Introduction

Much has changed since Joseph Aspdin patented grey Portland cement in 1824 with his son William, making it commercially viable by 1842. In the 21st Century, those who require cement have a vast array of solutions to choose from, depending on the particular properties they require. Everyday options, other than portland cement, include white cement (low ferrite), rapid hardening (fine ground), masonry, and sulphate resisting cements. This list continues to expand to over 20 common types of cement, as manufacturers and research institutions work on new

Furnace, Furnace, Burning Bright

Robert Prior, Carbolite Gero, provides an overview of oven and furnace requirements in cement testing.

varieties and blends that can meet the challenges of a construction industry that continues to push the boundaries of the possible.

In fact, by 2016, it was reported by the Mineral Commodity Survey that the total global production of cement had reached 4.2 million tpy, with advanced production techniques delivering improved performance, increased volume capabilities, reduced energy requirements, and a substantial decrease in the environmental impact that manufacturing has on its locality.

At the heart of all modern cements are rigorous BS, EN, and ASTM testing regimes that cover physical properties such as setting times, density, fineness, and flex strength, along with chemical tests, including PFA content, chromate VI levels, and conformity to BS EN 197.

Many will have visions of cement analysis taking place in bubbling flasks mounted on tripods in a laboratory, but for the main those old techniques have been made obsolete with the new techniques of x-ray fluorescence (XRF), x-ray diffraction (XRD), and microscopy.

XRF provides almost instantaneous compositional data at any stage of the production cycle by testing either a specimen formed in to a glass bead or a pellet pressed from uniform ground powder of under 100 µm. Consequently, XRF is at the centre of the 21st Century production process in a cement works, ensuring quality, consistency, and adherence to recognised national and international standards.

Production of cement in a laboratory pilot plant

Cement manufacturers and research organisations that wish to create cement using a pilot plant can invest in laboratory-scale equipment to grind and process the initial raw materials (limestone circa 80% and clay circa 20%), along with a rotating split tube furnace from a specialist manufacturer, such as Carbolite Gero, to sinter these ground raw materials.

While the real-world combustion fuel of an industrial cement kiln cannot be mimicked, the remainder of the manufacturing process can be simply achieved, enabling the laboratory creation of cement clinker. This is usually characterised using three parameters:

1. Lime saturation factor.
2. Silica ratio (SR or S/R).
3. Alumina ratio.

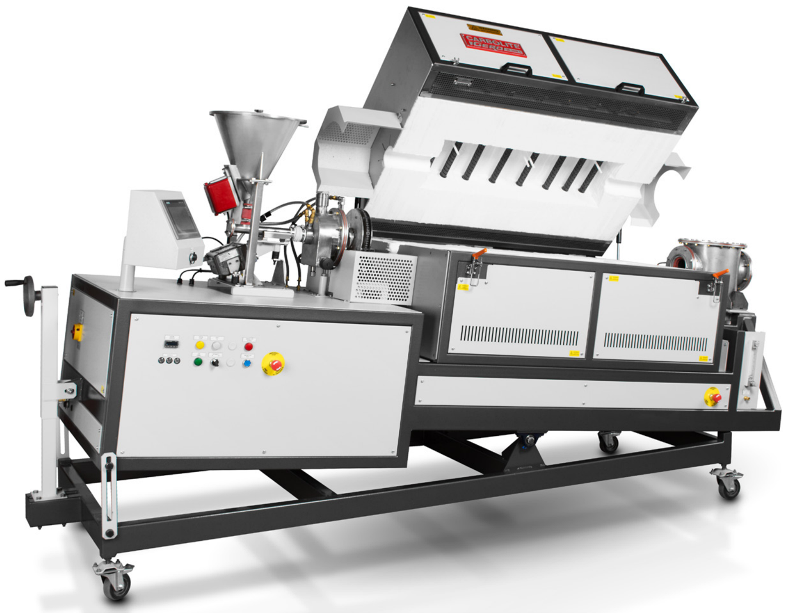
There is no simple answer as to which clinker composition is best; it really does depend on the requirements of the final concrete and the aggregates used to manufacture it.

A typical pilot plant furnace has a 75 mm maximum work tube diameter, with possible tube lengths of 600 mm, 900 mm, or even 1000 mm. Time in the heated zone for the raw material will be determined by the angle of inclination that is set for the tube and can be varied between horizontal and 10°. These laboratory furnaces should also be capable of creating a controlled/modified atmosphere, using inert gases with a gas flow rate, where required, of up to 5 l/min.

The chosen pilot plant furnace should also encompass observation port holes to allow the operator to view production of the cement clinker as it proceeds through the work tube. Where required, data logging should be provided to record essential information, such as furnace temperature cycles and material flow rates.

For further convenience, the rotating laboratory furnace might be equipped with a vibrating feed hopper capable of holding around 5 l of raw material. On activation, the raw material is passed through the rotating tube at speeds of between 1.5 and 10 rpm (with a feed rate of between 2 – 5 kg/hour) depending on production requirements. As the raw materials move along the quartz work tube, they are simultaneously mixed and sintered at a temperature between 1350°C and 1450°C. It should be noted that above 1350°C the proportion of liquid begins to increase and, by 1450°C, as much as 30% of the mixture may be liquid. This additional liquid causes particles to stick together and porous clinker nodules then form. As the clinker passes the burning zone, it begins to cool. Fast cooling is considered advantageous, due to its resulting structure and the fact that it is easier to grind.

The resulting clinker drops into a collection container, where it can be tested for composition and compliance, or ground to a fine powder in a cement mill and then



Rotating tube furnace from Carbolite Gero, used in pilot plant creation of cement clinker.

mixed with a small quantity of gypsum. This controls the subsequent setting properties of the resulting cement powder. It is this cement powder that is mixed with water and aggregates to create concrete.

Compliance testing of cement

Compliance of cement with recognised international standards is essential for a product that is ultimately shaping the built environment of the 21st Century. With continual improvements to construction materials, an architectural profession determined to push new boundaries, and a desire to use the most cost-effective materials, there continues to be massive scope for pilot plant production of clinker variants.

Cement producers will rightly insist on quality assurance for raw materials used in the manufacturing process, and production methodologies that will deliver a final compliant product. The following international standards will typically be tested for during the manufacturing process:

- BS EN 196-2:2013: Chemical analysis of cement.
- BS EN 13639:2002: Determination of total organic carbon.
- BS EN 15414-3:2001: Moisture content of solid recovered fuel.
- BS EN 15403:2011: Determination of ash content.
- BS EN 450-1:2012: Flyash for concrete (chemical and physical requirements).
- BS 7929:2016: Moisture content.

Testing for the above standards typically employs laboratory ovens at lower temperatures for compliance with standards such as BS 7929:2016. However, where testing above 250°C is required there will be a need for a laboratory furnace.

Loss-on-ignition testing, which forms part of BS EN 196-2:2013, requires a temperature of 950°C, while the determination of pure silica (part of chemical analysis testing) requires a temperature of 1175°C. A chamber furnace will be required facilitate these tests.

Another furnace that will be required in a fully-specified test environment is a tube furnace to determine the total organic carbon present in limestone to satisfy the requirements laid out in BS EN 13639:2002. When operating, the CO_2 in the limestone is driven off by using hydrochloric acid at 130°C. The residue is transferred to a platinum vessel and is oxidised in an oxygen atmosphere at approximately 900°C. The liberated CO_2 is absorbed by an inorganic carrier in an absorption tube and the increase in mass is directly proportional to the organic carbon content.

Laboratory furnace manufacturers playing their part in ensuring international standards

To produce high-quality cement, the mineralogical and chemical composition of raw materials, as well as intermediate and finished products, must be determined. At every stage of production, samples are required to be taken, processed, and analysed to ensure quality control without any gaps.

An 1100°C ashing furnace is the ideal solution for loss-on-ignition testing. Of course this testing would be further enhanced, if a furnace with integrated balance was employed. However, where necessary, loss on ignition tests can also be carried out in a general purpose furnace, with either a 1200°C or 1300°C temperature capability.

For organisations that also have a requirement to measure the burnability of free lime content in cement clinker, the purchase of a high-temperature furnace capable of heating to a temperature of around 1600°C might be considered.

Whichever cement-related standard is required to test for compliance, Carbolite Gero and other oven and furnace manufacturers will have a range of solutions that should meet specific requirements. A quick overview of typical oven and furnace solutions used by laboratories in the modern cement plant is set out in Table 1 and has been organised by the international standard to which it is related.

About the author

Robert Prior is Marketing Assistant at Carbolite Gero, a manufacturer of high-temperature furnaces and ovens for laboratory, research, and process applications.

Table 1. **Typical oven and furnace solutions used in cement plant laboratories by international standard.**

Test Standard	Testing	Material	Test Temperature (°C)
BS EN 196-2:2013	Chemical analysis of cement	Dried pumice stone cement	110 – 1175
BS EN 450-1:2012	Fly ash for concrete chemical and physical requirements	Fly ash	500 – 550
BS EN 13639:2002	Determination of total organic carbon	Limestone	75 – 900
BS 7929:2016	Moisture content	Limestone fines	105
BS EN 15403:2011	Determination of ash content	Solid recovered fuels	250 – 550
BS EN 15414-3:2011	Moisture content of solid recovered fuels.	Solid recovered fuels	105