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High temperature behaviour of a wide petrographic range of siliceous and calcareous aggregates for concretes

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HIGHLIGHTS

- Twenty-one aggregates of various mineralogical compositions are exposed to heating.
- For siliceous aggregates, quartz crystallinity has an influence on spalling sensitivity.
- Thermal expansion of siliceous aggregates is related to quartz content, especially after 450 °C.
- Thermal expansion of calcareous aggregates varies between 1 and 1.33% at 750 °C.
- Decarbonation occurs for dolomite at a lower temperature than for calcium carbonate.

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ABSTRACT

This study aims to characterize the physical and chemical transformations of aggregates undergoing high thermal solicitation in order to better understand their deterioration in this context. Twenty one (21) aggregates of various mineralogical compositions are exposed to heating/cooling cycle up to 750 °C. The thermal behaviour of siliceous and calcareous aggregates depends on several parameters related to mineralogical structure including granular texture, crystallinity of minerals, porosity and density. Thermal properties like expansion and weight loss related to chemical reactions show important variations among the siliceous aggregate category. The thermal behaviour of aggregate is dependant of its mineralogical and chemical compositions.

1. Introduction

Concrete is a thermal resistant material used in building construction. Its thermal properties are solicited when the concrete undergoes thermal loading that may occur in specific structures like in nuclear power plant, radioactive waste store or factories and laboratories using heating processes. Fire is an accidental thermal load that must be considered and included in building construction conception. The temperature may reach 1100 °C in buildings and more than 1200–1350 °C in tunnels or parking areas where hydrocarbon products may increase heating energy and cause severe damage to concrete structures [1,2].

Aggregates constitute about 70–80 % of the volume of the concrete. This suggests that concrete properties are very likely influenced, if not dependent, by those of the constituting aggregates. In the literature, pore pressure vapour implying tensile stresses is one of the main reasons for concrete spalling [3–7]. High temperatures related changes in the petrological and petrophysical aggregates properties can also lead to concrete instability. Moreover, thermal expansion of aggregates has an influence on the mismatch of deformation between aggregate and cement paste and therefore on concrete cracking occurrence. It seems important to establish the effect of high temperature on aggregates. The recommendation of Part 1–2 of Eurocode 2 [8] concerning aggregates is a

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distinction between concrete made with siliceous or calcareous aggregates. This distinction focuses on thermal expansion, thermal properties such as conductivity and heat capacity and thermal mechanical resistance.

Some studies have been made for describing the influence of the mineralogical properties of aggregates on concrete thermal behaviour [3,9–16]. Siliceous aggregates undergo a thermal degradation that is essentially attributed to the quartz α–β transformation occurring at 573 °C which induces internal stresses [10]. Among siliceous aggregates, different behaviours occur [17,18]: whereas flints – composed of silica (SiO_2) – are subjected to spalling (thermal instability) from 300 °C (depending on water content [19]), quartzite aggregates that are also composed of quartz SiO_2 can endure high temperature up to 750 °C without any instability. The influence of grain size and porosity was reported [20]: flints are composed of cryptocrystalline silica containing silanols groups [21–23] and the very low porosity and likely the fineness of the pores explain the increase of thermo-hydric stresses [24,25]. It confirms the fact that dry flints are less vulnerable to overheating [19,20]. Some authors [20,26,27] assumed that the thermal stability was proportional to the dimension of the element: larger volume where moisture evacuation is more hampered loses less water than smaller volume. For polymineralic stones such as granite, thermal incompatibility of its components may result on additional microcracks.

The decarbonation of calcium carbonate ($CaCO_3$) from 700 °C may constitute the main effect for calcareous aggregates in the subsequent hydration of calcium oxide (CaO). This phenomenon involves important volume changes (44% expansion) that alter the structure of aggregates and then concretes [28,29]. The texture of the rock combined with porosity has a strong influence on the thermal change response. Mineral expansion fills the intragranular porosity and generates less microcrackings [30,31]: a high porosity allows absorbing stresses [32]. Moreover crystals of calcite are known to undergo a marked anisotropic thermal expansion, expanding parallel and contracting perpendicular to crystallographic c-axis. Such anisotropic deformations are responsible for stresses than can lead to the opening of cracks at grain boundaries [33].

Based on literature review, high temperature behaviour of aggregates cannot be explained by means of chemical composition only (siliceous vs calcareous). Other parameters (type of silica, porosity, etc.) may interfere within the same category of aggregates. It was necessary to investigate the wide range of petrographic aggregates to determine the relative influence of these parameters. The goal of this study is to distinguish aggregates by their thermal stability in order to explain their influence on concrete's behaviour at high temperatures.

Several results in the literature use a mix of different types of aggregate to overcome all ambiguities coming from the denomination of aggregates (e.g. "silico-calcareous" aggregates composed of X% of siliceous and Y% of calcareous). This study takes into account the uniqueness of facies and each of them is analysed individually. Moreover, the poorly known high temperature behaviour of some aggregates such as rhyolite and basalts is investigated. The study also focuses on the measurement of aggregate thermal expansion that is one of the most important parameter explaining concrete damage at high temperatures.

Coming from ten different quarries involving different origins, twenty-one facies have been identified and distributed as fifteen siliceous aggregates and six calcareous aggregates. The experimental program describes sample preparation, heating parameters, devices and data acquisition. The results of this program are coupled with petrographic and thermal stability at high temperatures. These parts include physical properties, mineralogical and chemical compositions associated to thermal stability, crack evolution, thermal expansion, ThermoGravimetric Analysis and Differential Scanning Calorimetric (TGA/DSC) measurements.

2. Experimental program

Samples used in this study were crushed or rounded (alluvial) aggregates in the range of 4/22.4 mm. This study takes into account twenty-one different petrographic aggregates. Three devices were used for heat treatment: an electric furnace (Fig. 1), a furnace integrated with the thermal expansion device and a furnace integrated with the TGA/DSC device.

2.1. Petrophysical study of aggregates before heating

To characterize the initial state of aggregates (i.e. initial properties), petrophysical study was performed before applying any experimental thermal stress. A macroscopic recognition was made in order to identify the petrographic facies. A thin section was prepared for each studied aggregate. This microscopic preparation is made of a 30 μm thick slice of aggregate mounted between two slides of glass. Thin sections were then observed under polarized light microscope. Microscopic observations were completed by X-ray Diffraction (XRD) analysis in order to obtain precise mineralogical compositions. XRD analysis is completed on ground coarse

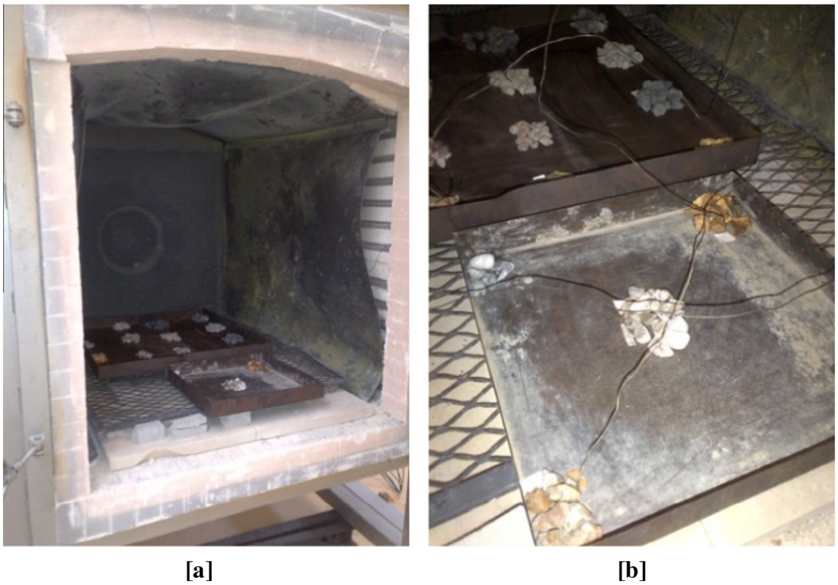
[a] **[b]**

Fig. 1. Electrical furnace and specimens. [a] General view inside furnace and sample batch disposition; [b] Disposition of thermocouples on samples.