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Concretes Containing Hematite for Use as Shielding Barriers

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Heavyweight concrete is widely used for radiation shielding of nuclear reactors and other structures that require radiation impermeability. Aggregates play here an important role – while hematite and other iron ores are commonly used for the purpose. However, little information on properties of hematite-containing concrete other than radiation shielding data has been reported. We have studied effects of different concentrations of hematite (10 to 50 volume % at 10 % intervals) on physical and mechanical properties of concrete. A unique water-to-cement ratio of 0.42 kg/m^3 and 400 kg/m^3 cement content was selected. Addition of hematite increases the unit weight (density) so that a smaller thickness of concrete is required to provide radiation shielding. After 30 freeze-thaw cycles the plain concrete looses 21.3 % of its compressive strength while the composite containing 10 % hematite looses only 7.8 % of the strength. Concrete and hematite composites have lower drying shrinkage than plain concrete, thus lowering stresses resulting from the shrinkage.

Keywords: hematite, heavyweight concrete, radiation shielding, concrete aggregate.

1. INTRODUCTION

Concrete is considered to be an excellent and versatile shielding material; it is widely used for shielding nuclear power plants, particle accelerators, research reactors, laboratory hot cells and medical facilities. Concrete is a relatively inexpensive material, it can be easily handled and cast into complex shapes. It contains a mixture of various light and heavy elements and a capability for attenuation of photons and neutrons [1].

Various ways of reinforcing concrete are in use. Thus, fibers of various kinds [2 – 7], silica fume and fly ash [8] or $CaCO_3$ [9] as well as nanoparticles such as zinc and iron oxide [10] are added as a dispersed phase. A different option consists in making concretes with a polymeric matrix than can be strengthened further by irradiation and by inclusion of polymeric fibers [6, 8, 11, 12].

When protection from irradiation is required, a good candidate is a class of materials called heavyweight concretes, all inorganic. The commonly used types in this category are those based on barites (density 2.5 – 3.5 g/cm^3), magnetite (3.5 – 4.0 g/cm^3) and hematite (4.0 – 4.5 g/cm^3). Occasionally, even denser aggregates such as iron are incorporated. Density of those concretes can reach up to 5.0 g/cm^3 to enhance the shielding properties [13 – 17].

Apart from radiation shielding, other physical and mechanical properties of concretes are important issue in point of engineering properties. Thus, according to the Turkish Code, conventional concrete should be durable against harmful water, fluids and gases [18]. There are no such requirements for heavyweight concrete – unless it is used for protection against radiation [15 – 17]. But, structures like hospitals and nuclear power plant buildings etc. using these concretes must have some engineering properties like compressive strength, importantly durability and workability. According to Akkurt and coworkers [19], in building construction two features are the most important: resistance against earthquake represented by strength of the building and resistance against radiation expressed as radiation attenuation. Since environmental protection is now a significant issue, more efforts must be made to understand heavyweight concretes and their structural behavior [20].

Improvement of durability and extension of the service life of concretes is a worldwide problem [10]. A concrete radiation shield has both structural and shielding functions. Pertinent here is the workability of fresh concrete; it affects the compaction and therefore the density and strength of concrete. If segregation of components occurs in the composite, the loss of homogeneity affects negatively the properties. The main factor, which affects the workability of concrete, is the water content of the mixture [21].

There are several positive reports on radiation shielding properties of concretes containing hematite [22 – 24]. However, there is little information on physical and mechanical properties of these systems. In this situation we have investigated physical and mechanical properties of concrete + hematite materials, paying particular attention to workability and durability.

2. MATERIALS and METHODS

2.1. Aggregates

Given that aggregates typically constitute (70 – 80) wt. % of concrete, aggregate types and sizes play an essential role in modifying concrete properties. We have

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produced plain concrete (PC) using limestone-based aggregates with three different grain sizes: crushed stone (CSt-I), natural river stone (NRS) crushed stone II (CSt-II). The aggregates were obtained from Atabey, Isparta, Turkey, graded, washed and cleaned of clay and silts. To reduce difficulties of producing, mixing and placing of concretes and to prevent segregation of heavyweight aggregate in the fresh concretes, the maximum aggregate size was selected as 16 mm diameter. Results of sieve analysis of fine and coarse aggregates used are presented in Fig. 1.

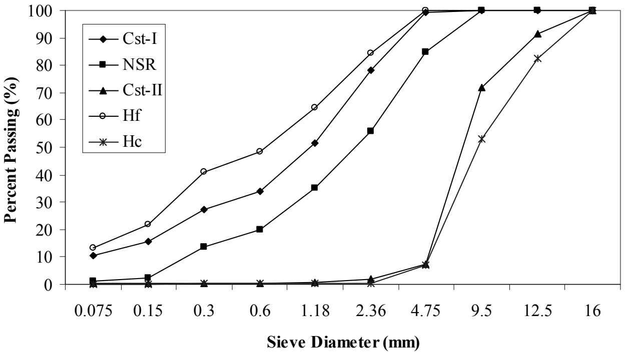

Fig. 1. Grading curves of aggregates

Unlike PC, the main feature of heavyweight concrete is the inclusion of metallic fillers – usually ilmenite, magnetite, hematite etc. In this study hematite was adopted as a replacement for concrete aggregates. It was obtained from Hekimhan region in Malatya, Turkey.

We recall that hematite, a natural red rock that contains iron oxide, when pure has the Mohs hardness between 5.5 and 6.5 and the specific gravity between 4.9 g/cm^3 and 5.5 g/cm^3. However, physical properties of rocks in which hematite is the main constituent may vary considerably; the specific gravity of hematite ores can range between 3.2 and 4.3. Some ores are soft and produce dust in the course of being handled, what would make them a poor aggregate for heavy concrete. Hematite particles tend to be flaky, which is undesirable in regard to the workability of concrete [21].

Hematite was prepared as aggregate by crushing and grounding the ore in a laboratory mill, then sorting it via sieves into two groups of coarse (H_c) and fine (H_f) aggregates. Specific gravity, water absorption and loose unit weight were determined according to ASTM C 127, ASTM C 128 and ASTM C 129 standards. Physical and mechanical properties of all aggregates are presented in Table 1. Chemical composition of hematite (Table 2) were determined by an inductively coupled plasma optical emission spectrometer.

Table 1. Physical and mechanical properties of aggregates

Aggregate codes	Specific gravity (g/cm^3)	Water absorption (%)	Loose unit weight (kg/m^3)	Fineness modulus
CSt-I	2.61	2.91	1913	3.8
NRS	2.63	3.13	1830	4.9
CSt-II	2.70	0.83	1676	7.3
Hf	3.75	2.35	1956	3.3
Hc	4.00	1.27	1733	7.8

Table 2. Chemical composition of hematite in weight %

Fe_2O_3	81.13
MnO	0.14
MgO	1.55
TiO_2	0.03
Al_2O_3	0.57
CaO	4.80
SiO_2	4.20
LOI*	5.82

*Loss of ignition.

2.2. Cement

The cement used in all the concrete mixtures was a normal Portland cement, which corresponds to CEM II/A-M (P-LL) 42.5N Portland cement. It fulfills the requirements of European Norms EN 196-1 code [25] and EN 197-2 code [26], which are adopted in Turkey as valid standards. The cement was manufactured at Goltas cement factory in Isparta, Turkey, and is suitable for use in heavyweight concrete. Physical and mechanical properties and chemical analysis of cement are presented respectively in Tables 3 and 4. We note more than doubling of the compressive strength between 2 and 28 days and nearly doubling the flexural strength in the same period.

2.3. Mix Proportions

To investigate the effect of heavyweight aggregate on the physical and mechanical properties of concrete, the mixture of both concrete using hematite and plain concrete were designed. Heavyweight concrete for radiation

Table 3. Physical and mechanical properties of Portland cement

Compressive strength (MPa)			Flexural strength (MPa)			Initial setting time (hour)	Final setting time (hour)	Le Chatelier (mm)	Specific gravity (g/cm^3)	Blaine (cm^2/g)
2 Days	7 Days	28 Days	2 Days	7 Days	28 Days	2.25	3.15	1	3.15	4150
22.5	36.6	47.8	3.7	5.6	6.9					

Table 4. Chemical analysis of Portland cement (weight %)

Total SiO_2	Al_2O_3	Fe_2O_3	CaO	MgO	SO_3	Cl	LOI*	Free CaO	Total Admixture
22.9	5.32	3.63	55.83	1.99	2.62	0	4.20	0.82	19.45

*Loss of ignition.

shielding can be proportioned using the ACI Method of absolute volumes developed for normal concrete [5]. The absolute volume method is generally accepted and is considered to be more convenient for heavyweight concrete [21]. Hence, the absolute volume method to obtain denser concrete was used in the calculation of the concrete mixtures.

After extensive trials, we have settled on the water/cement ratio = 0.42 and the cement content of 400 kg/m^3 for all mixtures. The American Concrete Institute Manual of Concrete Practices [27] advises not to use Type III cement nor accelerators so as to avoid high and rapid hydration heat and potential consequent cracking [18]. In heavy concrete the cement content is generally quite high, more than 350 kg/m^3. This helps to improve the shielding characteristics of the concrete because of the high bound water content of the paste [12 – 15].

We have used 10 % (H10), 20 % (H20), 30 % (H30), 40 % (H40) and 50 % (H50) of hematite aggregate to examine the effect of metallic aggregate instead of limestone-based aggregates (L). Because of high density of hematite, segregation is a danger. To avoid it, we have used the preplaced aggregate method. The weights and volumes of used materials in the final mix design to obtain 1 m^3 of concrete are displayed in Tables 5 and 6.

Table 5. Compositions of mixtures of heavyweight concrete

	PC	H10	H20	H30	H40	H50
Cement (kg)	400	400	400	400	400	400
Water (kg)	168	168	168	168	168	168
Water/cement	0.42	0.42	0.42	0.42	0.42	0.42
Air content (%)	1.5	1.5	1.5	1.5	1.5	1.5
CSt-I (kg)	445	391.17	347.70	304.23	260.77	217.30
NRS (kg)	447.5	387.40	344.37	301.30	258.27	215.23
CSt-II (kg)	920	826.43	734.60	642.77	550.97	459.13
H_f (kg)	0	121.57	243.13	364.67	486.23	607.80
H_c (kg)	0	129.37	258.77	388.13	517.53	646.90

Table 6. Volumes of aggregates in the mixtures in %

	CSt-I	NRS	CSt-II	H_f	H_c
PC	25.0	25.0	50.0	-	-
H10	22.5	22.5	45.0	5.0	5.0
H20	20.0	20.0	40.0	10.0	10.0
H30	17.5	17.5	35.0	15.0	15.0
H40	15.0	15.0	30.0	20.0	20.0
H50	12.5	12.5	25.0	25.0	25.0

2.4. Mixing, Curing and Testing Specimens

The procedure for mixing heavy concrete is similar to that for conventional concrete. In a typical mixing procedure, the materials were placed in the mixer with capacity of 56 dm^3 in the following sequence: first course aggregates, fine aggregates followed by cement, initially dry material mixed for 1 minute and finally addition of 80 % of water. After 1.5 minutes of mixing, the rest of the mixing water was added. All batches were mixed for a total time of 5 minutes; in order to prevent fresh concrete from segregation, the mixing duration was kept as low as possible.

For each mixture, a good workability and sufficient strength gain were achieved. Slump test (ASTM C 143) and air content test (ASTM C 231-04) of the fresh concrete were also performed for each mix, providing useful data to determine the amount of voids. Then all concrete specimens were cast in molds and the molds subjected to vibration. However, the high specific gravity of hematite is such that excess compacting vibration, which can cause segregation, must be avoided. After 24 h, the specimens were demolded and then cured in lime-saturated water at 20 ºC ±2 ºC temperature for 28 days prior to testing. It is well recognized that adequate curing of concrete is very important not only to achieve the desired compressive strength but also to make durable concrete.

Three series of specimens made from the above six mixtures were fabricated. The (150×150×150) cm cubic specimens were primarily used for compressive strength, Schmidt test and pulse velocity determination. Cylinders of 150 mm in diameter and 300 mm in height were used to determine the modulus of elasticity, stress-strain curves and splitting tensile strength. For shrinkage tests (TS 3453), prism specimens with (25×25×285) mm size were used and early shrinkage of concrete was tested after 28 days.

We have used test procedures: compressive strength according to ASTM C 39-86, splitting tensile strength according to ASTM C 496-87, modulus of elasticity following ASTM C 469-87 (secant method) and_ACI 318-92 (Eq. (1) below) with European Concrete Committee (CEB) (Eq. (2) below). Freeze-thaw durability (FTCs) were performed according to ASTM C 66. We have subjected all our specimens to 30 FTCs, 2 hours freezing and 1 hour thawing. Schmidt surface hardness was determined according to EN 12504-2 and pulse velocity according to ASTM C 597-02. The last two tests were performed for cubic samples with the side length of 15 cm. We have

$$E = 0.043\, W^{3/2}\, \sigma^{1/2}\ ; \qquad (1)$$

$$E = 9500\, (\sigma + 8)^{1/3}\ ; \qquad (2)$$

here E is the modulus of elasticity in MPa; W is the unit density in kg/m^3; σ is the compressive strenghth after 28 days in MPa.

Polarization microscope and image analysis system with a 5.1 megapixel digital camera, objective by 4×, 10×, 20×, 100× and 15× ocular lens were used for petrographic and ore microscopy, bottom and top illumination, for thin and bright sections.

3. PHYSICAL PROPERTIES of CONCRETES

Along with physical properties of concrete, we have also studied hematite as a mineral. Fig. 2, A, shows gradually increasing redness of the samples with increasing hematite concentration – an expected effect. This is because of those intensive Ho parts of hematite aggregates, which desquamated transferred to the matrix during the process of mix. All mixtures have shown a wide homogenized distribution of aggregates. This is due to great gradation of aggregation and mixing process. Although all aggregates were covered by the matrix (M), it can be seen that aggregates composed a form, in which

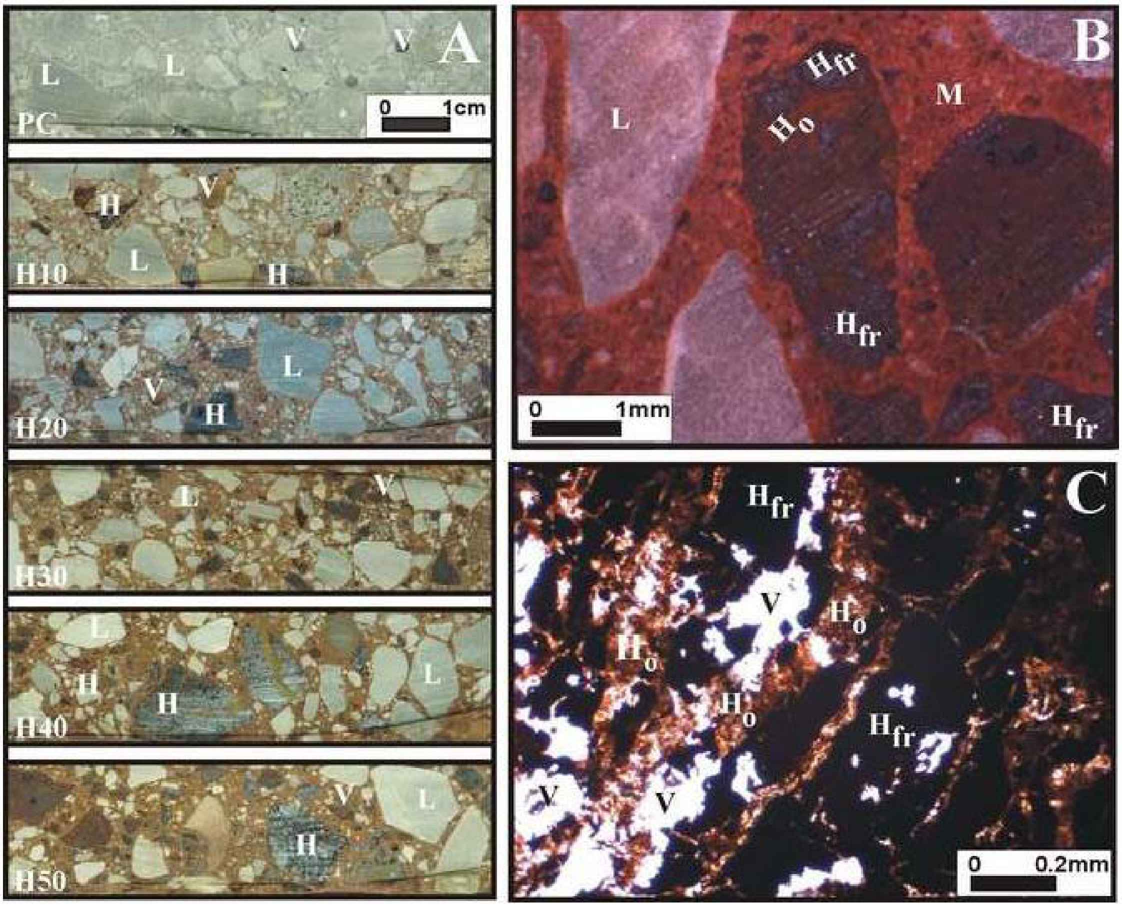

Fig. 2. Cross-section views of concretes

aggregates weren't touchable or close to each other. This may be interrupted like that the concrete is the matrix-based, not aggregate-based in respect of load bearing.

We show in Fig. 2, B, a cross-section of concrete seen in a stereo microscope and in Fig. 2, C, a thin section seen through a polarizing microscope.

We find in Fig. 2, B, that our hematite consists of two zones, oxide (H_O) and fresh (H_{fr}). Hematite aggregates have high porosity (Fig. 2, C) – a consequence of voids (V) which have appeared during formation of hematite. We also note that hematite has the form of flakes.

As already noted in Section 1, workability is essential for strength and durability after hardening. However, workability includes mixing, transporting, placing and segregation of freshly mixed concrete and there is no single test to evaluate workability. A widely used one is the so-called slump test. The slump test, which is simple, quick and cheap, is almost universally used for nearly all types of medium and high workability concrete. It measures a flow property of concrete under self-weight after standard compaction. And it is sensitive to small changes in water content. In this test, there are there steps of its application. First, the cone is filled with concrete in three equal layers, and each layer is compacted with twenty-five tamps of the tamping rod. Second, the cone is slowly raised and the concrete is allowed to slump under its own weight and finally, the slump is measured using the upturned cone and slump rod as a guide. And the slump value of 50 mm is considered a minimum acceptable [28]. Results of the slump testing are presented in Fig. 3.

Fig. 3 tells us that all materials studied including the plain concrete (PC) without hematite fulfill the condition of minimum 50 mm. We also note that the effect of hematite addition is periodic and not very strong.

Slump value depends on the density values of the aggregate, cement dosage and water/cement ratio. The re-rise of slump at 50 % replaced by hematite accepting segregation in the beginning turned out to be a good approach. Using plain concrete as the reference, the changes caused by addition of hematite are as as follows: 6.85 % for H10, 5.48 % for H20, –1.37 % for H30, –1.37 % for H40 and 5.48 % for H50.

We now consider the issue of air content in concretes. Although the slump test provides some idea about uniformity of concrete, the air content test does that also.

Concrete consists of a graded mix of aggregate particles in a cement paste matrix and the cement paste consists of unhydrated cement, hydration products and the residue of the water-filled space which gives rise to capillary porosity. Capillary pores are up to 1 µm in diameter, whereas gel pores are around 2 nm. Concrete may also contain entrained air, entrapped air and other voids. Intentionally entrained air voids are bubbles typically 0.1 mm in diameter and are distributed evenly throughout the cement paste. Accidentally entrapped air usually forms very much larger voids, often up to several millimeters in diameter. This will typically account for 2 per cent of the volume of the concrete [29]. Differences between air content values determined should not exceed 1 % to be able to accept concrete with uniformity [30]. The results are presented in Fig. 3.

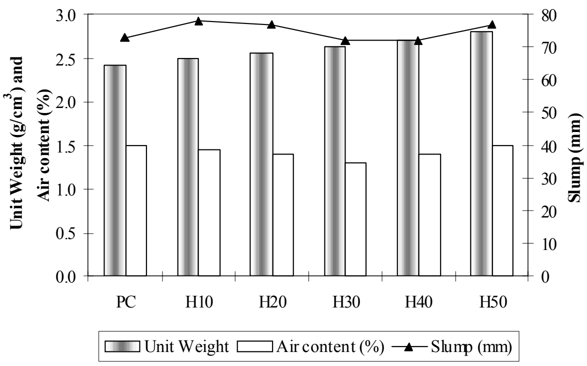

Fig. 3. Comparison of slump values, unit weight and air contents of fresh concretes

As shown in Fig. 3, air contents slightly decreased up to 40 % of hematite replaced. The reason may be more compact packing of concretes since the unit weight of increased. However, at 50 % hematite, air content has increased; apparently the aggregate carrying capacity of mortar matrix has reached the maximum and the beginning of segregation takes place.

Return now to the microscopy result shown in Fig. 2, A. We see a reasonably uniform distribution of the dispersed phase in the matrix. Thus, microscopy agrees with our inference from Fig. 3.

4. MECHANICAL PROPERTIES

4.1. Unit weight

The probability of an incoming photon interacting with a given material per unit path length is usually represented by the linear attenuation (also called linear attenuation coefficient) – clearly pertinent for radiation shielding. The attenuation depends on the density of the material [31]. Thus, unit weight of concretes is important. We have determined unit weights and present the results in Fig. 4.

Since hematite has higher density than plain concrete, addition of hematite increases the unit weight – an expected but also desired result. The higher the density, the smaller the thickness of concrete is required to provide radiation shielding.

4.2. Compression strength

There is no need to argue that the compressive strength is the most important property of concrete. It was expected that addition of hematite – a material with higher density and higher hardness than cement – will increase the compressive strength. The results are presented in Fig. 4.

We see in Fig. 4 that hematite increases the compressive strength of plain concrete for 10 % hematite and only slightly for 20 % hematite. The reason behind it may be the porosity of hematite discussed above in connection with Fig. 2. The more hematite we have, the more pores inside of hematite regions will appear. Using plain concrete again as the reference, changes in the compressive strength are: 4.33 % for H10, 0.48 % for H20, –1.77 % for H30, –2.57 % for H40 and –2.41 % for H50.

4.3. Splitting tensile strength

The results are presented in Fig. 4. Effects of hematite addition are not large. Aggregate quality rather than mortar matrix is important for the splitting tensile strength test results. At 10 % hematite, splitting strength is lower with respect to plain concrete because of larger voids between aggregates. Splitting strength is increasing because gap between aggregates is decreasing at 20 % – 30 % replaced of hematite. At 40 % – 50 % hematite, even thought gaps between aggregates are smaller, splitting strength values are significantly reduced because more weak points appear – due to oxidation at mortar-aggregate interfaces. Splitting strength with respect to PC decreases as follows: 8.78 % for H10, 4.26 % for H20, 3.52 % for H30, 35.35 % for H40 and 34.97 % for H50.

4.3. Elastic modulus

The modulus of elasticity *E* values were determined after 28 days. A strain-gage with the sensitivity of 0.002 designed for cylindrical specimens was used.

E modulae were obtained from $\sigma(\varepsilon)$ curves. The results are shown in Fig. 5.

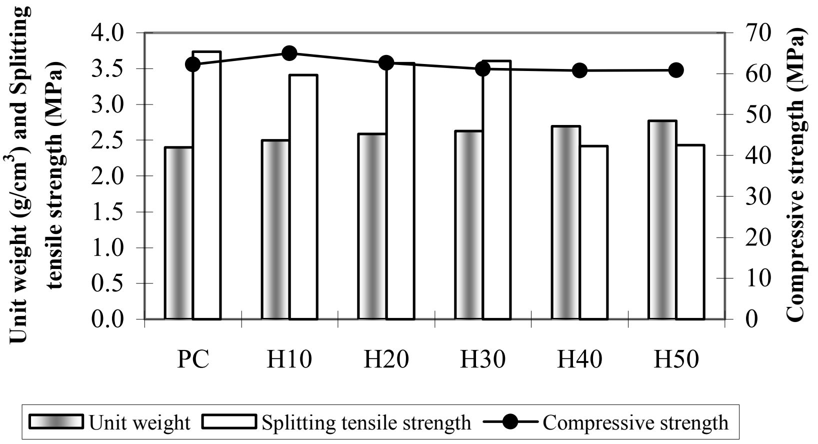

Fig. 4. Comparison of unit weight compressive and splitting tensile strengths of hardened concretes

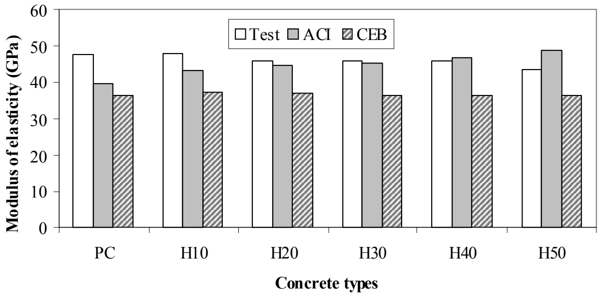

Fig. 5. Variations of concrete *E* modulae with hematite content

E modulae as a function of concentration of hematite behave similarly to compressive strength when we use the CEB method. However, the ACI method provides oppposite results. This is due to the fact that the ACI method takes into account compressive strength as well as the unit weight.

We note differences between the three methods used. Within each method, the effects of hematite addition are not large. In the first method the range of *E* values is between 43 GPa and 48 GPa.

4.4. Pulse velocity

The experimental results of pulse velocity for different types of concrete are presented in Fig. 6. Pulse velocity values as a function of concentration of hematite increase due to porosity of hematite; the effect is smaller that for Cst-I, Cst-II or NSR.

As seen in Fig. 6, pulse velocity values range from 4600 m/s to 5100 m/s. The PC had the highest value and addition of hematite decreases the velocity. Long ago Whitehurst [32] classified the concretes as excellent, good, doubtful, poor and very poor for pulse velocity values of 4500 m/s and above, 3500 – 4500, 3000 – 3500, 2000 – 3000, and 2000 m/s, respectively. Thus, all our concretes produced are excellent according to the Whitehurst classification.

4.5. Schmidt hardness

The Schmidt hardness test is a popular nondestructive method [33 – 35]. A uniform compressive stress of 2.5 MPa is applied to the test specimen along the vertical direction (the same as the casting direction) before striking it with a hammer; this prevents dissipation of the hammer striking energy due to lateral movement of the specimen.

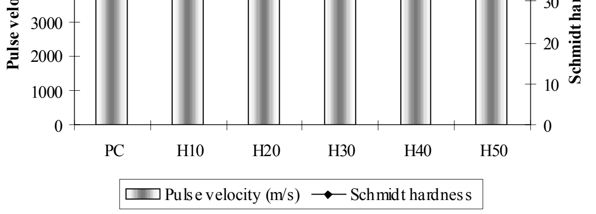

Fig. 6. Comparison of Schmidt hardness and Pulse velocity results of concretes

Striking points were uniformly distributed to reduce the influence of local aggregates distribution and averages of the rebound energy calculated. The results are presented in Fig. 6.

Schmidt hardness is a method related to compressive behavior since it is based on the rebound ratio from surfaces of samples. Therefore, similar behavior is expected as in Fig. 4. Fig. 6 shows similar behavior patterns as Figure 4, except for H10 in Figure 4. Thus, the Schmidt hardness values decrease when hematite is added to the PC. The effects are small.

4.6. Freeze-thaw durability

Micro-cracks mainly exist at cement paste-aggregate interfaces within concrete even prior to any loading and environmental effects. When the number of freeze-thaw cycles (FTCs) increases, the degree of saturation in pore structures increases by sucking in water near the concrete surface during the thawing process at temperatures above 0 °C. Some of the pore structures are filled fully with water. Below the freezing point of those pores, the volume increase of ice causes tension in the surrounding concrete. If the tensile stress exceeds the tensile strength of concrete, micro-cracks occur. By continuing FTCs, more water can penetrate the existing cracks during thawing, causing higher expansion and more cracks during freezing. The load carrying area will decrease with the initiation and growth of every new crack. Necessarily the compressive strength will decrease with FTCs [36]. The results of Freeze-thaw durability tests are presented in Fig. 7.

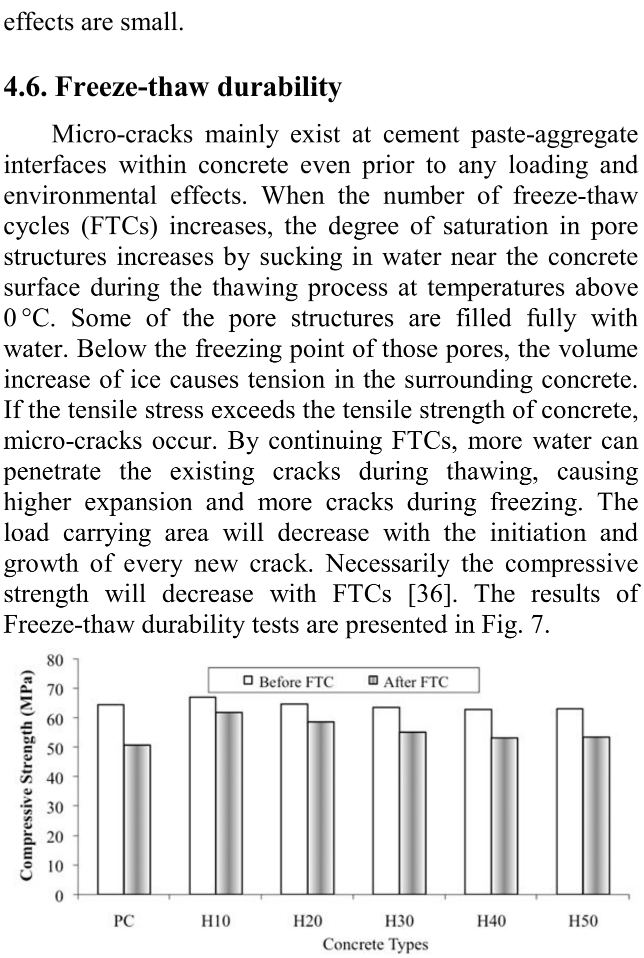

Fig. 7. Freeze-thaw durability of concretes

We see in Fig. 7 that all concrete types had lost strength in cycling. However, for hematite containing materials the losses are lower. Apparently, specimens containing hematite absorb less water and are thus less affected by FTCs. The strength loss for PC amounts to 21.3 % while for the H10 composite only 7.8 %. Still, the effect in pure PC is acceptable according to the ASTM C 666 code.

4.7. Shrinkage

Drying shrinkage is by far the major portion of volume change of concrete [37]. Although several types of volume change due to moisture movement can occur in concrete, volume change due to drying shrinkage is particularly important in radiation-shielding concrete. Stresses resulting from drying shrinkage cause cracking. Although cracking tends to be largely a surface effect, large cracks could affect the effectiveness of the radiation

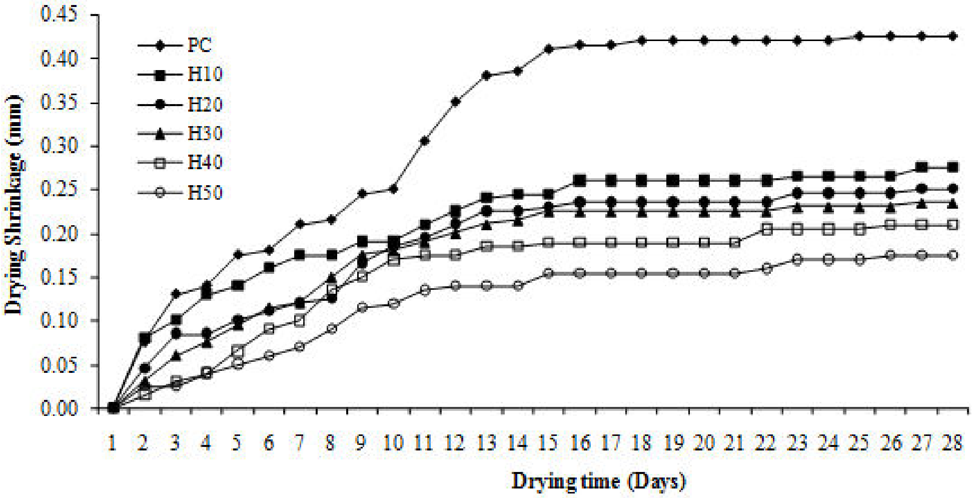

Fig. 8. Shrinkages of concretes

shield while extensive microcracking would reduce the effective density of the shield [21]. We have obtained the appropriate results which are presented in Fig. 8.

We see in Fig. 8 that after 15 days or so the drying process is largely completed.

And by dependent on hematite content in concrete, shrinkage has decreased as PC (0.43) > H10 (0.10) > > H20 (0.09) > H30 (0.08) > H40 (0.07) > H50 (0.06).

Finally, we recall we have not used a plasticizer – which would reduce the volume of voids and thus also enhance the unit weight and other desirable properties.

5. CONCLUSIONS

We have found that there is only a minor effect of hematite added to concrete on its essential properties. Mechanical properties, especially the compressive strength of concrete with hematite, do not differ from those of plain concrete. Using PC as the reference, compressive strength by 4.33 % – 0.48 % by replacement with hematite. At the 40 % – 50 % replaced, weak points is increasing due to oxidation in mortar-aggregate interface.

Concretes with hematite have sufficient workability. With respect to the PC, the slump value changes by 1.37 % – 6.85 % as a consequence of replacement with hematite. Unit weight of concrete for shielding increases with replacement ratio of hematite to concrete. Water content affects unit weight while the volumetric concentration of voids is related to water content. Voids can also be decreased by an increase in cement dosage. However, at 50 % hematite – again as pointed out in Section 3 – there is beginning of segregation. Freeze thaw durability of concrete with hematite is sufficient in terms of compressive strength according to ASTM C 666 code. It should be considered that because hematite includes high iron ore volume, it tends to undergo undesirable oxidation. Shrinkage values for hematite containing concrete have been reduced by increasing hematite ratios in concrete. Concrete + hematite composites have lower drying shrinkage than plain concrete, thus lowering stresses resulting from the shrinkage.

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