DHA 27

27th MEETING OF DYES IN HISTORY AND ARCHAELOGY

8th – 11th October 2008
Istanbul –Turkey

Turkish
Cultural
Foundation

27th Meeting of Dyes in History and Archaeology
Istanbul, 8 - 11 October 2008

Dyes in History and Archaelogy

Dyes in History and Archaelogy (DHA), an international group of experts with multi-disciplinary background, meets every year, since 1982, to discuss chemical, analytical, biological, historical and technological aspects of natural and synthetic dyestuffs. More information on preceding DHA meetings and publications can be found at
http://www.chriscooksey.demon.co.uk/dha/meetings.html

27th Meeting of Dyes in History and Archaelogy

The 27th Meeting of Dyes in History and Archaelogy (DHA27), including a welcome reception, a gala diner, an extra diner and post conference visits to museums takes place from 8th to 11th of October 2008 in Istanbul, Turkey. The meeting will be jointly organised by the Marmara University.

In the evening of 8th October a welcome reception will take place at the Turkish and Islamic Arts Museum. The lecture and poster sessions will be held at the Marmara University historical Conference Hall in Sultanahmet, on Thursday the 9th and Friday the 10th of October 2008. On Saturday the 11th of October there will be post conference visits to museums (Turkish and Islamic Arts Museum, Topkapi Palace Museum, St. Sophie Museum, Blue Mosque etc).

36 oral lectures and 21 poster presentations from 31 countries will be performed at the meeting.

Wellcome to Istanbul for all participants.

On behalf of the Organizing Committee

Prof. Dr. Emre DÖLEN

DHA 27 **27th Meeting of Dyes in History and Archaeology**
Istanbul, 8 - 11 October 2008

PATRONAGE

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PROGRAMME

WEDNESDAY, 8th OCTOBER 2008

15.00 – 19.00 **REGISTRATION AND INFORMATION** : Main entrance of the Marmara University Rectorate Building in Sultanahmet

19.00 – 21.00 **WELCOME COCKTAIL :** Turkish and Islamic Arts Museum in Sultanahmet

THURSDAY, 9th OCTOBER 2008

08.30 – 09.30 **REGISTRATION AND COFFEE :** Entrance of the Conference Hall of the Marmara University Rectorate Building in Sultanahmet

08.30 – 09.30 **AFFIXING THE POSTERS :** Entrance of the Conference Hall

09.30 – 10.30 **WELCOMING SPEECHES :** Conference Hall

10.30 – 12.30 **SESSION I**
Chair : *Jo Kirby ATKINSON*

10.30 – 10.50 ***David PYBUS***
Interaction of 16th to 18th century English Yorkshire alum producers, salters and London dyers

10.50 – 11.10 ***David MITCHELL***
An 18th century London dyer's working notebooks : How they produced pastel colours 'out of grain'

11.10 – 11.30 ***Roger FELDMAN, David PYBUS and David MITCHELL***
The working life span of 17th and 18th century London dyers

11.30 – 11.50 ***Matthijs de KEIJZER, Maarten R. van BOMMEL and Regina Hofmann-DE KEIJZER***
The early synthetic organic dyestuffs : The Ponceaus

11.50 – 12.10	***François DELAMARE*** *Prussian blue, such a French dye*
12.10 – 12.30	**DISCUSSION**
12.30 – 13.30	**LUNCH**
13.30 – 15.10	**SESSION II** **Chair : *Evangelia A. VARELLA***
13.30 – 13.50	***Lamya HAYAT*** *Red color, water insoluble medicinal extracts of shikonin dyes from Arnebia decumbens (Boraginaceae)*
13.50 – 14.10	***Katarzyna SCHMIDT – PRZEWOZNA*** *Surface improvement on linen naturally dyed fabrics to increase dyeing effciency*
14.10 – 14.30	***Shaukat ALI, Saima UMBREEN, Muhammad ASIF and Tanveer HUSSAIN*** *Dyeing properties of alkaline extracts of Eucalyptus's bark and their comparison with reactive dyeings*
14.30 – 14.50	***Shaukat ALI, Saima UMBREEN, Muhammad ASIF and Tanveer HUSSAIN*** *Optimization of alkaline extraction of natural dye from Acacia's bark and its dyeing on cotton by exhaust method*
14.50 – 15.10	**DISCUSSION**
15.10 – 15.30	**TEA AND COFFEE BREAK**
15.30 – 17.10	**SESSION III** **Chair : *André VERHECKEN***
15.30 – 15.50	***Bernard VERHILLE*** *Indigo plants and woad in ancient Asia Minor*
15.50 – 16.10	***Adeola V. POPOOLA*** *Indigo dyeing among the Yorubas of South Western Nigeria : A historical perspective*
16.10 – 16.30	***Maj RINGGAARD and Annemette Bruselius SCHARFF*** *Indigo in archaeological textiles*

16.30 – 16.50 ***Naceur AYED***
Traditional recipes for dyeing with natural mixed dyestuffs in Tunisia : The last artisan dyeing with indigo and madder

16.50 – 17.10 **DISCUSSION**

17.10 – 17.30 **TEA AND COFFEE BREAK**

17.30 – 18.45 **SESSION IV**
Chair : *Ioannis KARAPANAGIOTIS*

17.30 – 17.50 ***Chris COOKSEY***
The identification of historic dyes – Past, present and future

17.50 – 18.10 ***Türkan YURDUN, Recep KARADAG and Emre DOLEN***
Determination method for the dye sources of historical dyestuffs

18.10 – 18.30 ***Irina PETROVICIU, Andrei MEDVEDOVICI and Florin ALBU***
Analytical protocol for the identification of natural dyes and organic pigments in museum objects, mainly based on MS detection

18.30 – 18.45 **DISCUSSION**

20.00 – 24.00 **GALA DINNER : Historical Galata Tower**

FRIDAY, 10th OCTOBER 2008

09.00 – 10.40 **SESSION V**
Chair : *Regina Hofmann-DE KEIJZER*

09.00 – 09.20 ***Erwin ROSENBERG***
On the feasibility of building LC - MS spectral libraries for natural organic dyestuffs

09.20 – 09.40 ***Aldo ROMANI, C. CLEMENTI, G. BASCONI, and C. MILIANI***
Carthamus tinctorius : An organic dyestuff characterized by luminescence spectroscopy

09.40 – 10.00 ***Carole MATHE and Cathy VIEILLESCAZES***
Chromatographic study of a yellow matter historically employed in Cultural Heritage : Reseda luteola (weld)

10.00 – 10.20 ***Katarzyna LECH, Ewa MIANOWSKA and Maciej JAROSZ***
Yellow multiplicity – Attempt to identify dyestuffs by applying mass spectrometry

10.20 – 10.40 **DISCUSSION**

10.40 – 11.00 **TEA AND COFFEE BREAK**

11.00 – 12.40 **SESSION VI**
Chair : *Karen Diadick CASSELMAN*

11.00 – 11.20 ***Cecily M. GRZYWACZ, Jan WOUTERS, Michel BOUCHARD, Maria Vega CAÑAMARES, Ana CLARO and Maria João MELO***
GCI'S Asian organic colorants project : a study of Gardenia augusta colorants by HPLC-PDA-MS, Raman, SERS and microSPEX

11.20 – 11.40 ***Margarita GLEBA, Ulla MANNERING and Ina Vanden BERGHE***
Colours and dyes of early iron age Danish textiles

11.40 – 12.00 ***Ileana CRETU, Irina PETROVICIU and Ina Vanden BERGHE***
Some more dye analysis on Romanian archaeological textiles from Bucovina, 16th century

12.00 – 12.20 ***Lemonia VALIANOU, Kostantina STATHOPOULOU, Ioannis KARAPANAGIOTIS, Prokopios MAGIATIS and Yannis CHRYSSOULAKIS***
Young fustic: Chemical composition and identification in post-Byzantine textiles

12.20 – 12.40 **DISCUSSION**

12.40 – 13.30 **LUNCH**

13.30 – 15.10 SESSION VII
Chair : *Richard A. LAURSEN*

13.30 – 13.50 *Yoshiko SASAKI and Ken SASAKI*
Dye analysis using portable spectroscopic systems for Japanese historical kimono

13.50 – 14.10 Evangelia A. VARELLA and Zoe Eirini PAPLIAKA
The effect of humidity, temperature and ultraviolet radiation on the colour and molecular structure of contemporary pictorial artworks

14.10 – 14.30 *Omar ABDEL-KAREEM*
The role of natural dyes in preservation of Ancient Egyptian textiles

14.30 – 14.50 *Leopold PUCHINGER, Friedrich SAUTER and R. PALTRAM*
Optimization of pigment production based on madder roots

14.50 – 15.10 DISCUSSION

15.10 – 15.30 TEA AND COFFEE BREAK

15.30 – 17.10 SESSION VIII
Chair : *Leopold PUCHINGER*

***15.30 – 15.50* Leonel Carvalho da SILVA, A. CLARO, M. J. MELO, E. J. CABRITA and L. MAFRA**
Using solid and liquid state NMR techniques to unveil the secrets of alizarin lakes

15.50 – 16.10 *M. M. SOUSA, M. J. MELO, A. J. PAROLA, F. E. COOK and J. A. LOPES*
On natural flavylium chromophores as species markers for dragon's blood resins from Dracaena and Daemonorops trees

16.10 – 16.30 *Karen Diadick CASSELMAN and Takako TERADA*
Murex and orchil methods and technique : Part 2

16.30 – 16.50 *Zahra AHMADI*
Effect of ammonia solution in the natural dyes hue

16.50 – 17.10 DISCUSSION

17.10 – 17.30 TEA AND COFFEE BREAK

17.30 – 18.45 SESSION IX
Chair : *Chris COOKSEY*

17.30 – 17.50 *Cheryl PORTER*
The use of organic red in Mamluk manuscripts

17.50 – 18.10 *Mustafa ARLI, Nuran KAYABASI and H. Sinem SANLI*
Colours used in Turkish carpets and rugs and the plants yielding these colours

18.10 – 18.30 *Barbara BIGLER, Ina VANDEN BERGHE and Andre VERHECKEN*
The colour gamut of madder

18.30 – 18.45 DISCUSSION
18.45 – 19.00 CLOSING SESSION

20.00 – 24.00 DINNER : Bosphorus boot tour

INTERACTION OF 16TH TO 18TH CENTURY ENGLISH YORKSHIRE ALUM PRODUCERS, SALTERS AND LONDON DYERS

David PYBUS

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5000 tons per annum of English alum, produced at nine centres in North Yorkshire in the 1790s, utilised a cumbersome empirical process that changed little since its introduction in the 1600s, and North Yorkshire production only declined when superseded by a cheaper alum production process in the mid 19thC.

The complex family and social interactions between three apparently disparate groups, the North Yorkshire alum producers, salters and London dyers, suggest there was a much closer, and smaller, group of people involved in the trade than was hitherto thought.

Data concerning the North Yorkshire alum producers, amounts produced, and customer demand have been analysed from suppliers records, in association with records concerning London salters and dyers.

AN 18TH CENTURY LONDON DYER'S WORKING NOTEBOOKS : HOW THEY PRODUCED PASTEL COLOURS 'OUT OF GRAIN'

David MITCHELL FSA

David Mitchell, 22 Sydney Street, London, SW 3. England

In 1999, fifteen books relating to the trade of the Crutchley family - red dyers in Southwark, London from the late seventeenth to the early nineteenth century - were lent for a short period to the Victoria and Albert Museum in London.

They included soft covered notebooks often stained with splodges of hot dyestuffs, clearly used in the dye house to record the 'recipe' used to dye a particular cloth. One of these was written in Dutch, and two others, although primarily in English, contained many technical words in Dutch. At the back of two other notebooks were accounts in Spanish. In addition to these documents and two cash book, were three large counting house 'pattern books', bound in vellum. These had entries specifying the owner of the cloth, a fair copy of the dyeing process and one, two or three samples of dyed cloth representing each stage of the process.

This paper will concentrate on two particular aspects of the Crutchley's trade during the first half of the eighteenth century: Its organisation in terms of both personnel and documentation; and the technique of adding dyestuffs to used red vats to produce a range of pastel colours 'out of grain'.

THE WORKING LIFE SPAN OF 17TH AND 18TH CENTURY LONDON DYERS

***Roger FELDMAN*, David PYBUS and David MITCHELL**

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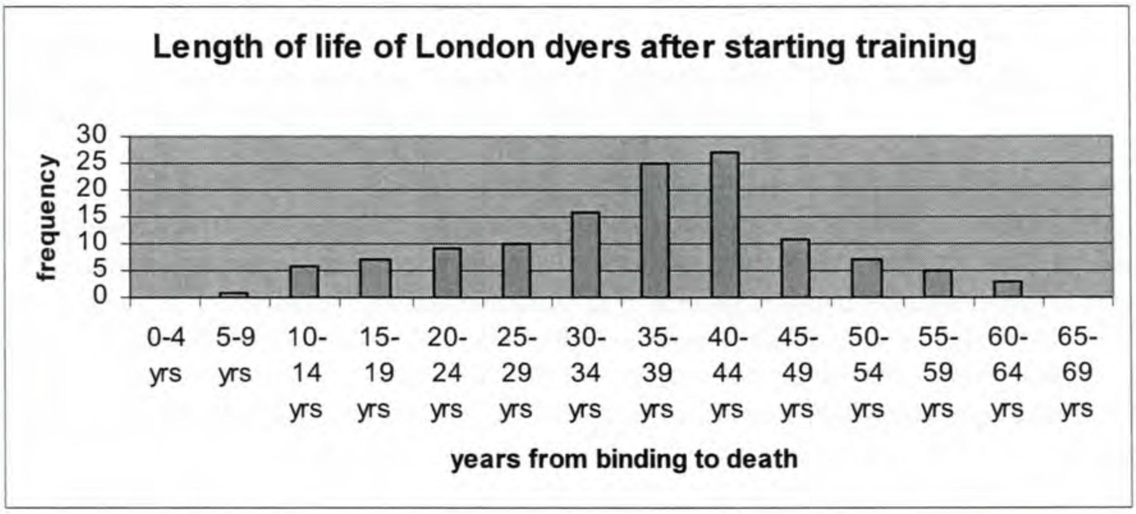
The length of time practicing as dyers has be followed using wills available in the UK Public Records Office, in association with apprenticeship and membership data from the Dyers' Company of London. The information will be presented showing time between beginning training (usually around age 14-15) and setting up a business; the number of apprentices bound by a master during a lifetime, and time between last apprentice bound and death

In the late 17th century, training in silk dyeing became more frequent. Information about the changes in frequencies of specialty occupations in dyeing will be presented.



THE EARLY SYNTHETIC ORGANIC DYESTUFFS : THE PONCEAUS

Matthijs de KEIJZER [1], ***Maarten R. van BOMMEL*** [1] ***and Regina Hofmann-DE KEIJZER*** [2]

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The lecture discusses one group of synthetic organic dyestuffs, the Ponceaus. The French word ponceau means 'wild corn poppy'. This dye class was discovered and patented by the German factory Farbwerke Meister, Lucius & Brüning in 1878. The dye company was founded as Meister, Lucius & Co. by Carl Friedrich Wilhelm Meister, Eugen Lucius and Ludwig August Müller in the village of Hoechst, near Frankfurt am Main, in 1862. At the end of 1864 Müller retired and Adolf Brüning was admitted as a partner. The name was changed to Meister, Lucius & Brüning in 1867. The first dyes produced were fuchsine and aldehyde green. In 1880 it became a stock company: Farbwerke vorm. Meister, Lucius & Brüning.

In 1871 Heinrich Baum (1849-1923) started to work as a chemist in the laboratory of the firm at Hoechst. In the years 1875-'76 he was inspired by the work of Johann Peter Griess, Zacharie Roussin and August Wilhelm Hofmann and he started to investigate the azo dyes. Baum discovered the ß-naphthol-disulfonic acids R (2-naphthol-3,6-disulfonic acid) and G (2-naphthol-6,8-disulfonic acid) in 1878. The dye firm received their first German Reichs Patent 3229 for the discovery of the Ponceaus on the 24th of April 1878.

This patent is the basis of the Ponceau dyes and describes the production and the separation of the ß-naphthol-disulfonic acids R and G; further dyestuffs prepared from both acids. Two other important patents for the Ponceaus are the German Reichs Patent 7217 of the 3rd of December 1878 and the German Reichs Patent 36.491 of the 1st of March 1884, which led to an extension of this dyestuff class. Dyestuffs made from R acid are Ponceau 2G, Ponceau 2R, Bordeaux R and Amaranth. Ponceau dyes prepared from G acid are Orange GG, Crystal Ponceau 6R and Cochineal Red A.

The Ponceau dyes on textiles (wool and silk) show different light-fastnesses. The dyes produced from R acid, such as Ponceau 2G, Ponceau 2R and Bordeaux R, have a poor light-fastness; they are strongly fading within several weeks. The dyes made from G acid, such as Orange GG, Crystal Ponceau 6R and Cochineal Red A, show a good light-fastness and they hardly changed within several weeks.

The history, the chemical constitution, the production and the names of the different Ponceau dyestuffs will be presented. Additionally the identification of these dyes will be demonstrated by case-studies on different art-objects.

Name	C.I. Name	C.I. Number	Constitution
Ponceau 2G	Acid Orange 14	16100	aniline and R acid
Ponceau 2R	Acid Red 26	16150	2,4-xylidine and R acid
Bordeaux R	Acid Red 17	16180	1-naphthylamine and R acid
Amaranth	Acid Red 27	16185	naphthionic acid and R acid
Orange GG	Acid Orange 10	16230	aniline and G acid
Crystal Ponceau 6R	Acid Red 44	16250	1-naphthylamine and G acid
Cochineal Red A	Acid Red 18	16255	naphthionic acid and G acid
Ponceau 6R	Acid Red 41	16290	naphthionic acid and 2-naphthol-3,6,8-trisulfonic acid

PRUSSIAN BLUE, SUCH A FRENCH DYE

François DELAMARE

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While Prussian blue did indeed begin its European career as a *pigment*, it was as a *dye* that it spread in France during the 19th century.

As early as the 18th century, various French chemists (Macquer, Le Pileur d'Appligny) had foreseen and proved that Prussian blue could be used as a dye. This discovery did not interest French dyers, who then used indigo from the French Antilles which was very cheap. It took the break in international trade due to the Continental System (1806) for the French government to have a replacement blue urgently sought. J.-M. Raymond perfected the Prussian blue dyeing of silk (*Raymond blue*) in 1811 and his son, P. Raymond, that of wool cloth in 1822.

The shades obtained were so highly prised that these dyes would be retained after the Empire's fall, when France would import anew indigo, English this time, from the Carolinas.

The fashion for Prussian blue dyeing was then in France the cause for the development of an industry for the production of cyanides and yellow potash prussiate, the major part of which was used for dyeing. Manufacturing processes evolved, but this flourishing situation was brutally brought to an end in the 1860's by the apparition of the first blue aniline dyes.

This was a catastrophe for that industry, which the growth in the market for inks or carbon paper was not enough to sustain. It would take the development of silver and gold recovery technique, through washing their ores in alkali cyanide solutions (1887), for this industry to fully recover.

RED COLOR, WATER INSOLUBLE MEDICINAL EXTRACTS OF SHIKONIN DYES FROM *ARNEBIAE DECUMBENS (BARAGINOCEAE)*

Lamya HAYAT

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The air dried roots of *Arnebia decumbens (Baraginoceae*) were powdered and extracted using continuous soxhlet with different solvents to obtain maximum yield.

Solvent Selection

Solvent	***Percentage leached***
Ethanol	**95.22**
Acetone	**54.08**
Methanol	**51.06**
Chloroform	**47.64**
Toluene	**40.56**
Ethyl acetate	**39.74**
n- hexane	**33.78**
Petroleum ether	**26.82**

The table above shows that the color extracted is water insoluble.The color is maximally obtained by ethanol.

The content of the crude material were specified by acid washed silica gel chromatography. Four different dyes have been identified : Shikonin ; deoxyshikonin acetylshikonin and isovaleryl shikonin. The red crude extract color could be changed to **brownish – red** by adding methanol ; **bluish purple** by adding ferric chloride or **dark blue** solution upon addition of alkaline solution.

The ethanol extract was used to color cotton and wool threads used for weaving rugs in Al- Sado society of Kuwait.

The color is fixed, it did not fade away washing with detergents or exposing it to the sun for a long period of time . This is because of the hydrophobic nature of the dye..

The anti microbial activity of the 4 compounds of the crude extract of Arnebia decumbens were tested against 5 microorganisms. The test resulted in positive effects on *Bacillus subtilis* ; *Sarcina lutea* ; *Eacherichia coli* ; *Saccharomyces pastorianus* and *Candida albicans.* The crude extract of Arnebia decumbens not only works as a coloring material but also protect the woolen rugs against moth destruction functions.

SURFACE IMPROVEMENT ON LINEN NATURALLY DYED FABRICS TO INCREASE DYEING EFFCIENCY

Katarzyna SCHMIDT – PRZEWOZNA

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The Institute of Natural Fibres has participated in a EU research project CORTEX, which was coordinated at the University of Minho in Portugal. One of the Institute's task was to investigate efficiency of CORONA treatment applied to linen fabrics and its effect on results of dyeing the fabrics with natural dyes. The paper presents the analysis of differences of colour obtained on linen samples with and without CORONA treatment dyed with natural dyestuffs. We compared the colour differences obtained on 120 samples dyed with: Annatto *Bixa orellana* L., Tumeric *Curcuma Longa* L., Weld *Reseda luteola* L., Cochineal *Dactylopius coccus* Costa, Cutch *Acacia catechu* Willd,, Madder *Rubia tinctorium* L., Woad *Isatis tinctoria* L

The samples after the process of natural dyeing shown a wide colour range of colours with big palette of shades. The technology of natural dyeing investigated in INF is an example of CORONA treatment applied during cellulosic fabrics finishing process, give better conditions for dyeing and bleaching, as a result of improvement of hydrophilic properties.

Thanks to application of CORONA treatment, it is possible to obtain "GREEN" product with less chemicals, especially for natural dyed fabrics. Main focus of Eco product lies in finishing. Natural colour data obtained using this method are compared with measurements taken with colour spectophotometer.

In our research we are interested in comparing of eco-friendly technology in natural dyeing and creation of modern, ecological textile products.

DYEING PROPERTIES OF ALKALINE EXTRACTS OF EUCALYPTUS'S BARK AND THEIR COMPARISON WITH REACTIVE DYEINGS

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Natural dyes were extracted from Eucalyptus bark using both aqueous and alkaline media. The optimum extract was used to dye cotton fabric by exhaust method. Premordanting with alum while postmordanting with iron resulted in increase colour strength. Fastness properties of mordanted fabric was not much effected except light fastness which showed some improvement. Lastly naturally dyed fabric was compared with dyed with synthetic dye of matching shade. In most of the cases, the washing, light and rubbing properties were equal.

OPTIMIZATION OF ALKALINE EXTRACTION OF NATURAL DYE FROM ACACIA'S BARK AND ITS DYEING ON COTTON BY EXHAUST METHOD

Shaukat ALI **[1,*], *Saima UMBREEN*** **[1], *Muhammad ASIF*** **[1] *and Tanveer HUSSAIN*** **[2]**

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Acacia can be successfully used as ecofriendly natural dye of vegetable origin, to dye cotton material. Dye was extracted in alkaline media for maximum colour extraction using environment friendly extraction methods, without extensive application of organic solvents. If such a fabric is pre and post treated with alum and iron mordant the dyeing showed improvement in depth and performance properties e.g light fastness increased whereas washing and rubbing fastness of sample dyed with natural dye were almost comparable with samples dyed with reactive dyes. The natural dye shades (dyed in absence and presence of mordant) are cost effective in comparison to reactive dyeing. Thus this natural dye could be used as co-partner with synthetic reactive dyes.

INDIGO PLANTS AND WOAD IN ANCIENT ASIA MINOR

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There are many indigo producing plants; however only a few species are known today compared to those used in the past. In her book Dominique Cardon has proposed eleven species from all around the globe. The three main ones are *Indigofera, Isatis* and *Polygonum*. Anatolia and the Caucasus would appear to be possible originating regions for *Isatis*. Moreover, Greek authors and Assyrian or Persian texts propose possible hypotheses for the plants used for indigo blue dyeing. From the Aegean to the Caspian sea and the Euphrates, blue was widely used for wall painting and dyeing; indigo has its part here as well as lapis lazuli and azurite.

Ancient texts and the most recent list of plants present in Asia Minor seem to confirm the importance of Anatolia for the origin of *Isatis* plants, but other texts imply two others species: *Indigofera* and *Polygonum*.

The extraction of insoluble indigo (called *anthrax* in preChristian Egypt and "black powder from the mountain" in the Fertile Crescent) confirms that indigo paste was certainly well-known BCE in these regions. The analysis of pigments in wall painting or dyed tissues is too difficult to enable archaeologists to give a very precise date of the usage of indigo after or during the Neolithic era.

The region of Anatolia and the shores of the Black Sea are essential to the analysis of the spreading of the technology of indigo dyeing throughout the globe, and especially from Asia to Europe.

INDIGO DYEING AMONG THE YORUBAS OF SOUTH WESTERN NIGERIA : A HISTORICAL PERSPECTIVE

Adeola V. POPOOLA

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Dyeing with indigo has become a symbol of Nigerian textile craft. Although other natural and synthetic dyestuffs are available and used, cloths dyed with indigo, having intricate patterns stand out vividly as a legacy of Nigerian contribution to textile arts of the world and will be discussed in this paper. The dye is widely used for two reasons, first, it is found naturally and readily across the entire length and breadth of the country and second, it is used on cotton which is the most convenient form of textile available in the country.

The Yorubas in the south-western region of the country are well known for their skills in dyeing using indigo. In most cases, fermentation vatting technique is the preferred method of dyeing. The essential features of the application process as is practised in the ancient Yoruba art and the socio-cultural relevance of the cloth among the people will be highlighted in this paper.

INDIGO IN ARCHAEOLOGICAL TEXTILES

Maj RINGGAARD [1] ***and Annemette Bruselius SCHARFF*** [2]

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This article will focus on observations and analysis that points towards indigo (*Indigofera* sp.) or woad (*Isatis tinctoria*) in non blue archaeological textiles from wet and waterlogged sites.

Archaeological textiles from wet and waterlogged sites are often brown and yellowish in colour when excavated. They may have a reddish or bluish tint, but dyestuff identifications are necessary in order to visualize the original splendour of these textile. However dyestuff analysis of archaeological textiles does not always give a positive result due to various reasons, and the analysis are also costly for museums, therefore it is important to pre-examine the textile thoroughly prior to a potential sampling for dyestuff analysis.

During archaeological excavations textiles are occasionally found with a characteristic shimmering yellowish - bluish tint, which are similar to the colours observed during indigo dyeing when a textile has just emerged from the dye bath and start to oxidize into its blue hue. They appear as if they were frozen in this partly "reduced stage". An example of this can be seen in the textiles from Lønne Hede (100 AD). Some fragments are yellow in colour which resembles the leuco-indigo stage of the dying process. This poses the question if indigo during burial has been reduced to its water-soluble leuco-form? To investigate this question an experiment was carried out. White wool and urine fermented indigo dyed wool were buried in slightly acidic peat imitating the conditions in a bog. One set of textiles were kept waterlogged and covered with a lid to get an anaerobic condition, the other set were kept in wet peat allowing a small amount of oxygen in the soil.

After excavation the textiles were examined. Although the preservation of the textile fibres from the waterlogged conditions was better than fibres from the wet peat, the colours of both indigo dyed and white wool had changed into light brownish yellow. The indigo dyed textiles in the wet peat had only changed slightly into a greenish blue colour.

During the excavation the colour of the brownish yellow did not change, thus the air did not oxidise the apparently reduced indigotin in the fibres. The anaerobic conditions in the soil have reduced the indigo and the water-soluble leuco-indigo can have migrated into the soil.

The textiles from the experiment and the Iron Age fragments from Lønne Hede of varying brownish blue to yellow colour have been analyzed to see if it is possible to identify indigotin in threads that now are yellow. Pre examination of fibres such as observation in a transmitted light microscope is compared to spot tests, visual spectroscopy and HPLC. The results show that it is possible to identify indigotin in the some of the yellow threads and that a pre examination of these fibres in a transmitted light microscope clearly indicates the presence of indigotin, which is observed as blue spots in the central part of these fibres.

TRADITIONAL RECIPES FOR DYEING WITH NATURAL MIXED DYESTUFFS IN TUNISIA: THE LAST ARTISAN DYEING WITH INDIGO AND MADDER

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Natural dyeing in Tunisia has been preserved until nowadays mainly for the « Fez » (a masculine headdress also called "Chechia") made with kermes or cochineal at Tunis and for the black-bleu blanket made with madder at Tunisian region called the Sahel. This later technique is still practiced by only one family with whom I have carried out inquiry and reconstituted a traditional costume in dyed wool. This work constitutes a safeguard of this ancestral know-how which faces a large number of risks due the difficulty to acquire raw materials and the sale price which remains not very attractive for the artisan. The "Tekhlila" and "El Kenaâ", made at two villages of the Sahel (Kalâa Seghira and kalâa Kebira, respectively) are wool blankets used by women as a veil whenever they go outside their homes in these villages. These blankets are traditionally dyed first with indigo (henna leaves, raisins and indigo) then with madder (natural tartar, alum and madder). A washing with ashes of some plants of the region ensures the fixation of the black-blue colour. This colour, widely used by the Copts in Egypt, could be an influence characterizing these villages of the Tunisian Sahel. The same Hue is found in the parchment's Manuscript of Kairouan, Tunisia (Xth century).This Specificity can be more explored to explain the exchange with different civilisations.

THE IDENTIFICATION OF HISTORIC DYES – PAST, PRESENT AND FUTURE

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This presentation will cover the identification of historic dyes and pigments on ancient and not so ancient artefacts using a variety of physical and chemical techniques. Up to the mid-20th century, analytical tools were too blunt to resolve many of the identification problems. Since then, advances in chromatographic and spectroscopic methods have yielded a wealth of data about the identity of colorants. This allows us, for example, to determine which species of madder was used in dyeing and consequently, sometimes, to determine the geographic origin of the plant. Some of the detection techniques currently in use will be illustrated and some predictions about future developments will be made.

DETERMINATION METHOD FOR THE DYE SOURCES OF HISTORICAL DYESTUFFS

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There is a great importance in determining the type of the insects and the plants which were used to dye historical textile products. Identification the types of the endemic insects and plants give us important clues about where and in which era those textile products were produced.

Nowadays, HPLC method is used widely in dyestuff analyses. Generally, pure dyestuffs are used as a reference substance in those analyses where they cause some major problems. When analyzing historical textiles, the structure of fibers and dyestuff sources peculiar to that era show great differences. To minimize those problems, the analysis steps mentioned below are highly recommended.

1. When analyzing different types of fiber, a determined solution system is to be used.
2. Analyzing different fibers used in historical textiles. Example: Different types of wool samples are used for wool analyses.
3. According to the colour and the specification of the historical object, dye process is held with various insects and plants with different mordants by using historical recipes.
4. HPLC method is used after extraction.

Possessed chromatograms and reference objects are compared to determine the impurity caused by fibers and the dyestuff source which is used to dyed the object. Example: For red color, by comparing different examples dyed with various *Rubia* species, the type of *Rubia* used in dyeing process of the historical object can be determined.

ANALYTICAL PROTOCOL FOR THE IDENTIFICATION OF NATURAL DYES AND ORGANIC PIGMENTS IN MUSEUM OBJECTS, MAINLY BASED ON MS DETECTION

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Subject of many publications, textiles in Romanian museums and monasteries were mostly studied based on artistic and historic criteria. Even if, especially in the last years, an interest in scientific investigation of these objects existed, identification of natural dyes and organic pigments in museum objects from Romanian collections was only possible due to collaborative efforts of Romanian institutions (INCCR, MNAR, Village Museum) and KIK/IRPA Brussels.

Based on the information accumulated within this joint research, the opportunities offered by EU programs (Eu-Artech, COST G8) and due to the access in the high specialized analytical laboratory of LaborMed Pharma Bucharest, it was for the first time possible to build an analytical protocol for the identification of natural dyes and organic pigments by HPLC-DAD-Ion Trap MS, in Romania.

The contribution presents the experiments performed in order to elaborate the database (dyes and standard dyed textiles) together some results obtained for museum objects, with an emphasize on the comparison between Diode Array and Mass Spectrometric Detections.

ON THE FEASIBILITY OF BUILDING LC-MS SPECTRAL LIBRARIES FOR NATURAL ORGANIC DYESTUFFS

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Liquid chromatography (LC) is one of the most common and important techniques for the characterisation of natural organic dyestuffs [1,2]. Further to uv/vis absorbance detection, mass spectrometric (MS) detection is being increasingly used, since it allows to tentatively identify the individual compounds separated by liquid chromatography [3]. The current limitations of this technique compared to gas chromatography coupled to MS detection, however, are the relatively limited structural information that the nowadays most-employed soft ionisation techniques for LC-MS (electrospray and atmospheric pressure chemical ionisation) provide, and the lack of libraries which can be searched.

We will discuss in this presentation which practical approaches can be taken to overcome both limitations, and to produce mass spectra which are richer in information than typically encountered when using standard acquisition parameters. With this higher degree of information, even obtainable on single quadrupole (or ion trap) MS instruments, spectra can be obtained that, after appropriate processing can be used to create spectral libraries that are searchable. This development, illustrated by some representative examples, further increases the usefulness of LC-MS in the characterisation of natural organic dyestuffs.

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CHARTAMUS TINCTORIUS : AN ORGANIC DYESTUFF CHARACTERIZED BY LUMINESCENCE SPECTROSCOPY

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Safflower (*Carthamus tinctorius* L.) is an annual herb, native to southern Asia, which since antiquity has been used, in oriental countries and later in Europe, for dyeing textiles and for producing a deep red pigment. The flower petals contain several water soluble yellow dyes and a red insoluble compound (carthamin) whose chemical structure has already been disclosed and classified into the quinochalcone family of flavonoids (1). The major yellow components are reported to be hydroxysafflor yellow A and safflor yellow B together with a number of kaempferol and quercetin derivatives. All of these molecules belong to the group of mordant dyes which require a pre-treatment of textile fibres with a mordant solution. The mordant is a salt whose metal cation can be complexed to appropriate functional groups of the fibre and, during the dyeing process, it interacts with dye molecules to form an insoluble coloured species, acting thus as a chemical bridge between the fibre and the dye. Carthamin is instead a direct dye which can be directly applied to the textile fibres without special pre-treatments.

In this study yellow and red dyestuffs were extracted from the dried petals of safflower and used both for dyeing wool and silk following documented historical recipes. Since the absorption and fluorescence properties of dyes are highly affected by the chemical and physical micro-environment, for diagnostic and conservation purposes, it is important to investigate the chromatic and spectral changes they undergo under environmental changes. For this reason a preliminary spectrophotometric and fluorimetric investigation was carried out on the petal extracts and on some coloured components, isolated by preparative HPLC. The effect of pH and Al^{3+} cation, which is the most extensively metal used as mordant, on the absorption and fluorescence spectra was also investigated. The spectral behaviour observed in solution was then compared with those detected on dyed wool and silk. The emission of safflower yellow on dyed textiles is shifted to longer wavelength, compared to the solution, due to the effect of complexation with the mordant. Almost no changes were instead detected for the red dyestuff in agreement with its direct dye nature which implies only a weak interaction with the fibre.

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CHROMATOGRAPHIC STUDY OF A YELLOW MATTER HISTORICALLY EMPLOYED IN CULTURAL HERITAGE : *RESEDA LUTEOLA* (WELD)

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Weld (*Reseda luteola L.*, Family Resedaceae) is a plant growing in great abundance and in wide geographic distribution over the world (Europe, Western Asia and North America)[1]. It is one of the first and most used yellow dyes in particular for the natural textiles production [2]. Weld produces flavonoids, in the aerial part of the plant, the most important being luteolin (5,7,3',4' tetrahydroxyflavone).

Main flavones of weld are analysed and quantified by High Performance Liquid Chromatography (HPLC). Several extraction techniques are tested (maceration, reflux and ultrasounds) and an optimal process is defined using ultrasounds.

Weld was used under lake form *i.e* by precipitation on an inert mineral support or charge (aluminium in particular) of yellow dyes, in order to make a mineral pigment. The extraction of colorants from their aluminium complex is realized by using strong acid conditions [3-9] followed by extraction into a suitable solvent
and analysis by HPLC. So, a new simple and sensitive method is performed using only an acetic buffer solution for lake sample treatement before direct analysis in HPLC.

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YELLOW MULTIPLICITY - ATTEMPT TO IDENTIFY DYESTUFFS BY APPLYING MASS SPECTROMETRY

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Yellow dyestuffs are widely represented in nature. What is more, many of them contain similar coloring compounds, *e.g.* flavonoids, present in all plants. Even a finding of colorant arrangement does not often give an unequivocal answer about used dyes. Weld, dyer's broom or sawwort have pretty much the same colorant composition. However, proper study of dyestuffs (insightful analyze and further exploration) can facilitate an. In order to do that, sensitive and selective techniques appropriate identification have to be applied, what is especially important in case of historical objects, when colorants can be degraded under the influence of time, storage or weather conditions. Coupling of high performance liquid chromatography with electrospray mass spectrometry (HPLC–ESI MS) is powerful tool in such research. However, the difficulties in dye identification arise not only from diversity of flavonoid dyestuffs but also from problems with electrospray ionization of other colorants, such as some carotenoids present in saffron or annato. Therefore UV-Vis spectrophotometry is handy and widely applied apart from MS detector.

The coupling of HPLC–UV-Vis–ESI MS allowed to develope method for the identification of wide range of yellow colorants from polar flavonoid glycosides, their less polar aglycones, and curcuminoids, to non-polar carotenoids, such as crocetin, bixin or norbixin. This new method was used to the examination of model wool samples dyed with various dyes as well as fibers taken from a few textiles belonged to the collection of the National Museum in Warsaw (Poland). Two fabrics attributed to Persian weaving centre were analyzed at first, both fragments are dated from the 17th century and depict flower bush. The second group of samples, a few Italian brocatelles were dated from the 16th century. These textiles, rich in decorative wefts, turned out to be valuable research materials and a source of important information for art historians. The obtained results allowed to identify and compare dyestuffs used in different weaving centres.

GCI'S ASIAN ORGANIC COLORANTS PROJECT : A STUDY OF *GARDENIA AUGUSTA* COLORANTS BY HPLC-PDA-MS, RAMAN, SERS AND MICROSPEX

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Getty Conservation Institute's Asian Organic Colorant (AOC) project began in 2006 and was presented at DHA25. This multi-year project systematically investigates biological sources used to prepare dyestuffs and organic lake pigments used in Asia, especially China. The five components of the research are: 1) an extensive literature search that has identified the top 25 AOC biological sources at the Genus level. 2) Acquisition of these biological sources, from China when possible. 3) Preparation of dyed silk dyed wool, dried extract cakes, organic lake pigment and paint in rabbit skin glue binder reference samples. The paint will be applied to mock-up wall painting test coupons and glass slides. 4) Development of an analytical strategy. 5) Analysis of historical samples. This presentation will discuss component 4 with respect to the reference samples prepared from *Gardenia augusta* L.

The goal of the analytical strategy is to use non-invasive techniques to identify organic colorants. If that is not possible, then instrumental methodologies that require minimal sample size will be investigated. As with any strategy, the trade off between invasiveness and sample size will be critically evaluated. Then an integrated analytical strategy for the identification of organic colorants will be designed. This presentation will describe the first such study for the carotenoid colorant, *Gardenia augusta*, using Raman, SERS [1], and microSPEX [2].

All prepared reference samples will be analyzed by HPLC-PDA-MS^n to characterize the components of the biological source and determine any differences between the various substrates: silk, wool, alum, and rabbit's skin glue. While this technique can be used to identify most biological sources, there are a few that pose a significant challenge to even the best liquid chromatographer such as the carotenoid structures. These contain long-chain conjugated double bonds. Most of these compounds do not survive the hydrolysis conditions required to release them from their matrix. Raman spectroscopy, Surface-Enhanced Raman spectroscopy (SERS) and newly developed microSPEX offer exciting identification and detected capabilities. These results will be presented.

This research demonstrates the networking effectiveness of DHA meetings to communicate new techniques such as microSPEX which was introduced at DHA26. (If time permits, an 8-minute video on the Asian Organic Colorants project will be shown.)

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COLOURS AND DYES OF EARLY IRON AGE DANISH TEXTILES

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Denmark possesses a unique collection of prehistoric garments recovered from bogs and burials. They provide an outstanding contribution to the understanding of the prehistoric textile and costume development. For a long time it has been accepted that particularly pre-Roman Early Iron Age textiles of Denmark were uncoloured. Patterns were achieved by combining different shades of naturally pigmented wool: white, black, grey and brown. Consequently all the reconstructions of Iron Age costumes have been conceived in 'black-and-white'. The most recent results of dye analyses conducted by the Danish National Research Foundation's Centre for Textile Research in Copenhagen in collaboration with the Royal Institute for Cultural Heritage (KIK/IRPA) in Belgium, within the framework of the research programme *Textiles and Costumes from the Bronze and Early Iron Ages in Danish Collections* have changed this picture dramatically. Over 200 samples from about 50 textiles dated 800 BC-AD 400 were analysed using high performance liquid chromatography with photo diode array detection, currently the most appropriate technique for natural organic dye analysis. Dye components were detected in the majority of the analysed samples. Most of the textiles included in this study have never been tested for dyes.

The paper will focus on the detected dye components found in the complete series of the Iron Age textile fragments and the interpretation of these results in terms of the archaeological, geographical and cultural context of these finds. Special highlights will be given to some of the most important textiles of the collection. Thus, one of the surprising results is that the famous Huldremose Woman's costume can now be seen in colour: the skirt was blue/purple and the scarf was probably red. Likewise, the leg wrappers of the Søgards Mose Man were blue, while the woman from Krogens Mølle Mose had a skirt with three blue stripes just below the knees.

SOME MORE DYE ANALYSIS ON ROMANIAN ARCHAEOLOGICAL TEXTILES FROM BUCOVINA, 16TH CENTURY

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The presentation is part of a larger project on "Dyes in Textiles from Romanian Collection", developed for more than 10 years between Romanian institutions (INCCR, MNAR, Village Museum) and KIK/IRPA Brussels and continues the research on dyes in archaeological textiles from North Bucovina (15^{th}-16^{th} century).

From the richest archaeological textile heritage preserved in Romania, the results obtained for the group of Mirauti were presented at DHA 26 while some others, discovered in Voronet, Probota, Balinesti Monasteries, St. John the Baptist Church in Siret and belonging to the National Museal Complex in Suceava (CMNB) are included in the present contribution.
The results obtained for the archeological textiles in the two studies, together with those previously detected in medieval textiles (religious embroideries and brocaded velvets) confirm the connections between the Orthodox Romanian space with both the Catholic West and the Ottoman Empire, in perfect accordance with the commercial roads crossing this area.

YOUNG FUSTIC : CHEMICAL COMPOSITION AND IDENTIFICATION IN POST - BYZANTINE TEXTILES

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Microsamples extracted from seven ecclesiastical garments of the post-Byzantine period (15th – 19th cent.) were analyzed by HPLC-PDA. The following dyestuffs were identified: dyer's broom, young fustic, an indigoid dye source (either indigo or woad), madder, cochineal, lac and soluble redwood. The results have been previously published and discussed [1]. Here we focus on young fustic (*Cotinus coggygria* Scop., *Rhus cotinus* L.). Continuation of phytochemical investigation [2] of the methanol extract of the heartwood has led to the isolation of several compounds including sulfuretin and fisetin as well as 7,3´,4´-trihydroxy-flavanone, 5,7,4´-trihydroxy-flavanone, 4,2´,4´-trihydroxychalcone, 2,3 dihydro-fisetin, 2,3-dihydro-quercetin, methyl gallate, 3,4,2',4'-tetrahydroxy-chalcone, quercetin, 4',7-dihydroxy-flavanone, 4',7-dihydroxy-2,3-dihydroflavonol. The aforementioned compounds were identified by HPLC-PDA in extracts of textile (silk and wool) fibers dyed with young fustic (reference samples) and some of them in the historic samples. The effect of degradation processes developed by artificial ageing on the chemical composition of the reference samples is currently investigated.

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DYE ANALYSIS USING PORTABLE SPECTROSCOPIC SYSTEMS FOR JAPANESE HISTORICAL *KIMONO*

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Spectroscopic approach is important for the Non-destructive characterization of natural dyestuffs used for the precious historical textiles. Especially, development of the on-site analytical methods for Japanese historical "*Kimono*" in the warehouses of museums or temples has been required.

The compact and portable spectrometers with optical fiber will be powerful devices for the on-site analysis, although their performances were limited in comparison with those of the bench top instruments. We have developed a portable system combined with visible reflectance and multi-excited fluorescence spectra, and applied it for on-site dye analysis of Japanese *Kimono* and their fragments (17^{th}C to 18^{th}C). The efficiency of this system was verified comparing with the results by the bench-top instruments. In consequence, it was found that the information of minor component of the multi-dyeing was easily obtained by combining visible reflectance and fluorescence emission spectra with multiple excitation wavelengths.

Here we will report efficiency and limitation of these on-site analytical methods and introduce historical dyeing in Japanese *Kimono*.

THE EFFECT OF HUMIDITY, TEMPERATURE AND ULTRAVIOLET RADIATION ON THE COLOUR AND MOLECULAR STRUCTURE OF CONTEMPORARY PICTORIAL ARTWORKS

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Pictorial artworks exhibited or store in museums are affected from many environmental factors – humidity, temperature, ultraviolet radiation and pollutants – leading to gradual degradation of their materials. Thus, for better prevention of damages and efficient conservation, it is necessary to study the behavious of all materials, be it substrate, binding medium or pigments.

In the present article, three organic colorants – alizarin, Hansa yellow PY3 and Hansa yellow PY74 – are investigated concerning the effects of temperature, humidity and ultraviolet radiation on experimental simulations of paintings, prepared as layers with the binding medium polystyrene on an inert substrate. Moreover, since titanium dioxide is broadly used in modern and contemporary pictorial art, acting both as a pure white pigment or a moderator of hue and saturation, the dioxide's role in the inherent protection of polychrome works of art is being investigated.. The experimental simulations are periodically removed from the ageing chamber, and are subjected to colorimetric and spectroscopic measurements. Chromatic changes are related to molecular changes by means of Fourier transform infrared spectroscopy (diffuse reflectance).

A systematic comparative study of all data permits evaluating the materials used as to their stability towards extrinsic factors, and is proposing degradation routes, in order to assist museum curators and conservators in every concrete case related to the pigments examined.

THE ROLE OF NATURAL DYES IN PRESERVATION OF ANCIENT EGYPTIAN TEXTILES

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Dyed textile is one of the most important materials in the Egyptian Museum in Cairo. These dyed textiles seem in a good condition than undyed ones. This study aims to investigate the role of ancient dyes in prevention, reducing or inhibiting the deterioration of ancient Egyptian textiles. Various ancient Egyptian textile samples were collected from the Egyptian museum in Cairo. New textile samples simulating ancient ones were prepared in the lab. to be used as experimental samples in the evaluation of the role of dyes in preservation of textile objects. In this study linen textile samples were experimentally dyed by three natural dyes which were popular in the most of historical periods in the Egyptian civilization. The blue dye was used as an example for vat dyes, the yellow dye was used as an example for direct dyes and the red dye was used as an example for mordant dyes. Unbleached, bleached and dyed linen samples were artificially aged by light to prepare samples simulated to ancient ones. The changes in the color of dyed linen textile samples after aged by light were observed visually. Also the color components (parameters L, a, and b values were recorded). The changes in the parameters L, a, and b values (ΔL, Δa, and Δb) were calculated. Finally the total changes in the color differences (ΔE) were calculated. The extent of the deterioration of the artificially aged textile samples and the ancient samples were determined by various methods such as tensile strength and elongation, X-ray diffraction and FTIR spectroscopy. These methods were used to assess the change in the physical and chemical properties of the undyed and dyed linen textiles. By comparing and discussing all results, it is confirmed that all tested dyes decrease the deterioration of linen textiles. These results confirm that all tested dyes played as protective layers from light deterioration of textile samples. The blue dye is the most protective dye. That may explain why Egyptian Museum in Cairo has dyed textiles in great quantities and in a good statement than undyed one. This study should be completed on different natural dyes and on various types of textile fabrics.

OPTIMIZATION OF PIGMENT PRODUCTION BASED ON MADDER ROOTS

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Since prehistoric times mankind invested a lot of work to develop new and better pigments needed for the production of various kinds of art objects. Lake pigments are produced traditionally by extracting the dyestuff from the plant or animal source, followed by precipitation with e.g. alum, copper sulphate, ferrous sulphate or tin chloride, thus producing insoluble complexes.

One of our tasks within a multinational EU-project [1] was to optimize the production process of madder lake from the roots of *Rubia tinctorum* L. from Turkey in laboratory scale before an industrial pilot plant will be set up.

Starting-point of our own work was a recipe described in [2]. In our studies we determined the optimal parameters for madder lake production by variation of the amount of powdered madder roots in a constant quantity of water, of extraction temperature, extraction time, alum concentration and pH-value. Nearly all our investigations were carried out with powdered material, but the influence of different length of madder root pieces on pigment quantity and quality was also checked. The field of application of the madder pigment decides on the optimal set of parameters to be used. Colour measurements with CIELAB, HPLC of the most important colorants of madder (alizarin, xanthopurpurin, purpurin and rubiadin) and ATR were additionally done to determine the optimal conditions.

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[2] J. Sanyova and J. Reisse. J. of Cult. Heritage 7 (2006) 229-235

USING SOLID AND LIQUID STATE NMR TECHNIQUES TO UNVEIL THE SECRETS OF ALIZARIN LAKES

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The most stable reds used in antiquity are based on the 1,2-dihydroxyanthraquinone chromophore, also known as alizarin (highlighted in Figure 1). In order to be used as pigments in medieval illuminations, oil paintings and other works of art,[1] the extracts were precipitaded in solution with aluminium salts or other inorganic substrates, forming the so-called "lakes" (Figure 1).[2]

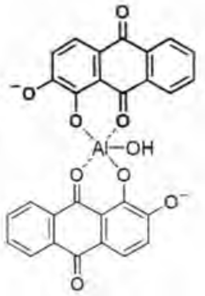

Figure 1

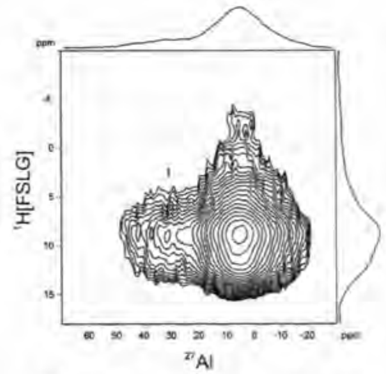

Figure 2 : High-resolution Solid State ^{1}H-^{27}Al HETCOR

These lakes are complex structures and only very recently have been studied by X-ray diffraction[3] and solid state NMR.[4] However the alizarin complexes studied were not made according to ancient recipes. In this work, we present a structural comparison, using liquid and solid state NMR, between those lakes and others obtained by us following methods more close to those described in ancient treatises. Since pigments are used as solid dispersions in a liquid vehicle, we will discuss the advantages of solid state NMR in the characterization of these historic red lakes.

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ON NATURAL FLAVYLIUM CHROMOPHORES AS SPECIES MARKERS FOR DRAGON'S BLOOD RESINS FROM *DRACAENA* AND *DAEMONOROPS* TREES

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Dragon's blood is a natural resin, presenting a rich deep red colour, obtained from trees of the genus *Dracaena* (Dracaenaceae)[1] and fruits of climbing rattans in the genus *Daemonorops* (Palmae)[2]. It has been used for centuries for different medical[3] and artistic purposes[4]. A fingerprint study of the red chromophores in dragon's blood resins from *Dracaena* and *Daemonorops* trees was performed using high performance liquid chromatography with diode array detector (HPLC-DAD) and principal component analysis (PCA). The 7,4'-dihydroxyflavylium was, for the first time, identified as the red natural flavylium in *Dracaena cinnabari* species. From circa fifty samples of *known* dragon's blood sources it was possible to select 7,6-dihydroxy-5-methoxyflavylium (dracorhodin),7,4'-dihydroxy-5-methoxyflavylium (dracoflavylium) and 7,4'-dihydroxyflavylium as species markers for *Daemonorops* spp. [Note 1], *Dracaena draco* and *Dracaena cinnabari*, respectively.

With these natural flavylium markers it was possible to confirm identities of 24 of the 37 samples from the Economic Botany Collection of the Royal Botanic Gardens, Kew (EBC, K), elaborate on 6 and question identities of 6. These natural markers provide a straightforward and quick method for identification of dragon's blood resins species by HPLC-DAD. This was further confirmed by global analysis of the chromatograms by PCA[5].

Note : The 13 species of *Daemonorops* family are currently under study and new results are planned to be presented in this conference.

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MUREX AND ORCHIL METHODS AND TECHNIQUE : Part 2

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Molluscs and lichens yield purple dyes identical in colour. Part 1 of this study investigated the validity of historical references to combined dyes of murex (molluscs purples) and orchil (an AM lichen dye). Our results from Part 1 demonstrated that murex over dyed with orchil, and orchil over dyed with murex, produced purples with superior colour as well as excellent wash and light fastness. Another goal of Part 1 of this study was to establish the best dye technique to use for future experiments. We found that immersion dyeing using a natural murex vat, and orchil dyeing without chemical mordants, resulted in a range of purples on a variety of natural fibres including wool, silk, and cotton. Part 2 of this study will extend the research in three ways:

1. Further investigate dye methods
2. Analyze the chemical basis of the excellent light and wash fastness we have obtained in Part 1
3. Introduce a Japanese orchil based on Asian lichens

Part 2 of the study will compare and contrast dye methods in order to substantiate the most efficient techniques beyond those already used in Part 1 of the study. As we obtained superior light and washfastness in Part 1, in Part 2 we will examine the chemical basis for the superior results. Additionally, we will introduce a vernacular form of orchil based on Japanese lichens. This component of the project will enable us to compare Japanese species to North American and European lichens in order to arrive at a more complete understanding of cultural preferences.

EFFECT OF AMMONIA SOLUTION IN THE NATURAL DYES HUE

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In this paper the hue change of dyed woolen yarn by some natural dyes in ammonia solution has been examined. Ammonia solution improve luster of dyed woolen yarns. Also the level of luster changes by kind of mordant that used in natural dyeing. Benefit and detriment of ammonia solution used for increasing of luster of woolen yarns has been evaluated.

THE USE OF ORGANIC RED IN MAMLUK MANUSCRIPTS

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The paper will briefly examine the notion of colour and its meaning in Islamic art. In particular, the paper will deal with the analysis of organic red colour safflower (Carthamus tinctoria) identified in Mamluk manuscript painting. The method of its manufacture will be examined in the light of Islamic medieval technical treatises, and the method of its application in manuscripts will be described.

COLOURS USED IN TURKISH CARPETS AND RUGS AND THE PLANTS YIELDING THESE COLOURS

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Natural dyeing has been carried out as an ancestral art for hundreds and hundreds years in Turkey. Natural dyeing has been applied at high level by Turks and it has thus constituted a sample for natural dyeing all around the world. It is mentioned that 2/3 of the root dye was exported from Anatolia in 1700s. Although nowadays natural painting is decreasing more and more, it is still maintained in some of our regions and natural dyes are used especially for dyeing wraps and laces in carpets and rugs. That natural dyes are used for rugs and carpets is a reason for preferring these traditional rugs and carpets that have artistic value. Especially these rugs and carpets can easily be marketed in domestic and international markets and sold at special prices and they yield to income. When rugs and carpets are to be dyed, some plants, bodies are used as a whole and some plants' flowers, grains, leaves, shells of trunks, fruit shells, shoots growing inwards, roots etc are used. The color range of the plants used in dyeing is quite large. Besides, mordants used in dyeing also widen the color range further.

In this study, the historical importance of natural dyeing as applied by Turks will be mentioned, colors used in the laces of Turkish rugs and carpets will be categorized, plants yielding to these colors and which parts of these plants are used for dyeing will be detailed.

THE COLOUR GAMUT OF MADDER

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It is common knowledge that evidence for madder dyeing is found in textiles from many different, geographically wide spread cultures and over a very wide time span. The colour gamut of madder dyeing found in historical objects, going from yellow, orange, red, over pink to purple and brown shades is even more intriguing and certainly the reflection of the skills of the local craftsmen.

The rich colour palette of an Anatolian kilim, dated approximately 1850 is used as the concrete starting point for a series of practical dye experiments on wool with the objective to mimic this very wide range of colours, using different madder origins, mordants and post treatments under varying extraction and dye conditions.

These dye experiments will be discussed in relation to their colorimetric characteristics and their influence on the ratio of alizarin and purpurin found after chromatographic analysis.

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The plants of using for dye in Kürecik (Akçadag / Malatya), eastern Anatolia of Turkey

P – 2 : ***Gizem Emre BULUT and Ertan TUZLACI***
Traditional dye plants of Bayramic (Canakkale-Turkey)

P – 3 : ***Aysen SOYSALDI***
The necessity of the education on natural dying in Türkiye, beginning 2000's

P – 4 : ***Fatma Nur BAŞARAN and Nuran KAYABAŞI***
Hand-woven carpets in Konya city Eregli district and yarn dyeing applications

P – 5 : ***Gulzade ABDULOVA***
National methods of dyeing in Azerbaijan

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Textile and dyeing in 4th-14th centuries in Azerbaijan

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Study on historical textile samples from central European collections : Research on type of dyes application period and its geographical distribution and places of origin

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P – 9 : ***Iulia Claudia TEODORESCU and Irina PETROVICIU***
The Transylvanian collection of Anatolian rugs : A permanent challenge

P – 10 : ***Lemonia VALIANOU, Ioannis KARAPANAGIOTIS, Mohammad S. MUBARAK, sister DANIILIA and Yannis CHRYSSOULAKIS***
A brief HPLC-PDA-MS study for the identification of dyestuffs in icons of the Cretan school

P – 11 : ***Lemonia VALIANOU, Recep KARADAG, Ioannis KARAPANAGIOTIS, Turkan YURDUN, Yannis CHRYSSOULAKIS and Emre DOLEN***
The dyes of a silk brocade of the Ottoman period

P – 12 : Seher KARSLI, Türkan YURDUN, Recep KARADAG and Emre DOLEN
Anthraquinones in wool fibres dyed with some *Rubia* species

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Liquid chromatographic study of weld dyes

P – 14 : ***Friedrich SAUTER and Leopold PUCHINGER***
Direct identification of indigo on textiles by means of PY-CGC/MS

P – 15 : ***Ozan DEVEOGLU, Recep KARADAG, Turkan YURDUN and Emre DOLEN***
Pigments obtained with Al, Fe, Sn and Ca metals from buckthorn (*Rhamnus petiolaris* Boiss) plant

P – 16 : ***Emine TORGAN, Recep KARADAG, Turkan YURDUN and Hikmet SAVCI***
Production and analysis of the pigment from madder plant (*Rubia tinctorum* L.)

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P – 20 : ***Skrdlantova MARKETA***
Degradation of dyed textile

P – 21 : Omar BATURAY and Yusuf YILDIZ
General status in solvent dyeing

THE PLANTS OF USING FOR DYE IN KÜRECIK (AKÇADAG / MALATYA), EASTERN ANATOLIA OF TURKEY

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In this study natural plants that used as dye wool and etc. have carried out in Kürecik district. At the same time dye method will be presented. The dye plants are:

Allium cepa L. ; *Armeniaca vulgaris* Lam. ; *Arnebia densiflora* (Nordm.) Ledeb. ; *Berberis crataegina* DC. ; *Crataegus x bornmuelleri* Zabel ; *Crataegus orientalis* Palas ex Bieb. var. *Orientalis ; Euphorbia macroclada* Boiss. ; *Juglans regia* L. ; *Papaver dubium* L. subsp. *laevigatum* (M. Bieb.) Kadereit ; *Papaver macrostomum* Boiss. et Huet ex Boiss. ; *Plumbago europae* L. ; *Quercus cerris* L. var.*cerris ; Rosa canina* L. ; *Triticum aestivum ; Tripleurospermum sevanense* (Manden.) Pobed.

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TRADITIONAL DYE PLANTS OF BAYRAMIÇ (ÇANAKKALE-TURKEY)

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As a result of our ethnobotanical investigations in Bayramiç, twelve plants (cultivated or wild) are used as traditional dye. These are presented below according to their local usages:

a. Dyeing of fibers of wool carpets and rugs (*Alnus glutinosa* subsp. *gutinosa, Juglans regia, Quercus cerris* var. *cerris, Quercus infectoria* subsp. *infectoria, Plumbago europaea, Rubia tinctorum, Vitex agnus-castus).*

b. Dyeing of wear (*Alnus glutinosa* subsp. *gutinosa, Juglans regia, Quercus cerris* var. *cerris, Quercus infectoria* subsp. *infectoria).*

c. Eye-liner *(Juglans regia, Amygdalus communis, Paliurusspina-christi)*

d. Hair dye *(Quercus cerris* var. *cerris, Anthemis tinctoria* var. *tinctoria)*

e. Non permanent tattoo (*Carthamus dentatus*)

f. In a special beverage (*Papaver rhoeas*)

THE NECESSITY OF THE EDUCATION ON NATURAL DYING IN TURKIYE, BEGINNING 2000'S

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In 1950's, the importance and necessity of natural dying for carpet and kilim production, has become awared of. To the studies that has been initiated by the akademicians of Ankara University in those years, in the leading of the DOBAG project of Marmara University, bachelor, master, doctorate course or thesis and books are added in other universities.

The DOBAG project is constituted and put into practice where the tradational rug production have been to introduce the traditional dying techniques. Thanks to these project, Turkish people become aware of the continuability of natural dying. Started in 2005 at İçel-Mersin, Natural Dying Project (DOBOP) that put into practice by Society of Handcrafts and Education (İÇEV), has trained the first natural dying workmans. In the same time, Research and Practice Center of Natural Dying was founded at Tokat Gaziosmanpaşa University.

Also, natural dying is a course in most universities and there are plenty of textbooks for these courses. These courses are tought as theory and in practice. Natural dying experiments are carried out with the traditional method in laboratory workshops.

However, considering time and cost, natural dying is not economic in Turkey, for the reasons of dye plants is not cultivated and the natural dye industry is not developed. Thus, natural dyes which are quite healthy aren't accepted as an valid and easily applicable dying method.

In spite of these conditions, natural dying is getting developed in Turkey, because of some conscious, well-trained people will be carrying on this issue.

HAND-WOVEN CARPETS IN KONYA CITY EREGLI DISTRICT AND YARN DYEING APPLICATIONS

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Anatolian woman, evaluating with her taste and understanding according to her socio-economical and traditional structure expresses her feelings, believes and desire, over the carpets she has woven till her earliest ages. Carpet weaving, which has an important place in art dimension and in country's economy, is defined as little handicraft technically and the force that keeps the art alive depends on the family bases. All family members working in the hand-woven process have information about the preparation of the yarns used in carpets. After the wool spinning process is finished and the yarns used are prepared, the coloration which improves the meaning and value, adds the real pattern property to the carpet, is done. For this process generally the dye plants found in wide variety in Anatolia are used.

The regional difference of the carpets woven for centuries in Anatolia, is both because of pattern and composition properties and because the colors used in carpets. Therefore, the plants cultivated in the region where the carpet is woven, the mordant used and dyeing techniques are the defining factors of the regional properties. This research has importance in order to define the regions where the natural dyeing techniques are used - though decreasing day by day, scientifically and to keep their continuity and to add these techniques to the records. In this paper, the hand-woven carpets from Konya city Ereğli district and applied dyeing process will be introduced.

NATIONAL METHODS OF DYEING IN AZERBAIJAN

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Dyeing of threads takes the important stage in weaving. The important condition in carpet weaving played dyeing by the expert dyer. There are data which proof that dyeing has an ancient history in Azerbaijan. If the weaver weaved a carpet or an oriental carpet for the personal use he necessarily used natural paints. Among people there are 2 ways of dyeing natural dyeing refers to "hasa", chemical dyeing named "emeli".

In empirical experience of people there were different methods of natural dyeing. Some painting plants were used in the dried up kind and some of them were used by people in a natural kind. Time of gathering of plants (in the morning or in the evening, in spring, in summer or in autumn) rendered the big influence on quality of paint. In dyeing of threads basically were engaged in autumn and in spring.

In many processes dyeing of threads mixed. This method of adulteration (mixing) helped at dyeing and added to a thread shine and density. Process of dyeing thread should occur quickly. Experts on craft of dyeing used the empirical knowledge, habits and even personal experience as a result of a plant past test in dyeing in a result was received with beautiful natural paints colors.

With this purpose for reception the most optimum method of reception of natural dye was involved with this purpose all plants and other plants in own "laboratories". Dark blue, blue and green colors it was impossible to receive in domestic conditions. All methods of dyeing were not identical used different technologies.

Traditional stage of weaving was dyeing woolen thread by national way was last chord of preparation of weaving. The big work skill, the habits, required experience and result of the future quality of a material, a combination of colors, tenderness depend on the big work.

TEXTILE AND DYEING IN 4^{TH}-14^{TH} CENTURIES IN AZERBAIJAN

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Different textile patterns belonging to 4^{th}-14^{th} centuries are protected in the National Museum of History of Azerbaijan. According to periods of discover we can divide this textile patterns into three groups:

1. Linen and silk textiles founding from graves belonging to Early Middle Ages in Mingachevir (central Azerbaijan). Remainders of ancient textiles are made from linen in Mingachevir. Linen textiles of this periods are white. Woolen, silk and cotton textiles were widely spread in Early Middle Ages. This kind of textiles were dyed with natural dyes. Natural dyes get from madder (red), wormwood (green), St.-John's wort, walnut, pomegranate, onion-skin (yellow).

2. Silk textiles founding from mongolian graves in Mingachevir. In Mingachevir in mineral area number 48/54 of ancient settlement number 3 in the depth of 1,5 m were found three mongolian graves which are belonging to 13th-14th centuries. Two silk oriental robe and different models of silk were carried out from this graves. Oriental robes were green and there were different ornaments on them.

3. Silk and woolen textiles founding from vault belonging to dynastys of Ilhanid in Kharabagilan (Nakhchivan). This textile patterns were found in 1980 in the grave-yard of Kharabagilan in tomb of the cube shape vault number 2 by the "Kharabagilan" archaeological expedition. Copper coins (belonging to Ilhanid period) found around the tomb, kufic writtings from portal and architectural style of vault shows that it belongs to the end of 13^{th} century. This textiles mainly were maid from silk, some of them were decorated with golden and silver threads some of them there were decorated with writings in different dyes. One of the textiles have also fringes on it. On the piece of woolen textiles we can see many-coloured tracery that are similar to ornaments of carpets.

STUDY ON HISTORICAL TEXTILE SAMPLES FROM CENTRAL EUROPEAN COLLECTIONS : RESEARCH ON TYPE OF DYES APPLICATION PERIOD AND ITS GEOGRAPHICAL DISTRIBUTION AND PLACES OF ORIGIN

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The textile sample collection of the famous late textile dye specialist Dr. Helmut Schweppe contains all his textile samples and his original analytical data. All this information is made available through a data base which we created.

This data base allows an overall view of all used dyes, the used fibres, types of dyes, place of origin of the textile samples and the period of application. The data compilation make interesting diagrams available.

CHARACTERIZATION OF ARARAT COCHINEAL : A MODEL STUDY

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Natural dyes may be classified to different criteria; the most important of which for analytical purposes is their chemical composition.
Most important among these was a red dye, the highly prized "Armenian red", which rivalled royal purple in value. This dye was prepared from the Armenian cochineal insect called *vordan* in Armenian, late known by its name of *kirmiz* (Turkish). The insect was found in the Armenian cities of Artashat and Dvin were particularly renowned for its production. In this paper we report the examination of some naturally occurring organic dyes performed by Pyrolysis gas chromatography mass spectrometry, Laser desorption FTICR and FTIR techniques. Experimental dyes were prepared using old recipes from Armenian fonts.

THE TRANSYLVANIAN COLLECTION OF ANATOLIAN RUGS – A PERMANENT CHALLENGE

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Anatolian carpets have been a part of European culture since the 14th Century and they are among the most sought-after of all carpets by museums and collectors today.

Transylvania is a well know region in north western part of Romania and Lutheran Parishes here preserve a valuable and important collection of Anatolian carpets – over 300 items – dating with 15th and 18th century. Entered in Transylvania in the second half of 15th century as a commercial merchandise, they quickly become an emblem of welfare and proud, a status symbol for a privileged social class. The acquisition detail of that time shows us that they were bought for weddings, gifts or for special occasions.

The high prestige of Anatolian carpets remains during the centuries and we can see them today adorning and warming with their beauty, abstract ornaments and vivid colors the austere walls of Protestant Churches in Transylvania. The art historians consider this "an interesting, complex and unique cultural phenomenon in Europe".

Considering the huge number of Ottoman rugs, the precious designee of the carpets and the fact that a distinctive group of carpets are named "Transylvania rugs" we decided that a scientific examination is necessarily in order to find out more about the process of creation and to correlate and evaluate those results with what we know about the other collections.
Part of this work, the results obtained by HPLC-DAD-MS analysis for red, yellow and brown samples from five carpets belonging to Black Church (Brasov) are discussed in the present poster.

A BRIEF HPLC-PDA-MS STUDY FOR THE IDENTIFICATION OF DYESTUFFS IN ICONS OF THE CRETAN SCHOOL

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The goal of this investigation is to identify the organic colouring matters contained in thirty (30) icons (Benaki museum, Athens, Greece) of the Cretan School of Iconography. Microsamples were extracted from the artworks, which are linked to some prominent representatives of the Cretan School of Iconography such as Dominikos Theotokopoulos. Sample analysis and dyestuff identification were performed by high performance liquid chromatography (HPLC) coupled to photo-diode array (PDA) and mass spectrometry (MS); investigations were carried out after a preliminary validation of the chromatographic method which included a comparison of the signal-to-noise ratios obtained by the PDA and MS detectors. This was achieved using standard materials such as carminic acid (CA), alizarin (AL), purpurin (PU), laccaic acid A (LA), indigotin (IND), kermesic acid (KA), rubiadin (RU) and flavokermesic acid (FL). Most of the standard materials were obtained from the market except for KA and FL which were extracted from their natural source and were purified using TLC, and RU which was synthesized and purified according to literature procedures.

The following organic dyes were identified in the tested artworks: cochineal, kermes, an indigoid dye source (either indigo or woad), a soluble redwood and madder (most likely *Rubia tinctorum* L.)

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THE DYES OF A SILK BROCADE OF THE OTTOMAN PERIOD

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The goal of this study was to identify the organic colouring compounds contained in a silk brocade of the Ottoman period (~16th cent.) from the Topkapi Palace Museum. To increase the credibility of the results, analysis was carried out by two institutions (Ormylia Centre and Marmara University) which used the same analytical technique (HPLC-PDA) but their own sample preparation protocols. Six samples of the following colours were investigated: red, light red, green, blue-black and yellow (two samples). The (major) colouring compound found in the red sample in large quantity was carminic acid (CA). In the light red sample, CA was detected in trace; components of soluble redwood (Bra' and type C compound) corresponded to the major peaks of the recorded chromatogram. Luteolin and apigenin were detected in both yellow samples indicating the use of weld during dyeing. Sulfuretin and fisetin (markers of young fustic) were also found to be present in the yellow samples but in substantially smaller quantities. Comparable peaks which corresponded to luetolin and indigotin were recorded in the chromatogram of the green sample. Indigotin was the major peak of the blue-black sample. Other minor compounds detected in the samples will be presented in the poster.

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ANTHRAQUINONES IN WOOL FIBRES DYED WITH SOME *RUBIA* SPECIES

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Natural dyes and colourants have been used throughout the history. With the popular use of the synthetic dyes, natural dyes and colourants are tend to be used less in most areas. However, dyes derived from natural sources have become a strong alternative to synthetic dyes for the synthetic dyes have been reported to have carcinogenic effects. Considering the fact that the worldwide concern has been focused on the use of eco-friendly and biogradable materials, natural dyes has once again attracted attention [1].

Rubia species are perennial plants which produce a red dye from its roots, naturally grow in Europe, the Middle East and India. Their colouring roots are found on anthraquinone dyes. The main ones are alizarin (1, 2-dihydroxy- anthraquinone) and purpirin (1, 2, 4-trihydroxy-anthraquinone), yet other anthraquinones such as pseudopurpurin, xanthopurpurin, rubiadin and munjistin are also present, mostly in glycoside forms [2,3].

Some metallic salts such as iron free alum which yields the best red; the alumina lake which is rose red or bluish red with calcium; the tin lake which is red-violet or red-brown; influenced the colour of the madder lakes by their amount of usage. The aluminum mordant of alizarin, which is a reputated dye named as Turkey Red, is used to dye cotton and wool with a great rapiditiy.

HPLC is the main analytical technique currently used for the detection and identification of dyes in textile samples [3]. In our study, we analyzed either plant saples of each *Rubia* species (*R. tictorum, R. cordifolia, R. peregrina, R. akane, R. davisiana, R. tenuifolia*) and wool fibres dyed with those plants by HPLC-DAD. Dyestuff substances as alizarin and purpirin, xanthopurpurin, rubiadin and *etc.* were detected in *Rubia* species.

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LIQUID CHROMATOGRAPHIC STUDY OF WELD DYES

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Among the many plants which yield a yellow dye, weld also called *Reseda luteola* L., belonging to the Resedaceae family is a plant widely growing in the world (Europe, Western Asia and North America) [1]. This tinctorial plant is used for textile dyeing process and lake manufacturing [2].

Weld produces flavonoids and more precisely flavone derivatives like luteolin (5,7,3',4' tetrahydroxyflavone) [3]. The main yellow dye constituents of weld were identified by High Performance Liquid Chromatography (HPLC). The calibration of heterosidic and aglycone compounds was realized in order to quantify these molecules in several extracts. Different dye extraction methods are performed in order to determine the optimal dyes extraction conditions from the vegetable matrix.

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DIRECT IDENTIFICATION OF INDIGO ON TEXTILES BY MEANS OF PY-CGC/MS

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A multinational EU-sponsored research project [1] is dealing with ancient colours applied to objects of art and archaeology, whereby the identification of the ancient dyestuffs is a major aspect, thus assisting the restoring of objects of cultural heritage. It is obvious that the methods applied for such analysis have to be as non-destructive as possible, i.e., they should use only tiny quantities of samples and - if possible – avoid prior extraction processes.

Within this project we approached this final goal by means of Py-CGC/MS, testing this method step by step:

In earlier studies we found out that crude ancient dyestuffs indeed could be identified by Py-CGC/FID, comparing their pyrograms to those of the corresponding pure reference compounds.

As the pyrograms alone would be by far too complicated for analyses applied to dyestuffs on textiles, we showed in a second step that the identification of crude dyestuffs plus reference compounds was perfectly feasible by linking Py-CGC to MS.

The present work is now dealing with the application of the Py-CGC/MS method to a dyestuff still being fixed to textile fibres: to start these studies, we used indigo from *Isatis tinctoria* on sheep´s wool as well as on cotton.

Results : The Py-CGC/MS method applied to sheep`s wool and to cotton, both of them dyed with indigo, allowed an unambiguous identification of the dyestuff.

[1] EU research project INCO CT 2005 015406 MED-COLOUR-TECH

PIGMENTS OBTAINED WITH Al, Fe, Sn AND Ca METALS FROM BUCKTHORN PLANT (*RHAMNUS PETIOLARIS* BOISS)

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In this study, aluminium(III), iron(II), tin(II), calcium(II) complexes of natural pigments have been prepared from the dyestuffs of buckthorn (*Rhamnus petiolaris* Boiss) plant berries.

One of the most important dyestuffs present in buckthorn berries is anthraquinone emodin and the other one is flavonoid rhamnetin. Dyestuffs in buckthorn berries were extracted with hot water. Al(III), Fe(II), Sn(II) and Ca(II) solutions were added separatel into the aqueous extract. This medium was neutralized with K_2CO_3 and the pigments involving these complexes were precipitated.

According to HPLC analysis of this pigments, it was determined that both rhamnetin and emodin present in the pigments were precipitated by Al(III) and Ca(II) whereas only emodin was precipitated by Sn(II) and Fe(II). Photographs of the pigments were taken with a stereoscopic microscope with 2625 times enlargement and, crystal structure of pigments were investigated.

L^* (brightness), a^* (red/green) and b^* (yellow/blue) values which constitute the CIELAB colour space of pigments were measured. By using L^*, a^* and b^* values, colour difference (distance) between pigments were calculated.

Buckthorn-aluminium(III), buckthorn-tin(II) and buckthorn-iron(II) pigments were applied on paper, paperboard and cotton together with a binder and their photographs were taken.

PRODUCTION AND ANALYSIS OF THE PIGMENT FROM MADDER PLANT (*RUBIA TINCTORUM* L.)

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Anthraquinones, naturally occurring in the madder roots (*Rubia tinctorum* L.), have been used for dying textile fibers especially to red color and it have also been used as a pigment rarely since ancient times. In addition to their antioxidant, antimicrobial, antifungal, hypotensive effects; anthraquinones have also been known for their various effects such as anticancer and skin diseases.

In the present study, complexes formed with anthraquinones and aluminum (III), iron(II), tin(II), calcium(II) and magnesium(II) were obtained as pigments. From solutions of each metal; 25, 50, 75, 100, 125 mL was added in the extract obtained from plant and pigments were formed. These pigments were analyzed qualitatively by reversed phase high performance liquid chromatography (RP-HPLC). It was determined by comparing standards that the dyestuff alizarin is in aluminum-anthraquinone and iron –anthraquinone pigments; the dyestuffs alizarin, xanthopurpurin and rubiadin were in tin-anthraquinone pigment; and the dyestuffs alizarin, purpurin and rubiadin were in calcium- anthraquinone and magnesium-anthraquinone pigments form pigment with metals. In addition to these dyestuffs which form pigment, other dyestuffs were found in the extract of hydrolyzed madder roots.

The luminance and color values of aluminum-anthraquinone, iron – anthraquinone, and tin-anthraquinone pigments were determined by CIELAB color space system. When both luminance and red color value for aluminum-anthraquinone pigment was examined, it was observed that the best luminance and red color were obtained in the pigment forming with 100 mL alum solution. The best luminance and red color value for iron – anthraquinone pigment was obtained in the pigment forming with 125 mL iron solution, and tin-anthraquinone pigment had the best luminance and red color value for the pigment forming with 100 mL tin solution. Dying properties of the aluminum-anthraquinone, iron–anthraquinone, and tin-anthraquinone pigments were investigated by application on paper, cardboard and cotton.

THE "TUCHLEIN" COLOURS IN THE STRASBOURG FAMILY TEXT : RECIPES FOR ANTHOCYANIN PIGMENTS

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Anthocyanin colorants are characterized by a great sensitivity to the slightest changes in pH and an instability in ordinary daylight. However, the study of the mediaeval German artists' recipes books have highlighted a great number of instructions for the preparation of dyes from anthocyanins. These colours are mentioned under the name "tuchlein" as they were conserved on little pieces of linen cloth. Among this artistic literature, the so-called Strasbourg Family is a corpus of manuscripts containing very similar contents and which are partly derived from an original, now-lost, manual dedicated to illumination. The possibility of collating different manuscripts of the same textual tradition allows us to study the evolution of anthocyanin recipes through time and through different stages of copying, each characterized by variations in their content. These changes are both due to *human factors*, which correspond to errors and missing or misunderstood words appearing during the copy, or *technical factors*, which go hand in hand with some technical or economical choices.

A first series of dummy samples of red and violet "tuchlein" has been prepared from juice of petals of *Papaver Rhoeas* L. The influence of the recipes variations on the properties of these samples has been studied by color spectrophotometry and HPLC .

INDIGO AND OTHER ORGANIC PIGMENTS DETECTED IN PAINTINGS INVESTIGATED AT THE NATIONAL MUSEUM OF ART, ROMANIA

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The contribution continues to present the organic pigments identified by microchemical analysis in the MNAR scientific investigation laboratory in the last 30 years.

If in the first part, presented at DHA 26, we only reffered to the identification of indigo in Romanian tempera painting – icons and painted wooden churches – while the present study shows the results obtained on some18th-19th century icons (tempera) and oil paintings.

Apart from indigo, the most widely identified organic pigment, some red lakes were also identified.

INTEGRATED METHODS IN STUDYING WOOD SUPPORT WEATHERING OF SOME SACRAL MODERATE PAINTS FROM STELEA-TARGOVISTE CHURCH

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The purpose of this study is clarifying the degradation type of artefacts wood support of ~450 years old, actually wethered by embedding rosin and bees wax 50 years ago. Thus, changes in the infrared spectra of wood samples due to the decay were recorded. Ass the sub-purpose, possibility of the application of the integrated methods: infrared and Raman spectroscopy, EDAX, XPS, AFM to the measurement of the degree of this decay were examined.

Changes in infrared spectra have been recorded with the comparison to wood belongs to different periods of time, fresh bees wax and rosin [1].

Consequently, it had been expected from this study that some changes in molecular structures of wood components would appear even at the initial stage of decay. The shift of the bands at 1730, 1380 and 1050 cm^{-1} in the FTIR spectra would be better correlated with FT-Raman spectra and the EDAX, XPS, AFM methods [2-4].

Considering the complex decay of rosin, bees wax and wood fiber of the support we developed a paralell the standard protocol of these integrated methods on different types of frash and laboratory weathered wood, bees wax and rosin samples. [3-4].

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DEGRADATION OF DYED TEXTILE

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The contribution deals with the study of dyed fiber degradation in dependence on fiber types and used dye types. The samples of dyed and colorless fibers were submitted to artificial ageing (simulation of accelerated ageing conditions in a climatic chamber with adjustable temperature, relative humidity (RH) and UV radiation). The textile fiber degradation was evaluated with the use of instrumental analytical techniques (FT-IR spectroscopy, X-ray diffraction) and laboratory techniques (copper number, intrinsic viscosity). The results gained from individual measurement methods are mutually compared and discussed.