

# Effect of interfacial transition zone on properties and microstructure of concrete

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## Abstract

The interfacial transition zone (ITZ) is a relatively heterogeneous region with a thickness of 50 micrometers, whose physical and mechanical properties are determined descriptively due to the fine length scale and heterogeneous characteristics. In this research, by focusing on the ratio of different elements forming the ITZ and simultaneous examination of microscopic images, it was possible to numerically investigate the effect of changes in the ratio of elements on the properties and microstructure of concrete. In order to investigate the effect of the ITZ on concrete properties by examining the rheology of fresh concrete, 30 standard cubic samples were examined in the form of 10 mixing plans, and then the selected mixing plan was determined by trial and error. Then, 54 cubic and cylindrical samples of aggregates with high silica and w/c as 0.45 were made, and tests were conducted to determine the mechanical properties and durability of concrete in the age range of 7 to 180 days; finally, the microstructure of the mixtures was investigated. With increasing age, hydrated calcium silicate and calcium carbonate increased by 24% and 21.5%, respectively, and accordingly, the compressive strength (CS) and specific electrical resistances also increased by 10.3% and 48.6%, respectively, and the accelerated penetration of chloride ions decreased by 5.2%. In the limit of 30 micrometers from the aggregate boundary, due to the disturbance in the boundary region, no acceptable correlation relations between the ratio of different constituent elements and concrete properties were obtained. From the limit of 30 to 50 micrometers, linear relationships with coefficients of determination of 0.85 were established between the weight ratio of calcium to silicon with CS and the accelerated penetration of chloride ions, so that by reducing the said weight ratio by 82%, the CS increased by 10.3% and penetration decreased by 5.2%.

**Keyword:** Interfacial Transition Zone (ITZ), Microstructure, X-ray spectroscopy, Atomic ratio of elements, Scanning Electron Micrograph (SEM)

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## 1- Introduction

The transition zone in concrete acts as a bridge between the cement paste and aggregates, and the presence of weak bonds in this zone can reduce the overall stiffness of the composite material [1, 2]. The atomic ratio of elements in the transition zone can affect the strength of the microstructure and, consequently, the compressive strength and durability of concrete. Extensive research and experiments have been conducted to understand the effect of these ratios on concrete [3-5]. The transition zone in concrete causes many negative effects due to the high concentration of ettringite and calcium hydroxide crystals and the lack of hydrated calcium silicate adhesive gel [6]. The factor that causes the cohesion between aggregates and cement paste is not fully understood, but part of it may be due to the mechanical entanglement between them due to the roughness of the aggregate surface and the cohesion under the influence of the physical and chemical properties of the aggregates, mineralogy and electrostatic conditions of the aggregate surface [7, 8]. Understanding the correlation between microstructural features and physical and mechanical properties of the transition zone is important for evaluating deformation and cracking. Researchers have focused on determining and quantifying these properties to better identify damage mechanisms in concrete [9]. Various identification methods, such as physical signal processing, have helped to evaluate the pore structures and microscopic

features of the transition zone. However, the limited resolution and accuracy of these techniques may prevent accurate detection of mineral phases and various elements [10]. This study investigates the effect of the transition zone on the mechanical properties and durability of conventional concrete. By preparing a laboratory mix design with native materials and performing standard tests, the relationship between the strength of this zone and the laboratory results was measured. Also, the internal structure of the concrete and the elements of the transition zone were analyzed using electron micrographs and non-destructive tests.

## 2- Laboratory Program

In this study, ten mix designs were made with a water to cement ratio of 0.45. The used sand was of the broken type and was obtained from the mines of Andimeshk city in Khuzestan province. The maximum nominal size of coarse aggregates was 19 mm. The specific gravity and water absorption of coarse aggregates in saturated state with dry surface were determined according to ASTM C127 to be 2.36 g/cm and 1.1%, respectively. Details of the reference mixing plan are given in Table 1 and descriptions of 54 samples according to the laboratory program for determining the mechanical properties, durability and microstructure of concrete are given in Table 2.

**Table 1 - Concrete Mixing Plan Details**

Unit weight (kg/m <sup>3</sup> )	Concrete components	
	Cement	350
	Water	157
	Superplasticizer	1.8
	Natural sand (0-5)mm	971
	coarse aggregate (5-9.5)mm	305
	coarse aggregate (9.5-19)mm	567

**Table 2 - Related tests and standards**

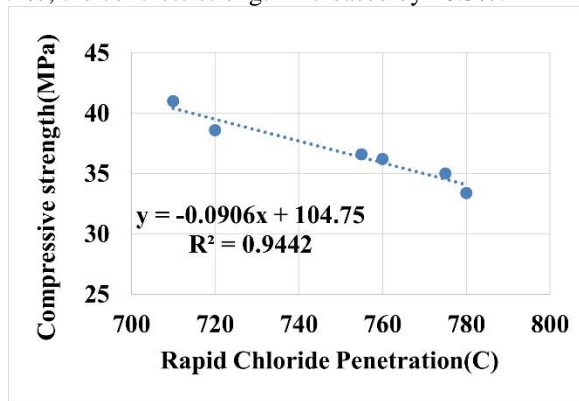
Test name	Standards and methods
Compressive strength	BS1881:Part 116
Static elasticity coefficient	ASTM C469
Water absorption for half an hour	BS1881:Part122
Long-term water absorption	ASTM C642
pressured Water penetration	EN BS12930-8
Rapid Chloride Penetration Test	ASTM C1202
Electrical resistance	AASHTO T398
Microstructure study of the transition zone (ITZ)	X-ray diffraction method(XRD)
	Scanning electron micrograph images (SEM)
	Energy dispersive X-ray spectroscopy (EDS)

To investigate the effect of the transition zone on the properties of concrete, tests were conducted on standard samples aged 7 to 180 days. X-ray diffraction (XRD) was used to analyze the crystalline phases at a distance of 0 to 50  $\mu\text{m}$  from the interface of aggregates and cement paste. After storage at 105 °C, the samples were analyzed in a vacuum chamber and the crystalline phases including CH, CSH, CaO, SiO<sub>2</sub> and CaCO<sub>3</sub> were identified. Also, the samples were coated with a layer of gold to increase the surface conductivity and microstructural images were obtained with a TESCAN Vega II electron micrograph. Finally, linear spectroscopic analysis was performed for different elements to investigate the quality of the transition zone and the relationship between the element ratios and the properties of concrete.

### 3- Analysis of Results

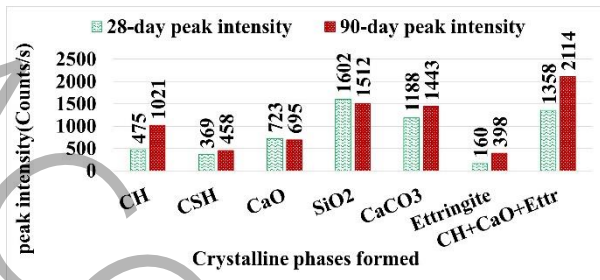
The test to determine the compressive strength of concrete was carried out at ages of 7, 28, 90 and 180 days according to BS1881:Part116. The results show that with increasing sample age, the strength increases and at 180 days it increases by about 13% compared to 28 days. Mineralogical compositions with silicon oxide

above 50% and the reaction of free silica with calcium hydroxide help to improve the transition zone. The elastic modulus increases faster than the strength at ages 28 to 90 days, which is related to the density of the transition zone. There is a linear relationship between strength and long-term water absorption with a correlation coefficient of 0.78, and at ages 28 to 180 days, with a decrease in water absorption of 31.4%, the strength increased by 18.3%. According to Figure 1, an inverse relationship was observed between chloride ion penetration and concrete strength with a correlation coefficient of 0.94; in the time interval from 28 to 90 days, with a decrease in chloride ion penetration of 5.2%, the concrete strength increased by 10.3%.

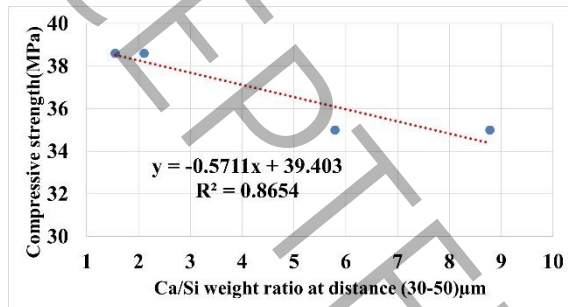


**Figure 1- changes Compressive strength of concrete in terms of accelerated chloride ion penetration**

Changes in the intensity of the peaks of hydration products at the ages of 28 and 90 days indicate a significant increase in the intensity of the CH and ettringite peaks by 2/15 and 2/49 times, respectively, due to the lack of use of microsilica. In Figure 2, the correlation of the intensity of the CSH and CaCO<sub>3</sub> peaks with the compressive strength indicates a direct relationship between these phases and the strength of concrete. An increase of 24% of hydrated calcium silicate and 21/5% of calcium carbonate led to an increase of 10/3% in the strength of concrete. The durability properties of concrete improved, and the electrical resistivity increased by 48.6% and the chloride ion penetration decreased by 5/2%. The atomic ratios of the transition zone elements affect the mechanical properties and durability of concrete. The weight ratio of Ca/Si increases with distance from the aggregate boundary up to a distance of 30  $\mu\text{m}$  and then decreases up to a distance of 50  $\mu\text{m}$ . The results obtained from the weight ratio of Ca/Si and changes in strength are presented in Figure 3.



**Figure 2- Phase diagram of concrete hydration products at 28 and 90 days**



**Figure 3- Changes in concrete strength depending on the Ca/Si weight ratio in the transition zone**

#### 4- Conclusions

The characteristic strength of concrete is limited to 35 MPa due to the strength of natural stone (more than 40) and failure occurs in the transition zone. With increasing sample age from 28 to 180 days, the immersion water absorption decreased by 31.4% and the compressive strength increased by 18.3%. An inverse relationship between chloride ion penetration and strength was observed at ages of 28 to 90 days with a correlation coefficient of 0.94; with a 5.2% decrease in chloride ion penetration, the strength increased by 10.3%. Examination of the intensity of CSH and CaCO<sub>3</sub> peaks showed that these phases have a direct relationship with the strength. An increase of 24% CSH and 21.5% CaCO<sub>3</sub> led to a 10.3% increase in strength; the static elastic modulus increased by 14.3% and the electrical resistivity increased by 48.6%, and the chloride ion penetration decreased by 5.2%. At 90 days of age, the Ca/Si ratio at the aggregate boundary is 5.6 times higher than that at the 10 μm distance, indicating more CH deposition at the aggregate boundary. At the (30-50) μm distance from the aggregate boundary, there is an inverse relationship between the weight ratio of Ca/Si and the strength, which decreases by 9.3% with an increase of 5.7 times the weight ratio; the accelerated infiltration rate increases by 5.5%; in addition, the weight ratio of Ca/(Al+Fe) fluctuates at the aggregate boundary up to the 30 μm distance and increases regularly from 0.2 to 0.37 at the (30-50) μm distance.

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