

# Investigation of autoclaved cement systems with reactive MgO and Al<sub>2</sub>O<sub>3</sub>-SiO<sub>2</sub> rich fired clay brick

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**Portland cement (PC) is one of the world's most important building materials, as it is a fundamental component of concrete. However, the manufacture of PC is highly energy intensive and leads to the emission of carbon dioxide (CO<sub>2</sub>). One promising control measure is the use of industrial wastes and by-products as supplementary cementing materials (SCMs) in order to minimise PC consumption, thereby producing greener cement-based products. This study investigates mechanical properties and phase development of hydrothermally treated cement–ground quartz sand blends with the incorporation of fired clay-brick (CB) waste and reactive magnesia (MgO). The addition of CB waste in autoclaved PC–quartz mortar mixes showed that the alumina–silica rich CB waste was pozzolanic when the Al<sub>2</sub>O<sub>3</sub> accelerated formation and increased crystallinity of Al substituted 1·1 nm tobermorite, resulting in the observed strength gain. Autoclaved mortar specimens incorporating reactive MgO showed a reduction in strength with increasing MgO addition. This was a result of dilution when the relative proportion of PC available for the formation of the strength contributing hydration products including tobermorite is decreased.**

## Introduction

The world's demand for Portland cement (PC) has increased substantially and global production rose from 594 Mt in 1970 to over 2550 Mt in 2006 (Worrell *et al.*, 2009), making the cement industry one of the largest single emitters of carbon dioxide (CO<sub>2</sub>). In addressing the issues of energy consumption and CO<sub>2</sub> emission, the use of industrial wastes as supplementary cementing materials (SCMs) is a promising control measure directed at minimising the consumption of PC, thereby producing greener cement-based products. The use of SCMs in the partial replacement of PC is a well-established practice in the production of concrete, mortar and other construction and building materials. These pozzolanic SCMs are designed to react with calcium hydroxide released from the hydration of PC to produce the principal cementing binder calcium–silicate–hydrate (C-S-H) phases. There are a number of benefits associated with the use of SCMs, which include improved mechanical properties, workability and durability (Malhotra and Kumar Mehta, 1996). The partial cement replacement by SCMs can result in substantial energy and cost savings as most of SCMs are industrial by-products, which require relatively little additional processing or no expenditure of energy. The utilisation of these industrial by-products also reduces pressure on waste management and dis-

posal. Common examples of SCMs include fly ash, blast furnace slag, silica fume, rice husk ash and fired clay brick (CB), all of which are industrial by-products or wastes.

Fired CB waste, investigated in this study, is generated in the order of million tonnes during manufacture, construction and demolition on a global scale. Although some of this waste material is recycled, a large amount ends up in landfills or reclamation sites for disposal. Fired CB waste is rich in Al<sub>2</sub>O<sub>3</sub> and SiO<sub>2</sub> with quartz, mullite, hematite and cristobalite as the major crystalline phases generally present in all brick types. The high firing temperature during the production of CBs is responsible for the thermal activation of clay minerals that renders them pozzolanic (Ambroise *et al.*, 1985). The pozzolanic activity of clay is highest when the calcination results in dehydroxylation and a collapsed and distorted clay structure (O'Farrell *et al.*, 2006). The feasibility of the partial replacement of cement in concrete with finely ground waste CB was rigorously investigated by Wild *et al.* (1998). Their research showed that CB waste exhibited high pozzolanic activity under ambient conditions when finely ground. Studies by Klimesch and Ray (2001, 2002) showed the formation of 1·1 nm tobermorite ( $5\text{CaO}\cdot6\text{SiO}_2\cdot5\text{H}_2\text{O}$ ) under autoclaving conditions is enhanced by the addition of finely ground CB waste

to blends of PC. More specifically, the presence of Al in CB waste, particularly mullite, accelerates the crystallisation of Al-bearing 1·1 nm tobermorite from calcium silicate hydrate.

An alternative approach to the minimisation of PC consumption is the addition of reactive or light-burned magnesia (MgO). Light-burned MgO (produced at calcination temperatures of 600°C to 900°C) is characterised by a small particle size, a range of surface areas, moderate to high chemical reactivity and undergoes rapid hydration to Mg(OH)<sub>2</sub> (brucite) in the presence of water. The relatively low calcination temperatures when compared to temperatures exceeding 1400°C required to manufacture PC, render reactive MgO an attractive material for the possible inclusion in cement-based building materials. Furthermore, unlike some industrial waste SCMs in which their quantities are finite, MgO is an abundant natural resource and hence large-scale utilisation may not be limited. Another advantage of reactive magnesia for the cement-based building industry is its potential to be used as a flame-retardant filler. The hydrated brucite releases water when decomposed to MgO at about 300°C (Xiang *et al.*, 2003). The utilisation of MgO had also been suggested as an expansive additive to produce a shrinkage-compensated cement (Buntoro, 2000; Taylor, 1997; Xu and Deng, 2005). The expansive properties of MgO originate from its hydration to Mg(OH)<sub>2</sub>, which involves a volume expansion of 118% (Rehs and Majumdar, 1969). This volume expansion for periclase (dead-burned MgO found in PC that is highly crystalline and low in reactivity) is, however, undesirable as its slow hydration to brucite can compromise long-term volume stability or soundness of hardened paste when its content exceeds 6% (Kasselouris *et al.*, 1985). By comparison, reactive magnesia is significantly more reactive and undergoes high rates of hydration, which results in a negligible effect on the soundness of hardened paste.

An important industrial technique used to manufacture cement-based building products is high-pressure steam curing (also known as autoclaving or hydrothermal curing). Hydrothermal curing is typically carried out between 160° and 200°C for several hours when, under such conditions, the pozzolanic materials are rendered highly active (Taylor, 1997). It is a well-established process to allow rapid strength development and afford superior durability properties. Under autoclaving conditions, C-S-H with a Ca/Si ratio of 0·9–1·0 tends to crystallise to give tobermorite (Mitsuda *et al.*, 1986). Tobermorite is regarded as one of the most important hydrous calcium silicate phases, as it serves as the principal binder of most autoclaved calcium silicate-based building materials. The presence of Al from Al-containing material, such as CB waste, accelerates the crystallisation of Al-bearing 1·1 nm tobermorite from C-S-H (Kalousek, 1957; Klimesch and Ray, 2001; Klimesch *et al.*, 2002). From the viewpoint of durability, Al-tobermorite is regarded as more beneficial than Al-free tobermorite (Šauman, 1971).

This study investigates the hydrothermal chemistry and mechanical (compressive strength) properties of autoclaved PC-quartz

blends when PC was partially replaced by reactive magnesia (MgO), a potential flame-retardant filler, and alumina–silica rich fired CB waste fines, an abundant industrial waste and a proven SCM (Wild *et al.*, 1998).

## Material and methods

### Raw materials

Raw materials used in this study were sourced from local suppliers. The chemical compositions of the raw materials used are given in Table 1. Details of the raw materials used are listed below.

- Goliath cement (ordinary Portland cement; OPC) produced by Australian Cement, Auburn, NSW, Australia, was used. Goliath cement is a general-purpose Portland cement with low shrinkage characteristics satisfying type SL requirements of Australian Standard AS3972 (SAA, 2010). The material has a fineness index (Blaine) of 350 m<sup>2</sup>/kg.
- Type 200G ground silica (quartz sand) was supplied by Unimin Australia Limited, Sydney, Australia, with a Blaine value of 360 m<sup>2</sup>/kg. The material has a particle size range of finer than 75 µm with a maximum of 5·5% greater than 75 µm.
- Natural magnesite (MgCO<sub>3</sub>), with surface area of 2·3 m<sup>2</sup>/g, was supplied by Unimin Australia Limited, Sydney, Australia. It has a bulk density of 1·6 g/cm<sup>3</sup> and a particle size range of less than 75 µm with a maximum of 2% greater than 75 µm. The magnesite was calcined to light-burned magnesia at 600°C for 2 h. The degree of decarbonation of calcined MgO, determined by using differential thermal analysis and thermogravimetric analysis (DTA/TGA), showed that 96·2%

Oxides	OPC	Silica	Magnesite	Brick waste
SiO <sub>2</sub>	20·0	98·9	6·15	69·71
Al <sub>2</sub> O <sub>3</sub>	4·5	0·6	1·60	18·64
Fe <sub>2</sub> O <sub>3</sub>	3·7	0·1	0·60	7·48
CaO	64·2	0·1	1·16	0·21
MgO	1·48	0	44·11 <sup>a</sup>	0·95
K <sub>2</sub> O	0·67	—	0·28	1·88
Na <sub>2</sub> O	0·052	—	0·42	0·44
TiO <sub>2</sub>	0·27	0·06	0·086	0·84
LOI	3·1	0·1	46·47	0·08

Oxides and LOI are in mass %.

<sup>a</sup> Magnesium carbonate equivalent = 92·3%

Chemical composition of magnesite and fired brick waste were determined by X-ray fluorescence spectroscopy. Chemical composition of OPC and 200G silica were obtained from the suppliers.

**Table 1.** Chemical composition of the raw materials used in this investigation

of the MgCO<sub>3</sub> had been calcined to MgO. The Brunauer–Emmet–Teller surface area of calcined MgO was 104·2 m<sup>2</sup>/g.

- Fired CB waste used was from a major CB manufacturer in Sydney, Australia. The pre-screened brick waste was ground by a dry ball milling method to produce a surface area comparable to that of the quartz sand. Crystalline phase identification of the CB waste was determined using X-ray diffraction (XRD) and these phases are listed in decreasing order of abundance as follows: quartz (SiO<sub>2</sub>); mullite (3Al<sub>2</sub>O<sub>3</sub>.2SiO<sub>2</sub>); hematite (Fe<sub>2</sub>O<sub>3</sub>); cristobalite (SiO<sub>2</sub>); rutile (TiO<sub>2</sub>). From a combination of XRD and X-ray fluorescence it is surmised that in addition to mullite, CB fines contain a quantity of reactive Al<sub>2</sub>O<sub>3</sub> in a disordered glassy state.

### Mix design

Three series of mortar mixtures were prepared using a water-to-total solid ratio (w/s) of 0·35 and a constant total Ca/Si oxide molar atom ratio of 0·41 (based on a 40:60 cement to quartz % mass ratio). A constant Ca/Si oxide ratio was achieved by varying the OPC and quartz sands contents to compensate for CB waste and MgO additions. The first series of mixtures is concerned with the addition of MgO (M series) at 0, 5, 10, 15 and 20 mass % and the second series with the addition of CB waste (C series) at the same amounts. The third series deals with the addition of both MgO and CB waste (CM series) in equal amounts having the total combined amount of 0, 5, 10, 15 and 20 mass %.

### Testing and analysis

Mechanical mixing was conducted in accordance with ASTM C 305-99 (ASTM, 1999) using a Hobart mixer. Pastes were cast into 50 × 50 × 50 mm<sup>3</sup> stainless steel moulds and compacted on a vibratory table, followed by a 24 h curing period in a moist cabinet (temperature 23 ± 1°C, 95% relative humidity as per ASTM 511-03 (ASTM, 2003)). Demoulded specimens were autoclaved for 7·5 h, 6 h of which were at 180°C under approximately 1 MPa of pressure in saturated steam. After autoclaving, specimens were allowed to air dry for 2 days followed by compressive strength testing as per ASTM C109/C (ASTM, 2002) using a Tinius Olsen compression testing machine. Three specimens per mix were loaded under compression until failure. The average compressive strength of all acceptable specimens (in accordance to clause 13.2 in ASTMC109/C (ASTM, 2002)) per mould is reported to the nearest 0·1 MPa. After testing, the specimens were immediately oven dried at approximately 105°C overnight. Small fractured pieces from the core of the cement cubes were ground using a tungsten carbide disc mill for 25 s followed by an additional drying at 105°C overnight. All samples were kept in sealed glass jars and stored in a desiccator over silica gel and soda lime for further characterisation.

XRD analyses were performed using a Siemens D5000 diffractometer operating at 40 kV and 30 mA with CuKα radiation scanning from 3 to 60° 2θ at intervals 0·02° using a step time of 8 s per interval. Peak intensity and integrated areas were determined by the Scherrer method using TOPAS 2·0 with correction

for line broadening using a fundamental parameters approach for X-ray line profile fitting (Cheary and Coelho, 1992). Thermal analyses were performed on 20 mg ± 0·2 mg samples using a TA Instruments' SDT 2960 simultaneous DTA/TGA at a heating rate of 10°C/min under flowing air (20 ml/min) from ambient to 1000°C.

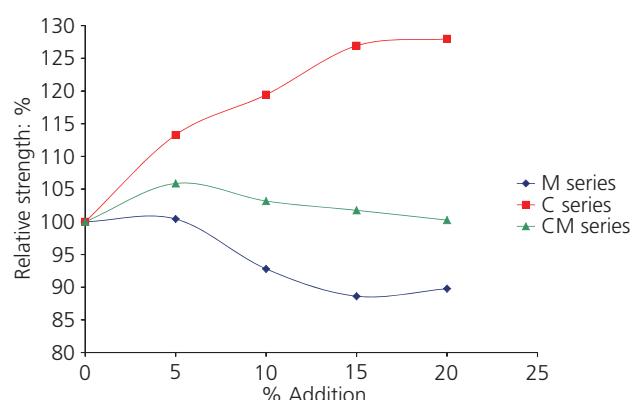
### Results and discussion

#### Compressive strength

The compressive strengths for each mix are listed in Table 2 and the relative strength, relative to the mix with no SCMs added, are plotted in Figure 1 as a function of the addition level. The C series specimens showed substantial strength increase with increasing CB waste additions. The strength gain is relatively linear up to 15% CB waste additions (C15 specimen) and stabilises for the C20 specimens in which the strength gain is approximately 28%. The M series specimens showed a general decrease in strength. The M5 specimens showed strength equivalent to that of the control. The maximum strength reduction is approximately 11% for the M15 specimens, while the M20 specimens showed ~10% reduction in strength. This reduction in strength is markedly less than that in the M20 specimens made with

% Addition	M series: MPa	C series: MPa	CM series: MPa
0	58·6 ± 3·2	58·6 ± 3·2	58·6 ± 3·2
5	58·9 ± 2·5	66·4 ± 3·4	62·1 ± 1·0
10	54·4 ± 1·0	70·0 ± 1·1	60·5 ± 3·2
15	51·9 ± 3·1	74·4 ± 1·7	59·7 ± 3·0
20	52·6 ± 1·4	75·0 ± 1·8	58·8 ± 2·2

**Table 2.** Average compressive strength values for autoclaved blends. The 0% addition (control specimens) values are used as a reference for the normalisation of the data in Figure 1



**Figure 1.** Normalised average compressive strength curves for autoclaved blends. The normalised data are calculated as a percentage relative to the control specimens for a particular autoclave run

relatively more cement-rich environments of Ca/Si = 0.58 and 0.49 (cement/quartz mass ratio of 50:50 and 45:55, respectively) (Liu *et al.*, 2007a, 2007b, 2007c). The strength values of the CM series specimens remain above the control throughout the whole series with ~6% strength gain for the CM5 (2.5% CB waste, 2.5% MgO) specimens.

### Phase analysis

Phase identification using XRD and DTA are summarised in Table 3. XRD results revealed that specimens containing MgO; that is, CM (Figure 2) and M series, showed increases in the amount of brucite with increasing MgO additions. Furthermore, magnesium silicate hydrate (M-S-H) phases were not detected in the MgO containing series. The absence of M-S-H phases indicates that the MgO is not reacting with the quartz or the CB waste under highly alkaline autoclaving conditions. The integrated intensity of the 1.1 nm tobermorite peak is plotted in Figure 3 and is indicative of the proportion of tobermorite present, which, for the M blends, decreases with increasing MgO addition, although this could be attributable to a dilution effect when the amounts of CaO, Al<sub>2</sub>O<sub>3</sub> and SiO<sub>2</sub> are progressively less, resulting in a proportion of tobermorite below that of the limit of detection. The tobermorite peak for the M15 specimens is present in trace amounts and is not detectable for the M20 specimens. This decrease in the proportion of tobermorite present in the M series with increasing MgO content, coupled with the lack of reactivity of the MgO in the production of new M-S-H phases, is likely to be the origin of the reduction in strength of the mortar cubes.

The integrated intensity of the tobermorite peak for the C series showed a significant increase with increasing addition (Figure 3). This observation is in accord with previous findings that CB is

pozzolanic (Connan, 2007; Klimesch and Ray, 2001). The CM series (Figures 2 and 3) showed a relatively stable trend with a relative independence of the proportion of tobermorite produced on the % addition. It is likely that the degree of dilution due to the MgO addition is compensated by the excess formed by the reactivity of the CB waste added. At the equivalent % CB waste addition (Figure 3), the CM series showed a lower degree of Al substitution owing to dilution of the blend by the addition of MgO, furthermore the presence of MgO appears to have chemically inhibited the reaction.

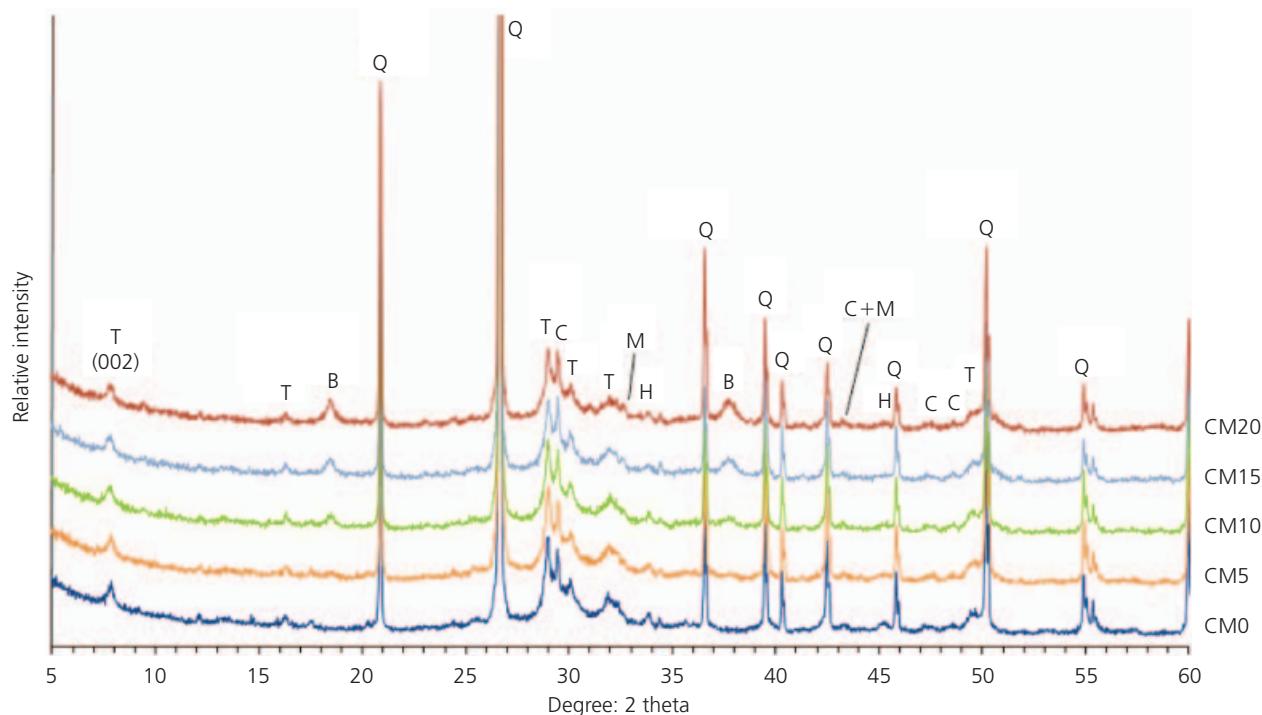
Figure 4 depicts the DTA traces of the CM series specimens while the relationship between the beta-wollastonite ( $\beta$ -CS) exotherm temperature and the total bulk Al<sub>2</sub>O<sub>3</sub> content is shown in Figure 5. The strong endotherm with peak max at approximately 400°C is ascribed to the loss of water from brucite. The strong exotherm observed in the range of approximately 850–900°C is attributable to the crystallisation of  $\beta$ -CS from C-S-Hs including tobermorite. The increase in Al substituted tobermorite with increasing Al<sub>2</sub>O<sub>3</sub> is indicated by the gradual reduction in height of the exothermic  $\beta$ -CS peak accompanied by a progressive shift of this peak towards higher temperatures (Klimesch and Ray, 2002). These observations may be compared to those of Kalousek (1957), who found that with increasing Al content in 1.1 nm tobermorite the  $\beta$ -CS formation temperature increased and the exotherm became flatter and broader. Furthermore, the exotherm is broadened owing to the stacking disorder caused by Al-Si replacement (Hara and Inoue, 1983). The C series showed an increase in the  $\beta$ -CS exotherm temperature with increasing CB waste addition. This is as a result of the overall increase in the amounts of Al<sub>2</sub>O<sub>3</sub> and SiO<sub>2</sub> and hence the Al/(Al + Si) ratio. This trend confirms that the aluminous source, including mullite, in CB is reactive under hydrothermal conditions with the release of

Sample	Al-Tob <sup>a</sup>	Quartz <sup>a,b</sup>	Brucite <sup>a,b</sup>	Magnesite <sup>a,b</sup>	Calcite <sup>a,b</sup>	C-S-H <sup>b</sup>	Hydrogarnet <sup>a</sup>
Control	✓	✓	✗	✗	✓	✓	✓
M5	✓	✓	Trace	Trace	✓	✓	✓
M10	✓	✓	✓	Trace	✓	✓	✓
M15	Trace	✓	✓	Trace	✓	✓	✓
M20	✗	✓	✓	Trace	✓	✓	✓
C5	✓	✓	✗	✗	✓	✓	✓
C10	✓	✓	✗	✗	✓	✓	✓
C15	✓	✓	✗	✗	✓	✓	✓
C20	✓	✓	✗	✗	✓	✓	✓
CM5	✓	✓	Trace	✗	✓	✓	✓
CM10	✓	✓	✓	Trace	✓	✓	✓
CM15	✓	✓	✓	Trace	✓	✓	✓
CM20	✓	✓	✓	Trace	✓	✓	✓

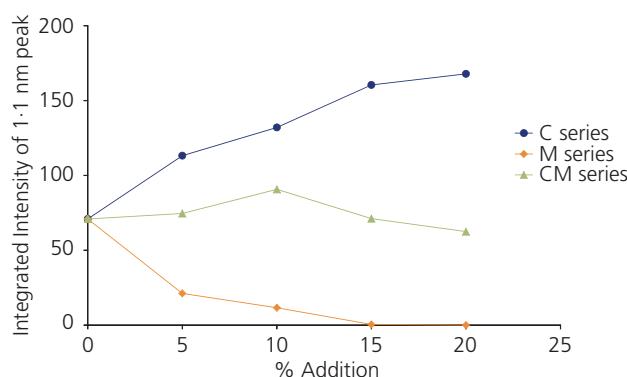
<sup>a</sup> Phase identifications using XRD

<sup>b</sup> Phase identifications using DTA/TGA

**Table 3.** Phases identified by XRD and DTA/TGA



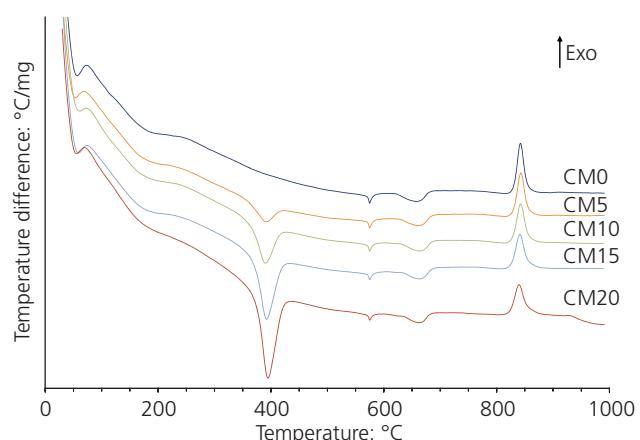
**Figure 2.** XRD patterns of CM series specimens with combined additions at 0, 5, 10, 15 and 20%. T = tobermorite, Q = quartz, B = brucite, C = calcite, M = magnesite, H = hydrogarnet



**Figure 3.** Integrated intensities of 1.1 nm Al-tobermorite peak plotted against percentage addition

Al<sub>2</sub>O<sub>3</sub> and SiO<sub>2</sub>. Moreover, unreacted mullite was not detected by XRD in any specimens with CB waste addition, which further suggests mullite is reactive under autoclaving conditions.

The M series showed a decrease in the exotherm temperature while maintaining relatively constant bulk Al<sub>2</sub>O<sub>3</sub> content (Figure 5). Reductions in Al<sub>2</sub>O<sub>3</sub> content with the replacement of PC is compensated for by the presence of Al<sub>2</sub>O<sub>3</sub> in the calcined reactive MgO (approximately 3.2%). This may again suggest the possible suppression of Al substitution reactions in the formation of Al-tobermorite by MgO. The CM series appeared to be an

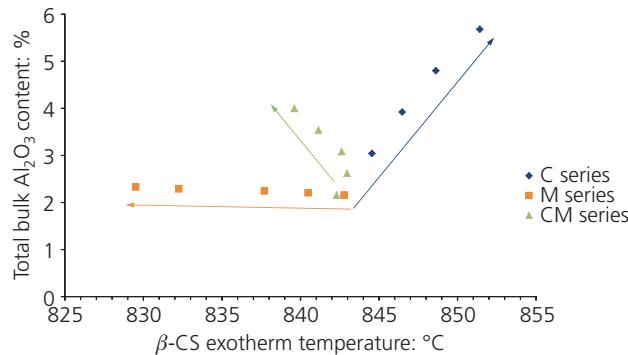


**Figure 4.** DTA curves of CM series specimens at 0, 5, 10, 15 and 20% combined CB fines and reactive MgO additions

intermediate of the C and M series, suggesting that the addition of MgO may suppress the Al substitution reaction of tobermorite chemically and through the effect of dilution.

## Conclusions

Examination of autoclaved PC-quartz mortar mixes revealed that alumina-silica rich CB waste was pozzolanic when the Al<sub>2</sub>O<sub>3</sub> provided a source of Al ions for the accelerated formation and increased crystallinity of Al substituted 1.1 nm tobermorite.



**Figure 5.** Total bulk Al<sub>2</sub>O<sub>3</sub> content in the initial raw mix plotted against the variation in the β-CS formation temperature (arrows depict the direction of increasing percentage addition)

The C series specimens showed substantial strength gain of approximately 28% for the C20 specimens. The strengths of the CM series specimens were maintained above the control throughout the whole series. The addition of reactive MgO to PC had a negative effect on the compressive strength. This was attributable to a relative decrease in the proportion of PC available for the formation of the strength contributing hydration products. The brucite phase was observed from XRD as a separate phase, indicating that MgO did not take part in the structure of the lime-based hydration products. XRD and DTA data suggested that there was, however, a chemical influence on the tobermorite formation and that MgO was not inert and might play a role in the possible retardation of the reaction.

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