



## Evaluation of microstructural and microchemical aspects of high density concrete exposed to sustained elevated temperature



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### ABSTRACT

High density concrete is widely used as a primary material for radiation shielding due to numerous advantageous properties such as, strength, durability and ease of fabrication in comparison with other construction materials. As concrete is subjected even to high temperature environment in a nuclear reactor, the selection of concrete aggregates depends upon the exact application of the concrete. ACI limits the normal operating temperature in concrete structures to 65 °C in general and 90 °C locally, as exposure of concrete to high temperature induces complex changes in the moisture content as well as chemical composition of the cement paste and mismatch in thermal expansion leads to internal stresses and microcracking in the concrete constituents (aggregate and cement paste). Present research work is to investigate the suitability of concrete ingredients for producing high-performance high density concrete, of different densities using different types of aggregates (hematite and steel shots) and studying the physicochemical changes in them due to a constant exposure at 120 °C as in a sodium cooled reactor. The results indicated heavy moisture loss in the first seven days of exposure and reduction in the mechanical properties, followed by increase in strength on continued exposure. Microstructure and morphological variations in thermally aged fractured specimens were used to explain the above variation in the mechanical properties.

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### 1. Introduction

Concrete is widely used as a primary structural material in construction due to numerous advantageous properties such as, strength, durability, ease of fabrication and non combustibility in comparison with other construction materials. Concrete is widely used in nuclear reactors as a shielding material for the prevention of radioactive leakage [1]. The aggregate component of concrete containing a mixture of many heavy elements has good shielding properties which attenuate gamma and neutrons, thus, playing an important role in improving concrete shielding properties [2,3]. Heavy density concrete with density higher than 2.4 g/cc is achieved by using different aggregates such as hematite, magnetite, limonite and barite with high density.

Concrete gets exposed to elevated temperature either due to fire or due to its proximity near the furnace or reactor environment [4]. Exposure of concrete to high temperature induces complex changes

in the moisture content as well as chemical composition of the cement paste. Therefore, factors such as changes in chemical composition of concrete and the mismatch in thermal expansion lead to internal stresses and micro cracking in the concrete constituents (aggregate and cement paste). The transient strain in the concrete [5] results due to two processes: (1) moisture movement and dehydration of concrete due to high temperature and (2) acceleration of the process of breakage of bond [6]. Concrete as a shielding material gets exposed to high temperature due to attenuation of neutrons and gamma rays as well as in-core reactor conditions [7]. In the case of concrete shields which function at temperatures less than 105 °C, free water remains in the concrete for 10–20 years. As a worst-case scenario, thermal effects due to radiation could result in the loss of almost two-thirds of the total original water content over the useful life of the concrete-shield [8]. ACI limits the normal operating temperature in concrete structures to 65 °C in general and 90 °C locally unless the concrete mix is evaluated for variation of its properties and structure is designed accordingly [9]. A sodium-cooled pool-type Prototype Fast Breeder Reactor (PFBR) is under construction in Kalpakkam, India. High density concrete is being used for vault

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**Table 1**  
Various ingredients of the high density concrete mixtures.

Specimen ID	Cement, kg/m <sup>3</sup> (lt/m <sup>3</sup> )	Water, kg/m <sup>3</sup> (lt/m <sup>3</sup> )	Coarse aggregate, kg/m <sup>3</sup> (lt/m <sup>3</sup> )		Fine aggregate, kg/m <sup>3</sup> (lt/m <sup>3</sup> )	Steel shots, kg/m <sup>3</sup> (lt/m <sup>3</sup> )		Admixture, kg/m <sup>3</sup> (lt/m <sup>3</sup> )
			20 mm	12 mm		4 mm	2 mm	
A	420 (135.09)	155.4 (155.4)	580 (211.50)	580 (211.50)	772 (283.82)	–	–	2.94 (2.69)
B	420 (135.09)	185.0 (185)	884 (184.62)	884 (184.62)	1274 (306.05)	–	–	5.04 (4.62)
C	420 (135.09)	176.4 (176.4)	740 (155.22)	740 (155.22)	1105 (264.63)	416 (54.59)	416 (54.59)	5.04 (4.62)

**Table 2**  
Physical Properties of Concrete ingredients.

S. no.	Ingredient	Specific gravity of		Water absorption % (saturated surface dry condition)	
		Coarse aggregate	Fine aggregate	Coarse aggregate	Fine aggregate
1	Normal density aggregate	2.7	2.66	0.3	1.5
2	High density aggregate	4.71	4.1	0.4	3.5
3	Cement	3.11	–	–	–
4	Admixture	1.09	–	–	–

and roof shielding of main reactor vessel [10] in PFBR. Roof slab made of steel, forms the top cover for main vessel, which is filled with concrete for the purpose of nuclear radiation shielding. During normal operation, hot pool within the reactor is at 550 °C while roof slab is maintained at 120 °C and reactor vault is maintained below 65 °C. Since concrete is exposed to varying temperature regime, it is required to study the effect of temperature on microstructure and composition of such concretes.

In the present work, locally available haematite aggregate and steel shots made of high carbon steel were added to concrete to achieve the required density for  $\gamma$ -ray attenuation. A few workers have studied [11] effects of different proportions of haematite on physical and mechanical properties of concrete and concluded that the concrete properties improved on adding haematite aggregate. In an earlier study [12] five different concrete specimens were prepared with varying haematite contents and evaluated for neutron shielding properties.

Divya Rani et al. [13] reported that normal density concretes when subjected to sustained thermal ageing at temperatures of 65°, 75° and 90 °C did not show any change in their density values up to 50 days exposure. The aim of this research work is to investigate the suitability of concrete components for producing high-performance heavy density concrete, with different densities (2.4, 3.6 and 3.9 g/cc) using different types of aggregates and studying the physicochemical changes in them due to the constant exposure at 120 °C, resembling the conditions of the roof slab of PFBR.

## 2. Experimental methods

### 2.1. Materials

Pure haematite, a natural red rock that contains iron oxide, has the Mohs hardness between 5.5 and 6.5 and the specific gravity between 4.1 g/cc and 4.8 g/cc. Haematite was prepared as aggregate by crushing and grinding the ore in a laboratory mill, followed by sorting it through sieves into two groups such as coarse and fine aggregates. Three types of concrete mixtures were prepared for this study using the Portland cement conforming to IS 8112-2013 [14]. Details of mix design used for concrete are presented in Tables 1 and 2. Mix design was done using the absolute volume method (IS 10262[15]) to obtain non air entrained high density concrete. For a given water-cement ratio, for radiation shielding concrete, cement content is generally quite high, greater than 0.35 g/cc which helps to improve the neutron shielding characteristics of the concrete because of the high bound water content in the paste [16,17]. Also, the increase in the cement content will also increase the water

content per unit volume of concrete increasing the workability of concrete. Apart from increasing cement content, admixture in the form of superplasticizers (naphthalene based) with a solid content of 30% by mass, is added to achieve a cohesive workable concrete mix which can be easily placed within reinforced concrete vault.

- Concrete A - siliceous sand and crushed granite were used as the fine aggregate for granite aggregate concrete with a density of 2.4 g/cc.
- Concrete B - haematite aggregates were used for designing concrete with high density of 3.6 g/cc.
- Concrete C - In addition to the haematite aggregate, high chromium high carbon steel shots of 2 and 4 mm diameters were used for designing concrete with high density of 3.9 g/cc.

The concrete specimens, concrete A, concrete B and concrete C have been designated and referred as A, B and C hereafter throughout the text.

### 2.2. Specimen preparation

Absolute volume method was used to obtain denser concrete specimens using suitable mix design. Three specimens each with dimensions, 150 mm × 150 mm × 150 mm as well as cylinders with height 200 mm and diameter 100 mm were prepared using each mixture (Table 1). The cubes and cylinders were cast in iron moulds and de-molded after 24 h. The specimens were then conserved in water bath (22 °C) for 28 days, then taken out and left at ambient temperature for three months. The measured density of the specimen is given in Table 3.

### 2.3. Test procedures

#### 2.3.1. Heat treatment

The specimens were subjected to heat treatment at 120 °C in a thermal cycling chamber, (dimensions 1500 × 1500 × 600 m<sup>3</sup>)

**Table 3**  
Measured density values of concrete specimens.

S. no.	Specimen ID	Type of concrete	Measured density, g/cc
1	A	Normal aggregate with River Sand	2.50
2	B	Haematite aggregates	3.65
3	C	Haematite aggregate with steel shots	4.02

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