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Iron-Manganese crystalline phases in ancient Lead Glazes

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Iron and Manganese oxides are commonly used to colour and decorated lead glazes since medieval times. Original raw pigments react, dissolve, and recrystallize into new crystalline compounds during firing. Their nature, size and distribution depend on the composition of the pigment, glaze and ceramic ground, method of application and thermal paths. Their presence gives direct information on the technology of production and may be related to the final colour and appearance. Therefore, the analysis of crystalline phases in ancient glasses has been demonstrated very useful for determining ancient techniques.
Hematite and melanotekite crystals were found inside iron lead glazes and kentrolite, bixbyite and braunite inside manganese lead glazes. Fe plus Mn combined compounds in glazes had not been studied in detail before. High-Temperature Synchrotron Radiation X-Ray Powder Diffraction experiment was performed to determine the iron and iron-manganese compounds formed during the heating and during cooling of a near eutectic high-lead glass composition. Calcite, dolomite and kaolinite were also added to study their role in the iron-manganese compounds formed. To expand the firing temperature range above the range accessible in the HT-PXRD experiment (928℃) the same mixtures were also fired in the laboratory following the same thermal protocol at temperatures (690℃-1020℃) and were analysed by Scanning Electron Microscopy (SEM). The kinetic profile of the experiment is designed to provide the right sequence of iron-manganese phases formed during heating and cooling. Bixbyite ((FeMn)2O3), barysilite ((Pb,Mn,Fe)Si2O7), kentrolite-melanotekite (Pb2(FeMn)2Si2O9), hematites (Fe2O3) and braunite (Mn7SiO12) are formed in Fe Mn lead Glazes. Kentrolite-melanotekite and braunite crystallise with different crystal habits during heating and cooling. If dolomite is present a pyroxene ((CaMg,Fe,Mn)2Si2O6) is formed. If calcite is present, ganomalite (Pb3(Ca,Mn)2Si3O11), margarosanite (Pb(Ca,Mn)2Si3O9) and wollastonite ((Ca,Mn)SiO3) are formed in Mn Lead glazes and andradite Ca3(Fe,Mn)2Si3O12 in Fe Mn lead glazes. Wollastonite can incorporate enough manganese to transform into bustamite ((Mn,Ca)3Si3O9) at high temperatures. This leaves less manganese available for the crystallisation of kentrolite and braunite.

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