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Effect of Boron Carbide Addition on the Physical, Mechanical and Microstructural Properties of Portland Cement Concrete

[1]Fatin Nabilah Tajul Ariffin, [2]Yusof Abdullah, [1]Roslinda Shamsudin,
[3]Roszilah Hamid and [1]Sahrim Haji Ahmad
[1]Faculty of Science and Technology, School of Applied Physics, University Kebangsaan Malaysia, 43600 UKM, Bangi, Selangor, Malaysia
[2]Malaysian Nuclear Agency, Bangi, 43000 Kajang, Selangor, Malaysia
[3]Department of Civil and Structural Engineering, Faculty of Engineering and Built Environment, University Kebangsaan Malaysia, 43600 UKM, Bangi, Selangor, Malaysia

Abstract: Concrete nuclear reactors could be improved in terms of life safety by adding boron carbide. This study presents an experimental investigation of the physical, mechanical and microstructural properties of Portland cement concrete containing boron carbide (B_4C) as a neutron radiation-absorbing material for nuclear reactor applications. The boron carbide powder additions were 5 and 20% of the cement weight. The water-to-cement ratio of the concrete design mix was 0.4. The results show that the concrete density decreased as the percentage of boron carbide content increased. The results also show that concrete with a 0% content of B_4C produced the highest compressive strength (32.73 MPa) and that the addition of 5 and 20% B_4C produced a negligible reduction of strength (<2% compared with 0% B_4C concrete). Scanning Electron Microscopy (SEM) results confirm that the addition of boron carbide to Portland cement concrete reduces the strength and density of concrete because the morphology of samples containing 5 and 20% B_4C by weight (wt.) shows a more porous concrete microstructure compared with the control samples. Energy Dispersive X-ray (EDX) analysis was used to conclude that the higher content of B_4C results in a lower percentage of calcium in the concrete which in turn reduces the strength. Up to 20% B_4C powder by weight can be added to concrete which produces minimal strength reduction.

Key words: Boron carbide, neutron absorption, Portland cement, compressive strength, nuclear reactor, concrete

INTRODUCTION

A nuclear reactor is a source of energy for heat and electricity generation (Morioka *et al*., 2002). Nuclear research in Malaysia began in 1983 with the development of a nuclear research reactor named Triga Mark II which generates 1 mW of power. Nuclear reactors are usually surrounded by thick layers of concrete which serves as biological shielding. The concrete protects the surroundings from the high levels of radiation emitted from the reactor and is used to shore up the reactor and its related equipment (Yousef *et al*., 2008). Therefore, a special radiation shielding concrete is a very important component of a nuclear reactor.

Boron carbide (B_4C) is the third hardest material after diamond and cubic boron nitride (McColm, 1990). Boron carbide has ideal characteristics for use as a radiation shielding material because of its high neutron absorption capacity. It can sustain high temperatures, has a low density and a high degree of chemical inertness (Unal *et al*., 2006). Fast neutrons are not easy to extinguish because they easily penetrate most materials. They should be slowed down by scattering within materials that have a high interaction-scattering cross section (Kharita *et al*., 2011). The lighter isotope of boron, boron-10, could cause low penetration from neutron capture by hydrogen in the gamma ray test (ASTM C 638-92, 2002). The neutron absorption cross section of boron-10 is 760 barns. The higher the neutron absorption cross section, the more stable the nucleus capturing the neutron. Li and Qiu (2007) reported that boron carbide is used as a reinforcement in ceramic matrix composites in high-technology industries to increase wear and corrosion resistance properties.

Corresponding Author: Fatin Nabilah Tajul Ariffin, Faculty of Science and Technology, School of Applied Physics, University Kebangsaan Malaysia, 43600 UKM, Bangi, Selangor, Malaysia
Tel: +603 8921 6447 Fax: +60389216147

Thevenot (1990) noted that nuclear reactors have taken extensive advantage of boron carbide because of its adaptable properties, such as its low density, low thermal conductivity and high neutron absorption area (Rao *et al.*, 2009). Boron carbide powder (15 μm in size) is added to concrete to produce Portland cement/B_4C concrete as a neutron-absorber concrete. Boron carbide is categorized as a non metallic ceramic material and the hardest compound of the boron-containing materials. The properties of boron carbide is a combination of unique characteristics, such as strength (H = 35 Gpa), low density (ρ = 2.51 g cm^{-3}) and dynamic modulus of elasticity which is the highest of ceramics. Boric oxide and petroleum coke were used as the raw materials to produce boron carbide in a heat-resistance furnace at a temperature of up to 2600°K (Rao *et al.*, 2009).

A combination of calcium and aluminum silicates is used to produce Portland cement. These two powdered crystalline minerals can be hardened with the addition of water which produces a stone-like material. The recrystallization process forms interlocking crystals which results in the production of cement gel. Once hardened, this material has a high compressive strength (Nawy, 2009).

Aggregates is also a key component in the formation of concrete. The size and quantity of aggregate influence the concrete mix design. The concrete strength increases as the size of the aggregate decreases (Abo-Qudais, 2005). However, a small-aggregate-concrete will have a higher cost. Sand and rock fragments are commonly used in concrete mix (Kamarudin, 1995).

In the process of forming the concrete, the water to cement ratio (w/c) is the most direct measure of the strength of the concrete (Nawy, 2009). Cement will harden when it reacts with water. The concrete strength is governed by its water content (Ohdaira and Masuzawa, 2000).

In this work, the main objective was to determine the maximum percentage possible of boron carbide that can be added to Portland cement concrete to improve the nuclear reactor concrete shielding capacity while at the same time maintaining the concrete strength.

MATERIALS AND METHODS

Twelve samples of concrete cube with dimensions of 100×100×100 mm^3 were prepared using ordinary Portland cement (Type I) from YTL Cement Berhad. Four cubes were prepared as control samples (0% B_4C content), while another eight cubes of concrete contained different contents of boron carbide from Sigma-Aldrich Corporation (5 and 20%). Details of the mix proportions for the concrete containing different weight percentages of boron carbide are provided in Table 1. Samples were prepared using the Department of Environment, United Kingdom method (Teychenne *et al.*, 2010) for concrete grade 20 in the laboratory. Coarse aggregate with a maximum size of 10 mm was obtained from Lafarge Aggregates Sdn Bhd.

Table 1: Quantity of Portland cement/B_4C concrete components

Samples	Cement (kg m^{-3})	Water (kg m^{-3})	Coarse aggregate (kg m^{-3})	Sand (kg m^{-3})	Boron carbide (kg m^{-3})
Mix 1 (0 wt. % B_4C)	532.5	213.0	914.5	690.0	0
Mix 2 (5 wt. % B_4C)	532.5	213.0	914.5	655.5	34.5
Mix 3 (20 wt. % B_4C)	532.5	213.0	914.5	552.0	138.0

The density of the samples is determined using the Archimedes' concept according to BSI (1983a) to obtain more accurate density values. The values of the compressive strength of the concrete cubes after curing for 28 days are obtained in accordance with BSI (1983b).

The morphology of Portland cement/B_4C concrete is examined using Scanning Electron Microscopy (SEM). Prior to the scan, the sample surface is coated with a thin layer of gold. The thickness of the gold layer is between 0.01 to 0.1 μm. The elements present in the sample are analyzed by the X-ray Energy Dispersion (EDX) method (Buhrke *et al.*, 1998) conducted in conjunction with Scanning Electron Microscopy (SEM).

RESULTS AND DISCUSSION

The densities of the concrete samples are shown in Table 2. The results show that the Portland cement/B_4C concrete sample with 0 wt.% of boron carbide has the highest density, 2255 g cm^{-3}. The lowest density is observed for Portland cement/B_4C concrete with 20 wt.% of boron carbide, 2096 g cm^{-3}. This indicates that the concrete cube without boron carbide is denser than Portland cement/B_4C concrete.

Table 2 shows that the density of Portland cement/B_4C concrete decreases with the increase in the boron carbide percentage. This is due to the density of boron carbide (2.51 g cm^{-3}) which is lower than the density of concrete. As a result, the higher the weight percentage of boron carbide added to the concrete, the lower the density of the Portland cement/B_4C concrete. To confirm these results, Albano *et al.* (2005) found that the addition of other materials, such as scrap rubber, can lower the density of concrete. The study showed that concrete mixes with scrap rubber can decrease the density of the concrete by 20.34% compared with only a 2% density loss by the addition of 5 wt. % boron carbide.

Table 3 shows that the compressive strength decreases with increasing boron carbide content. Portland cement/20% B_4C concrete had the lowest compressive strength of 29.7 MPa, while the Portland cement/0% B_4C concrete had the highest compressive strength of 32.73 MPa. The decrease is less than 2% compared with concrete with 0% boron carbide. It can be concluded that up to 20% B_4C content can be added to Portland cement concrete without affecting the strength significantly. Based on previous research by Kharita *et al*. (2009), the improvement of the compressive strength of shielding concrete made from hematite aggregates can be performed by adding the optimum weight of 6% of carbon powder. Their study showed that the addition of carbon powder can improve the mechanical properties of the concrete. However, the addition of boron carbide gives the opposite result: it did not enhance the concrete compressive strength.

Scanning Electron Microscopy (SEM) is capable of analyzing images and providing a quantitative analysis of the surface microstructure of the samples. SEM observations of Portland cement/0%B_4C concrete are shown in Fig. 1. This Fig. 1 clearly indicates that the sample grain-size is small and irregular. Therefore, the surface area of the sample in Fig. 1 is large and this causes the particles to become more compact, as indicated by the higher density of Portland cement/0% B_4C concrete. Figure 2 is the SEM image of the 5 % B_4C concrete mixture which has a more homogeneous structure (Unal *et al*., 2006).

The morphology of the boron carbide distribution was observed by Unal *et al*. (2006), who studied the grain size determination of boron carbide. The SEM test of the samples shows that the boron carbide compound contained large particles, with smaller particles separated evenly throughout the matrix with a low-porosity surface. According to Fig. 2, when boron carbide was added to concrete, the SEM image shows that the microstructure has a high porosity with large and small particles of boron carbide separated on the concrete surface. It is clearly visible that the boron carbide particles can be detected by their shape and size difference. This shows that the boron carbide particles are bound to the concrete surface and influence the morphology of the concrete.

Figure 3 shows a larger grain size and a more porous structure than that observed in Fig. 1 and 2. This is because of the higher content of boron carbide of 20%. The relationship between density and porosity is an indirect proportionality. The density of 20 wt.% B_4C concrete sample is the lowest because the surface area of the grains in the sample is the smallest. It is clearly observed that the B_4C particles can be recognized by their shape difference (Aitcin, 2003).

Table 2: Density of Portland cement/B_4C concrete

Sample	Weight of $cube_{dry}$(g)	Weight of $cube_{wet}$(g)	Volume, v (cm^3)	Density, ρ ($g\ cm^{-3}$)
0 wt.% B_4C	2092.50	1164.25	928.25	2.255
5 wt.% B_4C	2120.00	1160.50	959.50	2.210
20 wt.% B_4C	1995.00	1043.00	952.00	2.096

Table 3: Compressive strength of Portland cement/B_4C concrete

Sample	Surface area (mm^2)	Load (kN)	Strength (MPa)
0 wt.% B_4C	1000	327.33	32.73
5 wt.% B_4C	1000	321.60	32.16
20 wt.% B_4C	1000	290.73	29.07

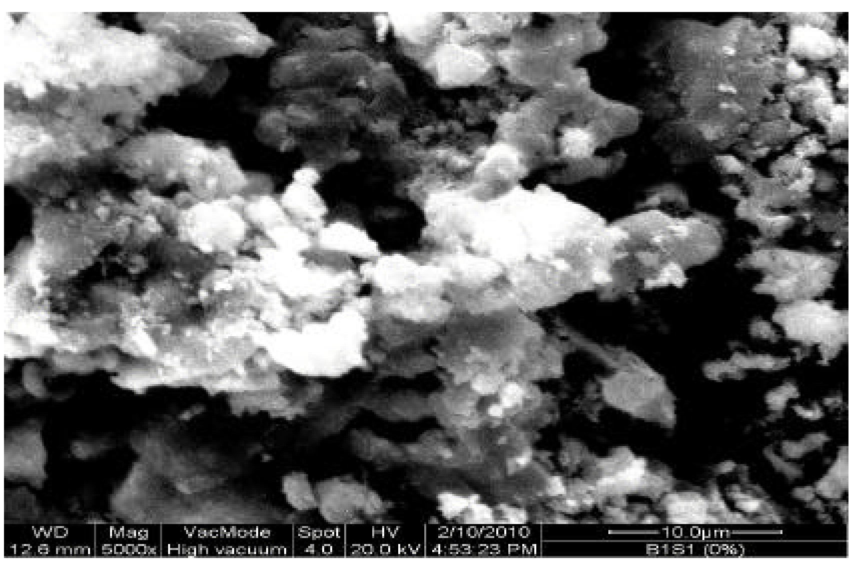

Fig. 1: SEM with 5000 times magnification of the 0 wt.% B_4C concrete

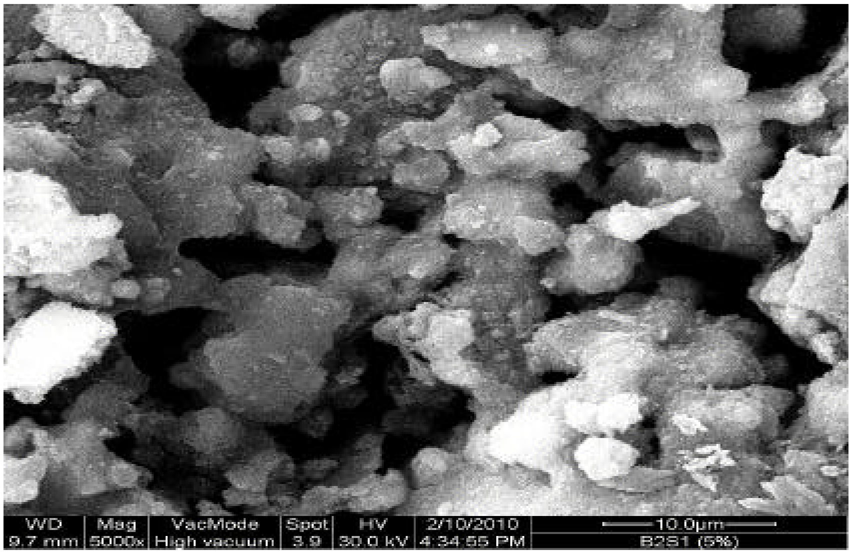

Fig. 2: SEM with 5000 times magnification of the 5 wt.% B_4C concrete

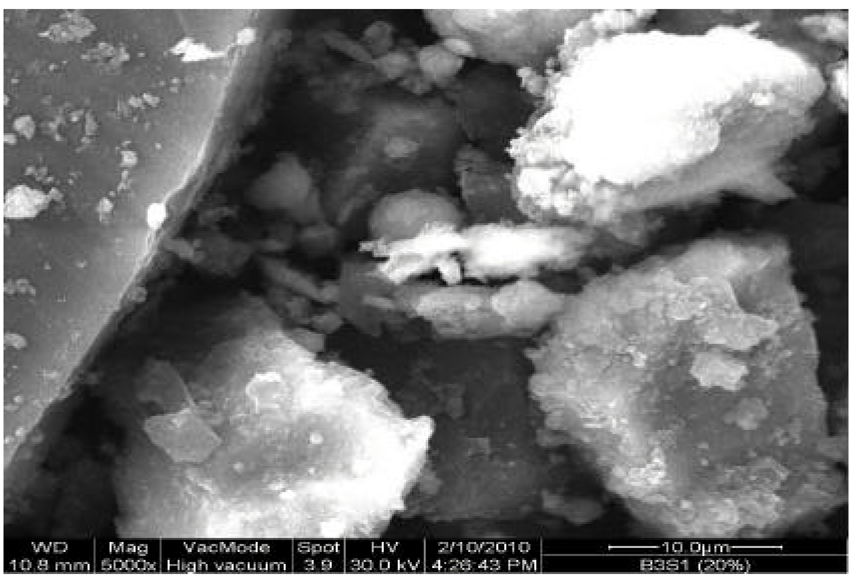

Fig. 3: SEM with 5000 times magnification of the 20 wt.% B_4C concrete

Energy Dispersive X-ray (EDX) analysis was performed to analyze the elements qualitatively. Previous study by Asokan *et al.* (2010) indicated that the EDX spectrum can show the elemental compositions of concrete. The results of EDX show that there are four main elements detected in the concrete: calcium, silicon, oxygen and boron. These elements are high-concentration elements and other trace elements are also present which include iron, aluminium, magnesium and sodium. The results confirmed the presence of boron in both spectra (Fig. 5 and 6). The relative percentage of calcium present in the 0 wt.% B_4C concrete sample (Fig. 4) is the highest (20.93%), followed by the 5 wt. % B_4C concrete sample (Fig. 5), which is 15.63%. The percentage of calcium in the 20 wt.% B_4C concrete sample shown in Fig. 6 is 12.16%. From this analysis, it can be concluded that the higher

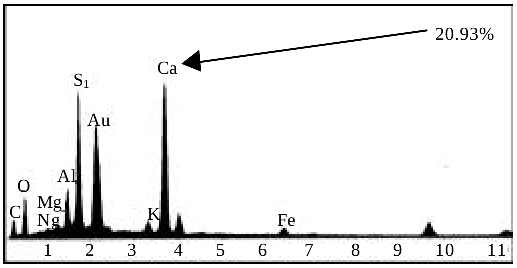

Fig. 4: EDX spectrum graph of a 0 wt.% B_4C concrete sample

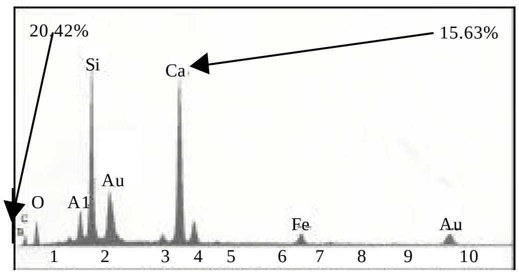

Fig. 5: EDX spectrum graph of a 5 wt.% B_4C concrete sample

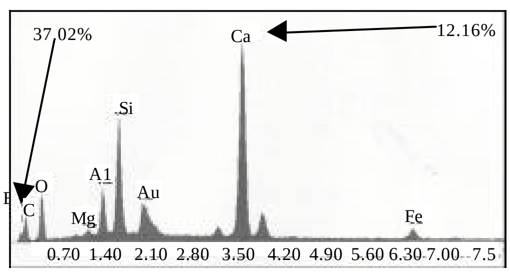

Fig. 6: EDX spectrum graph of a 20 wt.% B_4C concrete sample

content of B_4C results in a lower percentage of calcium in the concrete which in turn reduces the strength.

The peak of the boron element in Fig. 5 shows the presence of 20.42% boron. Meanwhile, Fig. 6 (20 wt.% B_4C) shows 37.02% boron which is higher than that in Fig. 5 (5 wt.% B_4C).

CONCLUSION

The density analyses prove that the density of the concrete with 0% boron carbide is higher than that of the Portland cement/B_4C concrete. Portland cement/5% B_4C concrete reduced the density by 2% which is significant. If strength is the major issue, 5 wt.% B_4C is the optimum value for the boron carbide addition in concrete since this mix strength is at par with the normal concrete. The addition of up to 20% boron carbide particles to Portland cement concrete reduces the strength of the concrete by 2%. The morphology of SEM observations also showed that the morphology of 0% B_4C is denser than the morphology of 5 and 20% B_4C concrete samples. Energy dispersive X-ray (EDX) analysis concluded that the higher content of B_4C results in a lower percentage of calcium in the concrete which in turn reduces the strength.

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