

# Effect of high temperature on mechanical and physical properties of lightweight cement based refractory including expanded vermiculite

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Four different composite mixtures with varying amounts of expanded vermiculite were exposed to high temperatures of 300, 600, 900 and 1100°C for 6 h. Physical and mechanical properties including unit weight, porosity, water absorption, residual compressive strength, residual splitting tensile strength and also ultrasonic pulse velocity were determined after air cooling. Microstructures were investigated by scanning electron microscopy. Lightweight concrete with vermiculite shows a good performance at elevated temperatures. Expanded vermiculite is a significant lightweight aggregate for cementitious materials which are used for fire resistance applications. Concrete with vermiculite can be used as cement based refractory.

**Keywords:** Expanded vermiculite, Lightweight concrete, Cement based refractory

## Introduction

Concrete generally provides adequate fire resistance for most applications. However, the strength and durability properties of concrete are significantly affected when subjected to elevated temperatures due to chemical and physical changes.<sup>1–5</sup> At temperatures above 300°C, evaporation of the bound water increases the deterioration of concrete and causes a decrease in compressive strength.<sup>2</sup> When temperatures exceed 400°C, calcium silica hydrates (C–S–H) undergo degradation, and the strength of concrete decreases rapidly; at ~900°C, the structure of C–S–H disintegrates.<sup>3,4</sup> Thus, the critical exposure temperature range is 400–800°C in terms of compressive strength loss. Most of the original strength is lost between 600 and 800°C.<sup>6</sup> The effect of high temperatures on the mechanical properties and durability of concrete has been investigated extensively in order to produce fire resistant materials.<sup>7–9</sup> Important factors here are properties of the aggregate, cement paste and aggregate–cement paste bonding, their relative compatibility, the presence of supplementary cementitious materials, high temperature conditioning affected by heating rates, cooling type, loading conditions and moisture regime.<sup>10–34</sup>

Within the factors affecting the high temperature resistance of concrete, the properties of aggregate play an important role in the degradation processes at high temperatures. Porosity and mineralogy of the aggregate have also significant effects. Mineralogy of aggregate determines differences in thermal expansivities  $\alpha$  between the aggregate and the cement paste, and thus bond strength at the interface.<sup>3</sup> Neville<sup>35</sup> stated that at temperatures approximately above 430°C, concretes with siliceous aggregates show significant strength loss when compared with those with lightweight aggregates. At 600°C, concrete can lose half of its strength. As already noted, above 800°C, the loss of strength reaches up to 80% as a consequence of the loss of the bound water in the hydrates. The difference between the strength loss in normal weight concretes and lightweight concretes becomes insignificant above 800°C. Lightweight concretes can easily be produced by utilising lightweight aggregate<sup>36–38</sup> and are generally classified into two types as natural (pumice, diatomite, volcanic cinders, etc.) and artificial (perlite, expanded vermiculite, expanded shale, clay, slate, sintered pulverised fuel ash, etc). Lightweight aggregates have inherent high resistance to fire since these aggregates were exposed to high temperatures during their formation and production process. Since such aggregates have low  $\alpha$  values and more thermal stability at elevated temperatures, lightweight concretes made from them have a better fire resistance than ordinary concrete.<sup>39</sup> Nekrasov and Tarasova<sup>40</sup> showed that concrete produced using burned chamotte as a light aggregate did not suffer from fire exposure up to 800°C. Turker *et al.*<sup>39</sup> reported that pumice aggregate mortar subjected to high temperatures up to 500°C does not show compressive strength loss and is more resistant to high temperatures

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than quartzite or limestone. Similar results were reported by Kong *et al.*<sup>41</sup> and Abeles and Bardhan-Roy;<sup>42</sup> they stated that concretes containing lightweight aggregate preserve their strength up to 500°C. Yazıcı *et al.*<sup>43</sup> pointed out that pumice aggregate mortar has a compressive strength value of 41% at 600°C, while conventional natural river sand mortar lost 39% of their strength, and the interface was strong when pumice was used. Poon *et al.*<sup>9,18</sup> reported that high strength concrete specimens show low permeability at 20°C. However, as the temperature was increased, impermeability is affected significantly at 800°C, probably due to internal cracking and pore structure coarsening.<sup>22</sup> Janotka and Bagel<sup>44</sup> reported that the permeability of concretes increases drastically with the increase in temperature. Hammer *et al.*,<sup>45</sup> Justnes and Hansen<sup>46</sup> and Gerard<sup>47</sup> pointed out three factors that cause spalling in lightweight aggregate concrete:

- (i) vapour pressure dependence on the moisture content and the permeability
- (ii) moisture clogging of capillary pores
- (iii) initial compressive stresses in the exposed layer.

Justnes and Hansen<sup>46</sup> noted that the impregnation of lightweight aggregates for preventing water absorption (WA) during storage or fresh concrete stage reduces the moisture content of hardened concrete and prevents spalling in lightweight aggregate concrete when exposed to high temperatures.

Vermiculites are naturally occurring minerals composed of shiny flakes, resembling mica in appearance. They are primarily formed by alteration of micaceous minerals as a result of weathering, hydrothermal action, percolating ground water or a combination of these three factors.<sup>47</sup> When heated to elevated temperatures, flakes of vermiculite expand as much as 8–30 times with respect to their original size due to the conversion of interlayer and structural water to steam.<sup>47</sup> Expanded vermiculite after cooling preserves its new volume with very thin streaks of air between the leaves. The particles of expanded vermiculite are viewed as thin plates separated by air gaps. Their shape, colour, luster and grain composition are closely related to the original raw material. The WA capacity of vermiculite increases drastically when the bulk density varies between 64 and 160 kg m<sup>-3</sup>, depending on particle size, after exfoliation. As a result, the annealed vermiculite possesses several valuable properties, such as low thermal conductivity, high fire resistance and strong sound absorption. Wetting of vermiculite by molten metals is

**Table 1** Chemical and physical properties of cement

Chemical properties	
Composition	%
SiO <sub>2</sub>	19.43
Al <sub>2</sub> O <sub>3</sub>	5.60
Fe <sub>2</sub> O <sub>3</sub>	2.44
CaO	63.01
MgO	2.59
SO <sub>3</sub>	2.90
Na <sub>2</sub> O	...
K <sub>2</sub> O	1.00
Cl	...
Insoluble material	3.29
Loss on ignition	3.30
Physical properties	
Specific gravity	3.06
Specific surface/m <sup>2</sup> kg <sup>-1</sup>	394

poor; hence, vermiculite is an effective high temperature (up to 1100°C) heat isolation material. Materials and products produced using vermiculite are incombustible, biostable and neutral to the action of acids and have stable strength with time and resistance to deformation. These facts make worthwhile the application of vermiculite in construction and simultaneously heat isolation and sound absorption materials. Using effective high temperature and thermal isolation materials allows to reduce the material capacity of the constructions. There are numerous heat resistant aggregates in different industry fields. Exfoliated vermiculite is a low density material and utilised as a constituent of lightweight concretes and plasters, a filler for plastics, paints and fertilizers and a soil modifier.<sup>48</sup>

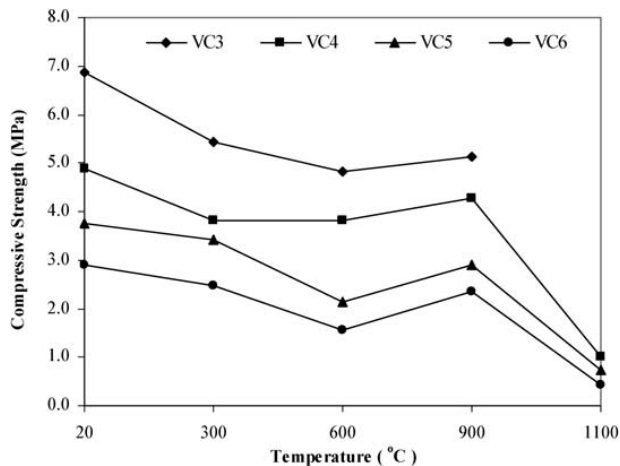
In this situation, we have decided to prepare lightweight concretes containing expanded vermiculite and to examine their residual compressive strength and splitting tensile strength at elevated temperatures. Temperatures of 20, 300, 600, 900 and 1100°C were chosen. The specimens of all series were exposed to the same temperatures. Compressive strength and splitting tensile strength of low strength lightweight concrete, which were exposed to high temperatures and cooled in air, were compared with each other and then compared with the samples that were not heated.

## Materials and experimentation

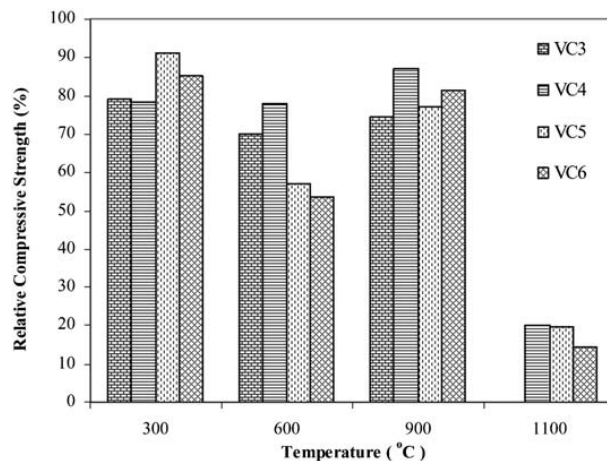
A CEM I 42.5R Portland cement produced by Cimpor Yibitaş Cement Plant, Yozgat, Turkey, was used. The chemical and physical properties of that cement are given in Table 1. Raw vermiculite was procured from the Demircilik vermiculite deposit in Yıldızeli, Sivas, Turkey. Expanded vermiculite was obtained by annealing raw vermiculite at ~600°C for 10 s. The chemical and physical properties of expanded vermiculite are presented in Table 2.

**Table 2** Properties of expanded vermiculite

Chemical properties	
Compositions	%
SiO <sub>2</sub>	34.1
Al <sub>2</sub> O <sub>3</sub>	17.2
K <sub>2</sub> O	4.52
CaO	6.4
MgO	16.3
Fe <sub>2</sub> O <sub>3</sub>	14.7
pH (in water)	6.1
Others	0.68
Physical properties	
Colour	Silver
Shape	Accordion shaped granule
Water holding capacity	240 wt-%
Cation exchange capacity	90 meg/100 g.
Thermal conductivity value	0.063 W m <sup>-1</sup> °C <sup>-1</sup>
Sintering temperature	1170°C
Combustibility	Non-combustible
Specific heat	0.92 kJ kg <sup>-1</sup> °C <sup>-1</sup>
Bulk density	140 kg m <sup>-3</sup>
Particle size distribution	
Sieve size/mm	Passing/%
8	100.0
4	53.3
2	14.1
1	3.8
0.5	2.4
0.25	1.7
0.125	1.0



1 Residual compressive strengths at several temperatures



2 Relative residual compressive strengths after heating (compared with strength at 20°C)

Cement and water were first put in the mixer and mixed for 1 min, and thus cement slurry was obtained. Expanded vermiculite was then added to the slurry and mixed for 3 min in order to achieve a homogenous structure. Expanded vermiculite aggregate was used dry, and trial batches were produced to determine the water content of each mix at which the slump would be zero. The expanded vermiculites/binder ratios were 3, 4, 5 and 6 by volume. For each test temperature, five series of specimens were produced, and thus, in total, 25 concrete series were investigated. The mix proportions of series are given in Table 3. For mix code of series, VC3, VC4, VC5 and VC6 refer to vermiculite/cement ratios of 3, 4, 5 and 6 by volume respectively.

Therefore, the prepared fresh concretes were cast in standard cylindrical moulds, 150 mm in diameter and 300 mm in height, in two layers, with each layer compacted by self-weight in a shaker for 20 s. All the specimens kept in moulds for 24 h at room temperature of ~20°C were demoulded and then cured in water at 23±2°C for 27 days. Unit weights (UWs), apparent porosity (AP) and WA were calculated for the specimens at the end of 28 days according to Archimedes' principle.

The compressive strength, splitting tensile strength and ultrasonic velocity (UV) tests were carried out on three specimens; the average values are reported below. Strength tests were made in accordance with European Standards.<sup>49,50</sup> Fractured surfaces of specimens exposed to predefined test temperatures were coated with gold in a vacuum evaporator and then examined using a scanning electron microscope to determine the morphological and mineralogical features. It is emphasised that the specimens were not dried out before testing. An electrically heated furnace, operating up to 1300°C, was used. Cylinders from each mix were first placed in an

Table 3 Mix proportions of concrete series

Mix code	Vermiculite/cement (by volume)	Cement/kg m <sup>-3</sup>	Vermiculite/kg m <sup>-3</sup>	Water/kg m <sup>-3</sup>
VC3	3	750	149	471
VC4	4	636	180	541
VC5	5	522	187	566
VC6	6	422	192	580

oven and heated from room temperature (20°C) to the target temperature at the rate of 5°C min<sup>-1</sup>. The specimens of each mix were then held at the desired test temperature for ~6 h; then, the furnace was turned off, and the specimens were left to cool in air until reaching room temperature. During the heating period, moisture in the test specimens was allowed to escape freely. The test results of specimens exposed to high temperatures were compared with those for unheated specimens.

### Physical properties

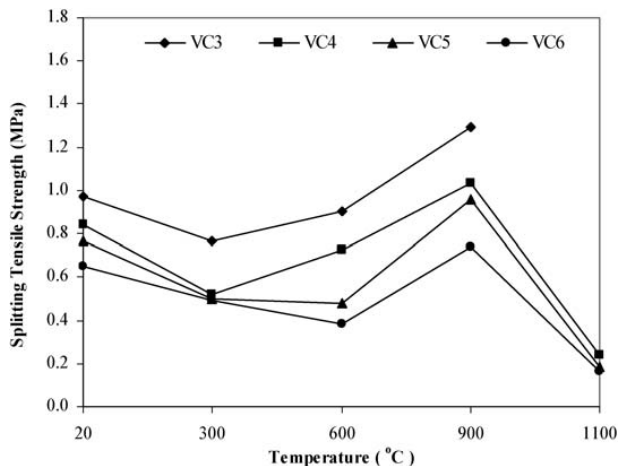
Some physical properties of unheated specimens after 28 days of curing, such as UW, AP and WA, are presented in Table 4. While UW was decreasing, it can be seen that AP and WA decreased with an increase in vermiculate/cement ratio. The UW ranged from 1.08 × 10<sup>3</sup> to 1.33 × 10<sup>3</sup> kg m<sup>-3</sup>. The AP changed between 30 and 37%, while WA varied between 24 and 35%.

### Residual strengths: compressive and splitting

For all the series, the compressive strengths of specimens decreased when increasing the heating temperature up to 600°C. At 900°C, a strength recovery was observed, and an increase is seen in the compressive strength values. This increment in strength may be due to filling or closing microcracks and voids by expansion of vermiculite at 900°C. Finally, a significant decrease in compressive strength is seen at the heating temperature of 1100°C for all series. Similar results were obtained in splitting tensile strength determination. Given the failure of specimens subjected to 1100°C with a high cement content or low vermiculite/cement ratio, the residual strengths were not determined. Graphical

Table 4 Test results of UW, AP and WA

Mix code	Vermiculite/cement (by volume)	UW/kg m <sup>-3</sup>	AP/%	WA/%
VC3	3	1332	29.9	24.4
VC4	4	1183	35.8	30.7
VC5	5	1116	36.2	31.1
VC6	6	1078	37.4	34.6

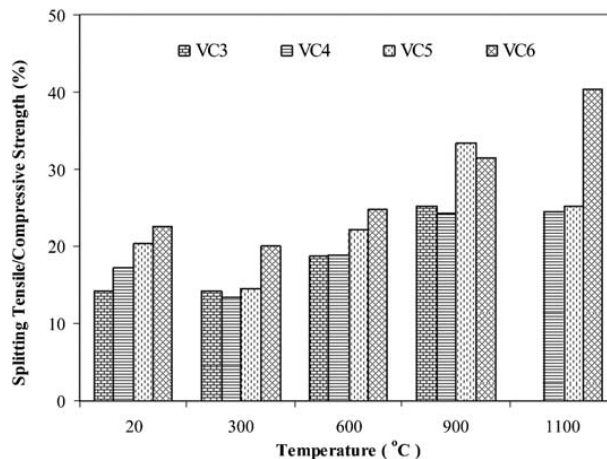


3 Residual splitting tensile strengths at several temperatures

representations of residual strengths are provided, as seen in Figs. 1–4.

We find that lightweight concrete produced using expanded vermiculite aggregate shows a good performance in terms of preservation of mechanical strength up to the temperature of 900°C. Thus, 900°C can be regarded as a critical temperature for strength loss of concrete. Ratios of the residual splitting tensile strength to compressive strength at different temperatures for all the concretes are presented in Fig. 5. It can be seen that these ratios are lower for the heating temperature of 300°C than for the room temperature. However, those ratios increase for temperatures of 600, 900 and 1100°C for all vermiculite/cement ratios. Expanded vermiculite thus turns out to be a good fire resistant material; it shows its effect on residual strengths by keeping the initial strengths of cementitious materials up to the temperature of 900°C.

For the prediction of residual strengths depending on the vermiculite/cement ratio  $V/C$  and heating temperature  $T$ , experimental data obtained were fitted to a polynomial using an analysis of variance commercially available (Design-Expert) software package. For the compressive strength  $f_c$  and splitting tensile strength  $f_{st}$ , the resulting regression models are respectively



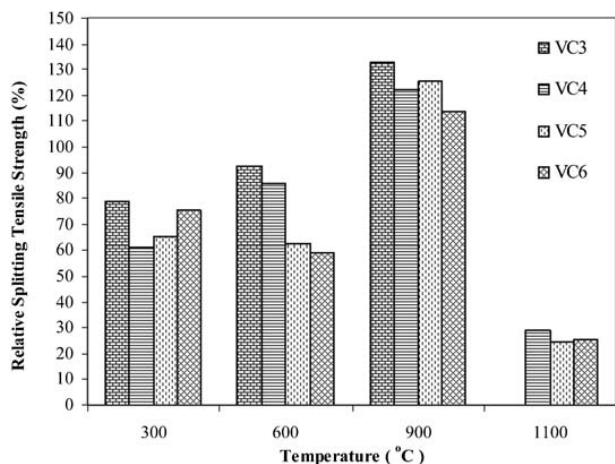
5 Ratio of splitting tensile strength to compressive strength of concretes at several temperatures

$$f_c = 14.359 - 3.077(V/C) - 0.012(T) + 0.204(V/C)^2 + 2.206 \times 10^{-5}(T)^2 + 3.739 \times 10^{-4}(V/C)(T) - 1.422 \times 10^{-8}(T)^3 \quad (2)$$

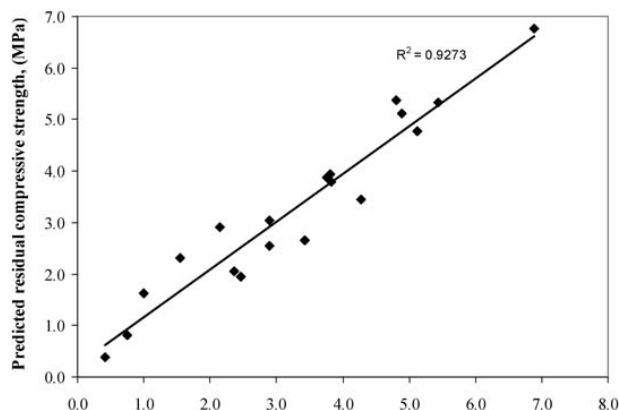
$$f_{st}^{-1} = 0.35 - 0.12(V/C) - 0.011(T) + 1.166 \times 10^{-3}(V/C)^2 + 3.492 \times 10^{-5}(T)^2 + 4.272 \times 10^{-4}(V/C)(T) - 2.40 \times 10^{-8}(T)^3 \quad (3)$$

The values of  $R^2$  (coefficient of determination) were 0.927 and 0.879 for equations (2) and (3) respectively.  $R^2$  is the correlation measure for testing the goodness of fit of the regression equation. A perfect agreement between the observed and predicted values would result in  $R^2=1$ . Comparisons between model predictions and experimental results for compressive and splitting tensile strengths are shown in Figs. 6 and 7 respectively.

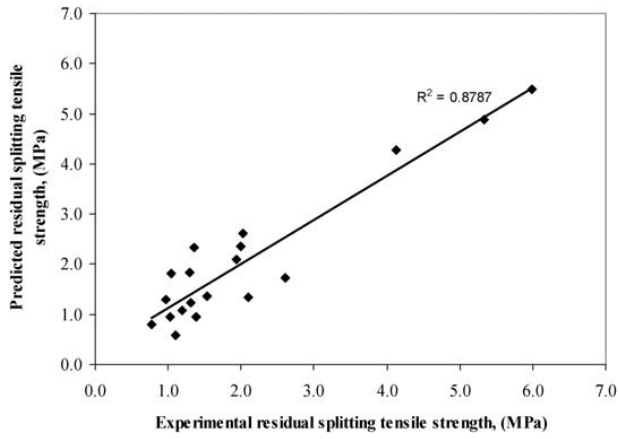
A tridimensional response surface is the graphical representation of a regression equation. It provides the visualisation of relationship between responses and experimental levels of each independent variable. The relationships between independent and dependent variables are shown as a three-dimensional representation of the response surfaces for compressive and splitting tensile strengths in Figs. 8 and 9 respectively. It can be



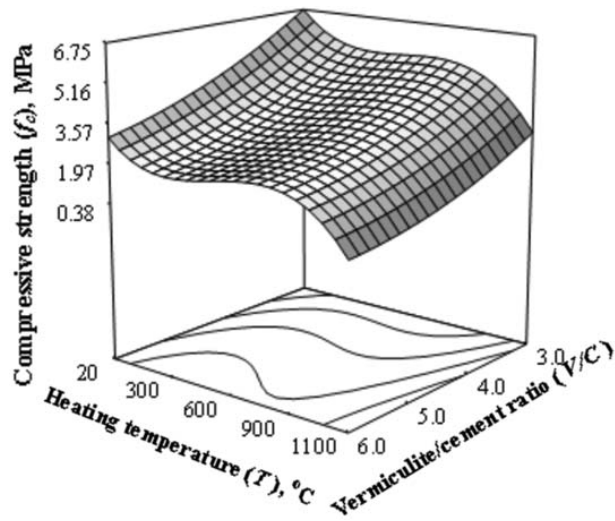
4 Relative residual splitting tensile strengths after heating (compared with strength at 20°C)



6 Relation of experimental and predicted residual compressive strengths

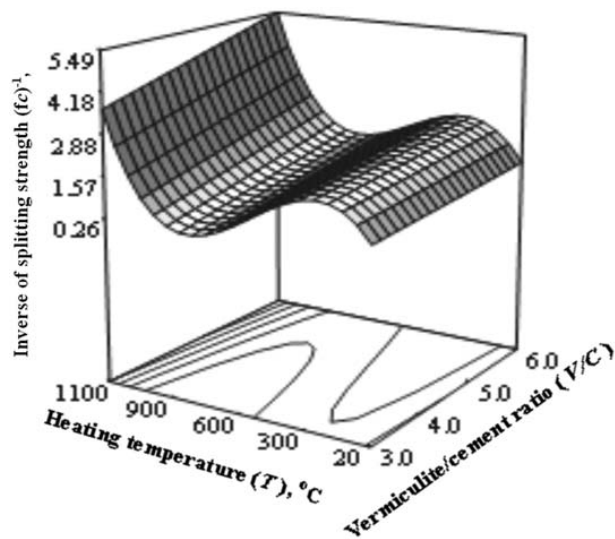


7 Relation of experimental and predicted residual splitting tensile strengths

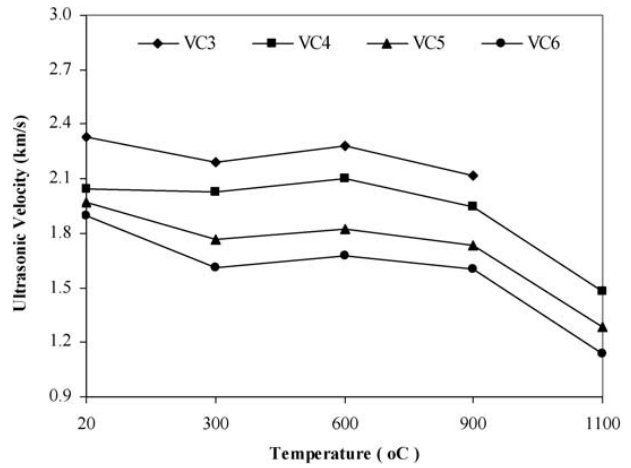


8 Response surface of residual compressive strength

found that the vermiculite/cement ratio and the heating temperature have significant influence on both residual compressive and splitting tensile strengths of lightweight concrete.



9 Response surface of residual splitting tensile strength



10 Residual UV at several temperatures

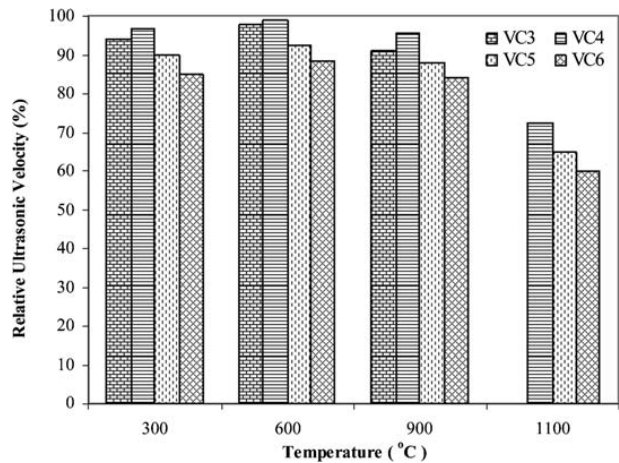
### Ultrasonic velocity

The initial pulse velocity values of concrete at 20°C and residual ultrasonic velocities after heating at our test temperatures are graphically presented in Figs. 10 and 11.

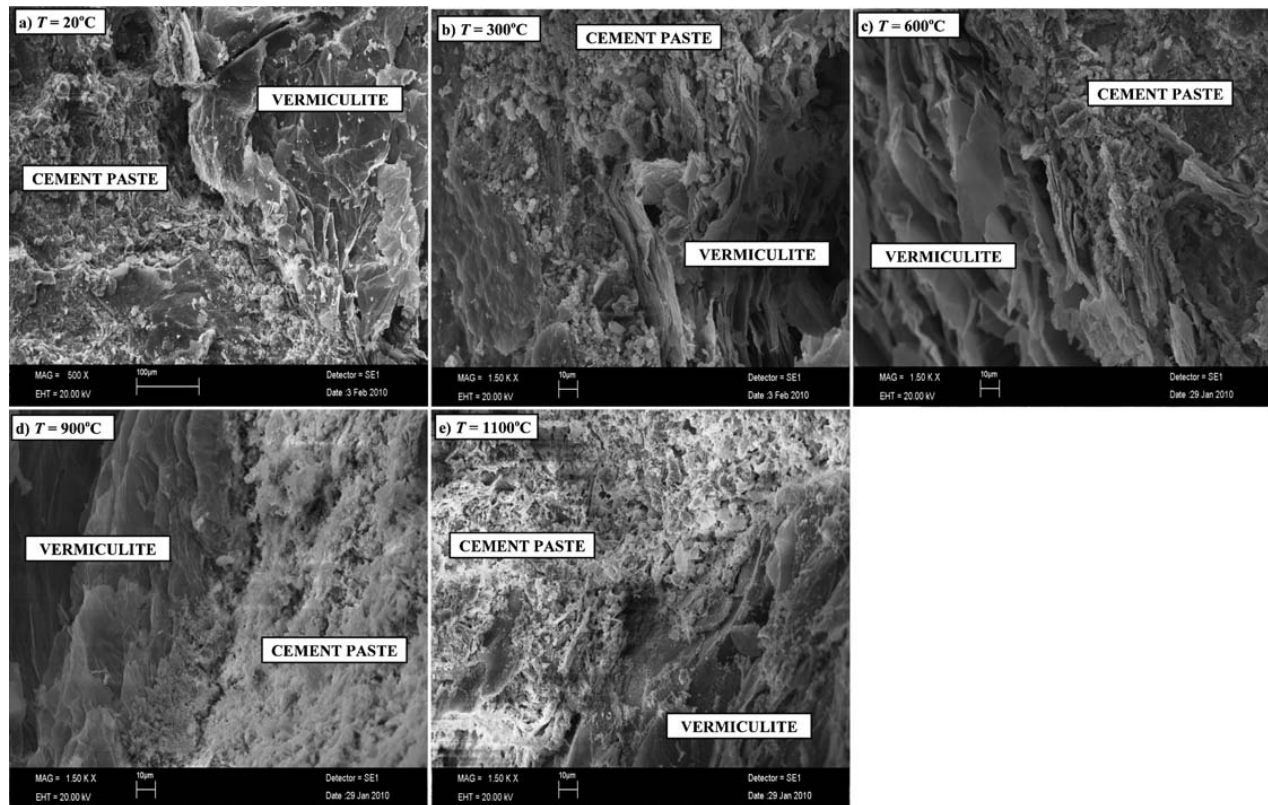
It can be found that UV drops as the ratio of expanded vermiculite to cement increases. This is the result of the high pore structure of expanded vermiculite. The UV is only slightly influenced by heating temperature up to 900°C. However, a significant reduction in UV is seen at 1100°C for all the mixtures. It is noted that a UV with increasing temperature is a sensitive measure of the progress of cracking in a material. Thus, degradation in microstructure of specimens subjected to heating temperature not higher than 900°C is insignificant.

### Microstructure investigation

Scanning electron microscopy investigations were performed on air cooled specimens of all the mixtures for each testing temperature. The results are presented in Fig. 12. It can be seen that a deterioration in microstructure starts at the heating temperature of 300°C. However, there is no significant additional damage in the matrix phase and bonding between cement paste and aggregate up to 900°C, although the void ratio in



11 Relative residual UV after heating (compared with UV at 20°C)



a  $T=20^{\circ}\text{C}$ ; b  $T=300^{\circ}\text{C}$ ; c  $T=600^{\circ}\text{C}$ ; d  $T=900^{\circ}\text{C}$ ; e  $T=1100^{\circ}\text{C}$

## 12 Scanning electron microscopy analysis

microstructure resulting from the expansion of vermiculite has increased. This can be explained by the temperature resistance of vermiculite and the strong bonding between cement paste and aggregate by resulting in differential thermal expansions between the aggregate and the cement paste. At  $1100^{\circ}\text{C}$ , it is observed that the structure of C–S–H disintegrated, a consequence of the disappearance of bond strength at the interface. A significant decrease in compressive and splitting tensile strengths is probably due to the increase in internal cracking and pore structure coarsening at  $1100^{\circ}\text{C}$ .

## Survey of results

The effects of high temperature and inclusion of vermiculite on the properties of lightweight concrete are summarised below.

The UW changed between  $1.08 \times 10^3$  and  $1.33 \times 10^3 \text{ kg m}^{-3}$  for specimens aged for 28 days. The weight values decrease as a function of increasing vermiculite volume fraction in the mix. The specific porosity changed between  $\sim 37$  and  $30\%$ , while WA changed between  $35$  and  $24\%$  for specimens aged for 28 days.

The expanded vermiculite shows a good performance as a lightweight aggregate. The critical exposure temperature for loss of strength is above  $900^{\circ}\text{C}$ . At the heating temperature of  $1100^{\circ}\text{C}$ , the loss in strength changes between  $73$  and  $100\%$  for compressive strength and between  $71$  and  $100\%$  for splitting tensile strength. In the microstructure of concrete with a high vermiculite/cement ratio, the degradation is more pronounced, and a loss in strength is higher with respect to the initial strength values.

It is noticed that the changes in UV values between  $20$  and  $900^{\circ}\text{C}$  are not large. However, a certain reduction in ultrasonic pulse velocity values above  $900^{\circ}\text{C}$  (at  $1100^{\circ}\text{C}$ ) indicates that the physical state of the concrete samples deteriorated rapidly beyond  $900^{\circ}\text{C}$ .

Expanded vermiculite enhances significantly the high temperature stability of concretes and can be a preferable material for the manufacturing of cement based refractory and heat insulation materials as an alternative to other lightweight aggregates. The performance or thermal resistance of lightweight concrete prepared with expanded vermiculite can be improved using high temperature resistant cements as binder.

Based on our earlier investigations,<sup>51–54</sup> future work is planned using fibres with admixtures (mineral and chemical) and possibly use of two different fibre kinds incorporated in the mixes in order to increase the mechanical strength of concrete containing vermiculite.

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