



## Technical note

## Evaluating resistance of fine bone china ceramic aggregate concrete to sulphate attack

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## ABSTRACT

This study deals with the results of sulphate attack on concrete containing fine bone china ceramic aggregate (BCFA). Natural river sand in concrete was substituted by 0%, 20%, 40%, 60%, 80% and 100% fine bone china aggregate. The concrete specimens were submerged in 3% MgSO<sub>4</sub> solution for 7, 28, 90 and 180 days. The influence of sulphate solution was evaluated by change in mass and change in compressive strength. Studies related to microstructure like FT-IR and XPS were carried out to monitor the influence of BCFA in sulphate resistance of concrete.

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## 1. Introduction

As one of the largest industries in the world the construction industry has a significant fingerprint on the consumption of raw materials and energy. To mitigate such negative impact on the environment and ecosystem several steps have been carried out: improved durability of concrete, use of industrial waste as aggregate, use of supplementary mineral admixtures and recycling and reusing materials [1–8].

The use of ceramic waste as aggregate is a positive step in conservation of natural resources and recycling of industrial waste. Most of the studies available have focused on the coarse and fine aggregate or as supplementary cementitious form of sanitary ware, tile waste, earthen ware and electrical insulator waste in concrete [9–14].

Medina et al. [15,16] reported that introduction of coarse sanitary ware ceramic aggregate enhances the mechanical and durability properties of concrete. The pozzolanic behaviour of ceramic aggregate resulted in dense microstructure which resulted in enhanced resistance to chloride based chemical agents. Viera et al. [17] reported lower durability characteristics of concrete mixes containing fine sanitary ware ceramic aggregates. The porous structure of aggregate was considered as probable reason for

higher ingress of chemical agents. Corominas and Etxeberria [12] showed that at 180 days of testing age the concrete mix with ceramic aggregate showed lowest permeability values. Halicka et al. [10] reported that use of both fine and coarse aggregate resulted in higher strength characteristics and ceramic concrete displayed better resistance against exposure to elevated temperature. Senthamarai et al. [14] reported that utilisation of coarse electrical insulator based aggregate resulted in slightly lower durability properties of concrete.

It can be seen from the current state of the art that different type and form of ceramic aggregate have diverse influence on the durability of concrete against chemical attack. Furthermore, as per best knowledge of author no study is available on the influence of BCA on sulphate attack resistance properties of concrete. The objective of this study is to identify and observe the physical and microstructural changes in magnesium sulphate exposed concrete containing varying percentage of BCA.

## 2. Experimental investigation

## 2.1. Materials and mix

The following materials were used in the study to assess the influence of fine bone china ceramic aggregate (BCFA) on sulphate resistance of concrete.

Ordinary Portland cement of 43 grade was used as binder. River based natural fine aggregate having water absorption and specific

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gravity as 1.0% and 2.67 respectively was used. Basalt based crushed stone having water absorption and specific gravity as 0.5% and 2.74 respectively was used as coarse aggregate. BCFA was manufactured by crushing ceramic waste from a local tableware producing unit. The water absorption and specific gravity of BCFA concrete was 2.5% and 2.40 respectively. The maximum diameter of natural fine aggregate, BCFA and natural coarse aggregate was 4.75 mm, 4.75 mm and 18 mm. Normal tap water was used in the mixing of concrete. To maintain a compaction factor of 0.90–0.95 a high range water reducer admixture was used and to compensate for water absorption of various aggregate extra water was provided in the mix to maintain uniform water/binder ratio throughout the study.

Chemical compositions of cement and natural sand are given in Table 1.

Table 2 shows the chemical composition of BCFA and other types of ceramic aggregate reported in other literature works. It can be observed that BCFA have nearly similar percentage of CaO, SiO<sub>2</sub> and Al<sub>2</sub>O<sub>3</sub>; whereas the other types of ceramic aggregate have dominance of SiO<sub>2</sub>.

The concrete mix design compositions based on the guidelines of BIS 10262 [18] are shown in Table 3. The 28 days compressive strength observed for different concrete mixes is also presented in Table 3.

## 2.2. Experimental programme

Twelve 100 mm cubic specimens for six concrete mixes were prepared and their resistance to sulphate attack was determined by immersing them in 3% MgSO<sub>4</sub> solution. The specimens were

**Table 1**  
Chemical composition of cement, natural fine aggregate and BCFA.

Chemical constituent	Ordinary Portland cement	Natural fine aggregate
CaO (%)	62.34	1.15
SiO <sub>2</sub> (%)	20.14	81.67
Al <sub>2</sub> O <sub>3</sub> (%)	4.65	9.81
Fe <sub>2</sub> O <sub>3</sub> (%)	3.29	2.11
SO <sub>3</sub> (%)	2.42	–
MgO (%)	2.23	0.75
K <sub>2</sub> O (%)	0.72	2.52
LOI (%)	1.96	0.2

**Table 2**  
Mix proportions.

Chemical constituent	BCFA	Sanitary ware aggregate [19]	Tile waste aggregate [20]	Electrical insulator waste [21]
CaO (%)	24.15	0.63	3.35	0.76
SiO <sub>2</sub> (%)	28.86	68.41	69.93	70.9
Al <sub>2</sub> O <sub>3</sub> (%)	23.86	24.46	15.80	21.1
Fe <sub>2</sub> O <sub>3</sub> (%)	5.41	0.94	1.98	0.81
MgO (%)	2.86	0.19	3.35	0.24
K <sub>2</sub> O (%)	1.58	2.80	2.68	3.57
LOI (%)	0.5	–	0.39	–

**Table 3**  
Mix proportions (For compressive strength average of three values is shown).

Mix	Cement (kg/m <sup>3</sup> )	Natural sand (kg/m <sup>3</sup> )	BCFA (kg/m <sup>3</sup> )	Coarse aggregate (kg/m <sup>3</sup> )	Water (kg/m <sup>3</sup> )	Superplasticiser (%)	28 days compressive strength (MPa)
CS0	383	821	0	1095	134.05	0.5	39
CS20	383	656.8	147.78	1095	134.05	1.0	40
CS40	383	492.6	295.56	1095	134.05	1.0	45
CS60	383	328.4	443.34	1095	134.05	1.0	47
CS80	383	164.2	591.12	1095	134.05	2.2	44
CS100	383	0	738.9	1095	134.05	2.4	44

**Table 4**  
Assigned peaks in FT-IR spectra.

Wave number (cm <sup>-1</sup> )	Functional bond	Assigned to	Reference
1641, 3000–3750	O–H	Ca(OH) <sub>2</sub>	[22–25]
1324–1576, 1640–1800, 2501–2550	CO	CaCO <sub>3</sub>	[23,26,27]
852, 890–1079	Si–O	CSH	[22,25]
450–650, 796	Si–O–Si	SiO <sub>2</sub>	[23,25]
1100–1150, 2330–2370	S–O	SO <sub>4</sub>	[28,29]
2850–2925	CH <sub>2</sub> /CH <sub>3</sub>	Methyl and methylene	[30]

properly marked and weighed for initial reference before allowing all the surfaces to be submerged in sulphate solution. The sulphate solution was replenished periodically to maintain the same concentration throughout the study. The exposure period to the sulphate solution was for 7, 28, 90 and 180 days.

The compressive strength after 7, 28, 90 and 180 days was carried out on three 100 mm cubic specimen after recording their mass. The change in mass and compressive strength is reported to compare the influence of BCFA concrete on resistance to sulphate attack.

The specimens exposed for 180 days were evaluated for microscopic changes due to sulphate attack. Sample preparation for FT-IR and XPS measurements was carried out by crushing a 100 mm cube specimen submerged in sulphate solution for 180 days and then dry grinding the bulk mortar pieces to obtain a uniform size passing 90 μm sieve size. The same procedure was followed for all the samples throughout the study.

The FT-IR analysis was performed by pellet method on the wavelength range of 4000–400 cm<sup>-1</sup> at 4 cm<sup>-1</sup> resolution. Linear baseline correction of the spectra was done automatically. The characteristics peaks observed at different wave number are presented in Table 4.

A pellet sample (5 mm × 5 mm × 1 mm) was used for XPS measurements. The XPS analysis was carried out on an XPS system using monochromatic Al K $\alpha$  radiation  $h\nu = 1486.7$  eV. The samples were deposited on a carbon tape and degassed overnight in XPS chamber to minimise air contamination. Pass energy of 50 eV was maintained to carry out the survey analysis.

## 3. Results and discussion

### 3.1. Change in mass

Fig. 1 shows the change in mass observed for specimens exposed to sulphate solution for 7, 28, 90 and 180 days. The values shown are average of three concrete specimens. For all concrete mixes an increase in mass was observed till 28 days of sulphate exposure. For concrete mixes CS80 and CS100 gain in mass continued till 90 days of exposure. The magnesium sulphate used to simulate the sulphate attack on concrete forms magnesium hydroxide which lowers the pH of the solution leading to formation of

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