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Mineralogy and SEM study of Reducting Conditions in the Fars Cement Factory Clinkers (SW Iran)

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ABSTRACT

Reducting conditions is one of the most important problems in the cement factories that decrease the cement quality and strength. Microscopical analyses are one of the easiest methods that can provide information about quality-control and trouble-shooting situations in kilns quickly. In this research reducting conditions in the Fars cement factory coarse clinkers were investigated by preparing 10 polished sections from rim layer to core, SEM pictures and Ono method. Based on these methods, because of high burning time and creation the reducting conditions in kiln, alite crystals alteration in to belite crystals and limestone that can be seen in clinker's core polished sections with abundant of belite crystals. More fractures were produced in clinker cores by Reducting conditions and cause the low strength in mortar and concrete. Also heating and cooling rates were slow and maximum temperature was long in the Fars cement factory clinkers generation.

Keywords: Reducting conditions, Microscope, Mineralogy, Fars clinkers

INTRODUCTION

Cement is one of the most important material used in industry and clinker microscopically examinations are the practical and fast methods for Cement Quality Control and kiln conditions that can be used in cement factory laboratories. The fundamental use of the microscope in Portland cement clinker analysis is to bring the observer visual appreciation of phase identities, size, conditions and mutual relationships. With only a basic assemblage of equipment, microscopically analysis can be easily performed, in many cases within a few minutes. The rapidity with which potentially energy saving information can be acquired clearly renders the analysis economically justifiable, especially in routine quality-control and trouble-shooting situations. In addition, the microscope has obvious value in scientific research in the manufacturing process. Study of polished section or thin section of Portland cement clinker, quickly reveal several details of crystal size, morphology, abundance, and distribution leading almost intuitively to interpretations relating these data to certain features of the raw material and burning conditions [1]. The microscopical method of analysis, using polished sections or thin sections of clinkers is uniquely advantageous because the investigator can see individual crystals, virtually undisturbed in their place of origin and can interpret these observations in terms of the microenvironment developed in that clinker nodule. These observations are related to characteristics of the raw feed particles and the burning conditions in the kiln. For example, nests of tightly packed belite crystals form in silica rich areas of the clinker suggest the possibility of coarse quartz grains in the raw feed and alite crystal size of 10 to 15 µ may indicate an undesirably rapid rate of temperature rise in the clinker as it passes through the kiln or large clusters of free lime suggest coarse limestone particles [1]. In this research the brown core clinkers were observed in Fars cement factory coarse clinkers and based on microscopical analysis methods. Ono method and SEM pictures the reducting conditions in this cement factory clinkers were investigated.

STUDY AREA

The study area is bounded by latitudes 52°30′:52°38′N and Longitudes 29°40′:30°05′E. It forms part of survey of Shiraz Topo sheet. This cement factory is located at the beginning of the Shiraz- Boshehr road and it has two cement

production lines with daily production capacity of 2500 tons cement per day. Lithological units in the study area including thick beds of white to cream dolomitic limestone and inter bedded with marly limestone and marl, dark gray ferrogenous, dolomitic limestone in upper portions (Jahrom Formation) also well bedded to thick bedded cream, brown and gray limestone and marly limestone inter bedded with marl (Asmari Formation). Figure 1 shows the simplified geological map of study area in the Shiraz Top sheet.

CLINKER PHASES IN POLISHED SECTIONS AND SEM PICTURES

According to Campbell method [1], clinker phases are diagnosed by their shapes in polished sections. Alite crystal is a solid solution series of trigonal, monoclinic, and triclinic modifications of impure tricalcium silicate [2], which is generally termed C3S in the cement industry. Substitution of magnesium and aluminum for silicon causes triclinic and pseudotrigonal forms to change to monoclinic and pseudotrigonal forms; other substitutions may involve iron and sodium [3]. It comprises 40 to 70 percent of normal Portland cement clinkers. Alite crystals are idiomorphous, vitreous, compact, usually six sides in cross section depending on the angle of cut, commonly perfect hexagons. Normal crystal sizes range from 25 to 65 µ. Alite crystallizes between 1200 to 1450°C from the melt, its birefringence ranges from 0.005 to 0.008, that pure tricalcium silicate birefringence is 0.003 [4] and alite crystals rarely show polysynthetic twinning [5] (Figure 2).

Belite is solid solution series of trigonal, orthorhombic, and monoclinic varieties of impure dicalcium silicate [2], normally termed C2S in the cement industry. Polymorphs of Dicalcium silicate are called alpha, alpha prime, beta and gamma, which the alpha prime and beta forms are said to compose approximately 10 to 30 percent of most Portland cement clinker. Substitutions may be magnesium, potassium, sodium, barium and sulfur. Belite grains are idiomorphous, vitreous and normally rounded with marked multidirectional lamellar structures, in part due to twinning. Its hardness is 4 to 5 on Mohs's scale. Belite is colorless, pale yellow, yellow, amber or shades of green (Figure 2). The crystal shows poor prismatic cleavage and its interference color in a thin section of 25 µ thickness is first order whitish yellow. Birefringence in thin section is 0.0153 [4]. Belite size is 1 to 4 µ below 1300°C but recrystallizes to 20 to 40 µ with treatment of high temperature (approximately 1500°C).

Ferrite is an orthorhombic solid solution series of $Ca_4Fe_4O_{10}$ to $Ca_4FeAl_3O_{10}$, ferrite crystal color is normally brown to yellow, elongation is positive and this phase is pleochroic with the Z direction showing the strongest brown absorption, Figure 2, it displays a distinct cleavage on 101 and can show twinning with 101 as the composition plane. Crystals

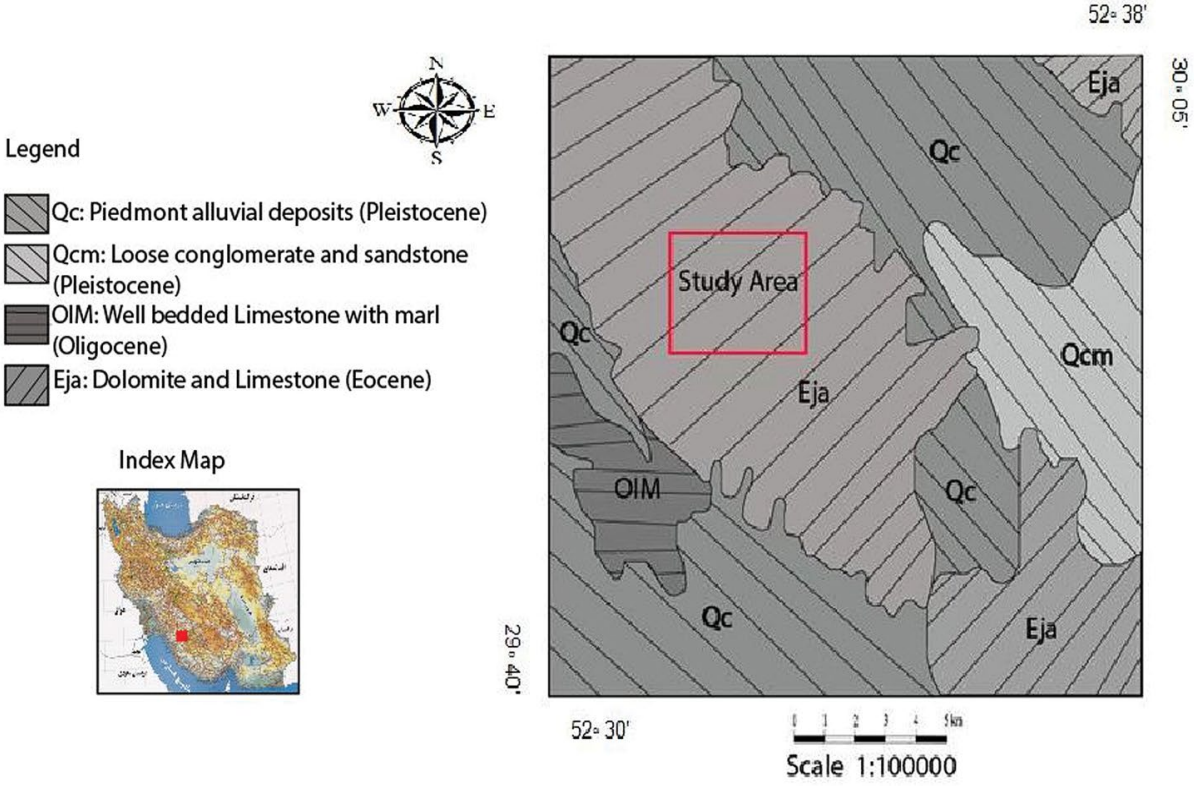

Figure 1: A part of Shiraz Geological map, showing the study area location (Geological survey of Iran)

are commonly tabular on 010 but can be idiomorphous interstitial filling. Ferrite can also be prismatic, dendritic or massive to fibrous [1].

Also in SEM pictures, Alite crystals showing Euhedral and Angular shapes, belite crystals are rounded and ferrite crystals showing typical box work pattern and lath like crystal form (Figures 3 and 4) [6].

THE ONO METHOD

Ono's method of cement kiln evaluation is based on observation of clinker or cement phases in polished or thin sections. The principal kiln conditions and microscopically parameters evaluated by Ono's technique are: heating rate (according to Alite size, AS), burning time at high temperature (according to Belite size, BS). Maximum Temperature (Alite Birefringence, AB), cooling rate (Belite color, BC). Table 1 shows the burning conditions based on Ono method [5]. According to this table and polished section picture's, heating and cooling rate were slow and Maximum temperature and Burning time were Long in Fars cement factory clinkers (Table 1).

REDUCTING CONDITION

Reducting conditions and their microscopical effects have received much attention recently and their effects largely involve iron and ferrite in clinker's composition [7]. In the Fars cement factory, some clinkers with coarse grading have brown core and gray rim that shows the high burning time and creation reducting conditions and reducing the amount of oxygen in the kiln.

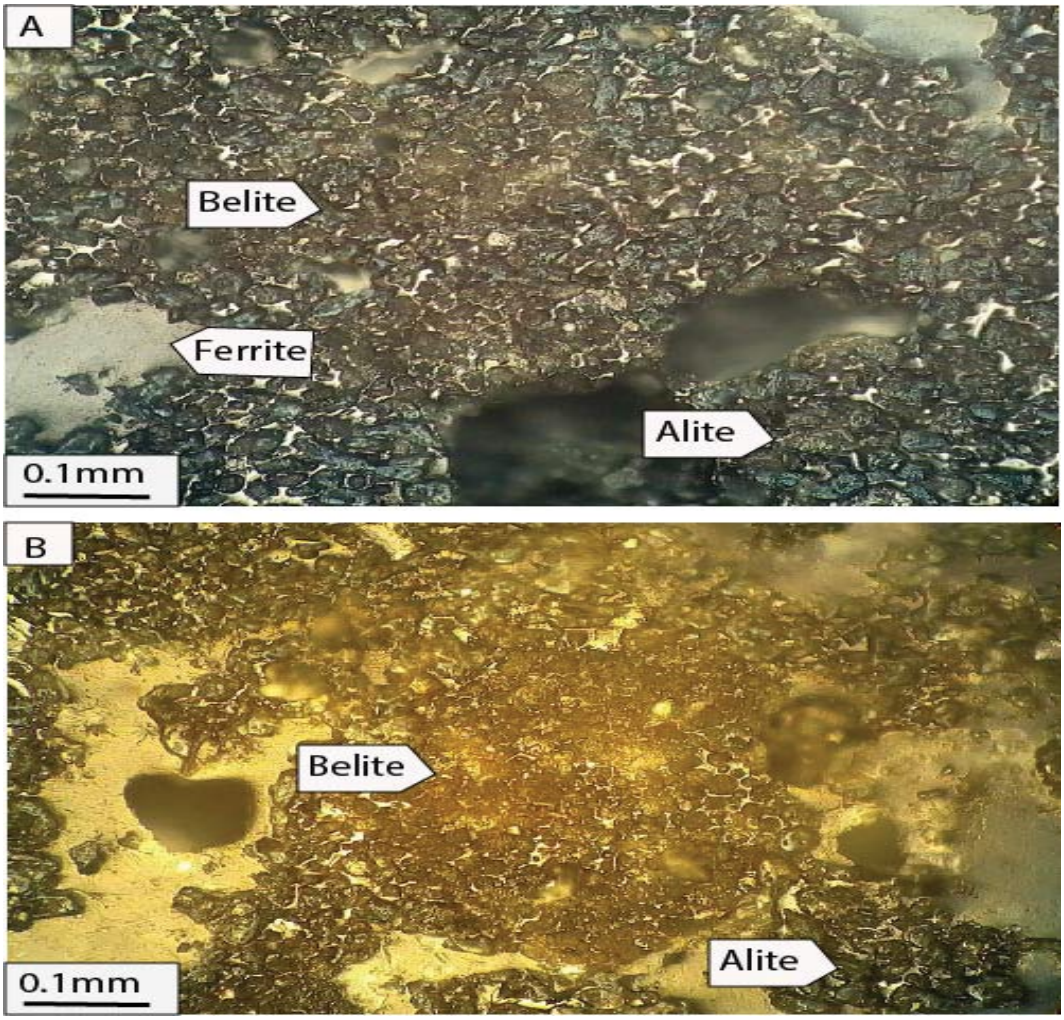

Figure 2: A and B) The presence of rounded belite and Euhedral alite in rim layer polished section. Alite crystals are more abundant than Belite crystals and the matrix is high content of ferrite. Alite crystals are fresh and did not show any alteration (PPL)

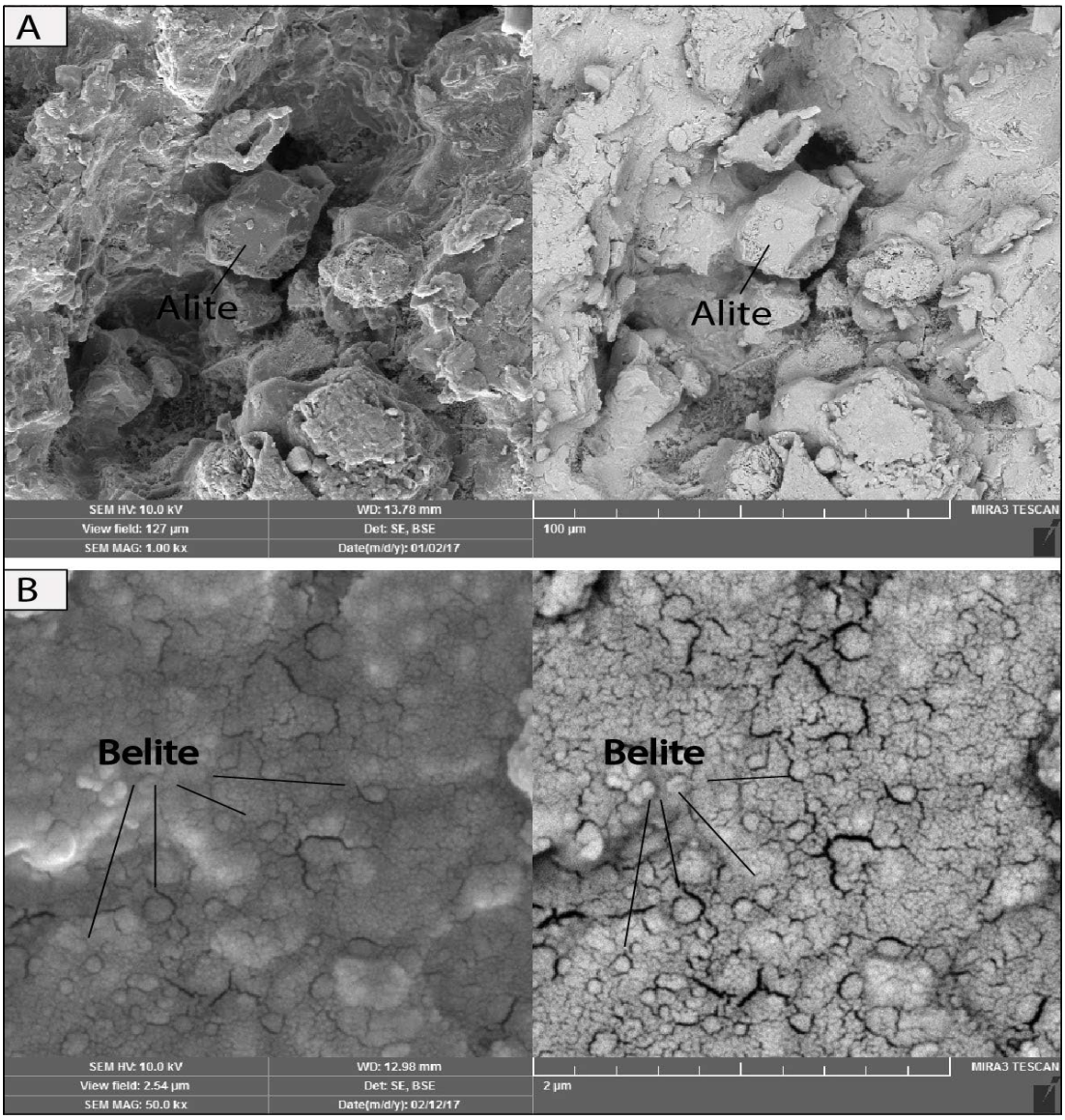

Figure 3: (A) The presence of euhedral alite in rim layer SEM picture. alite crystals are more abundant than belite crystals. (B) Rounded belite crystals in core SEM picture and belite crystals are more abundant than alite crystals

Reducting condition, high consumption of coal and over burning cause large clinkers (25 to 125 mm) to have less alite, more aluminate phases, large anhedral alite, large belite, alite and belite decomposition, high water demand, reduced setting time and lower mortar strength compared to small clinkers [8]. With regard to the observation of brown core clinkers, Pennell [9] concluded that this clinkers results from burning in a CO_2 atmosphere at typical burning temperature of 1400 to 1450°C or at high temperatures of 1500-1550°C in a highly reducting atmosphere under conditions such that semi-liquefaction and resultant vitrification of the clinker occurs, so that air cannot penetrate this vitrified mass upon exiting the burning zone. Brown core clinkers were produced in a laboratory kiln with a temperature of 1525°C [9].

Reducting conditions affected the composition of the ferrite, decrease of ferrite content, increased the content of aluminate phases and reduced the stability of alite crystals and conversion them to belite and free lime. So that the core polished section picture's show the alteration of alite crystals in to belite and some inclusions of free lime can be viewed in alite crystals (Figure 5). This state leading to lower strength and reactivity of cement mortars. Therefore the clinkers with brown core are associated with decrease oxygen content and creation reducting conditions because of high burning time in the kiln and decrease oxidation of Fe_2O_3 to FeO during cooling which tends to occur in large nodules that high in belite crystals [10].

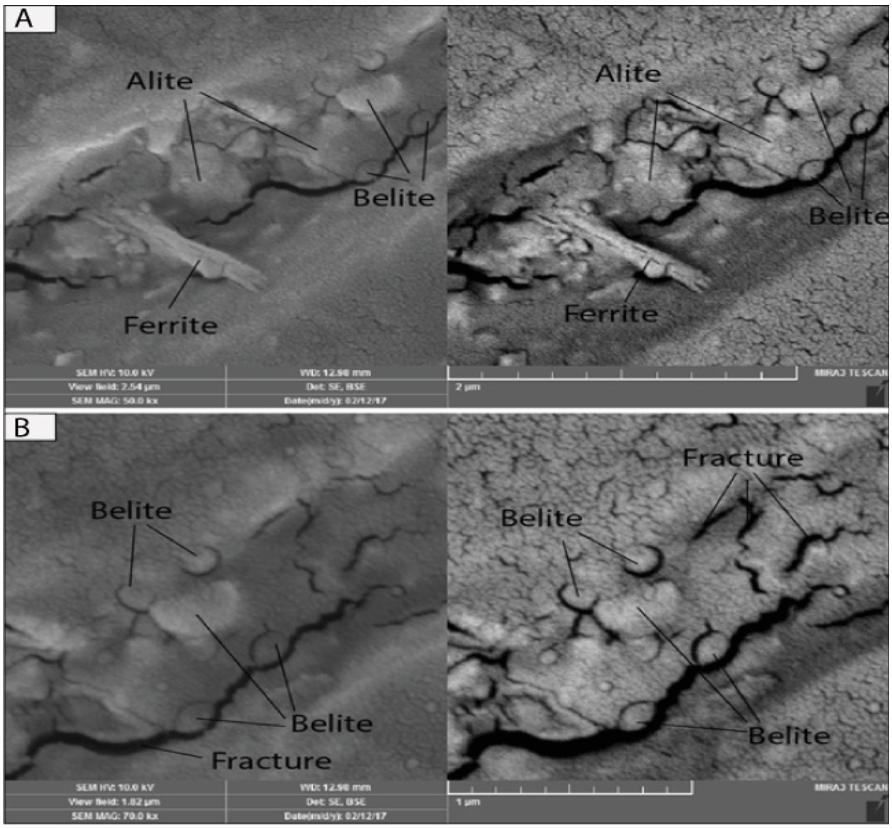

Figure 4: (A) The presence of alteration alite and rounded belite crystals in core layer SEM picture and the ferrite with lathlike crystal form. Belite crystals are more abundant than alite crystals. (B) Rounded belite crystals in core SEM picture and fractures are abundant in core SEM pictures

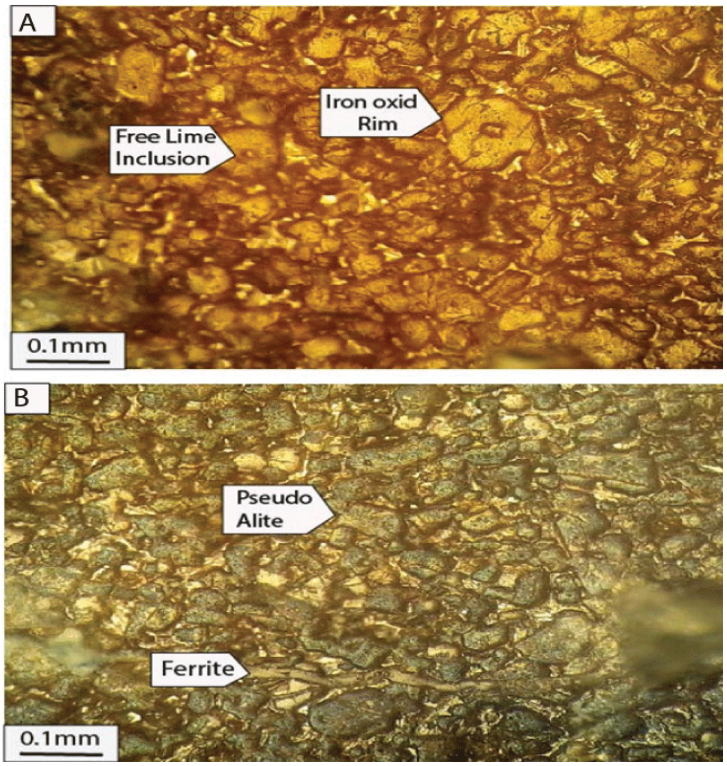

Figure 5: A and B) The presence of belite crystals in alite casts (pseudoalites) in core layer polished sections. Belite crystals are more abundant than alite crystals and the matrix is low content of ferrite. Alite crystals showing alteration in to belite, free lime was inclusion in pseudoalites and the iron oxide rims are abundant around the crystals (PPL)

Table 1: Burning condition and microscopically character of alite and belite [5]

Burning Condition	Excellent 4	Good 3	Average 2	Poor 1
Heating Rate)AS μ)	Quick 15-20	- 20-30	-(25) 30-40	Slow 40-60 (120)
Maximum Temperature (AB μ)	High 0.010-0.008	- 0.007-0.006	- 0.006-0.005	Low 0.005-0.002
Burning Time (BS μ)	Long 25-40 (60)	- 20-25	- 15-20	Short 5-10
Cooling Rate (BC)	Quick Clear (C)	- Faint Yellow (FY)	- Yellow (Y)	Slow Amber (A)

DISCUSSION

In this research 10 polished sections were prepared from rim layer to core of 5 Fars cement factory clinkers (SW Iran) and were investigated by reflected polarizing microscope at the geology laboratory, university of Tabriz and clinker phases were recognized by Campbell method in polished sections, also SEM pictures of this clinkers from rim layer to core were done in fundamental Research Center, University of Tabriz and for study the burning conditions, the Ono method [5] was used. Based on microscopically analysis and SEM pictures from rim layer to core of Fars cement factory clinkers, reducting conditions in this factory kilns were investigated and results show that alite and belite crystals with matrix of ferrite were abundant in rim polished section clinkers. Alite crystals were more abundant than belite crystals and did not show any alteration but in clinker's core polished sections, because of reducing the amount of oxygen. Alite crystals have been altered to belite crystals and lime stone so that belite crystals were found in alite casts (Pseudoalites) and free lime was inclusion in pseudoalites. Belite crystals were more abundant than Alite crystals and the amount of ferrite was reduced. Also the SEM pictures confirm this observation in polished sections and some fractures were observed in clinker's core SEM pictures and Alite crystals were lost their Euhedral shape. According to the Ono method, heating and cooling rates were slow and maximum temperature and Burning time were long in Fars cement factory clinkers.

CONCLUSION

Reducting conditions is one of the main problems in the cement industry that can be study by microscopically investigations so that this method can provide a lot of information about clinker burning conditions in the kiln and is the appropriate method for cement quality control investigations. Because of high burning time, the amount of oxygen Decreases in kiln and it causes the Reduction of Fe_2O_3 to FeO and creation the brown core in coarse clinkers.

Investigation of clinker phases in polished sections, according to Campbell method, showed that alite, belite and ferrite were the major phases in Fars cement factory clinkers.

In the rim layer polished sections alite crystals were more abundant than other phases and Ferrite was abundant in matrix. Alite crystals were euhedral and did not show any alteration but in core polished sections, because of reducing the amount of oxygen, slite crystals have been altered to belite crystals and lime stone so that belite crystals were found in alite casts (Pseudoalites) and free lime was inclusion in pseudoalites. Belite crystals were more abundant than alite crystals, the amount of ferrite was reduced, alite crystals lost their Euhedral shape and the iron oxide rims were created around the crystals.

SEM pictures confirm the observation in polished sections and some fractures were observed in clinker's core SEM pictures that these fractures may be created by crystal reactions during reducting conditions and can reduced the mortar and concrete strength.

According to Ono method, heating and cooling rates were slow, maximum temperature and burning time were long in the Fars cement factory clinker generation.

ACKNOWLEDGMENT

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