CHEMICAL CHARACTERISATION OF CERAMIC INKS USING SPECTROSCOPIC TECHNIQUES

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1. INTRODUCTION

The inks used in the manufacture of ceramic materials generally contain a mixture of organic compounds and an inorganic fraction consisting of a ceramic pigment. The mixture of organic compounds consists mainly of the following constituents: a solvent, which is usually a glycol or an ester, though a mineral or vegetal oil can also be used; a polymer, which may involve either an acrylic resin, butyl acetate, or PVC, etc.; and smaller quantities of certain additives: surfactants, dispersants, antifoamers, etc.

In order to control the composition of these materials and to optimise the product, it is of particular interest to have detailed information on the product constituents.

Spectroscopic techniques such as Fourier transform infrared (FT-IR) spectrometry, wavelength-dispersive X-ray fluorescence (WD-XRF) spectrometry, and elemental analysis by combustion and IR or thermal conductivity detection enable these types of materials (inks, vehicles, additives, etc.) to be characterised. On the one hand, a structural analysis is performed using FT-IR spectroscopy, which provides information on the functional groups that are present and, therefore, on the type of organic compound. On the other, WD-XRF spectrometry provides information on the elemental composition of the inorganic fraction, enabling the type of pigment used to be identified. Finally, the elemental analysis of C, H, N, and S complements the information obtained by FT-IR.

2. EXPERIMENTAL PART

In this study, a method has been developed that enables ceramic inks to be characterised using FT-IR and WD-XRF techniques, and C, H, N, and S elemental analysers. The method was fine-tuned by performing the relevant calibrations and validations. Since reference materials of ceramic inks are commercially unavailable, synthetic standards were prepared by mixing reference materials and reagents of high purity in order to have materials with a composition similar to that of ceramic inks.

Synthetic standards were prepared for the calibration and validation of the WD-XRF measurements using the following certified reference materials and chemical standards: BCS-CRM 313/1 High Purity Silica, EURONORM No. 782-1 Dolomite, BCS No. Grecian Chrome Ore, BAM 633/1 Manganese, GBW Wollastonite, BCS-CRM No. 388 Zircon, SRM 670 Rutile, SRM 25d Manganese Ore, SARM 34 Andalusite, SARM 13 Zirconium concentrate, Cr_2O_3 from Merck, ZnO from Aldrich, NiO from Aldrich, BCS No. 314 Silica Brick, EURONORM-CRM No. 682-1 Iron Ore, Cr_2O_3 from Fluka, ZnO from Merck, and NiO from Fluka.

For the FT-IR identification, the following reference materials of organic compounds were used: Isopropyl myristate from Alfa-Aesar, Ethylene glycol from Merck, Ethyl acetate from Sigma-Aldrich, Lauric acid from Acros Organics, Dipropylene glycol monobutyl ether from Sigma-Aldrich, and Polypropylene glycol from Sigma-Aldrich.

3. RESULTS

An example follows of the complete analysis of a ceramic ink using the three techniques mentioned above: FT-IR, WD-XRF, and elemental analysis by combustion and IR and thermal conductivity detection. The results obtained in the identification of the major compounds in the organic fraction of the ink are shown in Figure 1.

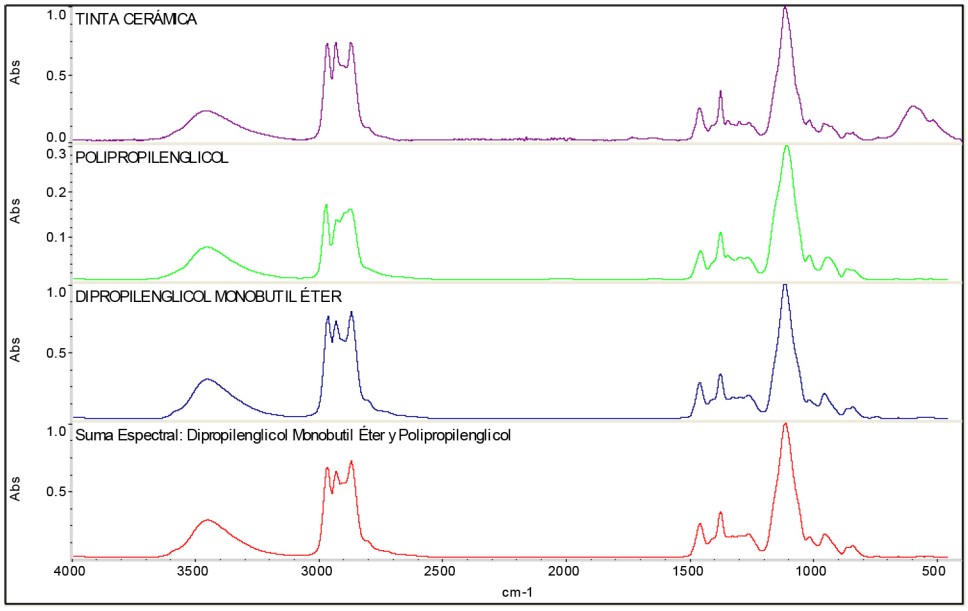

Figure 1 Identification of the compounds in the organic fraction of the ceramic ink using FT-IR

It may be observed that the organic fraction consisted of a mixture of glycols of the dipropylene glycol monobutyl ether and polypropylene glycol type because, when the spectra of the two compounds were added up in the appropriate proportions, a very similar spectrum to that of the ceramic ink was obtained, taking into account that the bands that appeared below 750 cm^{-1} corresponded to the inorganic fraction (brown pigment).

WD-XRF was used to determine the chemical composition of the inorganic fraction. The results show that this consisted of about 45% Fe_2O_3, about 40% Cr_2O_3, about 13% Al_2O_3, and about 1% SiO_2, which is the typical composition of a brown pigment.

Finally, C, H, N, and S were determined. The determination yielded 61% C and 11% H, whereas the N and S concentrations were below 0.1%. These values were consistent with the proportions obtained for both glycols in the FT-IR analysis.

4. CONCLUSIONS

The combination of FT-IR and WD-XRF spectroscopic techniques allowed most ink constituents to be characterised. However, for the detection and quantification of the minor compounds in the organic fraction of an ink, complementary separation techniques such as chromatography would be required, as these would provide better results in identifying and quantifying minor organic compounds.

5. ACKNOWLEDGEMENTS

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