



Chemical Reactions in Pozzolanic Concrete

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Abstract

Cement is the most utilized construction material, and the second most consumed commodity in the world after water. Its demand has soared proportionately with the exponential rise in population in a bid to match the required development. The heavily energy-intensive processes that are involved in its production contribute to about 7 to 10 per cent (%) of the total global emissions, with potentially adverse environmental implications, and are also economically expensive. These processes, and generally those of the production of concrete consume heavily on natural resources such as sand, gravel, water, coal and crushed rock, mining of which mars the environment. It is however possible, that energy and cost efficiency can be achieved by reducing on the amount of clinker, and in its place utilizing supplementary cementitious materials (SCMs) or pozzolans that require less process heating and emit fewer levels of carbon dioxide (CO₂). This paper elaborates on the different types of chemical reactions taking place in concrete containing pozzolans as a partial cement replacement. A pozzolanic material relies on the secondary reaction following the hydration of cement, whereby it reacts with the free calcium hydroxide to form the calcium silicate hydrates (C-S-H) phase which is the major contributor to strength in concrete; as a result there is usually long term strength development up to and beyond 91 days in pozzolanic concrete. Due to the depleted levels of calcium hydroxide, pozzolanic concrete impart superior sulfate resistance.

Keywords: Hydration of cement; Pozzolans; Pozzolanic reaction

Introduction

It is reported in the literature that apart from environmental friendliness, the use of supplementary cementitious materials (SCMs) such as pulverized fuel ash (PFA), ground granulated blast furnace slag (GGBS), silica fume (SF), metakaolin (MK) and rice husk ash (RHA) reduces the cost of concrete and improves the durability of hardened concrete, thereby enhancing the service life of structures [1,2]. This paper focusses on the chemical reactions which take place in pozzolanic concrete, i.e. concrete containing PFA, SF, MK and RHA.

Cement is deemed to have a considerably high carbon footprint, contributing immensely to global anthropogenic CO₂ [3]. Climate change is purported to be a phenomenon that can bring about a rise in global temperatures due to the presence of excessive carbon dioxide (CO₂) in the atmosphere and is cumulative and irreversible over timescales of centuries [4,5]. The burning of fossil fuels, in this

case for the production of cement contributes to the greenhouse gas effect, which is a major cause of climate change [6]. Even though heavily energy intensive, cement is pivotal to development and is produced in virtually all countries [7]. One ton of concrete on average is produced every year for each human being in the world, a population that currently stands above 7 billion [8,9]. The growing population, matched by a corresponding increase in demand for socio-economic infrastructure that is aimed at creating affluent societies, especially in the developing world and former socialist countries, has led to a gradual increase in the demand for cement in the past few decades, with construction investment directly linked to higher gross domestic product (GDP) [10,11]. Cement was described by Al-Salami and Salem [12] as the most utilized construction material in the world, its global consumption only seconding that of water. It constitutes between 7%-15% of the total mass of concrete mixes [13], yet according to Sakai and

Noguchi [11], the development of a nation is directly proportional to its consumption of concrete. Its yearly global production is approximately 2 billion tons, accounting for about 7 - 10% of the total global CO₂ loading in the atmosphere, a considerably high level of emissions when compared to 2% total global CO₂ emissions attributed to the aviation industry [14-16]. The production of a ton of cement emits approximately a corresponding ton of CO₂ [17], making it the most energy-intensive material produced after steel and aluminium [3,14]. The cement section elaborates on the production process. In as much as development is required to match increasing populations, it should also be sustainable [18]. The underlying principles of sustainability lie in the appropriate balance of economic, social and environmental impacts [19-21]. Steel et al. [18] defined sustainability as a road for society advancement in which progress must be in harmony with the natural world, rather than in conflict with it, while Gambhir [14], termed it as a regime in which endeavors are towards meeting the needs of the present generation without compromising those of future generations. With a heavy demand for concrete in the developing world and other major and equally populous economies such as China and India predicted, cement producing companies have not anticipated in the foreseeable future any major changes in production that will reduce on emissions [1]. However, energy efficiency can be achieved by reducing on the amount of clinker and utilizing SCMs, which require less process heating and emit fewer levels of CO₂ [14]. Table 1 shows the embodied CO₂ values for the main constituents of reinforced concrete. It shows the embodied CO₂ for both PFA (also known as Fly Ash) and GGBS is substantially less than CEM I. Industrial and agricultural waste products such as PFA, GGBS, SF, RHA and CCA unnecessarily occupy space when stored or create environmental hazards when dumped in landfill [1]. Their utilization in the construction industry reduces the overall cost of construction, mitigates on the technical and environmental nuisance that is associated with the production of cement, reduces solid waste, cuts on greenhouse gas emissions and conserves existing natural resources, thereby enhancing sustainability as well as improving the properties of fresh and hardened concrete [1,20,21].

Table 1: Embodied CO₂ for main constituents of reinforced concrete [23].

Material		Embodied CO ₂ (kg/tonne)
Portland Cement, CEM I		930
Addition or Cement constituent	Ground Granulated Blastfurnace Slag	52
	Fly Ash	4
	Limestone	32
	Minor Additional constituent	32
Aggregates		4
Reinforcement		427

Components of Concrete Mix

Concrete is a predominant material used in construction worldwide and widely used in all types of civil engineering

works, including infrastructure, buildings, housing, hospitals, and environment protection. Concrete is usually a mixture of cement powder, water, sand (fine aggregates) and gravel (coarse aggregates). Proportions of each ingredient/constituents are adjusted to produce a well-balanced concrete. Concrete is a versatile material with important properties such as strength, durability, versatility, affordability, and fire-resistance, and thermal mass. Furthermore, it is locally produced and utilized in all countries.

Cement

The most common type of cement, which is used in combination with other materials to form different types of concrete is Portland cement [22,23]. Portland cement is produced by burning a mixture of calcium carbonate (limestone or chalk) and an aluminosilicate (clay or shale) and then the product is ground with approximately 5% gypsum to produce cement [24]. Cement is a material which binds together solid bodies (aggregate) by hardening from a plastic state. The European Standard specification EN 197 [25] categorises cements generally available in Europe into five main types:

- I. CEMENT TYPE I, CEM I:** Portland cement -Containing 5% of secondary sand supplementary constituents
- II. CEMENT TYPE II, CEM II:** Portland-composite cement - Up to 35% of other ingredients (other than OPC), e.g. PFA or limestone
- III. CEMENT TYPE III, CEM III:** Blast furnace cement -Comprising Portland cement and higher percentages of Blast furnace slag than in CEM II cement
- IV. CEMENT TYPE IV, CEM IV:** Pozzolanic Cement - Includes Portland cement and greater proportions of pozzolana than in CEM II cement
- V. CEMENT TYPE V, CEM V:** Composite Cement - Mix of Portland cement and combinations of Blast furnace slag, fly ash or pozzolana

In the United Kingdom, concrete is specified using BS EN 197-1:2011 [25] Cement. Composition, specifications and conformity criteria for common cements.

Cement production

The manufacture of cement is an energy intensive process. The first step is to quarry the main raw materials such as limestone, clay and other materials. After quarrying the rock is crushed in different stages. The first crushing reduces the rock to a maximum size of about 15cm and the secondary crusher or hammer mills reduces the rock size to about 7.5cm or smaller. A mixture of crushed rock and other ingredients such as iron ore or fly ash and ground, is fed to a cement kiln. The cement kiln is heated to 1400-1600 °C, in large cylindrical steel rotary kilns lined with special firebricks, [24]. Kilns are about 365cm in diameter and large enough to accommodate an automobile. The large kilns are mounted with the axis inclined slightly from the horizontal. The fine ground raw material is fed into the higher end of the kiln and at the lower end is a powerful blast of flame produced by burning of coal, oil, fuel or gas. Certain elements of the material (that moves through the kiln) are driven off in form of gases and the remaining material that emerges from

it as grey balls are known as clinker [23]. During the manufacturing process, large amounts of CO₂ gas is emitted; hence given this is an energy intensive process, the production of cement has a very high embodied CO₂. The clinker is then cooled and ground to a fine powder in a tube or ball mill. A ball mill is a rotating drum filled with steel balls of different sizes (depending on the desired fineness of the cement) that crush and grind the clinker. Gypsum is added during the grinding process to provide means for controlling the setting of the cement. The cement is then bagged and transported for concrete production. According to Taylor [26,27], at the mixing phase some specification allows for the addition of other materials. Table 2 illustrates the main components and their percentage in the clinker.

Table 2: The main components in clinker and their percentages [28].

Components	Percentage
CaO (calcium oxide)	67%
SiO ₂ (silica)	22%
Al ₂ O ₃ (alumina)	5%
Fe ₂ O ₃ (iron III oxide)	3%
Other components	3%

Cement composition

There are four main chemical compounds present in Portland cement, whose proportions in the mix affect the properties of the resulting cement substantially [23]. These compounds are: Tricalcium Silicate, Dicalcium Silicate, Tricalcium Aluminate and Tetracalcium Aluminoferrite and its properties are shown in Table 3 below:

Table 3: Typical composition of Portland cement [23].

Chemical Name	Shorthand Notation	Chemical Formula	Approximate % in OCP (mass)
Tricalcium Silicate	C ₃ S	3CaO·SiO ₂	45.0 - 60.0
Dicalcium Silicate	C ₂ S	2CaO·SiO ₂	15.0 - 25.0
Tricalcium Aluminate	C ₃ A	3CaO·Al ₂ O ₃	7.0 - 12.0
Tetracalcium Aluminoferrite	C ₄ AF	4CaO·Al ₂ O ₃ ·Fe ₂ O ₃	6.0 - 10.0

The properties of the compounds are:

A. Tricalcium silicate (C₃S): Water reacts rapidly with C₃S to produce a large amount of heat (exothermic reaction) to form calcium silicate hydrates. It has high strength and contributes most to the early strength of cement hydrates.

B. Dicalcium silicate (C₂S): Water reacts slower with C₂S to form calcium silicate hydrates. Due to the slow rate of reaction, any heat developed is dissipated before sufficient temperature rise occurs.

C. Tricalcium aluminate (C₃A): Water reacts very rapidly with C₃A by developing a significant amount of heat and with a

very rapid set. This reaction is retarded by the addition of gypsum during the grinding stage, i.e. to prevent flash setting.

D. Tetracalcium aluminoferrite (C₄AF): Water reacts rapidly with C₄AF however the reaction does not produce much heat with minimal contribution to strength. Therefore, the hydration of C₃S and C₂S are the main exothermic reactions (a chemical reaction that releases heat).

Water

Water has an effective impact on the workability of concrete and thus on the water-cement ratio [14]. On one hand, using a low water-cement ratio will lead to better strength and higher durability [14]. However, the low water-cement ratio may result in difficulties in handling and working with the concrete mix; the workability of concrete can be enhanced by using super plasticizers [14]. It is important the water is relatively pure; sea water must not be used as the high salt content can induce rapid chloride attack in reinforced concrete [14].

Cement hydration

When Portland cement is mixed with water it reacts and its constituent compounds undergo a series of chemical reactions that are responsible for hardening of the concrete [1]. This process is called the hydration of cement [23]. Specifically, as previously mentioned water reacts with tricalcium silicate and dicalcium silicate, the most reactive compounds. During this first reaction cement, the calcium silicates react chemically with water to produce a hard paste - gel (that coats the aggregates) made up of Calcium Silicate Hydrates (CSH) phase which is the major contributor to strength in concrete.

The first reaction involves tricalcium silicate:

Tricalcium Silicate + Water → Calcium Silicate Hydrate + Calcium Hydroxide (lime) + Heat

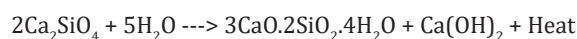


This is often written in shorthand form as follows in most civil engineering and construction literature:

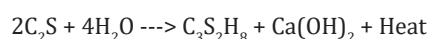


The initial reaction also involves dicalcium silicate:

Dicalcium Silicate + Water → Calcium Silicate Hydrate + Calcium Hydroxide + Heat



This is also often written in shorthand form as follows:



During the hydration of cement there is also the formation of lime (calcium hydroxide). Both reactions are exothermic, which means that heat is released during the reaction process. Tricalcium silicate and dicalcium silicate are the main contributors to compressive strength in concrete. According to Darwin et al. [23] when tricalcium silicate is first mixed with water, for the first 15 minutes a rapid evolution of heat occurs, known as stage

1. Then for the next 2 to 4 hours follows a relative inactivity, known as induction/dormant period (stage 2). Portland cement concrete remains in the plastic state for several hours due to this prolonged phase of inactivity. At the end of the induction period tricalcium silicate continues to rapidly hydrate and by 4 to 8 hours it has reached its maximum rate of heat evolution. This phase is acceleration period or stage 3. After 8 hours the early hardening has begun and during stage 4 the rate of reaction slows down again. Within 18 to 36 hours it reaches the steady state or stage 5. Figure 1 below shows the rate of heat evolution during hydration of

tricalcium silicate. Dicalcium silicate hydrates in the same way but because it is a less reactive compound the rate of reaction is slower. During hydration, tricalcium aluminate reacts with sulfate ions that are found in gypsum, to form calcium sulfoaluminate hydrate, commonly called ettringite. If the sulfate is all consumed before the C_3A has completely hydrated, then ettringite transforms to another calcium sulfoaluminate hydrate containing less sulfate, called tetra calcium aluminate monosulfate-12-hydrate or otherwise known as monosulfoaluminate [1,22-24]:

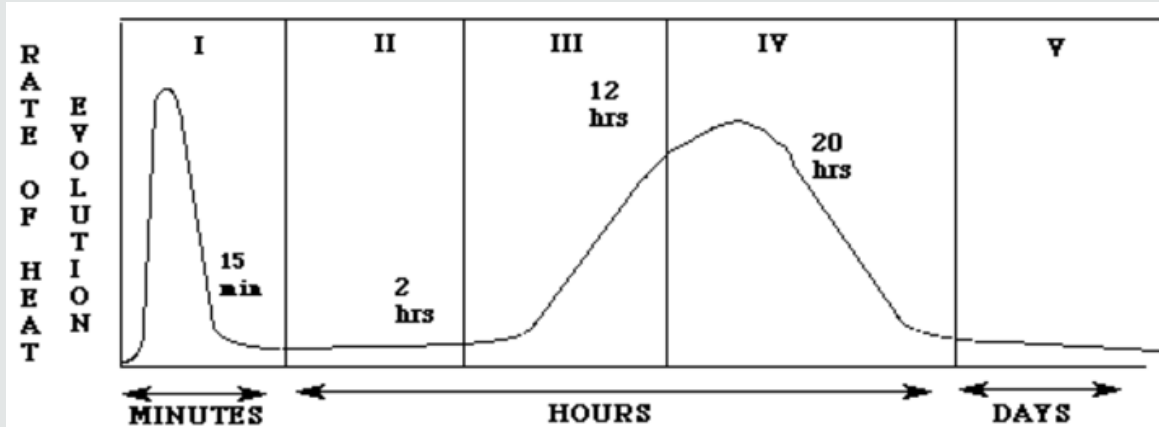
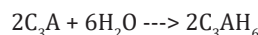


Figure 1: Rate of heat evolution during hydration of tricalcium silicate [1,22-24].

Ettringite does not contribute to compressive strength because of the sulfate environment. According to other researchers [1,22-24], during hydration, Tetracalcium aluminoferrite with or without gypsum forms similar hydration products to tricalcium aluminate, but the reactions are slower and require less heat. Concrete containing 100% cement usually reaches its maximum strength within 28 days, hence, this is usually taken as the reference point for most projects using concrete.

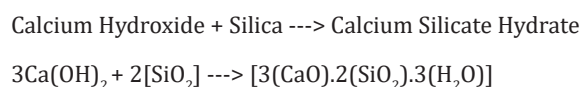
Pozzolanic reaction

Most SCMs are pozzolans, however, it is important to differentiate between a direct cement replacement and a pozzolan. A direct cement replacement is a type of cement substitute that can replace the Portland cement without requiring a pozzolanic activity/reaction discussed below [1]. The most common type of these replacements is Ground Granulated Blastfurnace Slag (GGBS), which is a by-product of the iron and steel industry, is a fine white powder. GGBS is chemically very similar to cement (Table 4) so can directly replace cement by up to 90% in some structural concrete [1]. However, the most common replacement is between 30 - 70% [1]. The reactions between Portland cement, GGBS and water are complex. Portland cement reacts first with water by forming insoluble hydration products such as calcium silicate hydrate and at the same time it forms a more soluble product, calcium hydroxide, which migrates through the pore solution [1]. The GGBS reacts with excess free calcium hydroxide to form calcium silicate hydrates (CSH) which reduces the size of capillary pores [1]. The reduction in free calcium hydroxide makes the concrete chemically more stable and it restricts the penetration of aggressive salts and sulfates to diffuse through the concrete (discussed later in this section) [1].

Table 4: The chemical compositions and physical properties of GGBS and Portland cement [29].

Oxide	Composition	
	GGBS	Portland cement
CaO	41%	65%
SiO ₂	35%	20%
Al ₂ O ₃	11%	6%
MgO	2%	8%
Colour	Off-white	Grey or White

Unlike GGBS (direct cement replacement), pozzolans, e.g. PFA, silica fume (SF), metakaolin (MK), rice husk ash (RHA) and corn cob ash (CCA) do not contain enough calcium to form cementitious materials, and need to combine with calcium hydroxide [Ca(OH)₂], the hydration product of cement, to form further strength giving Calcium Silicate Hydrate (C-S-H) [30]; most pozzolans e.g. PFA, SF, RHA contain a large amount of silica (SiO₂) which reacts with calcium hydroxide to form the strength forming C-S-H. As a result, pozzolans have been characterised with a delayed strength development, as they only act as fillers before Ca(OH)₂ becomes available, but have been reported to gain strengths that are close to or above those of 100% cement at a latter age which can be up to 365 days [30]. It has been reported [31-33], however, that concrete containing SF, RHA and MK exhibit impressive early age strength. The reaction between Ca(OH)₂ and SiO₂, also referred to as the pozzolanic reaction is shown below:



One thing to note is once all the calcium hydroxide has been completely depleted due to the pozzolanic reaction, any unreacted pozzolanic material in concrete will have minimal contribution to strength, merely fulfilling the role as filler [30]. Due to the limited availability of $\text{Ca}(\text{OH})_2$ in concrete, the cement substitute range using pozzolans is usually between 2.5-40% (by mass) depending on the type of pozzolan used and application of concrete, e.g. for PFA it is 20-40%, 5-20% for MK, and 2.5-10% for both SF and RHA [1,2,6,11,13,20,22,28,30-33]. As a result, most pozzolanic concrete contain highly depleted levels of calcium hydroxide which confers superior durability properties, e.g. sulfate resistance. Sulfate attack is a durability issue that causes concrete to lose its compressive strength, with severity depending on the type of sulfate [22-24]. All commonly available water-soluble sulfates are deleterious to concrete, with potentially severe effects. There are mainly two forms of sulfate attack of concrete, Magnesium Sulfate (MgSO_4) and Sodium Sulfate (Na_2SO_4) [1]. Higher sulfate concentrations in water are due to MgSO_4 , which is highly soluble in water at room temperature (20 °C) when compared to Na_2SO_4 [1]. Both Na_2SO_4 and MgSO_4 attacks have deleterious action on concrete but with different resultant effects [1]. In sodium sulfate attack, Na_2SO_4 reacts with $\text{Ca}(\text{OH})_2$ to produce gypsum [$\text{CaSO}_4 \cdot 2(\text{H}_2\text{O})$] and/or with aluminate (C_3A) and alumina-bearing hydration products to form ettringite. Gypsum and ettringite's precipitation in concrete could generate stresses within the concrete, which if greater than the tensile strength of the concrete may bring about loss in strength, expansion, spalling and degradation [34]. In pozzolanic concrete, the chemical reactions from pozzolans help in resisting sulfate attack as they refine pores, dilute C_3A and remove $\text{Ca}(\text{OH})_2$ by converting it into a cementitious material, thereby reducing the quantities of gypsum formed [1,12,35,36]. In contrast with cement hydration, $\text{Ca}(\text{OH})_2$ does not precipitate on the cement grain, but in the void space between the grains of SCMs [1]. Increased C-S-H content that results from pozzolanic reactions, consuming and reducing the amount of $\text{Ca}(\text{OH})_2$ and aluminate hydrate (CaAl_2O_4), and the filler effect of unreacted pozzolans can explain the ability of SCM / pozzolanic concretes to resist Na_2SO_4 attack [12,31,32,35-40].

Conclusion

- a) The cement industry is responsible for up to 10% of global CO_2 production.
- b) During the hydration of cement, the reactions of the calcium silicates and water leads to the formation of the calcium silicate hydrate (C-S-H) phase which is the strength forming compound in concrete.
- c) Calcium hydroxide ($\text{Ca}(\text{OH})_2$) is a by-product of the hydration of cement reaction.
- d) A pozzolanic reaction involves a secondary reaction whereby calcium hydroxide and usually silica (SiO_2) react to form the strength contributing C-S-H phase.
- e) Due to the secondary reactions, most pozzolanic concrete develop strength beyond 28 days and up to 365 days in some

cases, however, eventually reach strengths that are close to or above those of 100% cement.

- f) The lower calcium hydroxide content in pozzolanic concrete leads to improved sulfate resistance.

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