion in different environments and corrosion products and patinas,also the case studies about corrosion in bronze statues in the open spaces. The importance of metallography as a useful method in determining the micro structure of ancient metals and the technical studies, will be mentioned by a case study on a piece of historic bronze of Iran and a review a bout metal working in ancient Iran and Lurestan bronzes, dating problems. In articles review, the technical studies about ancient bronzes and modern analytical methods, corrosion products, deterioration process and metallurgy in Mesopotamian sites,is explained.

**Keywords:** Ancient bronzes, Historical study, Technical study

## Infrared reflection absorption (IRRAS) and Raman spectroscopy to study early stages of atmospheric silver corrosion

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Silver had a profound influence on the evolution of human societies and is still central to many technologies. Valued as a precious metal it was used for objects of our cultural heritage such as statues, jewellery, currency coins and silverware. Exposed to different atmospheres silver items can be affected or even destroyed by the interaction with the ambient atmosphere. Conventional atmospheric parameters that affect silver comprise weathering factors (temperature, moisture, solar radiation, wind velocity etc.), air pollutants (H<sub>2</sub>S, SO<sub>2</sub>, CO<sub>2</sub> etc.) and aerosols. As our cultural heritage is inherited from the past generations, unique and irreplaceable, it is our responsibility to protect this cultural property for future generations. Therefore, a fundamental understanding of the chemistry occurring is needed to control material degradation in the near future.

Highly surface sensitive methods have been applied to investigate in-situ the atmospheric corrosion behaviour of silver exposed to controlled atmospheres containing different amounts of relative humidity (%RH), SO<sub>2</sub>, H<sub>2</sub>S, CO<sub>2</sub> and O<sub>3</sub>. Additionally, the influence of photo catalytic reactions caused by UV-light has been investigated. In order to obtain information concerning the chemical reactions and the species formed on the surfaces, in-situ IRRAS and Raman spectroscopy were applied. A Quartz Crystal Microbalance (QCM) was used for gravimetric information of changing chemistry on the sample surface. Two of these in-situ methods (IRRAS and QCM) were already combined in a self-designed and self-built environmental cell allowing simultaneous time-resolved measurements. These degradation studies are indispensable for developing strategies to reduce or even avoid such degradation reactions.

**Keywords:** silver, atmospheric corrosion, in-situ, IRRAS, Raman

## Chemical simulation of a decaying process observed in weathering steel sculptures exposed to an urban atmosphere

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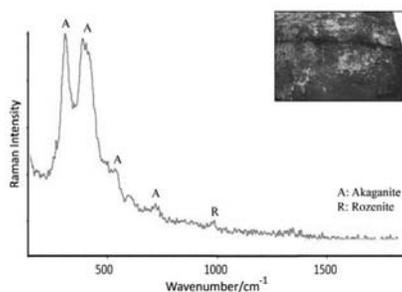
Weathering steel is a self-protecting material which develops a rust layer to achieve protection against the atmospheric corrosion. However, the protective iron oxide layer is not enough in some environmental conditions. In urban environments the surface of the steel can be attacked by acid gases, producing compositional changes and detachments on the structure and surface.

In this work it was modeled the detaching process (chemical degradation reactions) observed in some weathering steel sculptures exposed to the urban atmosphere of Bilbao city. An integrated analytical method was carried out. For elemental analysis a SEM-EDS system was used, whereas for molecular analysis a Renishaw RA100 Raman spectrometer provided with a 785 nm laser was used. The thermodynamic simulation was carried out with the MEDUSA software.

In the weathering steel surfaces by means of Raman spectroscopy, magnetite (Fe<sub>3</sub>O<sub>4</sub>) was detected, as well as calcium carbonate (CaCO<sub>3</sub>). In the EDS mapping it could be seen that the deposition of calcite over the steel surface was high. Applying the acid conditions of the Bilbao atmosphere, the chemical system was studied thermodynamically, and it could be seen that when the concentration of SO<sub>x</sub> increases, calcite starts to transform into different forms of calcium sulphate. Moreover, with long exposures to SO<sub>x</sub>, iron sulphate starts to form. Different hydrated calcium sulphate phases were determined by Raman spectroscopy, as well as iron sulphate. The role of these compounds in the detachments is discussed.

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**Keywords:** CORTEN Steel, pollution, Raman spectroscopy, rozenite, sculpture



**Figure 1**

Figure 1. A Raman spectrum of an iron oxide (akaganite) and an iron sulphate (rozenite). On the top, it could be seen a photograph of a white crust on a weathering steel surface.

## **Microbiodeterioration study of Anselmo Pineda collection saved by National Library of Colombia: Evaluation and Selection of Chemical Control Products**

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WePaper is subject to numerous factors of biotic and abiotic degradation, which can cause irreversible lost of important documents and works of graphic art, consolidated as heritage. Microorganisms, especially fungi play an important role on biodeterioration of paper and other materials of historical importance. This work aimed to study biodeterioration process caused by fungi in documentary units from Anselmo Pineda collection saved by National library of Colombia and to evaluate chemical agents for controlling biodeterioration in two phases: first in response to elimination of microorganisms and evaluating possible physico-chemical changes generated.

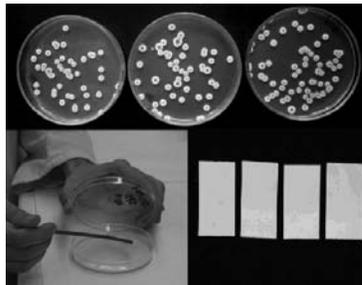
Documents were classified into four biodeterioration levels depending on the intensity and coverage of microorganism growth indicators. The isolation of filamentous fungi and yeasts followed sampling method in PDA and DG18 medium for xerophilic fungi. We obtained 57 isolates, identified by microscopy and taxonomic keys. The most common genera isolated were *Penicillium* and *Aspergillus* followed by *Cladosporium*, *Paecilomyces*, *Trichoderma*, and *Rhodotorula*. Evaluation of chemical agents followed dilution-neutralization methodology, chromatic change assessment and quantification of reducing sugars by technique of 3,5-dinitrosalicylic acid as an indicator of cellulose degradation. Laboratory tests showed Quaternary Ammonium (QAS) as antimicrobial agents effective against microorganisms tested. Enilconazole showed activity against filamentous fungi and Gram positive bacteria, and Ethanol 75% is only effective on fungi. Regarding the physical-chemical tests, we found that QAS generates color changes on manual paper and no chemical degradation of paper was detected. Results of this study contribute to development of documentary heritage conservation programs.

Keywords: Biodeterioration, Fungi, Paper, Control, Chemical products, Conservation.

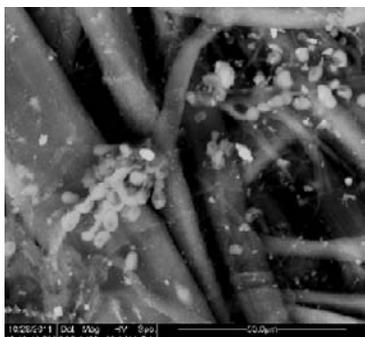
*Biodeterioration indicators on paper.*



*Evaluation of Chemical control Products*



*SEM (Scanning Electron Microscope) results.*



*Fungi structures on paper.*

## Investigation on some antioxidants for ink damaged paper

**Olga Darcanova, Aldona Beganskiene, Aivaras Kareiva**

*Vilnius University, Department of Chemistry*

A significant part of culture heritage was recorded with different types of inks. The large variety of different recipes and the compositional diversity of the natural raw materials result in a diversity of distinguishable degradation mechanisms leading to changes in color after time and, occasionally, resulting in ink corrosion. This process ends in a complete degradation of the paper or parchment. Two principal causes are usually considered to be responsible for the paper degradation: the high acidity of some inks that leads to hydrolytic splitting of the cellulose (formation of acid) and the presence of soluble and mobile metal ions that may act as catalysts for oxidative decomposition of the cellulose.

The stabilization procedure of ink damaged documents includes: deacidification process and antioxidant treatments of inked paper. The most common deacidification agents are calcium and magnesium compounds: hydroxides, carbonates, bicarbonates. However, deacidification treatment does not protect the historical manuscripts against the oxidative decay induced by several transition metals. The main goal of this study is to investigate some antioxidants for stabilization of ink damaged paper.

**Keywords:** paper, ink, antioxidants, stabilization

## Effects of atmospheric pollutants and meteorological factors on a column from a 19th century building (Palma, Spain)

**Catalina Genestar, Carmen Pons, Rafael Forteza**

*Department of Chemistry, University of the Balearic Islands, Palma, Spain*

The effects of atmospheric pollutants were studied in a decayed column from the Seminari de Sant Pere. This 19th century building is situated in the historic centre of Palma (Mallorca, Spain). Its distance from the sea is less than 1 Km. Predominant winds in Palma are from N and NW and from SE, S and SW in winter and summer, respectively.

The stone of the column is constituted by calcium carbonate, which is very susceptible to decay under the effect of marine aerosol and atmospheric pollutants.

Samples were collected from the internal and external part of the crusts formed in the four sides of the column. The samples were analyzed by means of thermal analysis, X-ray diffractometry, scanning electron microscopy, Fourier transform infrared spectroscopy and ion chromatography.

The results obtained allow to establish a relationship between the side of the column analysed, the source of weathering and the predominant winds.

**Keywords:** stone weathering, thermal analysis, FTIR, SEM-EDS, XRD, IC

## Stone weathering studies on the Portal des Mirador from the Cathedral of Palma

**Catalina Genestar, Francisca Alba, Víctor Cerdà**

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Portal des Mirador is located in the southern façade, facing to the sea, of the Cathedral of Palma. This 15th century entrance has undergone several restorations. This part of the building is made of pedra de Santanyi (i.e. oolitic limestone). In this sense, limestone is considered the most susceptible of building stones to salt decay.

Soluble salts are a principal agent of decay in porous building materials such as pedra de Santanyi and a major concern to those involved in the conservation of historic buildings. Among the sources of soluble salts in building materials, sea salt, fertilisers, acid gases from atmospheric pollutants, saline soils and groundwater, salt naturally occurring in the stone and microorganisms should be highlighted. As far as Portal des Mirador is concerned, marine aerosol, airborne pollutants and groundwater are the main deteriorating agents for the structural and decorative materials.

In this work, samples of decayed stone from Portal des Mirador were studied using thermal analysis, X-ray diffractometry, scanning electron microscopy (SEM), Fourier transform infrared spectroscopy and ion chromatography. The results have been essential to plan an efficient intervention with compatible restoration materials.

**Keywords:** soluble salts, stone weathering, FTIR, SEM-EDS, IC, XRD

## Portable non-destructive analytical techniques to diagnose the impacts of modern urban atmospheres and other environmental factors on archaeological sites: The case of Insula IX, 3 (Pompeii, Italy)

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Since the archaeological site of Pompeii was brought to light, Pompeian houses (including wall paintings) have suffered important deterioration processes. This fact can be attributed to rainfall, humidity, water infiltrations, as well as pollution and colonization of microorganisms.

In this work, walls and wall paintings exposed to the open air (around 150 years ago) and protected ones (ceilings of recent construction), from Insula IX, 3 (Houses 1,2 and the House of Marcus Lucretius) were analyzed using portable, non-destructive instrumentation (Raman, Diffuse Reflectance Infrared and X-ray fluorescence spectroscopies).

In protected walls, higher sulphur contents have been observed comparing to the ones exposed to the open air (rain washed walls). Apart from gypsum, other sulphates were also detected (see Figure 1B), formed as a consequence of SO<sub>x</sub> impact and also due to the use of modern Portland cement. Additionally, only on protected walls, nitrate salts were detected (see Figure 1B). In different areas, biological patinas of different colors (black, green, yellow, white, etc.), some of them related to biosynthesized pigments (carotenoids, scytonemin, etc.), were also identified, coming from both microorganisms and non-vascular plants (e.g. moss) colonization processes.

Portable, non-destructive instrumentation has been proven a valuable tool for the characterization of the original building and decorative materials, as well as the deterioration products of walls and wall painting remains in archaeological sites, without the need of sampling or sample preparation.

This work was partially funded by Spanish Research Project (DEMBUMIES, BIA2011-28148).

**Keywords:** portable non-destructive spectroscopic techniques, insula IX, 3 of Pompeii, walls and wall paintings, sulphation processes, colonization processes

Figure 1

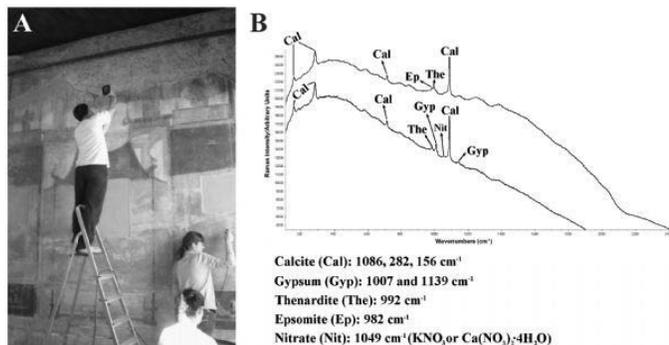


Figure 1. (A) Details of in situ measurements on the House of Marcus Lucretius in the insula IX 3 (B) Two Raman spectra obtained in situ on efflorescences of walls from the House of Marcus Lucretius showing calcite (CaCO<sub>3</sub>, Ca), gypsum (CaSO<sub>4</sub>·2H<sub>2</sub>O, Gyp), thenardite (Na<sub>2</sub>SO<sub>4</sub>, The), epsomite (MgSO<sub>4</sub>·7H<sub>2</sub>O, Ep) and nitrate (KNO<sub>3</sub>/Ca(NO<sub>3</sub>)<sub>2</sub>·4H<sub>2</sub>O, Nit) bands.

## Combined use of SEM-EDS, micro-XRF and Raman spectroscopies to evaluate the composition of black and salt crusts on building stone surfaces formed by natural and anthropogenic sources

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The major factors affecting stone constructions are atmospheric pollutants (acid aerosols, anthropogenic airborne particulate matter, etc.) and environmental natural stressors (marine aerosol, crustal particulate matter, etc.). Their impact, frequently promotes the formation of crusts on these building surfaces.

In the present work, black and salt crusts formed over sandstone and limestone, of several buildings located in the North of Spain (Getxo, Biscay) and affected by marine aerosol, road/railway traffic and industry, have been analyzed by means of non-destructive techniques; Raman spectroscopy, micro-X-Ray fluorescence (XRF) and Scanning electron microscopy/X-Ray energy-dispersive spectroscopy (SEM/EDS).

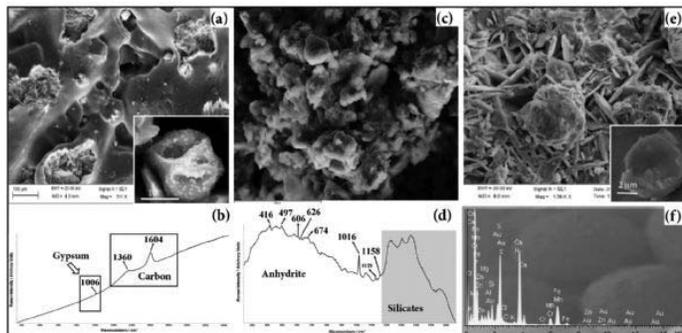
The micro-XRF analysis revealed the presence of S, K, Ca, Ti, Mn, Fe (stone components and/or exogenic) and Cr, Ni, Cu, Zn and Pb (exogenic) in different relative proportions in each sample. The EDS analysis confirmed the presence of these elements plus V. Furthermore, it allowed the identification of C (see details in Figure 1a), Al, Si (stone component and/or crustal particulate matter) and Na, Cl, F and Br (marine aerosol). Additionally, EDS maps were also obtained in order to evaluate the distribution of each element in the samples. Finally, by Raman spectroscopy, the components of the stone (e.g. calcite, hematite, magnetite and silicates) and the exogenic compounds (e.g. gypsum, anhydrite, aluminium silicates and carbon) were characterized.

The oldest constructions considered in this work show the presence of black crusts (carbon and gypsum), and the newest ones show the presence of salt crusts with trapped metals (see Figure 1).

This work was funded by Spanish Research Project (DEMBUMIES, BIA2011-28148).

**Keywords:** SEM-EDS, micro-XRF, Raman spectroscopy, black and salt crusts, carbon, sulphates, airborne particulate matter

Figure 1



(a) SEM microphotograph of a black crust over sandstone, showing gypsum crystals in the holes of a homogeneous surface of carbon covering the original stone surface and a magnification of a chloride particle. (b) Raman spectrum of the (a) black crust showing gypsum and carbon bands. (c) SEM microphotograph of salt crusts over limestone with its (d) representative Raman spectrum. (e) SEM microphotograph of a black crust over limestone and in the inset, magnification of an iron particle and (f) EDS spectrum showing several metals trapped on a black crust.

## Manufacturing techniques of Porcelain Bowls from Xing Kiln in 6th-7th centuries A.D.in China

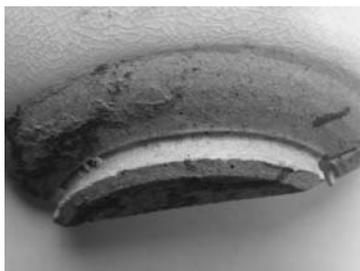
**Jiming Xu, Weidong Li**

*Shanghai Institute of Ceramics, Chinese Academy of Sciences*

Porcelain bowl is the most popular vessel used by Chinese people to eat food. The shapes of bowls have not changed a lot over time, such stability makes it easier for us to study the techniques for shaping and processing porcelain bowls using the experimental archaeology method, which is of great significance upon the research on the manufacturing techniques of porcelains in ancient China. In this project, 274 pieces of bowl sherds of late Northern Dynasty to Sui Dynasty (6th-7th centuries A.D.) which were excavated from Xing kiln site in Hebei province of China were observed and analyzed. It has been proved that the technique of fast-wheel shaping with counter-clockwise rotation was already widely used during that period. Metal tools began to be used for cutting and trimming green bodies of porcelains no later than the 6th centuries A.D.. Most of the techniques invented no later than the 6th centuries A.D. are still being used till today.

**Keywords:** Xing kiln, 6th-7th centuries A.D., porcelain bowl, manufacturing technique

**Fig.1 Porcelain bowl sherd from Xing kiln site**



*Sui Dynasty (581-618 A.D.), with distinct trace of cutting and trimming*

**Fig.2 Porcelain bowl sherd from Xing kiln site**



*Sui Dynasty (581-618 A.D.), with combing marks formed by cutting*



**Fig.3 Porcelain bowl sherd from Xing kiln site**

*Northern Qi Dynasty (550-577 A.D.), with arch lines formed by body trimming*

## Meybod Ziluo Museum: Preventive Conservation plan, Microclimate and Indoor Climate Control

**Faeze Sadat Asadi Firuozeabadi<sup>1</sup>, Mohammad Taghi Ashoori<sup>1</sup>, Farnak Bahrololumi<sup>2</sup>, Atefeh Sadat Asadi Firuozeabadi<sup>3</sup>, Parichehr Moradi<sup>1</sup>**

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Ziluo is a kind of rug which are produced by tightly interweaving the warp and weft strands of the weave to produce a flat surface with no pile. Its date back about to 14th century. Zilou is woven with cotton (except for a few items that have been woven with wool), colored with natural dye materials, without any lint and its scaffold is different from other rugs and textiles.

The zilou Museum is the only one in the world which the exhibited artifacts are belongs to 15th century.

The Museum is located in Meybod (central Iran), in a desert area which is regularly exposed to local winds and dust, therefore it is necessary to control regularly the indoor climate and also microclimate of objects. Seasonal fluctuation of temperature and relative humidity is very high in this area. Because of these difficult conditions the Indoor climate were adjusted with the measurement of environmental factors such as temperature, Relative Humidity (RH), light, dust.

The first step to fixing the set points was identification of fibers, mordant and dyes used in Zilou production with different methods include FTIR, PLM and wet chemistry. After the investigations for diagnostic of damages in Zilous, a preventive conservation plan is offered for this unique museum.

**Keywords:** Textile Museum, Ziluo, Preventive Conservation, Microclimate, FT-IR, PLM, Meybod

## Exhibition Method And Environment Condition Proposals For Yenikapı Shipwrecks

**Aslı Gökçe Gökçay**

*Istanbul University, Istanbul, Turkey*

The architectural finds and small finds revealed at the field, the Yenikapı excavation has the feature of having collection which includes the greatest number of Shipwrecks of the world with 35 units of Shipwrecks revealed. After the removal and documentation of the found ship wrecks, the protection and repair works have been started and also the projects for the exhibition which is the final step after the revealing of the artifacts and the conservation and restoration works have been started to be produced. Within the museums where the revealed artifacts will take place and especially for the museums where the Shipwrecks will be exhibited, the special exhibition method and environment conditions are needed. When the dimensions of the artifacts are considered and by considering the fact that the Shipwrecks which are organic artifacts cannot be exhibited inside the display windows, it is clear that special works are required for the environment in which the artifacts will be displayed. The appropriateness of the exhibition environment to be selected according to the realized conservation applications in terms of the parameters such as humidity, temperature and light are very important in terms of the condition of the artifacts.

With this study, the method and environment conditions of the wreck museums doing exhibitions in the world and the appropriateness of the data obtained as a result of this with the exhibition of the Shipwrecks revealed in Yenikapı will be analyzed

**Keywords:** Yenikapı Shipwrecks, conservation, exhibition, museology.

## Evaluation of the effectiveness of laser removal of graffiti on granite by means of ftir

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*<sup>2</sup>Center of Technological Investigations, University of A Coruña (Campus Ferrol)*

This paper presents the results of the application of FTIR technique to evaluate the efficacy of laser in the removal of graffiti in granitic stones. The study was conducted on a variety of granite widely used in the construction of buildings in Galicia, which weathered an intense deterioration.

Before the cleaning treatment, the different colour graffitis were characterized by scanning electron microscopy and Fourier transformed infrared spectroscopy by ATR (Nicolet 6700). The laser used in this work was a Nd:YVO<sub>4</sub> (Coherent AVIA Ultra 355–2000) at the wavelength of 355 nm and pulse duration 25 ns. Frequency was fixed at 10 kHz and the laser fluence varied in the range 0.1 to 2.5 J/cm<sup>2</sup>.

FTIR analysis of the laser treated samples allowed us to determine the fluence values for an effective removal of graffitis. FTIR technique not only helped to identify the presence of graffiti signals but provides useful information on the stability of minerals under different laser parameters and therefore to assess the damage of the granitic substrate.

**Keywords:** Granite; graffiti; Nd:YVO<sub>4</sub>; FTIR; removal.

## Novel material for stone protection with water-repellent and antibacterial properties

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The preparation of resistant, low cost materials for the treatment of monumental stones in order to protect them from the physical, chemical and biological attacks attracts the effort of many research teams.

In the present work a new silica-based material is reported, together with the investigation of protective properties for stone surfaces. Hybrid organic-inorganic silica films were prepared base on tetraethoxysilane, organomodified ethoxysilane derivative and various aminopropyl triethoxysilanes. The silica dispersions and the deposited films have been investigated by means of TG-DTA, FT-IR spectroscopy, DLS and SEM. The structure of the resulting materials consists of a SiO<sub>2</sub> network with pendant hydrocarbon chains and amino groups.

The changes in stone wettability were evaluated from the contact angle measurements using static and dynamic methods. The film from the novel material deposited on the stone surface exhibit ultrahydrophobic/ superhydrophobic properties and significantly reduces the penetration of water into the stone.

Travertine samples were used to investigate the protective ability of new coatings against acid rain attack. Both organomodified silica films, with and without amino groups, improve the resistance of the stone against acid rain.

Bacterial adhesion assays have been performed on the travertine samples subjected to the treatment with silica material and the development of biofilm on the stone surface was shown to be inhibited

**Keywords:** nanomaterials, stone protective materials, superhydrophobic, antibacterial

## Study of new pigment products for filling the red marble in the conservation works

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*<sup>2</sup>Ozan Deveoglu, Faculty of Fine Arts, Marmara University, Istanbul*

The filling process has been used powder of marbles for to give a novel touch to color in the marble in the conservation science since conservation science was born. However, some times the marble, mainly of local provenance, suffers of superficial decohesion and color distortion. Because, powder of marble some case (humidity, wrong ratio for filling plaster ecc) not stabile.

These scopes of this research include;

- i) Components of red marble (from lasos of Caria)
- ii) Product red stabilized pigment analysis for filling with plaster
- iii) FT-IR analyses both for detected organic compounds

In order to weight up the plaster mechanism between organic pigment and other component of plaster for the filling process. First produce of synthetic pigments; second add to in the plaster of filling which are principle and different ratio and after controlled dry with different conditions. All observe and try to understand; stability, color accordance with in the original marble color

**Keywords:** red pigments agents, FT-IR analyses, and red marble

## A multi-analytical investigation on samples from the Macedonian Tomb "Makridi", Thessaloniki, Greece

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 Panagiotis Manoudis<sup>2</sup>, Dimitrios Lampakis<sup>2</sup>, Haralambos Tsagalides<sup>1</sup>

<sup>1</sup>16th Ephorate of Prehistoric and Classical Antiquities, Ministry of Culture, Thessaloniki, Greece

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Within the framework of the conservation processes (supported by the National-European projects "Culture" and "NSRF") which is carried out on the Macedonian Tomb "Makridi" (Thessaloniki, Greece) a multi-analytical investigation is currently performed on selected samples to collect (i) valuable archaeological/historical information and (ii) select an appropriate conservation strategy.

Twenty two small samples (with diameters <10mm) from already damaged areas were extracted and investigated using optical polarized microscopy, Scanning Electron Microscopy (SEM), SEM equipped with an Energy Dispersive X-ray spectrometer (EDX), micro X-ray fluorescence ( $\mu$ XRF) and micro Raman spectroscopy ( $\mu$ Raman).

The study is under progress and so far gave the following RESULTS: the intense red colour appeared in some areas of the Tomb (e.g. Fig 1) was achieved using hematite while the grey colour was the result of mixing calcite with carbon. Typical Raman spectra are shown in Fig. 2.

Two layers were identified in the plaster: a coarse (calcite mixed with sand) and a fine (pure calcite) grained layer.

Detached plaster areas and effects of Fungi were revealed using microscopic techniques (e.g. SEM in Fig. 3).

Conservation process was focused on the consolidation of plaster, achieved using nanoparticles of calcium carbonate.

**Keywords:** Raman, XRF, SEM-EDX, Macedonian tomb

Fig 1



Fig.1

Fig 2

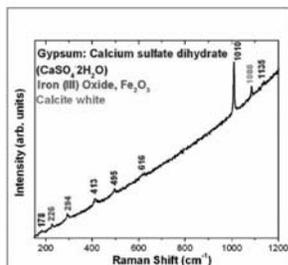


Fig 2.

Fig 3

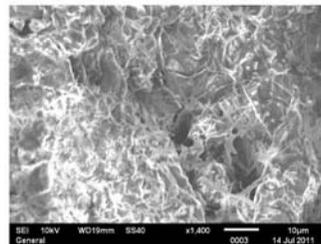


Fig 3.

## Novel Fully Conjugated 2H- and Metal- Phthalocyanine Network Polymers: Synthesis, Characterization and Dielectric Spectra Analysis

**Hamada Abdel Razik<sup>1</sup>, Khaled Mahmoud<sup>2</sup>**

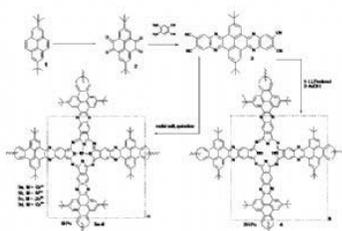
<sup>1</sup>Chemistry Department, Faculty of Science (Damietta), New Damietta 34517, Egypt

<sup>2</sup>Physics Department, Faculty of Science, Cairo University, Egypt

2,7-Di-tert-butylpyrene was oxidized to 2,7-di-tert-butylpyrene-4,5,9,10-tetraone. The latter through condensation reactions with vicinal diamine such as 4,5-diaminophthalonitrile produced heterocyclic monomer, 2,7-di-tert-butyl pyrene[4,5][9,10]bisquinoxaline-6,7-dinitrile, which was cyclo-tetramerised to the corresponding tetra[2,3-(1,4-diaza-6,6-di-tert-butylphenanthreno) [4,5] phthalocyanine]-based network polymer (2H-Pc), and tetra [2,3-(1,4-diaza-6,6-di-tert-butyl phenanthreno) [4,5]phthalocyanine metal II-based network polymers (M-Pc, M= Co, Ni, Zn or Cu). Elemental analytical results, IR and NMR spectral data of the new prepared molecules are consistent with their assigned formulations. Molecular masses and metal contents of the synthesized polymers proved to be of high molecular masses which confirm the efficiency of tetramerization polymerization and complexation reactions. The dielectric constant ( $\epsilon'$ ) and dielectric loss tangent ( $\tan \delta$ ) were studied as a function of temperature and frequency. The detailed analysis of the results showed that the dielectric dispersion consists of both dipolar and interfacial polarization. Measurements of ac conductivity as a function of frequency at different temperatures revealed that the non-overlapping small polaron tunneling (NSPT) is the most suitable mechanism for ac conduction behavior.

**Keywords:** Phthalocyanines, conjugated polymers, dielectric properties

*scheme*



## Improving paper mechanical properties and printing quality by using carboxymethyl cellulose as a strength agent

**Asmaa Mohamed El Shafey<sup>1</sup>, George Nubar Simonian<sup>2</sup>, Fouad Taha Abd El Halim<sup>3</sup>,  
Fathey Fahim Abd El Latif<sup>4</sup>, Abeer Mohamed Adel<sup>5</sup>**

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2Improving paper mechanical properties and printing quality by using carboxymethyl cellulose as a strength agent

Carboxymethyl cellulose (CMC) is an anionic water – soluble polymer that has been introduced in paper coating as a strength agent. One of the main objectives of this research is to investigate the influence of CMC concentration in improving the strength properties of paper fiber. In this work, we coated the paper sheets; Xerox paper sheets by different concentration of carboxymethyl cellulose solution (0.1, 0.5, 1, 1.5, 2, 3%) w/v. The mechanical properties; breaking length and tearing resistance (tear factor) were measured for the treated and untreated paper specimens. The retained polymer in the coated paper samples were also calculated. The more the concentration of the coating material; CMC increases, the more the mechanical properties; breaking length and tear factor increases.

It can be concluded that CMC enhance the improvement of the mechanical properties of paper sheets result in increasing paper stability. The aim of the present research was also to study the effects on the vessel element structure and vessel picking. Fragmentation of vessel elements through CMC modification was found to be important and results in a decreasing picking tendency which reflects in a good printability.

Moreover, Scanning – Electron Microscope (SEM) images are represented to specifically explain the improved bonding ability of vessels and fibers after CMC modification. Finally, CMC modification enhance paper mechanical properties and print quality.

**Keywords:** Carboxymethyl cellulose (CMC), breaking length, tear factor, vessel picking, printing, concentration.

## Disinfection of cultural property made of cellulose materials by herbs: Old materials, new technologies

**Narges Pedram, Shahmira Rahimi**

*Azad University*

Many Libraries, archive and storages for historical and cultural artifacts, do not have suitable air cleaner and conditioner. Considering fluctuation of temperature and humidity and also dispersion of fungi spores in these centers, these artifacts, especially artifacts made of plant materials are expose to attack or grow of microorganisms. Current methods, include chemical and physical disinfection, can be harmful for cultural properties and conservators too. For example fumigation with Thymol and Ethylen Oxide leave an impression on conservators and researchers and using Gamma ray with high energy causes photo chemical reaction in artifacts specially in illuminated manuscripts.

The purpose of this study is to investigate and determine the antifungal effect of these herbal extracts: "Thmus Vulgaris", "Ocimum Basilicum L", "Origanum Majorana L", "Origanum Vulgare L", "Salvia Officinalis L", from "Labiatae" and Cinnamum Zeylaicum Nees, "Zingiber Officinale Rose" from "Lauraceae" and "Hypericum Perforatum L" from "Hypericaceae". Hypericum did not have any fungicide property, for this reason we can compare the effects of other extracts with this extract.

Methods of research:

- preparing the extract of herbs by ethanol and acetone.
- investigate the effects of extracts on fungi by direct using and indirect on paper that mixed with one of extracts in that places of sample.

According to the results of the experimental studies, some of these plants and their extracts such as Thymus and Cinnamon and also combination of them can prevent the growth of many kinds of fungi especially ladosporium, Alternaria, Rhizopus, Penicillium, Aspergillus.

**Keywords:** disinfection, cellulose material, herbs, extracts, cultural property

## Evaluating the Application of Natural Pesticides on Korean Traditional Paper

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*Asarum sieboldii* Miquel was selected as candidate materials showed antimicrobial and insecticidal activity. Especially, *A. sieboldii* showed the most significant insecticidal activity adult of *Lasioderma serricorne* and termite. Also, This showed antifungal activity against fungi isolated from wooden cultural heritage, JanggyeongPanjeon of Haein temple. Such natural pesticides have its own color and include many organic compounds. Thus, these could cause color difference and physical and chemical changes on traditional paper. Therefore, this study performed that the influence of *A. sieboldii* against traditional paper evaluated for color, brightness, tensile strength, folding endurance and viscosity. As a result, color difference and brightness of traditional paper by *A. sieboldii* nearly unchanged, and also viscosity value increased in effective concentration of antifungal and insecticidal activity. The viscosity value was measured as index for paper component degradation by paper aging. And then, *A. sieboldii* was found to inhibit growth of microorganism caused the biological aging on the surface of traditional paper. For the future, *A. sieboldii* must be evaluated for safety against organic materials as well as metal, and finally prove to be a stable compound on the human body.

**Keywords:** Natural pesticides, *Asarum sieboldii* Miquel, conservation agent, traditional paper

## Sol-gel Method for the Conservation of Copper

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Sol-gel method is one of the most promising and innovative method that can be applied to metal conservation. Hydrophobic, anticorrosion and other coatings can be formed by sol-gel processing. Sol-gel protective coatings have shown excellent chemical stability, oxidation control and enhanced corrosion resistance for metal substrates. Moreover, the sol-gel method is an environmentally friendly technique for the preparation of protective coatings and had showed the potential for the replacement of toxic pretreatments and coatings which have traditionally been used for increasing corrosion resistance of metals.

In this study, the preparation of methyl-modified coatings by the sol-gel technique to protect the external surface of copper is discussed. Coatings were obtained by simple sol-gel method, in which the tetraethylorthosilicate (TEOS) has been used as a precursor to prepare sol-gel coatings for the surface treatment of copper. 3% SiO<sub>2</sub> sol, trimethylchlorosilane (TMCS) and hexamethyldisilozane (HMDS) were used for the preparation of methyl-modified silica sols. Paraloid B 72, Plexisol P 550-40 and polyvinyl butyral (PVB) coatings were also deposited on the copper substrate for comparison. Copper specimens were exposed to artificial ageing. The surface morphology changes of uncoated and coated specimens were investigated by atomic force microscopy (AFM) and scanning electron microscopy (SEM). The hydrophobicity and photochemical ageing effects were evaluated by contact angle measurements. Potentiodynamic measurements were obtained in order to compare corrosion parameters of the coatings. All the measurements were performed before and after the photochemical ageing.

**Keywords:** conservation of metals, copper, sol-gel method, TEOS, TMCS, HMDS, PVB, paraloid, plexisol.

## Physico-chemical characterizations of nickel substituted calcium hydroxyapatites

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Solid solutions of calcium nickel hydroxyapatites were synthesized by a hydrothermal process. Their structural and thermal properties were investigated. The substituting limit of nickel was estimated at about 18 atomic percentages. The replacement of calcium, by nickel, induces a small decrease of the crystallographic parameter "c" and an irregular change of the parameter "a". The distribution of the Ca and Ni ions between two non-equivalent crystallographic sites S(I) and S(II), were determined by the Rietveld method. The site-occupancy factors of atoms indicate a preference of nickel for site S(I) in the apatite structure in agreement with its smaller ionic radii. A progressive reduction of crystal size has been observed with increasing Ni content. The mixed apatites obtained show a thermal conversion into  $\beta$ -TCP above 700°C.

**Keywords:** hydroxyapatite hydrothermal properties Rietveld method

## Soltanieh dome tiles: scientific investigation and diagnostic for conservation

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Soltanieh dome is one of the most unique examples of Islamic architecture and the third largest dome after Santa Maria del Fiore (Italy) and Aya Sofia (Turkey) which has been built by Soltan Mohammad Khodabande (Oljaito) in Ilkhani period (13th century). This ancient monument has very valuable decoration at indoor and outdoor of its Facade that has been created in 2 steps. First step was tiling and the second one was fresco.

The present study describes the analytical investigation performed on the glazes of tiles mounted in different part of Soltanieh dome. The colors of these tiles are black, turquoise and azure. Two advanced instrumental techniques including scanning electron microscopy (SEM-EDX) and inductively coupled plasma (ICP) have been used to perform the characterization of the glazes. Results showed that these glazes were made by the traditional methods used by local craftsmen since the 13th century. They used lead as flux and a kind of plant named OSHNAN is used as flux too. These glazes can be considered as lead-alkali glazes opacified with tin oxide. According to the results of these tests, cobalt, copper and manganese are the main elements for the coloring agents. At the end, to improve the quality of these glazes, some solutions have been proposed.

**Keywords:** Tile, Soltanieh dome, SEM-EDX, ICP, Diagnostic, Conservation

## Two common adhesives in restoration of potteries (Researching and comparing)

**Roxana Minoueipour**

*Islamic Azad University*

In this project two adhesives that are used in restoration of potteries were chosen in order to inspect the PH, Transparency and colour, modulus of elasticity, tensile strength and flexibility both before and after aging in light, so that both study the changes generated in their specifications along with passing of time and via the results obtained from each test and the information existed regarding other specifications of each of the aforesaid the one most effective adhesive with a highest effective durability could be then determined.

Those chosen adhesives, include:

-Paraloid B72

-The combination of Paraloid B72 and Aerosil R-202(fumed silica hydrophobic 0.1% weight-based and 0.2% weight-based).

Due to the tests performed and the results obtained from such tests the combination of ParaloidB72 and fumed silica(0.1%weight-based)has a higher level of effective durability.

**Keywords:** Paraloid B72 and Combination of Paraloid B72 and Aerosil R-202

## Characterization of morphological and chemical changes at micro- and nano- scale in contemporary paintings treated with biocides

**Annette Suleika Ortiz Miranda<sup>1</sup>, Maria Teresa Doménech Carbó<sup>1</sup>  
Antonio Doménech<sup>2</sup>**

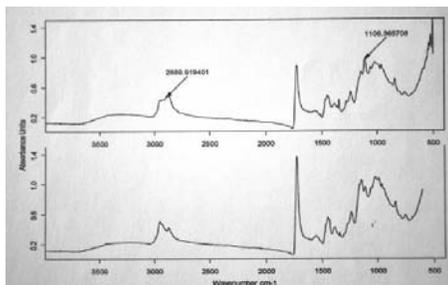
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The growing problems of biodeterioration undergone by commercial artists' paints have increasingly required the application of biocide treatments on contemporary artworks. In most cases, commercial biocides, which have not been created for the purpose of being used in the field of art conservation, are applied in dosages recommended by the manufacturer without control on the effects of their application on the artwork. From this, a study has been conducted aimed to evaluate the changes induced by the biocide on contemporary paintings of acrylic and PVAc type. Two biocides have been considered, Biotin T<sup>®</sup> and Preventol RI80<sup>®</sup>. Chemical changes have been identified by using FTIR spectroscopy and UV-VIS spectrophotometry. The morphological study at microscale has been performed by using optical microscopy and SEM-EDX. In a second step chemical and morphological changes at nanoscale have been characterized by using, at first time in the field of the analysis of artworks, the novel technique of electrochemical atomic force microscopy (ECAFM). Some of the most significant changes observed by microscopy were: appearance of spots and alteration of the brightness of the paint film, as well as, deposits of biocide. A notable delay in the coalescence phase of drying of the acrylic polymer used as binding media was recognized by means of ECAFM. Spectroscopic analysis results suggest that the application of the biocide causes a significant migration of additives to the surface from the core film. Acknowledgements.- Financial support is thanked to the Spanish (MICINN) R+D Project CTQ2011-28079-C03-01 and 02 also supported with ERDF funds.

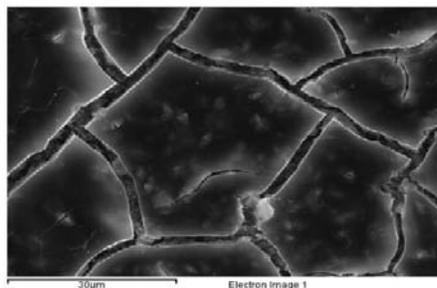
**Keywords:** biocide, acrylic artists' paint, PVAc paint, FTIR spectroscopy, electrochemical atomic force microscopy (ECAFM)

**FTIR**



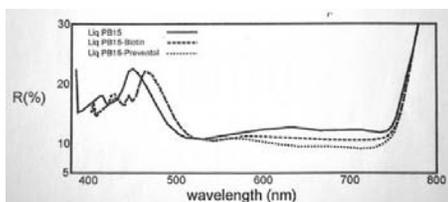
IR absorption spectrum obtained for: umber Liquitex specimen treated with Biotin T (up); untreated control specimen (bottom).

**SEM-EDX**



Surface image of the umber Flashe treated with Biotin T obtained by SEM-EDX, craquelure are appreciated in the thin layer created by the biocide in the film surface.

**UV-VIS**



Reflectance spectrum obtained for untreated Liquitex phthalocyanine blue and spraying with the two biocides in study.

## Application of surface analysis methods in historical artifacts

**Soodabeh Yousefnejad**

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Surface analysis is important in cultural heritage studies, because of many surface phenomena in the historical artifacts, such as corrosion, catalytic reactions on the surface, detergents and adhesives. All of the solid materials react with environment through the surface, so in this article the principles of surface chemistry will be discussed then some important methods in the surface analysis of organic and inorganic materials will be investigated by reviewing related articles. X-ray photoelectron spectroscopy (XPS), Auger electron spectroscopy(AES) and secondary ion mass spectroscopy(SIMS) are important methods of surface analysis with application in historical artifacts, such as pigments in the paintings, glass, pottery,ceramics and metals. Despite the undoubted importance of surfaces, only a very small proportion of the atoms of solids are found at the surface, that depend up on the shape and surfaces roughness of the material as well as it's composition. It simply illustrated that a successful technique for analyzing surfaces must have at least two characteristics; it must be extremely sensitive and efficient at filtering out signals from the vast majority of the atoms present in the sample. according to the theory of restoration and conservation of cultural heritage and the time line that the object has passed from past to present, should be noticed that it is an unrepeatable phenomena and must be conserved, so the sampling for studying it, is an important problem and the analytical methods which need to the minimum amount of sample are very useful in ancient objects studies.

**Keywords:** surface analysis, ancient objects, XPS, AES, SIMS

## **Occupational Safety of Chemicals Used in Restoration of Cultural Heritage Materials**

**Hazal Özlem Ersan**

*IBB KUDEB*

Wide variety of chemicals is used during the material analysis and restoration applications on cultural heritage materials. Chemical, especially those that are applied outside the laboratory conditions can cause hazards on the environment, on the workers as well as the building in question if they are used without control.

Many chemicals pose important risk factors on human health and environment during restoration and conservation applications, especially in cleaning of surface residues, removal of paint layers and storing of archaeological materials. Safety warning of all chemicals that are to be used should be in the work plan of the project and the personal that will use them should be informed about the risk factors and the application should be carried out with proper protective equipment. This way it will be possible to prevent irreversible health damages that may occur at the worksite.

The objective of this work is to create a minimum material safety understanding among the workers who are not actually chemist. We want to have the workers to have a broader understanding of the chemical that are used or stored. In this study the properties and risk factors of materials such as AB 57, Ethyl alcohol, acetone, toluene, that are widely used in restoration and conservation activities, are evaluated to minimize the health and environmental hazards of these chemicals.





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