

Microstructural changes in Concrete at Elevated Temperatures- A Review

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Abstract— *Microstructure of concrete plays most significant role in strength and durability of structure under normal circumstances and during fire exposure. When concrete is exposed to higher temperatures, it undergoes several microstructural changes that affects its durability and strength. The fire resistance of concrete depends on type of aggregate used, porosity, size of structural member etc. This paper discusses in detail the influence of these factors on concrete at elevated temperatures. Furthermore, influence of vermiculite on the fire resistance properties has been discussed.*

Keywords— *Concrete microstructure, Concrete behaviour, high temperature, fire exposure, vermiculite*

I. INTRODUCTION

Concrete offers good resistance to fire. However due to its composite nature, concrete suffers a certain degree of loss in strength. This loss can be accredited to the different thermal characteristics of its constituents. Also, the fire resistance of concrete depends on type of aggregate used, porosity, size of structural member etc. [1-2]. When concrete is exposed to high temperature, evaporation of free water takes place, followed by the evaporation of chemically bound water with further increase in temperature. At higher temperatures, aggregates begin to expand along with the shrinkage of cement due to difference in coefficient of thermal expansion between cement and aggregates. This induces thermal stresses in concrete which results in breakdown of interfacial bond between aggregate and surrounding cement paste [3-5]. Hence, when concrete is exposed to elevated temperature severe microstructural changes occur which affect the durability and strength of concrete. Therefore, higher temperature causes the physical deterioration, not only of cement but also of aggregates. To understand the effect of higher temperature on cement and aggregates, micro structural study is to be done. Significant number of researches have already been done in the field of microstructure of concrete at higher temperatures. Various methods are available to study the micro structural changes which occur in concrete at elevated temperatures such as Plane polarized transmitted light (PPTL) method, scanning electron-microscopy (SEM).

II. EFFECT OF ADHERENCE OF AGGREGATE- CEMENT MATRIX AT ELEVATED TEMPERATURES (PPTL METHOD)

An In PPTL method, concrete cube specimen is divided in thin sections. The use of these thin sections makes microscopical observation to a more accurate when light passes through them. K. Akcaozoglu has significant contribution in the field of micro structural changes in concrete when exposed to high temperature using PPTL method [6]. The PPTL method consists of preparation of two Cubic specimens of 71 X 71 X 71 mm for each temperature range, one for fast cooling (FC) process and other for slow cooling (SC) process and 5 thin sections were cut from each of the specimen of thickness 14 mm as shown in fig. 1. Out of these 5 thin sections, section 3 was taken into consideration for microstructural study as this section has least edge effects. For more accurate evaluation of the micro structural changes, thin sections were divided into three zone namely A, B and C (Fig. 2).

When thin sections were scrutinized at room temperature (22°C), no significant changes were observed in the aggregate-cement matrix. No cracks, fissures were observed. Similar types of results occurred when the thin section was exposed to 200°C and then was slowly cooled. In fact, the aggregate and cement matrix were adhered fairly well in SC thin sections. The microstructure was also found similar to the normal one. However, the effect was not same in FC thin sections when exposed to a temperature of 200°C [7]. The adherence of cement and aggregate matrix was clearly seen affected due to FC. Cracks and fissures were also observed in the cement-aggregate matrix interface. The reason of these formation of cracks is instantaneous temperature change in FC process which generated the stresses and adversely affected the adherence of cement- aggregate matrix. Also in various researches, it has been seen that adherence between cement and aggregate decreases with increase in temperature [6]. This decrement amount can be small or large depending upon the conditions like FC or SC processes. At 400°C, thin film layer which was observed in the SC thin section specimens when exposed to lower temperature became thicker, however this did not enhance their adherence.

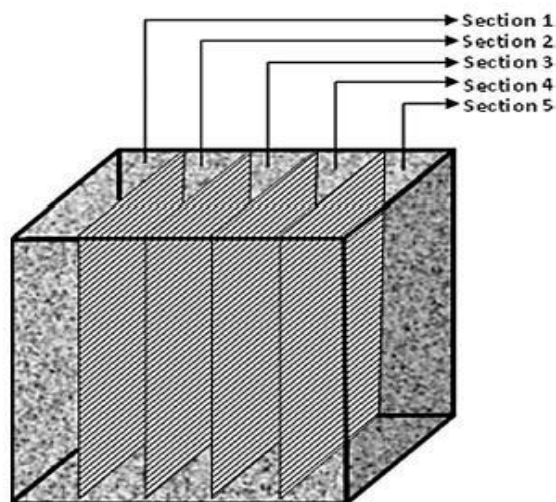


Fig. 1 Preparation of specimens of thin sections [6]

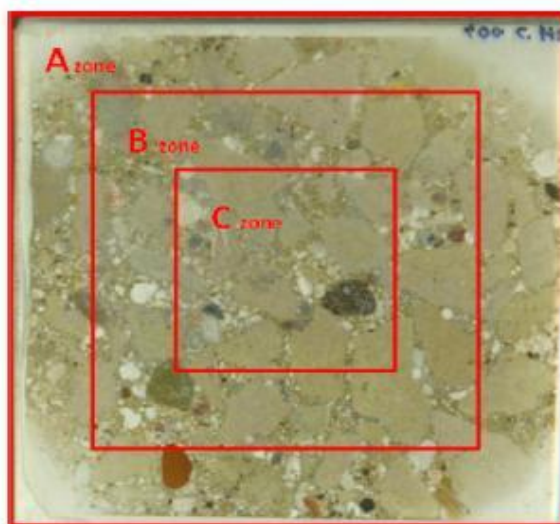
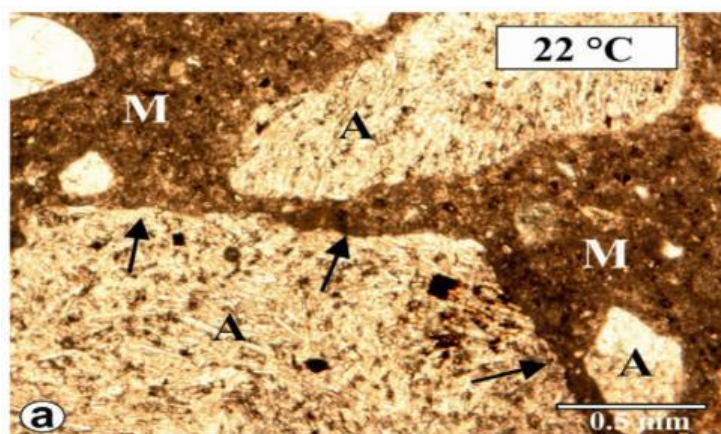


Fig. 2 Zone generation in the specimens [6]

Instead, these thick layers were not able to wrap the aggregates around completely like at lower temperatures. Similar effects were seen taking place in thin section FC specimens exposed to the 400°C. The layer became thicker than SC specimens hence making the wrapping of aggregate more poor than the SC specimen. On further increasing temperature to 600°C, the adherence of the aggregate–cement matrix interfaces became more poor as on increase in temperature the thicker layers were more expanding along with increase in their length, hence causing more discontinuity. At 600°C, almost all the interfaces became discontinuous. These results clearly states that the adherence of aggregate-cement matrix not only depends on the elevated temperature but it also depends on the cooling process [6].



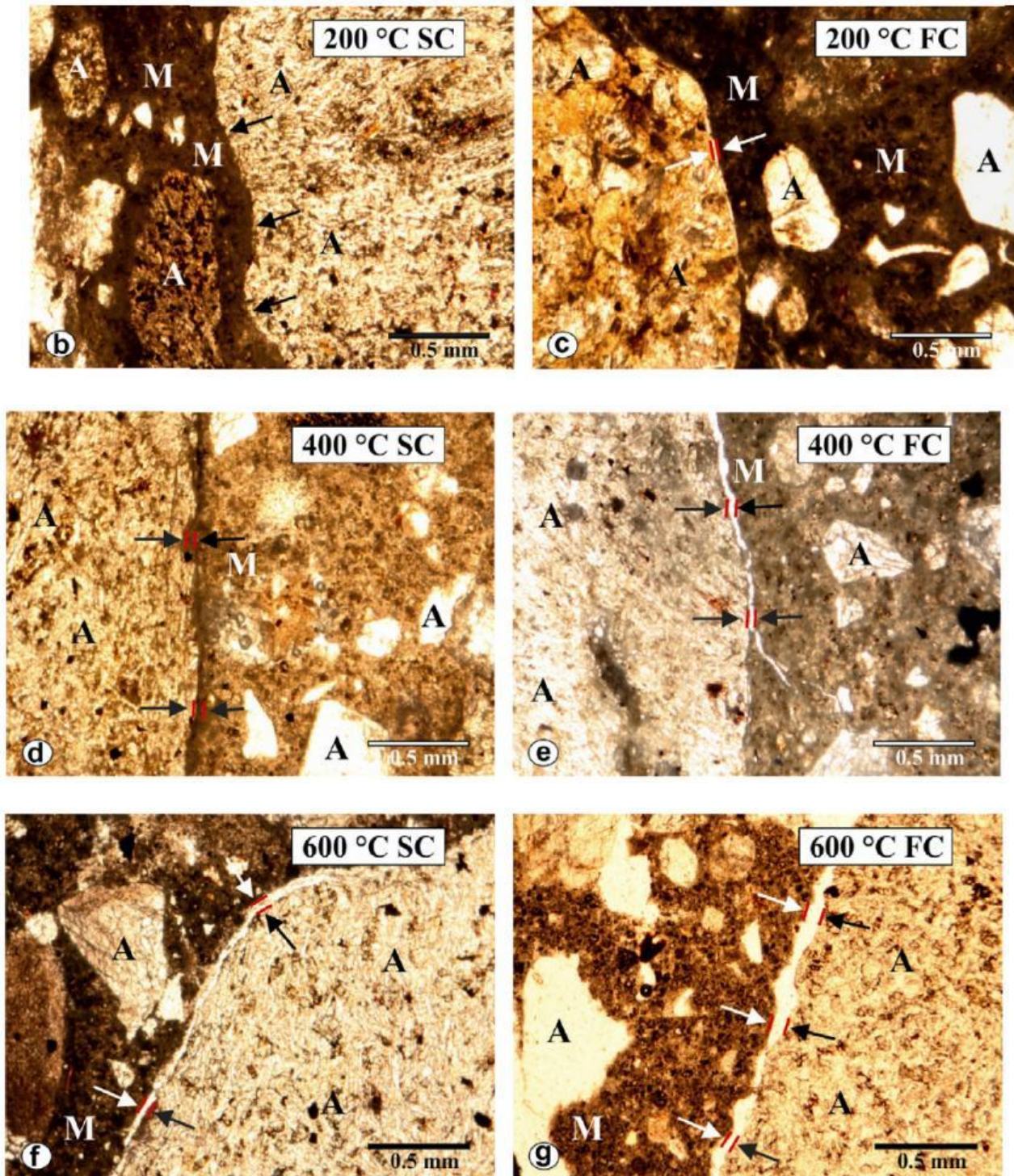


Fig. 3 The effect of elevated temperature on adherence of aggregate- cement matrix (PPTL Method, A: Aggregate, M: matrix) [6]

III. EFFECT OF ELEVATED TEMPERATURES ON THE STRUCTURE OF AGGREGATES (PPTL METHOD)

When the concrete specimen exposed to a temperature 22°C was inspected by PPTL method, some cracks were observed at the surface of the quartz aggregate. However the temperature effect on aggregate is not same for all types. This strength degradation is less in Basalt aggregate due to its high thermal stability [8]. Cracks, fissures and breakdowns in the quartz aggregates steadily increased depending on exposing to elevated temperatures. On increase in temperature, the strength was seen degrading irrespective of the type of aggregate. The only difference was in the extent of degradation. Practically on increasing temperature to 600°C, all of the quartz aggregates in the Fast cooled specimen were disintegrated, because quartz aggregates changes its constitution discretely at 570°C with a volume expansion and

subsequent damage [9]. The above results clearly states that the quartz aggregate was more affected from the elevated temperature and cooling process than the basalt aggregate [6].

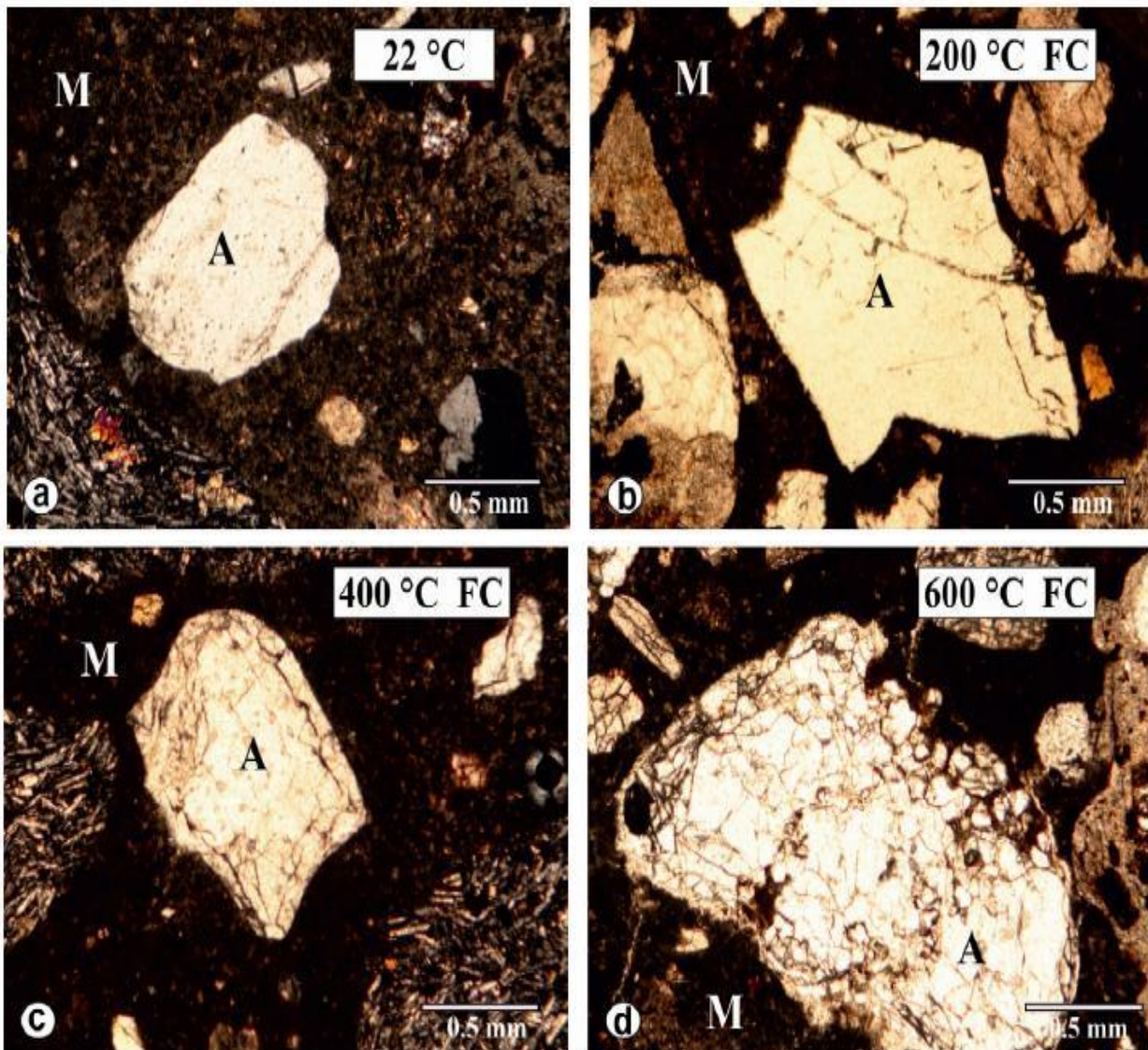


Fig. 4 Effect of elevated temperatures on the structure of aggregates (PPTL method) [6]

IV. EFFECT OF ELEVATED TEMPERATURES ON THE MICROSTRUCTURE OF CONCRETE (SEM METHOD)

Scanning electron microscope (SEM) method is another method of analyzing micro-structural changes occurring in concrete at elevated temperatures. Demirel and Kelestemur [10] has significant contribution in studying microstructural changes using SEM method. When concrete is exposed to high temperatures, it affects the microstructure, and undergoes some changes as shown in Fig.3-a. Proper inter-mixing of $\text{Ca}(\text{OH})_2$ crystals with C-S-H results in increasing voids to well hydrated phase initially (Mendes et al, 2012). With increase in temperature, formation of micro-cracks starts developing due to the deformation action of $\text{Ca}(\text{OH})_2$ crystals. On increase in temperature, the formation of micro-cracks becomes predominant due to disruption of C-S-H phase boundaries and due to formation of CaCO_3 (Fig. 3-b). On further rise in temperature, micro-cracks start to expand along with increase in their dimension-length (Fig.3-c). This expansion of micro-cracks was found to be important factor when micro-cracks get intermingled with the voids at relatively high temperature of range 600 to 800 °C. The intermingle relationship of micro-cracks with the voids is based on porosity of the concrete and concrete mix characteristics. For that reason, several research studies evidently show that on increase in temperature, concrete loses its strength due to loss of water at high temperature along with increase in porosity at elevated temperatures (Mendes et al., 2012). [10].

The above micro-structural studies of concrete exposed to elevated temperatures have clearly revealed that on increase in temperature, strength degradation of concrete takes place. Many studies conducted on concrete determined increase in strength of concrete on addition to mineral additives. The accuracy of these results will be ascertained only when the addition of mineral additives in concrete formation will also be increasing the strength at elevated temperatures. Numerous studies [11 -16] are performed on micro-structural changes in concrete formed with mineral additives like silica fumes, fly ash, blast furnace slag etc. at elevated temperatures and the results of those are presented in brief.

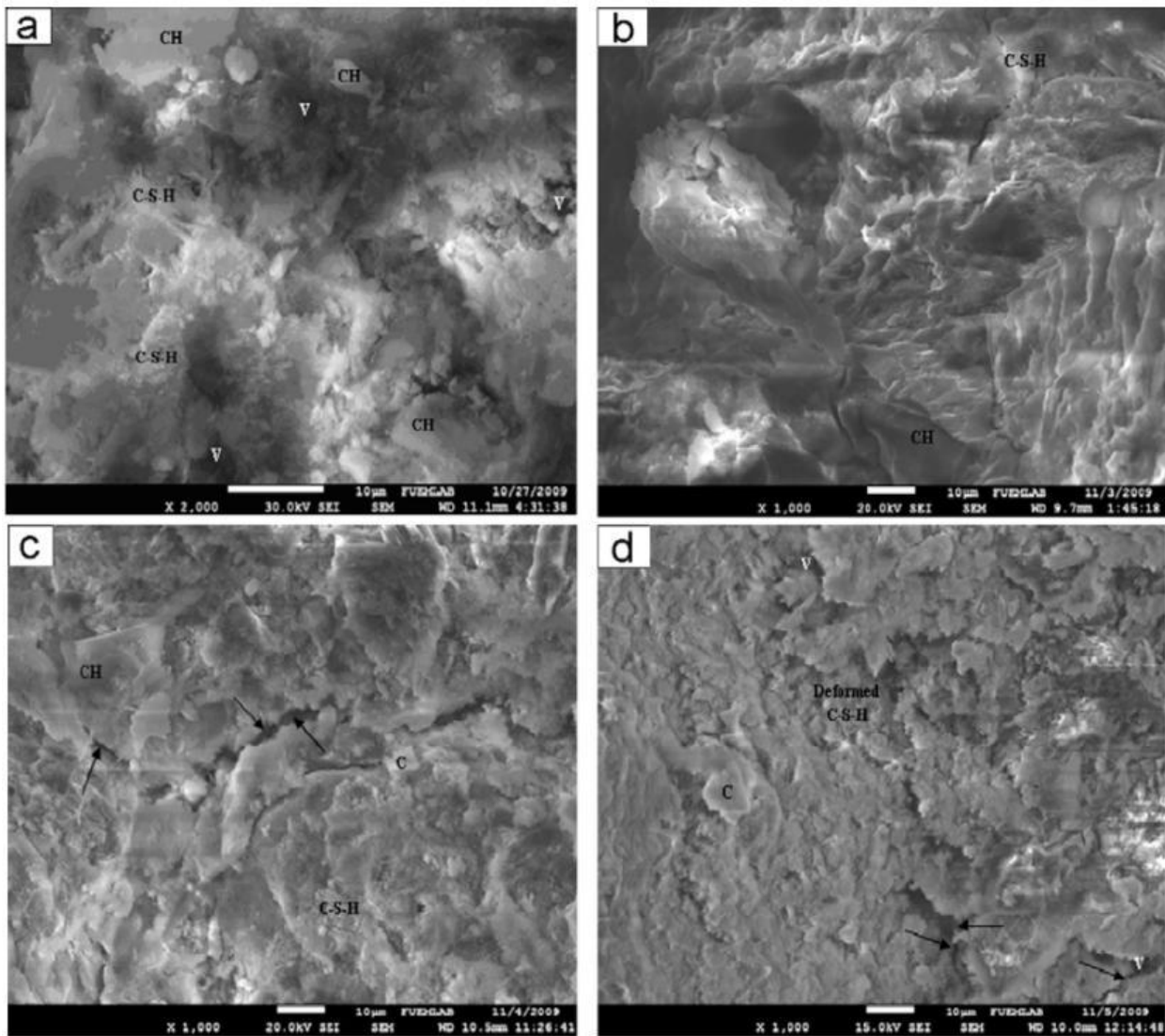


Fig. 5 SEM micrographs of Concrete specimens exposed to (a) 20°C, (b) 400°C, (c) 600°C, (d) 800°C [Ca(OH)_2 crystals – CH, CaCO_3 – C, Voids – V]

V. LIGHT WEIGHT CONCRETE MIXED WITH VERMICULITE

Light weight concrete [17-19] is made of light weight aggregates which have an inherent advantage of having high resistance to fire. The reason of their high resistance to fire is due to their exposure to high temperature during formation and production processes. Light weight concrete have high thermal stability than ordinary concrete. Hence these properties make it better fire resistant than the normal aggregates concrete. Vermiculite [20] is a hydrous phyllosilicate naturally occurring mineral. Its appearance resembles mica as it composes of shiny flakes. Vermiculite is a safe inert material and is light in colour. When heated, it exfoliates up to 8-30 times its original volume. This expansion is due to the conversion of interlayer and structural water to steam. The exfoliation process converts the dense flakes of ore into lightweight porous pellets containing numerous microscopic air layers. Exfoliated vermiculite is light and clean to handle, has a high insulation value, acoustic-insulating properties. The forged vermiculite possesses several valuable properties, such as low thermal conductivity, high fire resistance and strong sound absorption [21]. Materials produced using vermiculite are fire-resistant and neutral to the action of acids and have constant strength with time and resistance to deformation. These facts make meaningful the use of vermiculite in construction and concurrently heat isolation and sound absorption materials. To study the micro-structural changes occurring in concrete prepared with the addition of vermiculite, Scanning electron microscope (SEM) method was performed Koksai et al [17]. The SEM test was performed at each elevated temperature taken as reference in their study i.e. 20, 300, 600, 900 and 1100°C. Small deterioration was found to be started when the temperature was increased to 300°C. However, it was seen that there was no significant change in the aggregate cement matrix up to 900°C. Although due to the inherent property of expansion of vermiculite on higher temperatures caused increase in void ratio in micro-structure. On increasing temperature to 1100°C, the C-S-H phase boundaries disintegrated causing desertion of the bond strength at the interface [17].

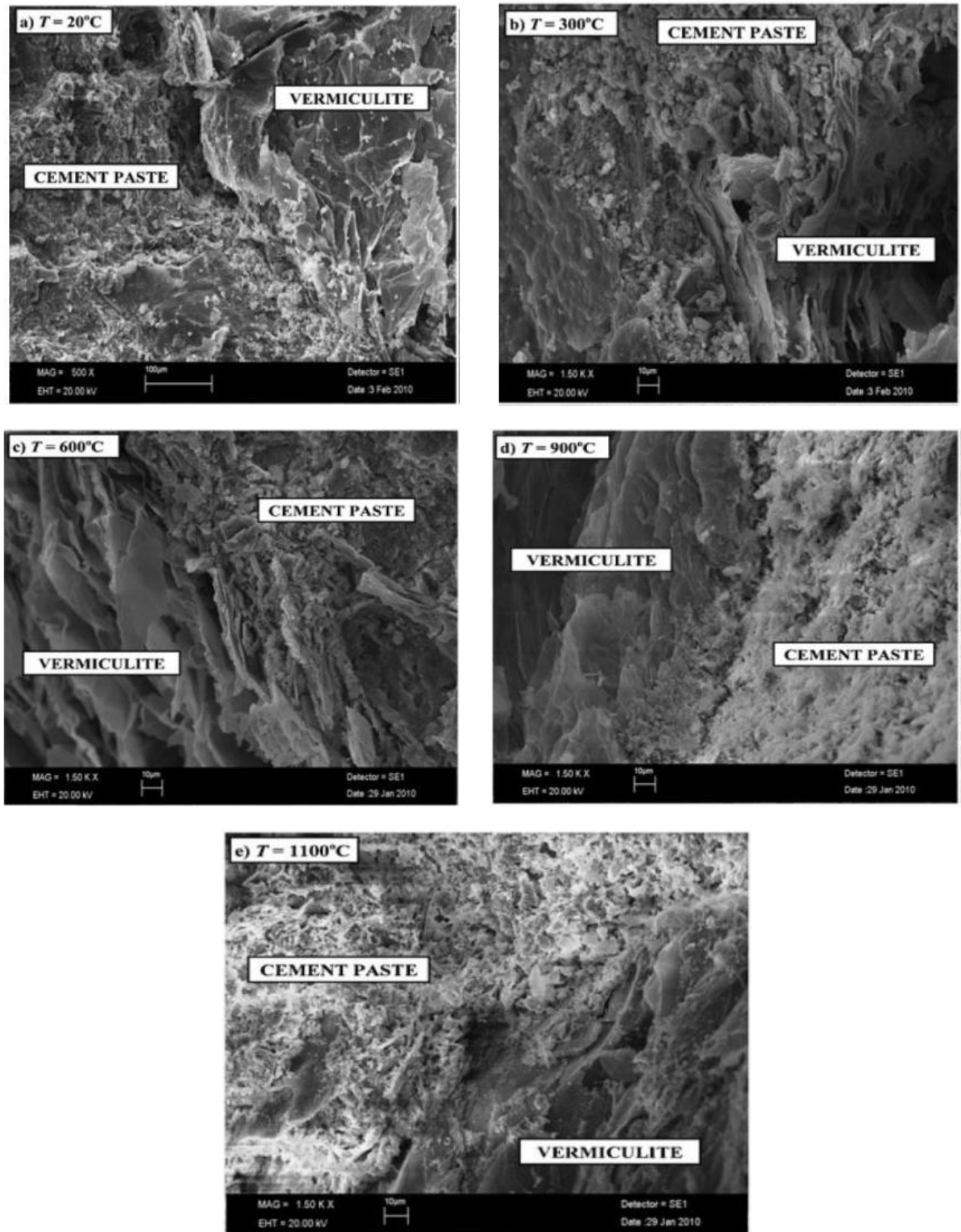


Fig.6- SEM micrographs of light weight concrete mixed with vermiculite at temperature (a) 20°C , (b) 300°C , (c) 600°C , (d) 900°C , (e) 1100°C

VI. CONCRETE CONTAINING CLINOPTILOLITE

As already discussed above, residual strength of concrete exposed to elevated temperature can be improved by the addition of supplementary strengthening materials in concrete. Clinoptilolite is a natural zeolite consisting of a microporous arrangement of silica and alumina tetrahedra. The structure of clinoptilolite consists of three-dimensional

grid which comprises of Silicate tetrahedrite (SiO_4)⁴⁻ each unified via oxygen atoms; the atoms of silicon is replaced by aluminum (AlO_4)⁵⁻, making a typical spatial structure with a substantial occurrence of cavities, interconnected by channels, in which metal cations, or water molecules are protected. Some of the main properties of clinoptilolite include ion exchange, their large surface area and their ability to act as molecular sieves [22]. Clinoptilolite is widely used in livestock farming, construction, agriculture, environmental protection. It is also used in cleaning water and gases and in various industrial uses. Clinoptilolite contributes to the strength and durability of concrete [24]. Due to large quantity of reactive SiO_2 and Al_2O_3 present in clinoptilolite, it undergoes pozzolanic activity. The SiO_2 further combines with Ca(OH)_2 to form supplementary C-S-H gel [25-26]. It also contributes to improve residual strength of concrete exposed to high temperatures. PPTL method was used by Akçaözog̃lu et al.[23] to study the microstructural changes occurring in concrete containing clinoptilolite at elevated temperatures. Similar to the previously discussed process, the concrete specimen were divided into thin sections. These thin sections were prepared from the C0, C10, C20, C30 and C40 specimens containing 0%, 10%, 20%, 30% and 40% by weight of cement replaced by clinoptilolite respectively. The thin sections were then divided into three different zones A, Band C to make more precise assessment about the effect of elevated temperature on the microstructure of the concrete.

VII. TEXTURAL DIFFERENTIATION BASED ON ZONES [23]

Almost all the specimen were observed undergoing noteworthy texture differentiation from the outer surface toward the center when exposed to elevated temperatures. As seen from Fig. 7, the area between the red lines was divided into three zones A, B and C in order to put the textural differentiation.

Zone A (Fig.7 A-C) - Cracks and disintegrations were observed extremely in the aggregate-cement matrix at the outer side of this zone. The cracks formed in outer side of zone A were parallel to the edge. In the outer part of zone A, cracks were not seen intersecting each other. More fissures and cracks were formed at quartz than basalt. The reason of this can be attributed to the fact that basalt has higher thermal stability than quartz crystals. When the center part of zone A was analyzed, the cracks and fissures were found of the same extent as at the outer side of zone A. In addition to it, the cracks were decreased in the aggregates. It was observed that on the portion of zone A far from the outer side, fissures and cracks were formed only at the interface of aggregate- cement matrix.

Zone B (Fig.7- D-E) - At zone B, fissures and cracks were observed to be excessively condensed. There were only a few singular cracks stretched into the center dividing it into two. Textural variations at this zone were in the form of thin alteration layers occurring at the interface of aggregate-cement matrix. Substantial changes were not seen in Basalt crystal aggregates at zone B. The changes in aggregate were restricted only to quartz crystals.

Zone C (Fig. 7-F-H) – The inner region of this zone did not show significant changes in aggregate-cement matrix. Quartz aggregates crystals remained in their well-developed form due to uncrowded growth. Basalt aggregates crystals bared a similar appearance of main rocks. No change was observed in the adherence of cement-aggregate matrix. At elevated temperatures, the region close to the outer surface was severely affected having dense cracks in the region. Both aggregate-cement matrix and aggregates were affected due to exposure to elevated temperatures. At inner region of this zone, there was not significant change in both the aggregate-cement matrix and aggregates. The inner changes were mostly in the form of singular cracks.

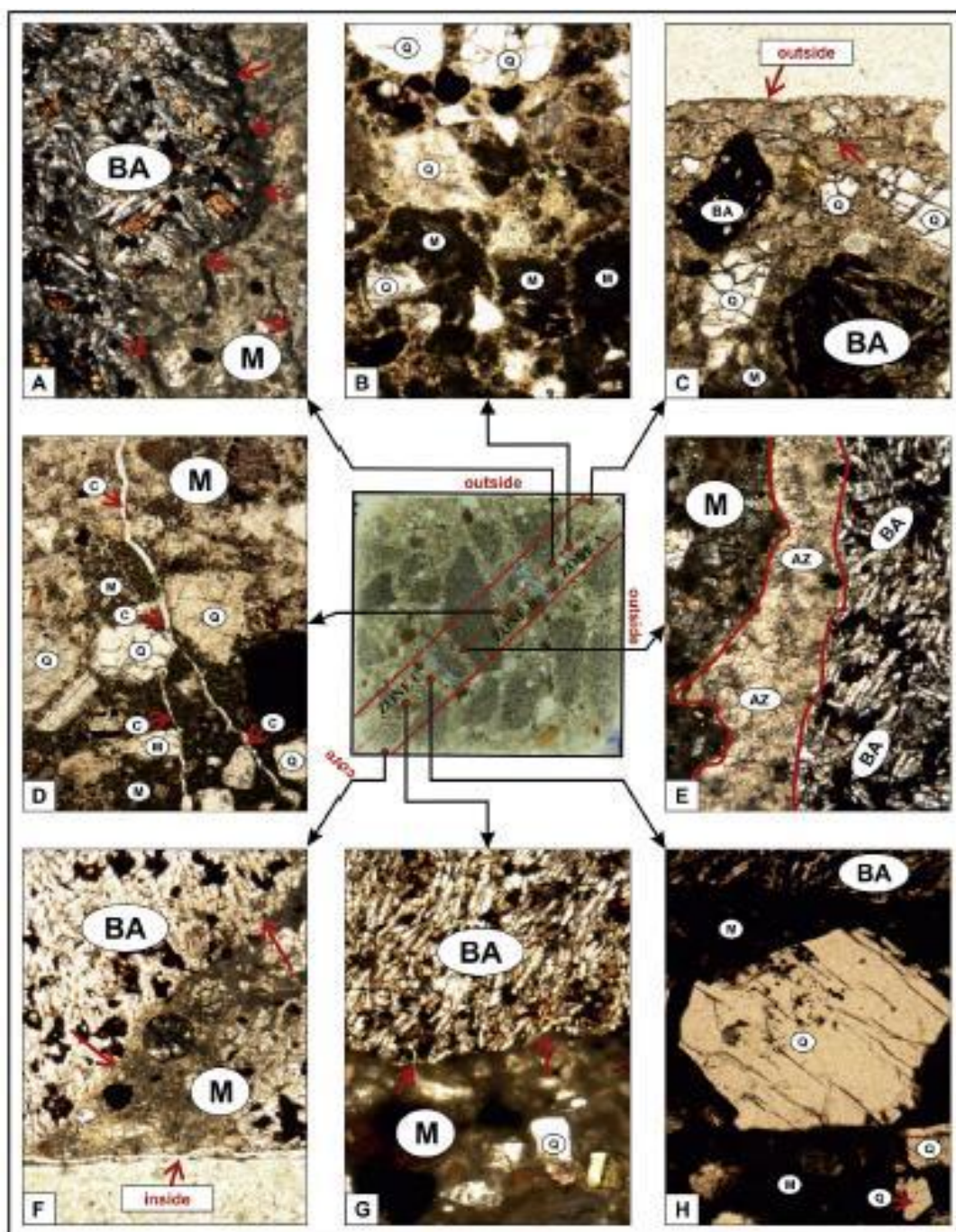


Fig.7- Textural differentiation of concrete containing clinoptilolite at elevated temperatures based on zones [23]
 (BA- Basalt, Q – Quartz, M- Matrix, C- Crack)

VIII. CONCLUSIONS

After the detailed study, Effect of elevated temperatures on the structure of aggregate using PPTL method and SEM method was discussed in this paper. It was observed that rate of cooling also has significant effect on deterioration of microstructure. Furthermore, concrete made with Basalt aggregate was found to have greater level of fire resistance than that of quartz aggregate. It can be observed from several studies that concrete loses its integrity at 600 °C - 800 °C because of the intermingling of cracks as a result of loss of bound water and increase in porosity at elevated temperatures. For concrete made with vermiculite, aggregate cement matrix was found to be stable upto 900 °C in comparison to 600 °C of normal concrete.

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