

Accepted Manuscript

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PII: S0026-265X(18)30539-3
DOI: [doi:10.1016/j.microc.2018.07.043](https://doi.org/10.1016/j.microc.2018.07.043)
Reference: MICROC 3284
To appear in: *Microchemical Journal*
Received date: 5 May 2018
Revised date: 30 July 2018
Accepted date: 30 July 2018

Please cite this article as: Gemma María Contreras Zamorano , The presence of iron in inks used in Valencian manuscripts from the 13th to 17th century. *Microc* (2018), doi:[10.1016/j.microc.2018.07.043](https://doi.org/10.1016/j.microc.2018.07.043)

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The presence of iron in inks used in Valencian manuscripts from the 13th to 17th century.

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Abstract: The significant artistic and documentary contribution of the manuscripts makes them unique instruments of knowledge that we must protect and conserve. Most of the manuscripts of Europe and America were written with iron gall inks, documented from the 4th century [1] to the 19th century [2]; we have evidence of the problems of conservation since the 17th century because the same scribes echoed similar problems through their writings[3].

The Valencian archives conserve numerous documentary collections from the period after the Conquest, but those in the stage between 1450 and 1600 stand out because of their deficient state of conservation.

For this reason we have endeavored further identify of the components in these inks in relation to their preservation. Given the high iron content of these inks, a systematic study of 989 micro-samples has been performed. The iron concentration ratio results obtained have been evaluated depending on the dates and the archives to which the micro-samples belong, with the semi-quantitative results offered by SEM / EDX analysis. These samples are not homogeneous; and the proportion of ink in the paper could vary, and the current filament values could also be different.

A statistical study has been carried out using an ANOVA analysis of iron concentration as a function of the archive. There is an increasing trend in the means of iron between the thirteenth and seventeenth century. Meanwhile there is no geographical difference. Ten homogeneous groups were identified. These values can be related to the incorporation of new organic and inorganic ingredients in the preparation of recipes, as well as the difference in preparation and concentration of iron sulphate according to the solvent used. There is significant evidence that the highest extremes of iron concentration belong to the years between 1450 and 1550, which corresponds with the most deteriorated documents.

The integrated study of historical sources and elementary analyses help to recognize both the materials and their state of conservation, which makes the process of conservation and restoration easier.

Keywords: iron gall ink; SEM-EDX; iron sulphate; manuscripts; recipes

1. Introduction.

Iron gall inks were the normal writing material used in European and American manuscripts from the 4th to the beginning of the 20th century. They are composed by elements that are degraded because of aging processes, acidification, and oxidation. They even go so far as piercing the writing box and putting the conservation and integrity of the manuscript, and the unique information it contains in danger.

Ink is a mixture made up of different organic and inorganic ingredients that react with each other to generate a liquid product that is consistent and also suitable for writing.

Considering the variety of materials that can be added to preserve, to create odour, to thicken, etc., the possible resulting chemical combinations are innumerable. The most important element is iron, as it produces the final compound.

The reaction between Gallic acid and iron sulphate creates the black coloured ink that we know. In order to obtain this acid it was essential that the vegetable polyphenols (galls, grenade crust, etc) were hydrolysed. This process was realized by maceration or by boiling the different components mainly in water or wine. As raising the temperature enhances the reaction, sometimes the maceration was exposed to the sun, or alcoholic products were used instead of

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