8th Meeting of the IUCR Commission on Crystallography in Art and Archaeology Saturday 24 August 2024 Aula Arduino, Department of Geosciences, University of Padova Satellite Meeting of ECM34

Ancient and Historical Glass: Order or Chaos?

Scientific Program

9:00-9:05 Koen Janssens, Ivana Angelini, meeting chairs; Gilberto Artioli, ECM34 president Welcome

9:05-9:50 Partha Pratim Das, NanoMEGAS SPRL, Brussels, Belgium Electron diffraction and related methods for the investigation of ancient glass

9:50-10:35 Myrtille Hunault, Synchrotron SOLEIL, Saint Aubin, France X-ray absorption spectroscopy for characterization of colored historical glass

10:35-10:50 Coffee Break

10:50-11:35 **Joost Caen**, University of Antwerp, Belgium *Imaging and analysis of stained-glass windows*

11:35-12:10 João Pedro Veiga, Universidade NOVA de Lisboa, Portugal Characterization of ancient and modern glazes

12:10-12:40 Alicja Rafalska-Lasocha, Wieslaw Lasocha, Jagiellonian University, Krakow, Poland XRPD research on historical glass and other cultural heritage objects

12:40-14:00 Lunch break

14:00-14:45 Dana Rohanová, Karolína Pánová, University of Chemistry and Technology Prague, Czech Republic

The development of glassmaking technology for potassium glass produced in transalpine Europe until the half of 18th century

14:40-15:30 Ana Franjic, Patrick Degryse, KU Leuven, Belgium Isotope ratios in provenance studies of ancient and historical glass

15:30-16:15 Ivana Angelini, Univ. Padova, Italy, Paolo Bellintani, Sopr. Archeologica Prov. di Trento, Italy, Andrea Cardarelli, Univ. Rome, Italy The protohistoric glass production in Frattesina di Rovigo, Italy: a review

16:15 Meeting chairs Closing remarks

* speakers to be confirmed

** preliminary title

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Saturday 24 August 2024 Aula Arduino, Department of Geosciences, University of Padova

Ancient and Historical Glass: Order or Chaos?

Scientific Program with Abstracts

9:00-9:05 **Koen Janssens, Ivana Angelini**, meeting chairs; **Gilberto Artioli**, ECM34 president *Welcome*

9:05-9:50 Partha Pratim Das, NanoMEGAS SPRL, Brussels, Belgium

Electron diffraction and related methods for the investigation of ancient glass

Understanding the properties, manufacturing processes, and conservation methods of archaeological artefacts necessitates detailed structural characterization. Transmission Electron Microscopy (TEM) offers significant advantages for studying artefacts at the nanometer scale using minimal material. This presentation explores the application of TEM techniques—such as Electron Diffraction (ED), Energy Dispersive X-ray Spectroscopy (EDX), and Electron Energy Loss Spectroscopy (EELS)—to investigate ancient artefacts, focusing on nanocrystalline phases in Greek amphoras, Roman glass tesserae, various ancient glass samples from around the world, and obsidian glasses from Mexico.

9:50-10:35 Myrtille Hunault, Synchrotron SOLEIL, Saint Aubin, France

X-ray absorption spectroscopy for characterization of colored historical glass

Most colors of medieval stained glass windows were obtained using transition metal (TM) elements. The stained glass windows of the Sainte-Chapelle in Paris are one example of the richness of the colour palette that could be obtained and how it has evolved from the 13th to 15th century as a testimony of the mastery of ancient glassmakers. The obtained color depends on the elaboration conditions: glass composition (raw materials and coloring elements) and melting conditions. The determination of the subtle relations between chemistry and optical properties of glasses provides tangible information about the history of art and glassmaking techniques. Optical absorption spectroscopy (OAS) enables to probe directly the origin of the glass color and its link with the chemical composition of medieval glasses. X-ray absorption spectroscopy (XAS) is a powerful complementary technique that allows also probing the invisible species and determining the TM redox and local coordination in glass. The combination of both techniques with other non-invasive techniques such as particle-based composition analysis provides quantitative chemical information on the origin of the glass color and allows inferring medieval glassmaking recipes.

This will be illustrated through several case studies of blue, green, purple glasses from the Sainte-Chapelle in Paris and other monuments as well as model glasses.

10:35-11:20 Joost Caen, University of Antwerp, Belgium

Imaging and analysis of stained-glass windows

Glass Conservation studies were started in Antwerp ca 35 years ago and from the start, there was a high interest in chemical analyses of glass vessels, stained glass windows and of vitreous paints. Initially our aim was to be able distinguish between original glass and later infills and reconstructions in stained glass windows. At first, only partial information could be obtained based on limited sampling in combination with Scanning Electron microscopy coupled to Energy Dispersive X-ray (SEM-EDX) analyses. The development of macroscopic X-ray fluorescence analysis (MA-XRF) scanning made it possible to obtain a much more detailed overall image of both sides of stained-glass panels. Moreover this technique is non-destructive and can be applied 'in situ'. In some cases the scans revealed important extra information about the glass and the paint layers, besides its origin and/or its authenticity. For example, special kinds of flashed glass were discovered (not visible with the naked eye), as well as later repainting and refiring of trace lines. The lecture will illustrate the importance of the technique for art historians and conservator-restorers dealing with present stained-glass research and conservation.

11:35-12:10 João Pedro Veiga, M.L. Coutinho, S. Coentro, T.P. Silva, Universidade NOVA de Lisboa, Portugal

Decoding Portuguese Azulejos: the intersection of art, history, and science

In recent decades, the production techniques of azulejos (Portuguese glazed tiles with predominantly blue color) remain a subject of ongoing research. Combining historical research with advanced analytical techniques, this communication aims to show that pigments and materials that define the azulejo colour palette, along with their production recipes and the chemical and structural composition of the glazed tiles. This communication highlights the application of synchrotron radiation to the analysis of Portuguese azulejos. Some examples will be presented, namely the Co and Cu K-edge X-ray absorption near-edge spectroscopy (XANES) results performed on glazed tiles from the Fronteira Palace, in Lisbon and the speciation of pigments from Pena National Palace, Sintra. The speciation state of certain chemical elements provides an understanding of their role as chromophores, glass network formers or modifiers, contributing to a deeper knowledge of the production techniques, provenance, and the tiles' history of use. The non-destructiveness nature allied to the high-resolution capabilities of synchrotron radiation chemical imaging offer a unique tool for investigating cultural heritage materials, enhancing our understanding of their composition, structure, and degradation processes, and contributing to their long-term preservation and interpretation for future conservation efforts.

12:10-12:40 **Alicja Rafalska-Lasocha, Wieslaw Lasocha**, Jagiellonian University, Krakow, Poland *XRPD research on historical glass and other cultural heritage objects*

X-ray powder diffraction (XPRD) is a widely spread quantitative method of phase analysis which can be applied to many problems of materials analysis. This lecture will provide an review of recent applications of XRPD in the cultural heritage sector related to the characterization on ancient materials, among which glass and glazes.

12:40-14:00 Lunch break

14:00-14:45 **Dana Rohanová, Karolína Pánová**, University of Chemistry and Technology Prague, Czech Republic

The development of glassmaking technology for potassium glass produced in transalpine Europe until the half of 18th century

The production of potassium glass in Western Europe began as early as the 8th century AD. It was heavily inspired by the sodium-ash glass production of the Near East and Southern Europe. The basic chemical composition of glass is determined by the type of ash used as an alkali source, which, for transalpine Europe, was mainly beech ash. Based on the archaeological glass finds from across Bohemia and Moravia, published European finds, and the knowledge of historically available raw materials, we were able to calculate the recipes for several basic glass types. These ranged from the one written by Theophilus Presbyter to the Bohemian Baroque so-called crystal. These theoretical recipes were then verified through experimental melting using historically authentic raw materials, such as beech ash and potash. The calculations and resulting model glasses demonstrated the simplicity and uniqueness of the old recipes, which were characteristic for each studied period: Gothic (14th – first half of the 16th century), Renaissance (second half of the 16th – end of the 17th century), and Baroque (end of the 17th – half of the 18th century).

14:45-15:30 Ana Franjic, Patrick Degryse, KU Leuven, Belgium

Isotope ratios in provenance studies of ancient and historical glass

Provenancing ancient and historical glass through stylistic, typological, and compositional analysis may prove challenging due to the wide diffusion of finished artefacts and the difficulty in differentiating between sources based solely on elemental analysis. Isotopic analyses, particularly involving strontium, neodymium but also lead, hafnium and oxygen, has provided a different entry into tracing the origins of glass artefacts. Ongoing research in this field demonstrates the potential of isotopic methods to differentiate between production centers, previously indistinguishable through 'traditional' compositional analysis.

Isotopic analysis certainly offers the capability to improve our understanding of ancient glass production and trade routes used, which can in turn shed more light on the cultural exchanges and social connections glass generated. However, this approach is not without challenges. This presentation will highlight the benefits of isotopic study for glass provenance research and discuss future prospects in the field. Looking ahead, we should focus on developing comparative isotopic databases, facilitating research into lesser-studied regions, and cross-validating findings with multiple isotope systems. As with many areas of archaeological research, interdisciplinary collaboration is crucial for advancing this discipline.

15:30-16:15 **Ivana Angelini**, Univ. Padova, Italy, **Paolo Bellintani**, Soprintendenza Archeologica Prov. di Trento, Italy, **Andrea Cardarelli**, Univ. Rome, Italy

The protohistoric glass production in Frattesina di Rovigo, Italy: a review

The well-known archaeological site of Frattesina di Rovigo is the only confirmed site of production of glass in Late Bronze Age Europe. The available data will be reviewed, with special focus on compositional data and some new evidence from recent investigations.

16:15 **Meeting chairs** *Closing remarks*