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Ceramic flaws: Laboratory tests and analysis using Scanning Electron Microscope to identify surface defects

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Abstract

The pollution of raw materials and ceramic products is a problem that affects all ceramic companies operating in the fields of traditional ceramics and thus needs to be further investigated. The presence of impurities in ceramic glaze and bodies, or between their contact surface, causes aesthetic defects.

The aim of this work is to analyse ceramic flaws generated during the processing phases. Analysis of the morphological and compositional imperfections was carried out on different samples by the use of a Scanning Electron Microscope. An efficient model of the flaws in the final ceramic product was later developed, correlating imperfections and pollutants which caused them. In this way the phases of the process, where the pollution might occur, are successfully identified. In order to reach this goal, ceramic samples were created and contaminated in laboratory by the use of potential pollutants, previously and carefully selected in the planning phase of the study.

Keywords: Pollutant; Ceramics flaws; Scanning Electron Microscope

1. Introduction

Pollution phenomena of bodies and glaze frequently affect sanitary-wares production during the ceramic process itself. Pollutants may derive from raw materials, such as organic compounds in clays, or may be introduced during the ceramic process (namely metals or alloys).[1] Moreover, since the production cycle involves different stages, attention has to be devoted to choosing appropriate raw materials for producing bodies and glaze. Thus, an extremely careful selection of clays, kaolins, feldspars, quartz, and other raw materials needs to be performed; this goal is successfully reached by chemical and mineralogical analysis which leads to minimize ceramic flaws.[1]

However, it has to be noted that pollution may be due to several phases of the cycle production.

For instance, the operation of slip casting – especially in plaster moulds-may cause residual impurities of gypsum on the final product. After drying and glazing the product, defects may emerge if any impurity occurs during the painting phase or within the paint plant. Furthermore, if we consider the firing phase – which is extremely expensive and crucial-temperature curves may vary depending on the body slip and glaze formulations (Fig. 1). During this step, well known phenomena occur, e.g. the quartz transition at 573 °C, the decomposing of organic matter (400–600 °C), carbonates (700–1050 °C) and sulfates (950–1100 °C). Some of these transformations lead degassing and formation of defects, such as pinholes or detachment of glaze from support.[2,3]

The present project involves an extensive study of aesthetic defects in ceramic products due to the pollution of body slip or glaze. They consist of spots with different morphology and colouration, pinholes phenomena, air bubbles due to degassing of impurities.

The aim of the survey is to identify the relationship between polluting materials (such as gypsum, coal, asphalt, iron, copper, aluminium, steel, brass, polycarbonate, PVC, tin, glue, oil, etc.) and defects generated on the final product. A ceramic defects database has been created, describing the tests results, showing the images of the flaws deriving from the samples pollution, and providing their chemical and morphological analysis through the

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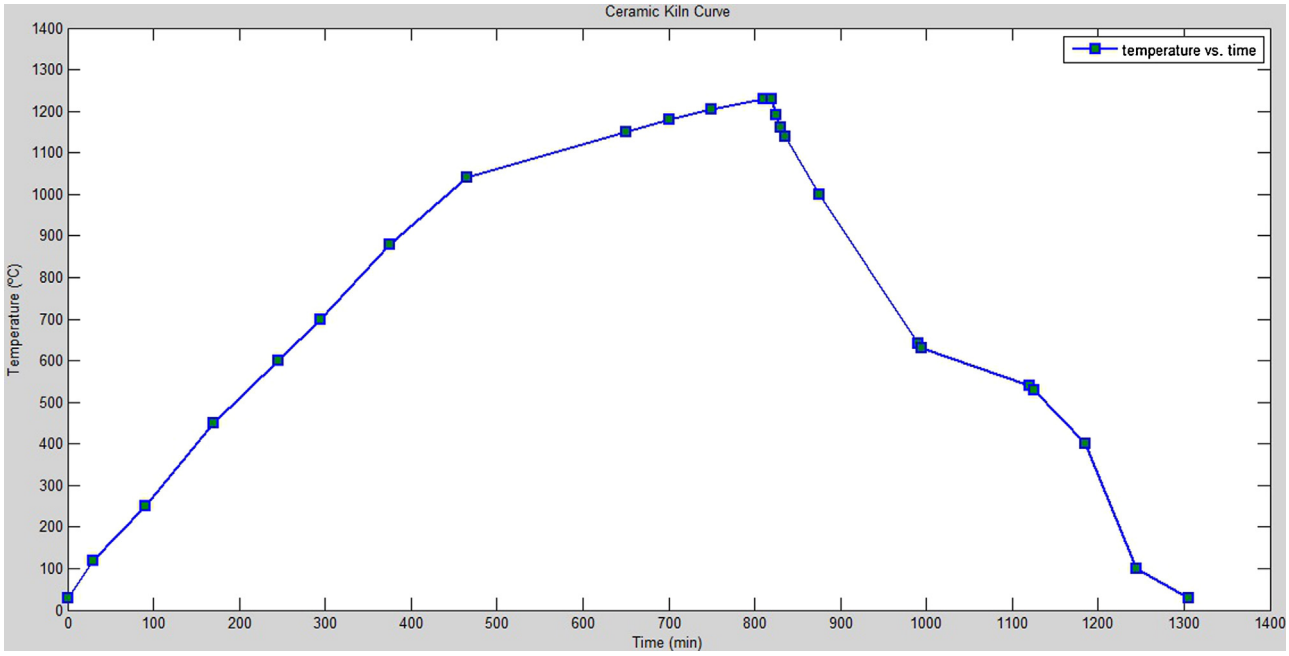

Fig. 1. Ceramic kiln curve.

use of a Scanning Electron Microscope (SEM). In order to reach this goal, a laboratory simulation procedure has been planned, addressed to reproduce the pollution phenomena of body and glaze inside the ceramic process for sanitary-wares production.

2. Materials and methods

An analysis of the surface defects of ceramic product has been carried out starting from samples made in laboratory. These samples have been polluted by substances that usually cause exterior defects, in order to assess their effect on the final product.[4]

Various tests have been carried out in order to define how the following parameters influence the final result:

- pollutant composition
- pollutant concentration
- method of application and positioning of the pollutant in the body slip or glaze.

According to these analyses the implementation procedures of the following tests have been planned. Initially the ceramic mixture has been characterized by internal procedures of laboratory in order to measure specific weight, viscosity and thixotropy. The studied ceramic mixture has been placed in a plaster mould to obtain small ceramic tiles. The tiles have been removed from the plaster mould and they have been dried, according to the following steps:

- drying at ambient temperature, 25–30 °C for about 15–20 h;
- drying in oven at 60 °C for about 24 h.

The residual moisture after the above mentioned phase has been around the 0.2–0.3% by weight.

After the drying, the samples have been glazed using an airbrush or a manual orifice, and fired in an electric oven. The firing curve reaches a maximum temperatures of about 1230 °C, with extremely low temperature gradients during prefiring stage and near the transformation temperature of quartz (573 °C: Quartz $\alpha \rightarrow$ Quartz β).[4]

During this small-scale reproduction of the industrial production cycle, a pollutant has been added in the body slip or in the glaze, respectively in the slip casting phase or during the glazing phase, making sure that the impurities have been trapped in the ceramic materials. As above explained, the ultimate goal has been to identify the types of defects caused by each kind of pollutants and the limit quantity of pollutants exceeded which the defects begin to appear. This result could be useful in order to previously identify, by X-ray Fluorescence chemical analysis, abnormal quantities of impurities in raw materials that could cause imperfections on the final product.

In order to make the laboratory test results suitable to the real industrial production, it is important to highlight the following concepts: the concentrations measured in the laboratory tests are meant as percentage of pollutant in the fluid body slip or glaze, as they appear before casting and glazing phases. Conversely, the chemical analysis through the X-Ray Fluorescence is carried out on dried sample.[5] Hence, in order to establish a correlation between the pollutants concentrations in the laboratory tests (on liquid suspensions) and the pollutants concentrations in a dry powder analysed by X-Ray Fluorescence, it is necessary to take into account the loss in weight to which the body slip and glaze are undergone during the drying phase; this loss in weight is approximately equal to 27% for Vitreous China bodies, 21% for