Reproducing 17th-18th century Dutch glaze recipes - what can the conservator learn?

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Reproducing 17th-18th century Dutch tin-glaze recipes
- what can the conservator learn?

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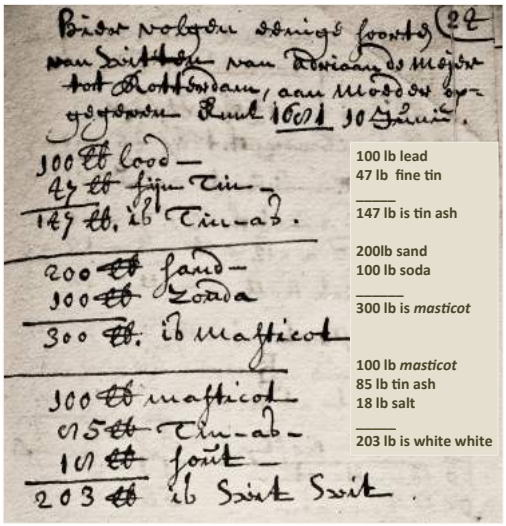
Research aim of pilot project

To assess the feasibility of reproducing 17th-18th century tin-glaze recipes as part of on-going PhD research that seeks to use archival sources to investigate the factors that influence early tin-glaze tile quality and glaze stability with relation to:

- Source and preparation of materials.
- technology of production.

Methodology

- Interpretation/formulation of recipes to creation reconstructions.
- Comparison of the reconstructions with original tiles using SEM-EDS, XRF and XRD analytical techniques.



Glaze recipe dated 1681 from the pottery notebook of Petrus Sijbeda. (Sijbeda 1712 - 23, Hannemahuis, Harlingen inv. num. 872, page 24.) Photo author, 2015.

Recipes

Two tin-glaze glaze recipes were tested:

1681: in **Petrus Sijbeda**, pottery notebook. Attributed to the Rotterdam tile-maker ***Adriaan de Meijer*** (*see left*).

1794: in **Gerrit Paape**, treatise on the Delft tin-glaze industry.

The **glaze** recipes were formulated from refined raw materials that chemically matched those described in the recipes.

The **ceramic tile** was made from commercial clays and raw materials that provided a compositional match to clay recipes in source documents.

1. The ceramic tile

Top: the clay mixes containing various $CaCO_3$ percentages; middle: Firing of test samples; bottom: colour and shrinkage tests of clay mixes with 20 – 60% $CaCO_3$ fired at 950°C, sample (a) was re- fired at 1100°C.

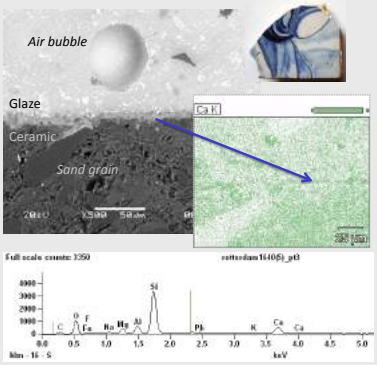
- $CaCO_3$- rich imported clay (marl) was significant in Dutch tin-glaze production.
- The influence of the $CaCO_3$% on the expansion coefficient and body-colour was tested with 8 recipes containing 20% – 60% $CaCO_3$.
- The test tiles were biscuit-fired at 950°C and 1100°C.

2. Reconstruction of 1681 Rotterdam glaze recipe

950°C 1000°C 1100°C
30% $CaCO_3$ biscuit tile
Rotterdam (above) Paape (below)

- Glaze compositions were calculated using the Segar formula, prepared, and applied to the test tiles.
- A modern lead-silica frit had to be used in place of the traditional *masticot* frit.
- samples were fired at 950°C, 1000°C and 1100°C.

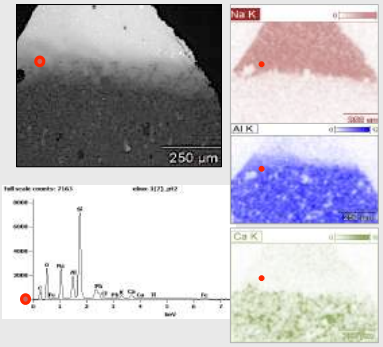
3. SEM-EDS analysis of Rotterdam sample tile dated 1640



Top left: SEM-EDS image of a sample taken from the tile; Right: Ca elemental map collected at 20.0 kV; bottom: EDS spectra of interface region. Analysis Luc Megens (RCE)

- The glaze composition matches closely that described in the Sijbeda recipe.
- The glaze-body interface is narrow and clearly defined in the Ca elemental mapping suggesting that it is dense and well-sintered.
- XRD analysis of the interface has yet to be undertaken.

4. SEM-EDS analysis of the 1681 Rotterdam reconstruction



Top left: SEM-EDS image of test sample (JEOL 591oLV, tile 25% $CaCO_3$ clay), glaze-fired 1000°C; right: Na, Al and Ca elemental mapping collected at 20.0 kV; bottom left: SEM-EDS spectra of interface region. Analysis Ineke Joosten(RCE)

- The glaze is more homogenous with fewer air bubbles than in the historic tile.
- The interface composition is higher in Na and lower in Mg.
- The glaze-ceramic interface is broader. Soda and lead in the glaze have clearly migrated into the ceramic body.

Provisional conclusions:

- While Sijbeda's Rotterdam recipe produced a working glaze, Paape's recipe proved to be deficient in flux and lacked gloss.
- Fritting of the glaze is important to prevent migration of components.
- The optimal firing temperature for tin-glaze is 1000 - 1050°C
- Increase in $CaCO_3$% results in a lighter body colour, notably around 1100°C and appears to influence the body-glaze interface.

Current research:

- At present the research focus is on the ceramic recipes. Synthetic clays mixes are being compared with mixes using real local and import marl clays that have been obtained from historically documented sources.
- The reconstructions are being compared with a selection of 16th – 17th Historic Dutch tiles showing varying levels of glaze quality and forms of deterioration using SEM-EDS and XRF analysis.
- Work has begun on the preparation of traditional glaze frits.

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