DB5.3 Guide for replicating project results

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Summary

This deliverable shows the guide prepared for the replication of project results.

This document describes the procedure that ceramic companies must follow in order to replicate LIFE REPLAY solution.

Index

1. Introduction

This guide outlines the technical procedures and best practices for replicating the results of the LIFE REPLAY project, which aims to promote the circular use of ceramic inkjet waste through the recovery and reuse of its by-products. The methodology has been developed and validated by ITC (Instituto de Tecnología Cerámica) and enables companies in the ceramic industry to convert waste materials—typically discarded during the inkjet printing process and during their manufacturing and storage—into valuable inputs for the production of ceramic tiles and inks.

The replication guide is designed for industrial stakeholders interested in implementing sustainable and innovative waste management practices. It provides a comprehensive step-by-step framework for:

- Processing ceramic inkjet waste to obtain reusable solid and liquid by-products.
- Incorporating these by-products into ceramic bodies for tiles or slabs.
- Formulating new ceramic inks from recovered materials.
- Testing the technical viability of these materials at both laboratory and industrial scale.

By following the procedures described in this document, companies can contribute to reducing their environmental impact, optimizing material use, and aligning with European sustainability and circular economy goals.

2. Recovery of LIFE REPLAY by-products from Ceramic Inkjet Waste

Once the ceramic inkjet waste has been collected, the first step is to sieve it (600 µm) to remove any coarse impurities, particularly if the waste comes from a residual ink pond.

After sieving, the waste batches should be homogenized, followed by the addition of specific additives to enable flocculation.

The resulting mixture must then undergo homogenization (at least 30 minutes), followed by centrifugation to separate it into two by-products: a solid cake and a liquid effluent.

Figure 1 Image of the by-products obtained Wet cake (left)and liquid effluent (right).

The collected cake should be dried in an oven at a minimum temperature of 150°C until constant weight is achieved.

Figure 2 Image of wet cake (left) and dry cake (right)

3. Production of coloured ceramic bodies from the solid residue

To assess the feasibility of incorporating the dried cake into ceramic support materials, laboratory testing must first be conducted to determine the maximum allowable percentage of inclusion for each atomized base material.

3.1. Laboratory Testing

- ✓ Moisture Content Measurement: Moisture must be measured in the incoming cake. If residual moisture is present, re-drying is required to ensure suitability for dry milling.
- ✓ Dry Milling and Sieving: The cake can be milled either manually (using a hammer) or with a planetary mill using alumina jars and grinding balls. The resulting powder should be sieved to <250 µm.

Figure 3 Image of manual grinding (left) and alumina jar and grinding balls (right)

Figure 4 Image of lid, 250 µm and base

- ✓ Blending with Atomized Material: The milled cake is then mixed with industrial atomized material in the proportions defined during testing. Manual or mechanical mixing (intensive mixer/granulator) may be used. Fluidizing additives can be added to improve flowability.

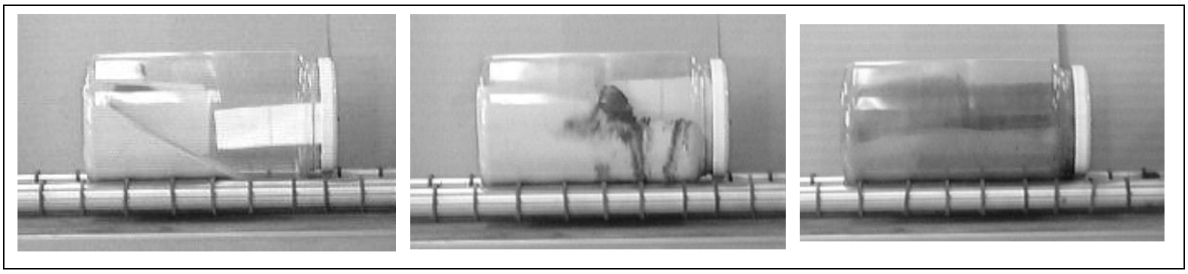

Figure 5 Time evolution of the surface coating coloration process.

Figure 6 Image of a high-intensity mixer

- ✓ Pressing and Sintering Performance: Tests should be performed to analyze the pressing behavior and sintering results. This includes compaction curves, dry mechanical strength (three-point flexural tests), and sintering behavior (densification diagrams, water absorption, and chromatic coordinates).

Figure 7 Image of the testing device for performing three-point bending

The maximum viable percentage of the pigmented residue is determined based on acceptable behavior during processing and resulting material properties.

3.2. Industrial Trials

The pigment material obtained from the milled and sieved cake (<250 µm) is mixed with the chosen atomized base in the optimal proportion determined during lab trials. Then, standard industrial tile or slab forming and firing processes are followed. Decorative printing can incorporate LIFE REPLAY-derived inks, followed by final firing.

Figure 8 Image of an industrial tile after the decoration process (left) and a fired industrial tile (right)

4. Production of ceramic inkjet inks from LIFE REPLAY by-products

4.1. Ink Formulation Using Liquid Effluent

- **Effluent Filtration**: Filter the liquid effluent at 1 µm using a Kitasato setup.

Figure 9 Kitasato flask for 1 µm filtration of the liquid effluent

- **Compatibility Testing**: Check for miscibility with solvents commonly used in inkjet formulations. Incompatibility implies that a stable pigment dispersion cannot be achieved. If incompatibility arises, the effluent can be used in traditional screen-printing ink formulations instead.

Screen-printing inks can be produced by milling inorganic pigments and frits with the effluent, achieving the required particle size according to the screen mesh.

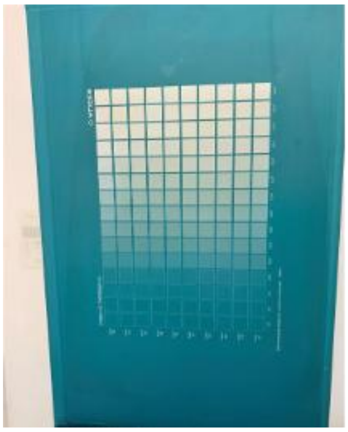

Figure 10 Image of screen printing

4.2. Ink Formulation Using Solid Residue (Cake)

- **Moisture Content Measurement**: Re-dry if needed. If solvent-based wet milling is preferred, ensure solvent compatibility.
- **Compatibility Testing**: Conduct solvent compatibility studies using alumina ball milling. Target $d_{97} < 0.8$ µm with 30-40% solids.
- **Ink Preparation**:
  - **Preconditioning**: The semi-dry cake is dispersed in solvent using a high-speed agitator. The resulting suspension is sieved at 200 µm.

Figure 11 Stirring tank/hopper

  - **Microbead Milling**: Ink production is completed via continuous microbead milling (e.g., using ZrO2-Y2O3 beads, 0.3-0.4 mm) to achieve particle sizes <1 µm.

Figure 12 Image of a Netzsch micro ball mill

4.3. Ink Characterization

- **Filtration**: Final ink must be filtered using 5 µm and 2 µm filters.

Figure 13 Image of Gesfilter filtration equipment

- **Particle Size**: Verify the ink's particle size is below 1 µm.
- **Density**: Measured with a pycnometer.

Figure 14 Image of a density measurement pycnometer

- **Viscosity**: Determined using a Brookfield or rotational viscometer. Viscosity should match printer head requirements.

Figure 15 Image of a Brookfield viscometer for measuring viscosity

- **Filterability**: Conduct vacuum filtration tests (100 ml, 1 µm filters). Assess filtration time and residue buildup.
- **Colloidal Stability**: Stability should be tested by:
  - **Sedimentation Test**: Place 50-100 ml of ink at 50°C for 7 days. Evaluate sedimentation behavior.
  - **Turbiscan**: Conduct analytical stability testing at 50°C for 7+ days, monitoring agglomeration, sedimentation, and compacting against a reference.

Figure 16 Image of colloidal stability equipment

- **Printability Testing**: If all properties meet specifications, the ink should be tested using lab-scale inktesters or semi-industrial plotters. Successful results confirm suitability for industrial application.

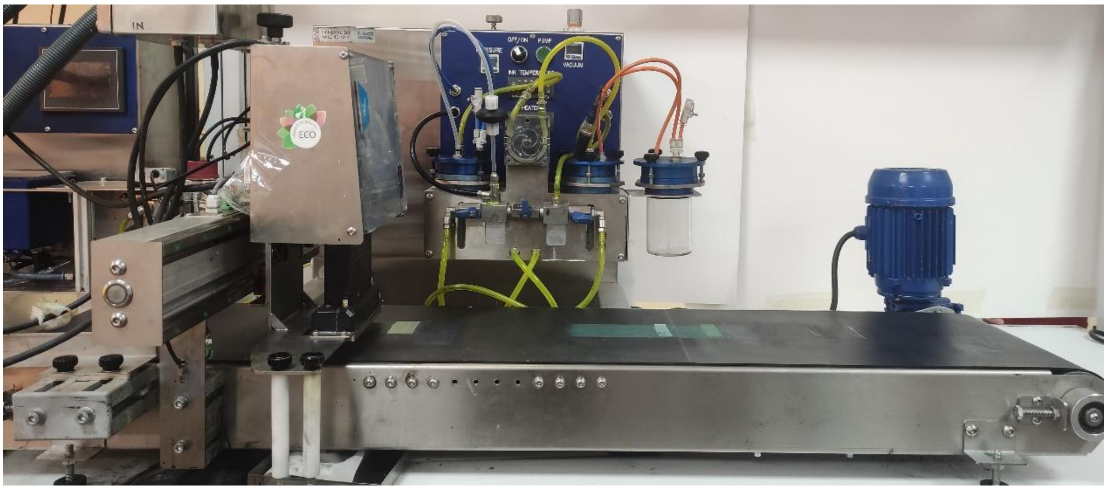

Figure 17 Image of laboratory inktester

Figure 18 Image of semi-industrial plotter