



Research paper

Estimation of the heat of reaction in traditional ceramic compositions

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ABSTRACT

Most energy studies on the traditional ceramic manufacturing process focus on the firing stage because this is the process stage that consumes the greatest amount of thermal energy. At present in Europe, using typical technologies, about 50% of the energy input in the firing stage is still lost through the kiln stacks. A key issue in energy studies is the accurate determination of the energy efficiency of the process, an issue that may become crucial in coming years to enable the energy management of different facilities and products to be compared. To reliably determine energy efficiency, accurate determination is required of the energy needed for the necessary physico-chemical transformations to develop in the material in the firing stage. This energy is also the only strictly unrecoverable energy, as the energy contained in other streams could, potentially at least, be partly recoverable. The present study was undertaken to develop an analytical methodology for estimating the heat of reaction of seven different traditional ceramic products, involving a broad spectrum of compositions, with peak firing temperatures ranging from 850 °C to 1200 °C. The following industrial ceramic compositions were studied: four ceramic tile compositions (red-body stoneware tile, porcelain tile, red-body earthenware wall tile, and white-body earthenware wall tile); two structural ceramic compositions (white brick and roof tile), and a porcelain tableware composition. To estimate the energy involved in the physico-chemical reactions in the firing stage, an analytical methodology was developed, based on the mineralogy data of the unfired body composition and on the enthalpy of formation of the minerals in the fired tiles. The methodology was validated by comparing the results with experimental data.

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1. Introduction

At present in Europe, using typical technologies, large quantities of thermal energy are consumed in traditional ceramic manufacturing processes. These processes are quite similar and consist of stages that differ as a function of the product made. The main production stages are as follows: raw material preparation (by dry or wet milling), forming (by casting, pressing, or extrusion), drying, firing of the body (in double-fired products), glazing and decorating, and firing (Agrafiotis and Tsoutsos, 2001; Bovea et al., 2010; IPTS. European Commission, 2007).

Thermal energy consumption takes place mainly in three process stages: wet milling (in which the ceramic slurry may be needed to be dried for semi-dry pressing); drying (after forming); and firing. In this manufacturing process, firing accounts for more than 50% and 70% of the overall energy consumption when wet milling and dry milling are used, respectively (Mezquita et al., 2009, 2014; Monfort et al., 2010).

In traditional ceramic manufacture, from an energy viewpoint, the firing stage is the most important process stage, as it needs to supply sufficient heat for the necessary physico-chemical transformations in the material to develop and to provide the product with the desired technical and aesthetic properties.

The thermal energy needed to fire glazed ceramics usually stems from the combustion of natural gas. However, in the manufacture of structural ceramics, which are mainly unglazed products, other fuels may be used, such as pet coke, coal, or biomass. These fossil fuels give rise to air emissions of CO₂, a greenhouse gas, and the emissions of which are subject to international control and mitigation measures.

The present study was undertaken to determine the quantity of energy consumed by the chemical reactions that develop in the ceramic firing stage, i.e. in the heat of reaction. At present, using today's technologies, the heat of reaction constitutes a relatively small fraction (5–20%) of the overall energy required in industrial firing. It is the only energy that cannot be recovered, in contrast for example to the sensible heat of fired ceramics (which is partially recoverable in the cooling phase) or to combustion gas heat (recoverable using heat exchangers).

From an energy viewpoint, in the firing cycle, the material undergoes both exothermic and endothermic transformations. These need to take place gradually and in a controlled way, as they could otherwise lead to permanent defects in the end product.

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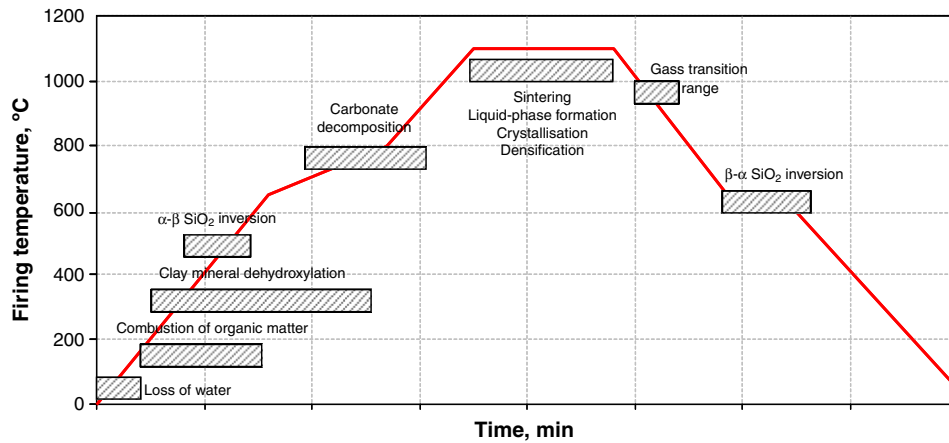


Fig. 1. Typical firing curve with the physico-chemical reactions that develop in traditional ceramic compositions.

The shape of the firing curve (temperature as a function of time) essentially depends on the body composition and, in glazed ceramics, on the nature of the glaze (glossy, opaque, matt, etc.). In general, every traditional ceramic firing cycle includes the following steps:

- **Heating:** In this step, the unfired products are heated from ambient temperature to about 800 °C (depending on the raw material composition). This is the step in which outgassing of the ceramic body needs to take place to avoid problems of bloating, bubbles, pinholing, glaze porosity, and colour differences at higher temperatures. Various transformations take place in the material during heating, such as the removal of free water, combustion of organic matter, allotropic transformation of α -quartz to β -quartz, loss of OH^- groups in the clays, and carbonate decomposition when the composition contains carbonates. All these chemical reactions lead to a series of internal stresses in the ceramic product owing to gas release, changes in volume, etc. If these stresses exceed the mechanical strength of the as yet “unfired” ceramic product, failure will occur.
- **Firing:** The actual firing step runs from about 800 °C to the programmed peak temperature, typically between 850 °C and 1350 °C, which depends on the product being made. The main physico-chemical transformations take place in this step. These reduce the ceramic material's porosity. They lead to the most important dimensional changes (shrinkage) in low porosity products (stoneware and porcelain ware) and to the formation of stable crystalline phases (calcium silicates) in porous products (earthenware wall tiles and structural ceramics).
- **Cooling:** This begins when the heat input ends. In the cooling step, product temperature decreases from peak temperature to near ambient temperature. In traditional ceramics the cooling rate is essentially

constrained by quartz phase transformation at 573 °C, as this produces a volume change that can cause cracking in the product if cooling does not take place uniformly throughout the material, owing to the low thermal conductivity of ceramic materials.

A typical traditional ceramic firing cycle is shown in Fig. 1. The figure summarises the most important physico-chemical changes that develop during traditional ceramic thermal treatment and the temperature ranges in which these changes occur (Pennisi, 1991). A ceramic firing schedule depends on several factors, the most important being body composition, thickness and geometry of the ceramic product, kiln loading density, and type of kiln. For instance, the schedule time in roller hearth kilns (low product thickness and low kiln loading density) is typically between 30 and 60 min, whereas in tunnel kilns (high loading density and thick products), this ranges from 18 to 24 h.

The raw material composition plays a very important role, not only in environmental emissions in the firing stage, but also in energy consumption in this stage. The quantities of carbonates, organic matter, and other raw materials in the ceramic compositions influence kiln thermal energy consumption, as the energy required to develop the physico-chemical reactions in firing depends on the ceramic composition involved (Mezquita et al., 2009).

Although numerous studies have been conducted on the firing of traditional ceramics (Bernardo et al., 2010; Castelein et al., 2001; Eliche et al., 2011; Macgee, 1926; Madivate et al., 2004; Njoya et al., 2012; Plante et al., 2009; Sánchez et al., 2001; Simpson, 1927; Vogt and Thomas, 2012), mainly addressing technical, environmental, or energy issues, few studies have focused on the energy involved in the physico-chemical transformations that take place (Macgee, 1926; Simpson, 1927; Madivate et al., 2004; Vogt and Thomas, 2012).

Table 1

Main processing details and end product characteristics of the studied compositions.

Reference	Product	Processing details					End product	
		Milling	Shaping	Firing			Glazed	Water absorption (%)
				Peak temperature (°C)	Total cycle (h)	Kiln		
RBT	Red-body stoneware tile	Wet	Pressing	1143	0.46	Roller	Yes	2.9
PCT	Porcelain tile	Wet	Pressing	1190	0.57	Roller	Yes	<0.5
RWT	Red-body earthenware wall tile	Wet	Pressing	1126	0.40	Roller	Yes	14
WWT	White-body earthenware wall tile	Wet	Pressing	1140	0.51	Roller	Yes	17
WBR	White brick	Dry	Extrusion	850	24	Tunnel	No	20
RFT	Roof tile	Dry	Pressing	1019	18	Tunnel	No	10
PTW	Porcelain tableware	Wet	Slip casting	1000	22	Tunnel	Yes	<0.5

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