Construction and Building Materials 72 (2014) 316–325

Contents lists available at ScienceDirect

Construction and Building Materials

journal homepage: www.elsevier.com/locate/conbuildmat

Microstructural examination of the effect of elevated temperature on the concrete containing clinoptilolite

Kubilay Akçaözoğlu [a,*], Mustafa Fener [b], Semiha Akçaözoğlu [c], Recep Öcal [a]

[a] Department of Civil Engineering, Faculty of Engineering, Nigde University, Nigde 51240, Turkey
[b] Department of Geological Engineering, Faculty of Engineering, Ankara University, Ankara 06100, Turkey
[c] Department of Architecture, Faculty of Architecture, Nigde University, Nigde 51240, Turkey

HIGHLIGHTS

- Microstructure of clinoptilolite concrete exposed to high temperature was examined.
- Thermal conductivity values decreased depending on increasing clinoptilolite amount.
- The textural changes showed variety depending on the amount of clinoptilolite.
- Texture differentiations were more significant at the outer surface of specimens.
- Textural changes in quartz crystals decreased depending on increasing clinoptilolite.

ARTICLE INFO

Article history:
Received 6 August 2014
Received in revised form 17 September 2014
Accepted 18 September 2014

Keywords:
Clinoptilolite
Concrete
Elevated temperature
Compressive strength
Thermal conductivity
Polarized microscope analysis
Microstructure

ABSTRACT

In this study, the effect of elevated temperature on the properties of concrete containing clinoptilolite was investigated by microscopic analyses. For this purpose, seven different mixtures were prepared (the control mixture and six mixtures including 5%, 10%, 15%, 20%, 30% and 40% clinoptilolite by weight). The water–binder (w/b) ratio used in the mixtures was 0.475. The dry unit weights, water absorption ratios, porosity ratios, compressive strengths and thermal conductivity coefficients of the mixtures were measured. In addition the specimens exposed to elevated temperatures of 250, 500, 750 and 1000 °C. Two different cooling methods were used (slow cooling and fast cooling). The residual compressive strengths of the specimens which were exposed to elevated temperatures were measured. In addition the mineral and texture changes of the specimens were examined by using plane polarized microscope. Test results indicated that, clinoptilolite substitution decreased the compressive strength of the specimens in early days, but increased at later days. The positive effects were observed about clinoptilolite substitution on the residual compressive strength of the specimens. It was observed from microscopic analyses that, as the amount of clinoptilolite increased in the mixtures, aggregates were less affected from elevated temperatures. Fast cooling (FC) method resulted in strength losses when compared to slow cooling (SC) method. Additionally, clinoptilolite substitution decreased the thermal conductivity coefficient of the concrete.

1. Introduction

Fire which its incidence has increased in recent years is one of the main threats of the modern buildings [1,2]. Concrete is a fire resistant material because of its properties such as being a non-flammable material, is not important damaged for a certain period and does not remove toxic fumes in high temperature [3]. However, this durability is valid for a limited period and specific temperatures [4]. When building materials are subjected to elevated temperatures some changes may occur in their characteristics, such as phase transformation, weight loss, and aggregate–cement bond which directly affect its chemical receptivity and mechanical properties [5–8]. Due to composite nature of concrete and different thermal characteristics of constituents, its performance is greatly affected by high temperature [9,10]. The type of aggregate and cement used in its composition, the porosity and moisture content of concrete, its thermal properties and sizes of structure members and their construction type are the other factors that determine the level of fire resistance of the concrete material [11].

* Corresponding author.

http://dx.doi.org/10.1016/j.conbuildmat.2014.09.023
0950-0618/

The use of supplementary cementing materials in concrete mixtures is an efficient technique to improve the residual strength of concrete material which is exposed to elevated temperature [12]. The beneficial effect of mineral admixtures with respect to high-temperature resistance arises from stabilizing of $Ca(OH)_2$ from cement by means of pozzolanic reaction [10].

Clinoptilolite is hydrated alumino-silicates which have been used in construction since ancient time [13]. It has a three dimensional structure which is composed by Si–O tetrahedroids and Al–O tetrahedroids. Its crystals have a honeycomb like structure with channels [14]. Some of the main properties of clinoptilolite include ion exchange, their large surface area and their ability to act as molecular sieves [15]. Due to their physical structures and chemical properties they play a significant role among industrial raw materials used in such fields as pollution management, energy, agriculture, stock farming and mine metallurgy [16]. Clinoptilolite, like other pozzolanic materials, contributes to the strength of concrete better than cement [17]. It undergoes pozzolanic activity due to its high quantity of reactive SiO_2 and Al_2O_3, which combines with $Ca(OH)_2$ to form additional C–S–H gel [18,19]. Besides the contribution to the strength and durability of concrete, it also contributes to improve residual strength of concrete exposed to high temperatures. In addition it has positive effects on the residual strength of concrete [16].

Plane polarized transmitted light (PPTL) method is one of the microscopical examinations to use determining fire damage of concrete. In this method, thin-section specimens are prepared for through which light will pass to allow microscopical observation. Thin-sections are examined in PPTL method using a high quality, medium to high-power petrological microscope at magnifications typically up to 600× [20].

In this paper the effect of elevated temperature on the residual compressive strength of the concrete specimens containing clinoptilolite was investigated. In addition, the microstructural examination of the clinoptilolite concrete specimens exposed to elevated temperatures was carried out by using PPLT method.

2. Materials

Ordinary Portland Cement CEM I 42.5 R (PC) conforming to requirements of TS EN 197-1 [21] was used for concrete mixture preparing. The specific surface area of PC measured with Blaine method was 3530 cm^2/g, and the specific weight of the PC was 3.11 g/cm^3. The initial and final setting times of the cement were 160 and 214 min, respectively. The 28-day compressive strength of PC according to TS EN 196-1 [22] was 49.9 MPa. Ground clinoptilolite used in the mixtures was supplied by the Rota Mining Company, Gördes region, Turkey. The particle size distribution of clinoptilolite which was obtained using a laser scattering technique is given in Fig. 1. The specific surface area and the specific weight of clinoptilolite were 3900 cm^2/g, and 2.18 g/cm^3, respectively. Chemical oxide composition of Portland cement and clinoptilolite are presented in Table 1.

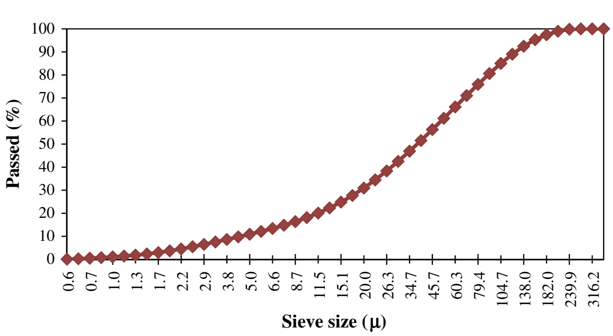

Fig. 1. Particle size distribution of clinoptilolite.

Table 1
Chemical properties of PC and clinoptilolite.

Oxide (%)	SiO_2	Al_2O_3	Fe_2O_3	CaO	MgO	SO_3	K_2O	Na_2O	LOI
PC	20.39	5.25	3.07	62.91	1.42	3.43	0.91	0.58	1.91
Clinoptilolite	68.43	10.91	1.32	3.12	1.10	–	2.92	0.53	10.0

Table 2
Aggregate grading with standard limit.

Sieve size (mm)	Passed (%)			
	TS 706 EN 12620+A1 lower limit	TS 706 EN 12620+A1 medium limit	TS 706 EN 12620+A1 upper limit	Aggregate used
31.5	100	100	100	100
22.4	98	99	100	100
16	85	92	99	96.7
11.2	68	79	90	80.3
8	48	63	77	58.9
4	33	49	64	50.3
2	22	37	52	41.1
1	15	28	41	30.9
0.5	10	20	30	16.8
0.25	6	13	20	10.9
0.15	3	7	11	3.6
0.063	1	3	5	1.2

Three type of aggregate were used in the mixtures. Uncrushed, quartzitic natural sand (0–3 mm and 0–6 mm) and crushed basaltic coarse aggregate (5–15 mm) in accordance with TS 706 EN 12620+A1 [23] were used in the mixtures. The specific weights of fine aggregates and coarse aggregate at saturated surface dry condition were 2.58, 2.54 and 2.75 g/cm^3, respectively. The water absorption values of fine and coarse aggregates were 1.35%, 1.57% and 1.70%, respectively. The grading of aggregates is presented in Table 2 with the standard specification. Polycarboxylic ether based hyper plasticizer (HP) according to TS EN 934-2 [24] was incorporated in the concrete mixture to improve workability. The specific weight of hyper plasticizer was 1.11 g/cm^3.

3. Mixture properties and testing methods

Mixture design was made with according to absolute volume method given by TS 802 [25]. The water–cement (w/c) ratio used in mixtures was 0.475. Binder content was kept constant as 400 kg/m^3 for per cubic meter. Clinoptilolite was used at 0%, 5%, 10%, 15%, 20%, 30% and 40% replacement by weight of cement. Seven different mixtures were prepared for experimental studies. Approximate concrete composition for concrete mixture of a cubic meter is given in Table 3.

Cubic specimens with a 100 mm side were prepared to use compressive strength tests. The test specimens were de-molded at 1 day, and then cured in lime saturated water at 20 ± 2 °C until the time of the testing. The compressive strength of concrete specimens were measured according to TS EN 12390-3 [26] at 1, 3, 7, 28, 90, 180 and 360 days. Experiments were carried out at uniaxial compression instrument with a capacity of 3000 kN and loading

Table 3
Approximate concrete mix design for a cubic meter.

Mixture	PC (kg)	Clinoptilolite (kg)	Water (kg)	Aggregate (kg)			HP (%)	Slump (cm)
				0–3 mm	0–6 mm	5–16 mm		
C0	400	0	190	344	536	869	0.35	13
C5	380	20	190	344	536	868	0.50	13
C10	360	40	190	343	534	866	1.00	15
C15	340	60	190	342	533	864	1.50	13
C20	320	80	190	341	531	861	2.00	12
C30	280	120	190	340	529	858	2.68	12
C40	240	160	190	337	525	851	4.00	11