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Grisaille Production: Some Examples from the 14th to the 20th Centuries

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Production de grisaille : quelques exemples du XIVe au XXe siècle – Résumé

La grisaille était traditionnellement fabriquée par les vitriers et les verriers à l'aide d'un verre à base de plomb mélangé à des métaux brûlés (fer ou cuivre). À partir de 1855 environ, ces matériaux ont commencé à être produits de façon industrielle, ce qui a conduit à la création de formules nouvelles et standardisées. L'objectif principal de ce travail est d'analyser les matières premières de cette peinture à base de verre pour mieux comprendre leur évolution au fil du temps.

Des fragments de vitraux de la cathédrale de Léon (Espagne), de la cathédrale de Nidaros (Norvège), de l'église du Saint-Esprit à Madrid (Espagne), de la cathédrale d'Uppsala (Suède), de l'église de Groziec (Pologne), de l'Atelier de Restauration de Canterbury (Royaume-Uni et France) et échantillons de vitraux belges, ont été étudiés. Les chronologies des échantillons se situent entre le XIVème et le XXème siècle. Les fragments ont été analysés par la technique PIXE (particle-induced X-ray emission) et les résultats ont été comparés avec les données disponibles dans la bibliographie.

Les résultats préliminaires concernant la composition des échantillons permettent d'identifier l'apparition de différents métaux au début du XIXe siècle, comme les oxydes de cobalt, de chrome et de manganèse. Ces nouveaux composés apparaissent généralement avec les oxydes de fer ou de cuivre, qui sont les principaux colorants identifiés dans les échantillons plus anciens. En général, la composition des verres de base présentait moins de PbO que prévu dans les recettes historiques, et les proportions entre le verre de base et les agents colorants n'ont pas beaucoup changé avec le temps.

Grisaille Production: Some Examples from the 14th to the 20th Centuries – Abstract

Grisaille was traditionally produced by glaziers and glass-painters using a lead-based glass mixed with burned metals (iron or copper). From around 1855, these painting materials started to be industrially produced leading to the creation of new and standardized formulas. The main objective of this work is to analyse the raw materials of this glass-based paint for better understanding their evolution throughout time.

Stained-glass window fragments from the Cathedral of Léon (Spain), Nidaros Cathedral (Norway), church of the Holy Spirit in Madrid (Spain), Uppsala Cathedral (Sweden), church of Groziec (Poland), Restoration atelier of Canterbury (UK and France) and samples from Belgian stained-glass windows, were studied. The chronology of the samples is between the 14th and 20th century. The fragments were analysed by PIXE (particle-induced X-ray emission) and the results compared with previous ones in the literature.

As preliminary results regarding the sample's composition, it was possible to identify the appearance of different metals, such as cobalt, chromium and manganese oxides in the samples from the beginning of 19th-century. These oxides usually appear together with iron or copper oxides, which are the main colouring agents in the older samples. In general, base glasses presented a lower content of PbO than expected from the historical recipes, and the proportions between the base glass and the colouring agents did not change much through time.

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1. Introduction

Grisaille is a glass-based paint applied in the production of stained-glass panels, generally used for the creation of outlines and shadows.[1] These paints appear typically in dark colours (black, brown) and are the oldest painting materials and the most used in stained-glass windows.[2] Written sources, such as the 12th-century *De diversibus* artibus by Theophilus,[3] describe the production of grisailles by mixing metal oxides (Fe_2O_3 and/or CuO), as colouring agents, with a ground lead-based glass, responsible for the

[1] Machado et al. 2019.
[2] Schalm 2000.
[3] Hawthorne & Smith 1979.

adhesion of the grisaille to the glass substrate.[4] The mixture thus obtained is applied on the glass substrate after being mixed with a vehicle, such as vinegar and water, which will give the necessary plasticity to paint, and gum Arabic as a temporary binding agent.[5] After being fired at temperatures between 650°C and 700°C, an opaque and heterogeneous layer of colourless glass with metal oxides dispersed is formed on the top of the glass panel.[6]

Table 1 – Data sources of the grisaille samples studied in the bibliography

Country	Chronology	References	Country	Chronology	References
France	10th century	Bettembourg 1991	Portugal	15th century	Vilarigues et al. 2009 Fernandes et al. 2008 Vilarigues et al. 2004
	13th century	Vilarigues et al. 2019 Bettembourg 1991 Perez y Jorba 1991 Perez y Jorba 1984 Verità 2003 Verità 2010		16th century	Vilarigues et al. 2009 Vilarigues et al. 2019 Fernandes et al. 2008
	16th century	Verità 1996		20th century	Vilarigues et al. 2009 Fernandes et al. 2008
Germany	13th century	Bettembourg 1991	Belgium	17th century	Schalm et al. 2010
	14th century	Bettembourg 1991 Verità 2010		19th century	Schalm 2000 Vilarigues et al. 2019
	15th century	Marschner 1996 Bettembourg 1991		20th century	Vilarigues et al. 2019
Italy	13th century	Verità 2010	Low Countries	15th century	Vilarigues et al. 2019
	14th century	Verità 2010 Verità 1996 Bollati et al. 2010		16th century	Vilarigues et al. 2019
	15th century	Verità 1996 Plaisier et al. 2017 Échard et al. 2008		17th century	Vilarigues et al. 2019
	16th century	Verità 1996.	Switzerland	16th century	Machado et al. 2017
	19th century	Alberta et al. 2011		17th century	Vilarigues et al. 2019 Machado et al. 2017
Spain	13th century	Carmona et al. 2006 Palomar 2013	Czech Republic	13th century	Cílová, et al. 2015
	15th century	Palomar 2013		14th century	Cílová, et al. 2015
	16th century	Pradell et al. 2016 Carmona et al. 2006		15th century	Cílová, et al. 2015
	17th century	Pradell et al. 2016	Poland	15th century	Bernady et al. 2018
	19th century	Pradell et al. 2016	United Kingdom	13th century	Spencer et al. 2018
	20th century	Pradell et al. 2016 García-Heras 2005 Palomar 2013 Carmona et al. 2010			

[4] Machado et al. 2019.
[5] Machado et al. 2019; Schalm 2000; Pradell et al. 2016.
[6] Schalm 2000; Machado 2016.

Investigations concerning stained-glass windows have been growing over the years;[7] however, they have mainly focused on the materials characterization of the glass substrate, with few studies of the painting materials. Since 1984, only 27 published studies were found on grisaille painting, shown in Table 1.

In these studies, the main metals responsible for the colour were iron and copper oxides (Fe_2O_3, CuO), used individually or in combination. Zinc was also found in older grisailles (15th century),[8] as well as cobalt, chromium and manganese in recent ones (19th and 20th centuries).[9] These metal oxides are mixed with a base glass described as a high lead-silica glass with different proportions between the lead oxide and silica content. Some authors also identified the use of borax since the 17th century.[10]

In Vilarigues et al. 2019, a first spatiotemporal study of the grisailles compositions was made. Some conclusions were drawn regarding the colouring agents used, being identified the iron oxide as the favourite used and with the highest proportions in central and western Europe. The eastern European countries were the ones where a higher content of copper was used together with iron, in a ratio iron:copper of 1:1 or 1:2. It was also compared the proportion between the base glass and the colouring agents, seeing that it is constant over the centuries in around 40-50 %, except on the 15th and 17th centuries, when the grisailles presented up to 80 % of base glass.

In this study, selected grisaille samples will be characterized and compared with grisaille compositions from the literature (Table 1), with the aim of tracing the evolution of this glass-based paint composition based on its provenance (country where it was produced) and chronology.

2. Materials and methods

2.1. Historical grisailles

Twenty-one grisaille samples were chosen to be chemically characterized from seven different collections: Joost Caen's private collection (JC), Belgium; Cathedral of León (CL), Spain; Holy Spirit Church of Madrid (HSC), Spain; Nidaros Cathedral (NC), Norway; Uppsala Cathedral (UC), Sweden; Church of Grodziec (CG), Poland; and from the Restoration atelier of Canterbury (LS), France and UK. The chronology of the samples is between the 14th and 20th centuries. The samples are represented in fig. 1.

To have a better spatiotemporal map of the grisaille compositions and a greater representativeness, grisaille compositions from the literature were also selected for comparison. Only the studies with specific compositions of the grisailles filling the spatiotemporal gaps were selected. The studies were selected from the ones shown in Table 1: Marschner 1996, Verità et al. 2003, Vilarigues et al. 2004, Fernandes et al. 2008, Verità, 2009, Cílová et al. 2015, Machado et al. 2017, Bernady et al. 2018, Spencer et al. 2018, and Vilarigues et al. 2019.

In this study (sample analysis and literature) compositions from the 13th to the 20th centuries were compared from thirteen different European countries (Portugal, Spain, France, Belgium, Germany, Low Countries, Switzerland, Italy, Czech Republic, Poland, Sweden, Norway, United Kingdom).

2.2. Analytical techniques

The chemical compositions from grisaille samples were obtained by µ-PIXE. Whenever possible the samples were cut with a steel wire with a diamond point and mounted in epoxy resin (Araldite 2020) from Huntsman™ and polished with up to 4000 grit with a SiC paper, to analyse by internal beam. An external beam was used for the samples that could not be mounted in resin. Further details on sample preparation and analysis can be found elsewhere.[11]

To better compared the results, as well as the compositions taken from the literature, concentration ratios were calculated: regarding the proportion between lead oxide and silica in the base glass (e.g. $PbO/(PbO+SiO_2)$), regarding the proportion of the different colouring agents (the colouring agents considered were Fe_2O_3, CuO, CoO, MnO, Cr_2O_3 (e.g. $Fe_2O_3/(Fe_2O_3+CuO+CoO+MnO+Cr_2O_3)$), and the proportion between the base glass (BG) and the colouring agents (CA) (e.g. $(PbO+SiO_2)/((PbO+SiO_2) +$

[7] Palomar 2018; Teresa Palomar et al. 2018; Corrêa et al. 2018; Adlington & Freestone 2017; Legrand et al. 2019.
[8] Vilarigues et al 2009; Marschner, 1996.
[9] Pradell et al. 2016; Vilarigues et al 2009; Schalm et al. 2003; Carmona et al. 2009; García-Heras et al. 2005.
[10] Pradell et al. 2016.
[11] Machado et al. 2020.

(Fe_2O_3+CuO+CoO+MnO+Cr_2O_3))). Afterwards, averages of the results for the different grisailles were calculated for each country and century.

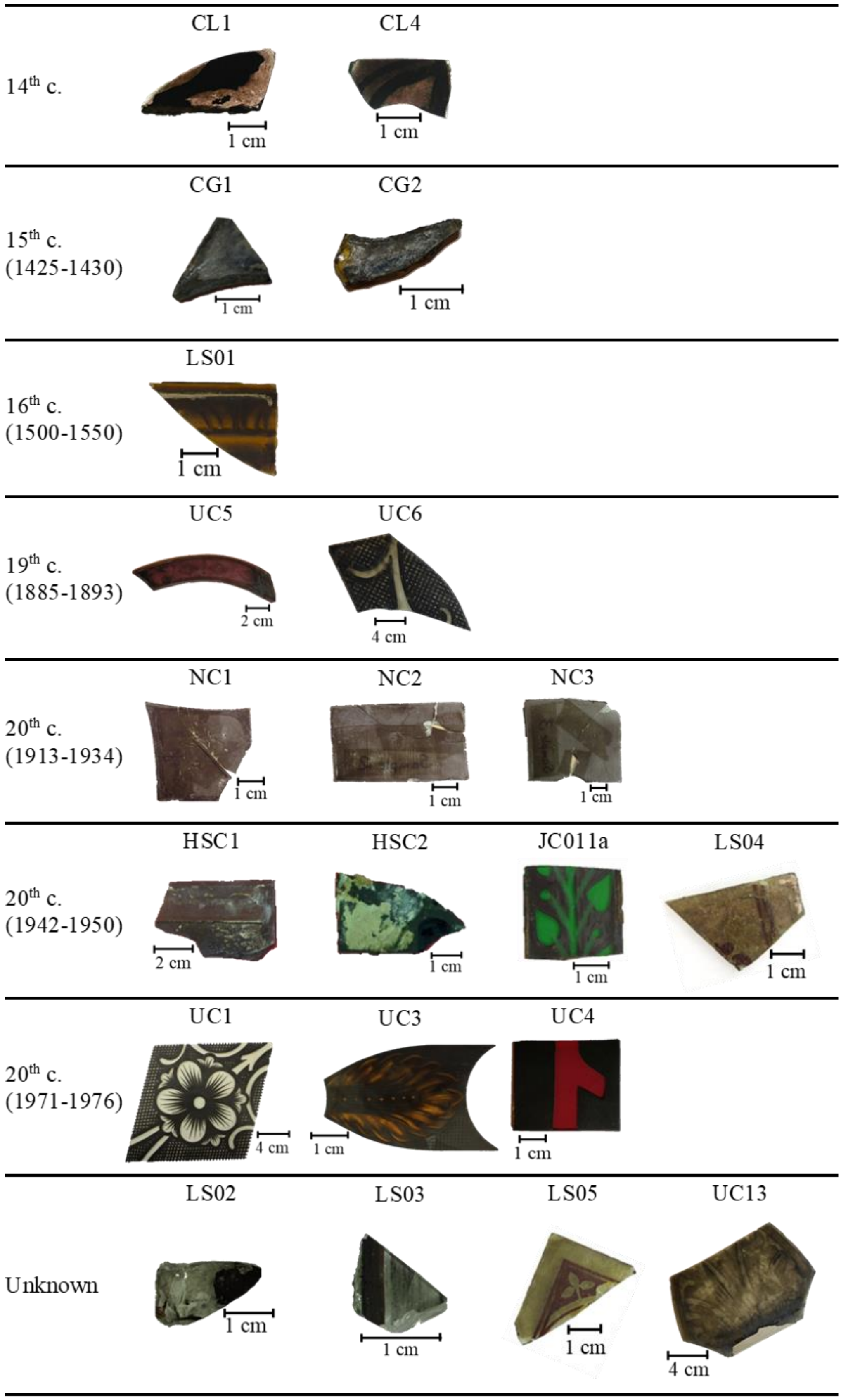

Fig. 1. Selected samples to be analysed in this study.

3. Results and discussion

The distribution of the different colouring agents in the grisailles according to their provenance and chronology is represented in fig. 2. It is possible to see that the iron oxide is the main colouring agent used in most of the countries and across the centuries. The iron presents a more than 50 wt. % concentration in the western and southern-western European countries (United Kingdom, Low Countries, Belgium, France, Switzerland, Spain, Portugal), as well as in the Polish grisailles. However, copper oxide was preferentially used in central and south central European countries (Germany, Italy, Czech Republic).

Further research may reveal how these geographical preferences reflect different relations between the countries, not only in commercial trade of raw materials but also in knowledge transfer between countries.

In the northern countries (Norway, Sweden) it was possible to see the appearance of other different colorants in higher proportions, for example, the chromium, cobalt and manganese oxides, together with iron and copper oxides. This is related to the chronology of the grisailles from this period as they are all from the 19th and 20th centuries where it is also possible to see the appearance of the use of these metal oxides.

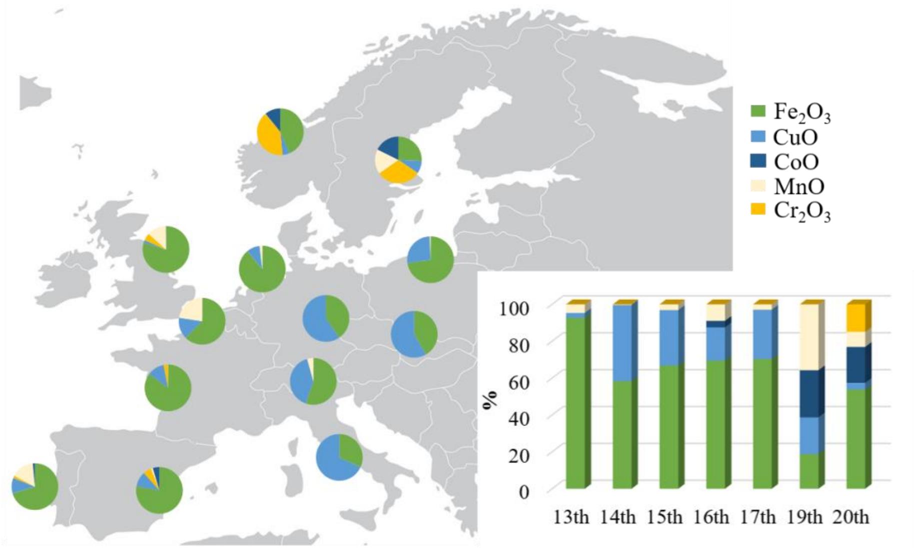

Fig. 2. Geographical and chronological distribution from the different colouring agents' proportions.

Regarding the base-glass compositions, fig. 3 shows the results from the proportions between the different base-glass components (PbO and SiO_2). In this case, they are not directly related to a geographical distribution showing a great variety of proportions.

Eight countries (Portugal, Belgium, Low Countries, Switzerland, Czech Republic, Poland, Norway, United Kingdom) have more than 50 wt. % of SiO_2, and only five countries (Spain, France, Germany, Italy, Sweden) have higher amounts of PbO. If these compositions are compared with the historical recipes from each country, it is possible to detect a higher amount of silica in the historical grisailles than the expected one. In the historical written sources, base glasses used to present proportions of lead oxide and silica of 1.5 to 3, but historical grisailles presented a ratio PbO:SiO_2 is around 1 (fig. 3).[12] Nevertheless, these results can be explained either by the volatilization of the lead during the firing process, leaving the final grisaille with a lower content of lead, or due to a degradation process by acid attack. In the latter, lead ions can interact with compounds from the atmosphere (CO_2, SO_2, etc.) to form soluble products that would be cleaned by the rain leaving a silica-enriched glass.[13]

From the chronological point of view, it is observed a higher proportion of SiO_2 (~60-70 wt. %) on 16th, 17th and 20th centuries. This can be related with addition of new components to the grisailles

[12] Machado et al. 2019.
[13] Verità 2010.

composition, such as the borax, that can decrease the melting temperature without a high amount of PbO.[14]

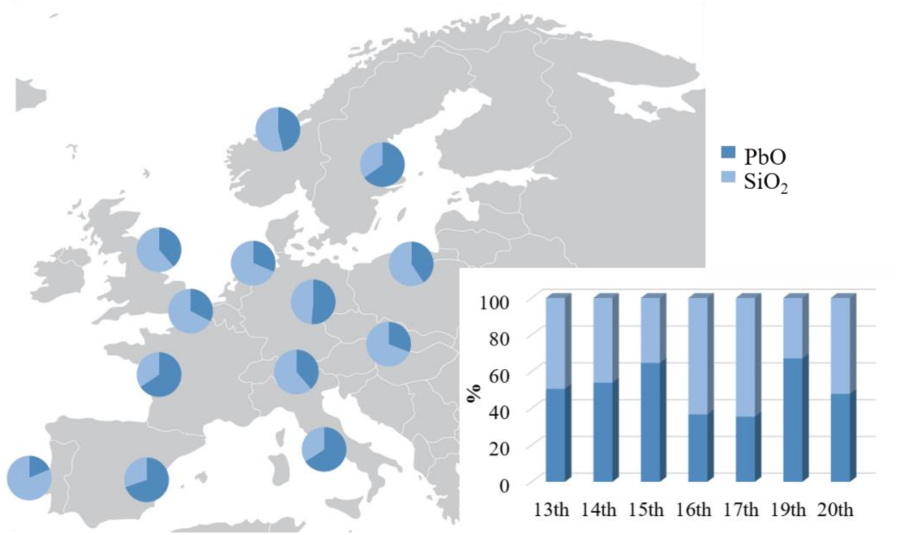

Fig. 3. Geographical and chronological distribution from the different base glass components proportions.

The comparison between the proportion of the two different elements of the grisailles, the base glass and the colouring agents, was also made. It is shown in fig. 4.

The graph shows that the average for the proportion of base glass to colouring agents is around 60-80 wt. % all over the different centuries, being higher on the 13th, 19th and 20th. These results are contrary with the ones obtained by Vilarigues et al. 2019, that showed the highest amount of base glass to colouring agents (up to 80 wt. %) in grisailles from the 15th and 17th centuries. The different result is related to the low number of samples analysed. To have a better representativeness, it is necessary to compare a higher number of samples.

Regarding the geographical distribution it is possible to observe that in most of the countries, except in the Czech Republic, the proportion of base glass to colouring agents is more than 50 wt. %. This implies a higher stability of the grisailles. Higher ratios of colouring agents induce a porous grisaille being more susceptible to abrasions, humidity and degradation processes.[15]

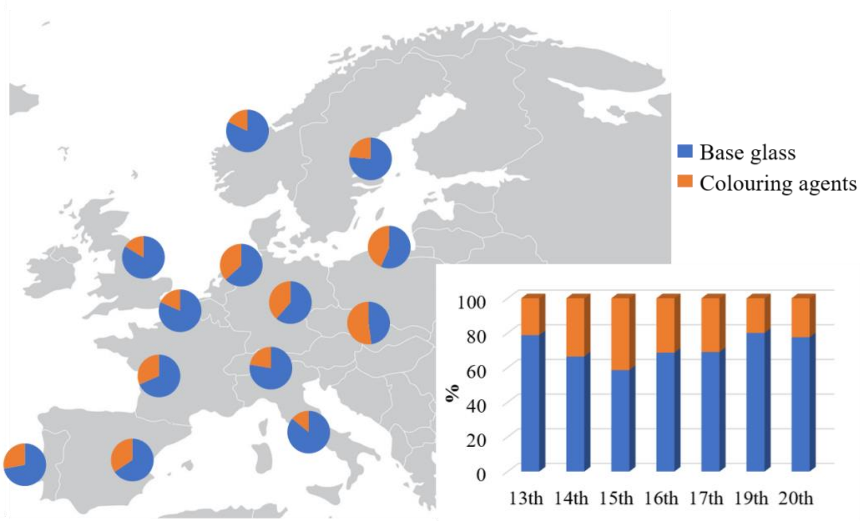

Fig. 4. Geographical and chronological distribution of the proportions between the base glass and colouring agents.

4. Conclusions

In the present study, it was possible to draw some conclusions regarding the spatiotemporal evolution of grisaille paint production, reflected in its changes in composition.

[14] Pradell et al. 2016.
[15] Palomar 2018; Bettembourg 1991.

The iron oxide was the most used colouring agent, except in central and south central European countries, where copper oxide was favoured. In the northern grisailles the cobalt, manganese and chromium oxides were also identified as colouring agents; however, this relates to modern grisailles (19th and 20th centuries). The base glass composition was generally uniform ($PbO:SiO_2$ 50-60 wt. %) except during the 16th, 17th and 20th centuries when the proportion was lower (35-50 wt. %). In the proportion between the base glass and the colouring agents, the differences between the different centuries were not remarkable (60-80 wt. %) except on the 15th-century samples when they were lower than 60%.

When comparing all the results, it was possible to observe that the samples from 16th and 17th centuries presented similar compositions. However, this happens because almost all studied samples from these centuries have the same provenance (Low Countries and Switzerland). For a more representative study, samples from each country with different chronologies would be needed, but their scarcity and high value of them makes this difficult to achieve.

To conclude, this study was the first step towards establishing patterns of evolution in the grisaille production and composition, as well as, towards the creation of a database of grisaille compositions that will be useful for a better understanding of stained-glass windows and their painting materials.

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