# Sensitivity of Brittleness of The Concrete To The Pore Structure of The Cement Paste



M.S.YATAGAN ISTANBUL TECHNİCAL UNIVERSITY, Taskisla Street.Taksim/Istanbul/Turkey serkanyatagan@ttnet.net.tr

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#### ABSTRACT

One of the essential factors which affects the performance of the concrete and reinforced concrete buildings under earthquake loads is the degree of brittleness shown by the concrete, essentially a quasibrittle material, against these effects. It is possible to investigate the brittleness of the concrete directly by dynamic experiments, as well as the static experiments which make it possible to get some numerical values for brittleness. In this work, the second way is choosen and the brittleness of the concrete is expressed as the ratio between the compressive strength and the tension strength of the concrete and the brittleness index.

In this work, the relationship between the brittleness and the grading of the aggregate mixture and the pore structure of the cement paste is investigated; the degree of sensitivity of the values determining the brittleness to the pore structure of the cement paste is also determined; then the relations between these values are found, and the factors determining the correlation constants of these relations are investigated.

The maximum size of aggregate used in concrete is 16 mm. The grading is held within the permitted limits. The cement content is constant as 350 kg/m3, whereas water/cement ratio changes between 0,50 and 0,70. Aggregate is normal concrete aggregate and the cement is Portland Cement 32,5.

These results are obtained from the investigation; (i) While the water/cement ratio increases, the unit weight, the tensile strength, the compressive strength, the brittleness index and the modulus of the elasticity decreases. (ii) The grading of the aggregate does not effect the unit weight and the brittleness index pratically. On the other hand, the tensile strength and the compressive strengths and the modulus of the elasticity have the lowest values compared to the others when the grading is between B16-C16. This situation is probably due to increased air content. (iii) While the compressive strength increases, the brittleness index and the ratio between the compressive strength and the tensile strength increases. It is said that while the strength increases, the concrete becomes more brittle.(iv)The correlations between properties are strong because the sensitivities of these properties to the pore structure of the cement paste are close.

# **KEYWORDS**

Brittleness, correlation coefficents, the pore structure

### **1.INTRODUCTION**

All concrete properties are related to the inner structure of the cement paste. In the modern engineering, understanding the effect of the inner structure of the cement paste or how the materials of the concrete change the inner structure of the cement paste or the effects of the interface between the cement paste and the aggregates is important that concrete is produced according to the requested conditions. The inner structure has an essential place for the determination the properties of the concrete because the properties of the concrete depend on the reactions of the hydration occurring in the cement paste.

From the past to the present, within the help of the developing technology, the inner structure of the cement paste is continuously researched. The first researches of the inner structure of the cement paste are the crystallization theory of Le Chatelier and the Michealis' colladial theory [1]. Powers and his friends proved that as a result of the cement hydration, the fibres like Labermant, forms in the cement paste and these fibres are in colladial dimension and they have a great inner surface. Also, the structure of these fibres is gel. Besides this gel, in the cement paste, there are calcium hydroxide crystals, the secondary components, unhydrated cement particles and the pores. These pores are surrounded by the gel. Since the size of these pores is capillary, they are called as capillary pores [2].

# 2.THE STRUCTURE OF THE CEMENT AND THE CEMENT PASTE

There are 4 main components of the cement;  $C_2S$ ,  $C_3S$ ,  $C_3A$  and  $C_4AF$ . After the hydration, when the cement is mixed with water, these components turn into the hydrated components. The hydrated components are C-S-H (Calcium Silicate Hydrate) and CH (Calcium Silicate). C-S-H supply the binding property of the cement.

The inner structure of the cement paste is porous. The pores form as a result of the hydration reactions because of the formed hydrated products. These pores are classified as the capillary and the gel pores. The capillary pores are formed after the hydration when used water evaporates. The capillary porosity depends on the water/cement ratio and the hydration degree. As the hydration degree increasaes, the more hydrated products are formed and they fill the pores so the numbers of the pores decrease. However, when the water/cement ratio increases, it is impossible that the hydrated products fill the pores because the amount of the water increases and then the numbers of the pores increases, too so the formed hydrated products are not enough to fill these pores. The gel pores are in the hydration products and the numbers and the total volume of the gel pores increase within the the hydration process. The dimension of the gel pores is smaller than the dimension of the capillary pores. Besides these pores, there are air voids, the dimension of which is more smaller . The air voids are formed because of the insufficient settlement of the concrete or some additives which drag air in the concrete.

#### 2.1 The inner structure of the interface between the cement paste and the aggregate

The interface between the cement paste and the aggregate is called as the weakest chain in the concrete. The inner structure of the interface between the cement paste and the aggregate differs from the inner structure of the cement paste. There are various models for the micro structure of the interface.

According to Barnes;

- 1. A film of CH acculumates vertically on the aggregates.
- 2. A film of C-S-H particles, like a hair brush, covers the aggregates.
- 3. Parallel to the aggregate surface, extensive CH crystals acculumate.
- 4. At the inteface zone, full volume formation of CH is seen.

According to Zimbelmann[3]; when the cement paste is mixed with water, in a few minutes, the etthrengite needles are formed. CH crystals vertically cover the etthrengite needles. The thickness of the level of CH crystals is 2-3 micron. After this level, hegzagonal CH crystals begin to develop. The dimension of these hegzagonal crystals changes between 10 and 30 micron.

At the interface, the porosity is high so the interface is weak and permeable. The thickness of the interface changes between 10 and 50 micron. The interface comprises the huge part of the concrete.

There are some ideas for the formation of the interface. During the mixture of the concrete, the aggregate particles are covered by a film of water. When the hydration starts, forming hydration products connect with this film of water so the water/cement ratio of the interface is higher than the original water/cement ratio.

## **3. THE PORE STRUCTURE OF THE HARDENED CEMENT PASTE**

In the fresh cement paste, the Portland cement paste is fluidy. However, in the hardened paste, there are hard hydration products and the pores. The hydration products form high dense masses (the gel pores) and they have C-S-H. Besides, there are capillary pores and unhydrated cement particles. The total porosity inolves the capillary and the gel pores. The dimension of these pores is microscopic and the gel pores are smaller than the capillary pores. The physical differences of the cement pastes depend on the difference between the capillary pores.

## 3.1 The water in the hardened cement paste

There are 3 types of water in the hardened cement paste[4],[5],[6];

1. The chemical water is an integral part of the cement gel. This water is not evaporated.

2. The pyhsically absorbed water is absorbed from the surface of the gel particles. The absorption is the surface reactivity of the gel. This water fills the gel pores.

3. The free water is the left water in the saturated paste and fills the capillary pores.

The physically absorbed water and the free water are able to evaporate.

#### **3.2** The properties of the pore structurte of the hardened cement paste

The total porosity decreases during the hydration process for the all water/cement ratios. At the first periods of the hydration, the decreasing ratio of the total porosity is high, but in 3 months, the ratio is linear. Also, the total porosity increases when the water/cement ratio increases because an increase in the amount of water between the unhydrated particles. The dimension of the distribution of the all pores gets better within the age of the hardened cement paste [7].

# **3.3** The effect of the pore structure of the cement paste on the mechanical properties of the concrete

The porosity is the most important factor which specifies the strength of the concrete. The radius of the pores which is smaller than 10 nm decreases the strength of the concrete. The constant porosity increases within the increasing amount of the hydration products.

# 4. THE QUASI-BRITTLENESS OF THE CONCRETE

Aitcin and Mehta proved that the thickness of the loading-unloading curves specify the strength of the the interface between the aggregate and the cement paste [8]. The concretes having the same water/cement ratios and different granulumetries give different results. For example, in the experiments, the elasticity modulus and the compressive strength of the granulumetry between A16-B16 are the highest, whereas the granulumetry between B16-C16 is the lowest. The compressive

strength results are shown in Figure 4.1. The right curve is for the granulumetry between A16-B16 and has the highest compressive strength.



Figure 4.1 The curves of the granulumetries between A16-B16, B16, B16-C16

The increasing thickness of the loading-unloading curves reveal that the bonds of the interface are weak. Also, according to the loading-uploading curves, when the compressive strength increases, the brittleness index increases. With the increasing strength of the concrete, the interface become unporous and homogen.

# 5. THE SENSITIVITY OF THE PROPERTIES OF THE CONCRETE TO THE PORE STRUCTURE OF THE CEMENT PASTE

If one of the properties of the concrete such as the compressive strength, the tensile strength or the elasticity modulus, only depends on the amount of the pores in the cement paste, the property of the concrete is insensitive to the pore structure of the ceement paste. If the type (or the geometry) of the pores also specifies the property of the concrete, the property of the concrete is sensitive to the pore structure of the correlation coefficients are useful to understand whether the property is sensitive or not sensitive to the pore structure of the cement paste.

# 5.1 The sensitivity of the properties of the fresh concrete to the pore structure of the cement paste- the sensitivity coefficient

If  $P_{fi}$  is one of the properties of the fresh concrete such as the unit weight or the slump and  $n_i$  is a parameter, the values of  $n_i$  are calculated with the obtained values of  $P_{fi}$  from the experiments according to the formulation;

 $n_i.w + a$  w: the amount of water in the fresh concrete a: the percentage of the air voids

 $n_i = n_i^*$ , the maximum correlation coefficient is calculated. In the Figure 5.1, the y-axis shows the values of  $P_{fi}$  which is obtianed from the experiments, on the other hand the x-axis shows the values of  $n_{i.w} + a$  which are calculated according to the values of  $P_{fi}$ . The graphic is drawn according to these values and then the peak point of the graphic is accepted as the maximum correlation coefficient of the property of the fresh concrete. Actually, after the graphic is drawn in Excel, the regration coefficient is found in the tendency of the graphic in Excel and then the square root of the regration coefficient of the graphic  $(\sqrt{R^2})$  is accepted as the maximum correlation coefficient  $(n_i^*)$ .

If  $n_i^* = 1$  The property is insensitive to the pore structure of the cement paste

 $n_i^* \neq 1$  The property is sensitive to the pore structure of the cement paste

Whether if the properties of the hardened concrete is sensitive or not,  $n_{i}$ .(  $w - 1,06.\alpha.c$ ) + a is used instead of  $n_{i}.w$  + a to consider the hydration of the cement paste. w is the amount of the water in the concrete,  $\alpha$  is the degree of the hydration, c is the amount of the cement (the doasge of the concrete) and



Figure 5.1 The relationship between P<sub>fi</sub> the property of the fresh concrete and n<sub>i</sub>.w + a

a is the percentage of the air voids in the concrete. The same processes are done to calculate the maximum correlation coefficient of the property of the hardened concrete.

Another way of calculating the maximum correlation coefficient is comparing with the unit weight of the concrete. The degree of the hydration is hard to be found so an approximate method should be used. The dry unit weight of the concret is calculated in the experiments. The unit weight is insensitive to the pore structure of the cement paste since the effect of the pores on the unit weight is just related to the volume of the pores, not the geometry and the dimension so  $n_i^* = 1$  for the unit weight. Therefore the formulation  $n_i.(w - 1,06.\alpha.c) + a$  changes into  $(1-1,06.\alpha.c/w).w + a$ . However, it is hard to calculate the correlation coefficients with these variants so the formulation is simplified as k.w + a. The sensitivity of the unit weight should be investigated according to this formulation and the maximum correlation coefficient (k=ko) of the unit weight is calculated. Moreover, k.w + a is used to investigate the sensitivity of a property of the hardened concrete and then a maximum correlation coefficient of the property of the hardened cement (k=ki<sup>\*</sup>) is calculated. Here, ko and ki<sup>\*</sup> are accepted as the central tendency parameter. The relationship between ko and ki<sup>\*</sup> determines the sensitivity of the property of the hardened concrete compared to the sensitivity of the dry unit weight.

If ko = ki<sup>\*</sup> The property is affected by the pore structure of the cement paste like the dry unit weight so the property is insensitive.

If  $ko \neq ki^*$  The property is sensitive.

# 5.2 The first type degree of the sensitivity of the properties of the concrete to the pore structure of the cement paste- (SD1)i

When the first type degree of the sensitivity of one of the property of the hardened concrete to the pore structure of the cement paste is investigated, the ratio of ki<sup>\*</sup> and ko is calculated [(SD1)i = ki<sup>\*</sup>/ko]. The respect of some reseraches, (SD1)i is close to the sensitivity coefficient,  $n_i^*$ .

# 5.3 The second type degree of the sensitivity of the properties of the concrete to the pore structure of the cement paste-(SD2)i

When the second type degree of the sensitivity of one of the property of the hardened concrete to the pore structure of the cement paste is investigated, the ratio of  $R(ki^*)$  and Ri(ko) is calculated.[(SD2)i =  $R(ki^*)/R(ko)$ ]. Here,  $R(ki^*)$  is the maximum correlation coefficient for the property of the hardened concrete which is calculated from k.w +a. R(ko) is the maximum correlation of the property of the hardened of k in k.w +a. The second type degree of the sensitivity is not more affected by the function of the inner structure so it is necessary to research the correlations between the properties in order to understand whether the secon type degree is significant or not.

### 5.4 The factors of the correlations between the properties of the concrete

The sensitivity similarity coefficient; tij = (SD)i/(SD)j and tij < 1. Here (SD)i is 1. or 2. type degree of the sensitivity of  $P_{hi}$ ,one of the property of the hardened concrete and (SD)j is 1. or 2. type degree of the  $P_{hj}$ , another property of the hardened concrete. tij changes between 0 and 1. If tij is close to 1,  $P_{hi}$  and  $P_{hj}$  are similar for the sensitivity to the pore structure of the cement paste.

There are other relationships of the similarity of the different sides between the correlations and the properties. The above formulation helps to understand the other relationships [10].

$$(P_{hi})' = A(c + w + a)' + B(c/[k.w + a])' + C(m)' + D$$

Here ()' are the ratio of the average in the serie of the variants in the paranthesis and how  $P_{hi}$ , the property of the hardened cement is affected by relatively changes in the volume of the cmenet paste, the structure of the cmenet paste and the granulumetry of the aggregates. In order to understand the similarity of the coefficients A, B and C of two different properties of the hardened concrete, the slope of the coefficients should be calculated in T(A)ij = |Ai-Aj|, T(B)ij = |Bi-Bj|, T(C)ij = |Ci-Cj|. If T is more bigger, A, B and C are too different.

# 6. EXPERIMENTS

The Portland Cement 32,5 is used and the specific gravity of the Portland Cement is 2,97 gr/cm<sup>3</sup>. The dosage of the concrete is  $350 \text{ kg/m}^3$ . The aggregates are broken stone 1, the sand and the stone powder. the specific gravity of the aggregates are relatively 2,72 gr/cm<sup>3</sup>, 2,60 gr/cm<sup>3</sup> and 2,70 gr/cm<sup>3</sup>. For the granulumetry between A16-B16, the mixture ratios are 70 % the broken stone 1, 20 % the sand and 10 % the stone powder. For B16 granulumetry, the mixture ratios are 65 % the broken stone 1, 25 % the sand and 10 % the stone powder. For the granulumetry between B16-C16, the mixture ratios are 60 % the broken stone 1, 30 % the sand and 10 % the stone powder. For every granulumetry, water/cement ratio begins at 0,50 and it is increased by 0,05 to 0,70. 15 types of the concrete are produced. The beginning value of the water/cement ratio is determined with the help of the VeBe experiment. The water/cement ratio of the fresh concrete which gives 7-8 sn in VeBe machine is accepted as the original water/cement ratio. For one water/cement ratio of every granulumetry, 150\*150 mm 4 cubes, 150\*300 mm 8 cylinders and 60\*150 mm 6 discs are produced.

After 24 hours, the samples are taken out from the moulds and then the samples are held in  $23+2^{\circ}C$ water for 21 days. At 21 day, the samples are taken out from the water and waited in the room to the 28.day. At 28.day, the compressive strengths of the cylinders and the splitting strengths of the discs are measured. The cubes are held in 105°C oven for 4 hours at 35.day and they are weighed and the dimensions of the cubes are measured in order to calculate the dry unit weight. The discs are used to measure the splitting strengths. The capacity of Amsler pressure instrument for the splitting tests is 500 kN. To distribute the load uniformly on the discs, two laths are mutually put on the upper and the lower part of the discs. The half of the cylinders are used to measure the compressive strengths and the capacity of Amsler pressure instrument for the compressive strength is 1000 kN. The speed of the loading is constant and for 2,5 tone, longitunal deformations are measured with the help of a frame. The elasticity moduluses are calculated within the help of these longitunal deformations. The other half of the cylinders are used to obtain the brittleness indexes. Firstly, the samples are loaded to a certain value and then the samples are unloaded. After unloading, the samples are loaded to the fracture. During this process, longitunal deformations are measured. The certain value is theoratically 80-90 % of the compressive strength of the sample. The graphic is drawn according to these results are shown in Figure 6.1. In Figure 6.1, S<sub>1</sub> is the permanent deformation energy related to the damage and  $S_{\rm II}$  is the elastic deformation energy. The x-axis shows the longitunal deformation (mm) and the y-axis shows the strength of the samples (MPa). The brittleness index is the ratio between  $S_I$  and  $S_{II}$  ( $S_{II}/S_I$ ).



Figure 6.1 The strength-deformation curves of the repetition loading of the concrete

In Table 6.1, the brittleness indexes obtained from the graphics of the tests. The brittleness indexes decrease when the water/cement raito increases. For the lowest water/cement ratios, the compressive strength and the brittleness index are high so it means that the concrete becomes brittle.

The granulumetries	The water/cement ratios						
	0.50	0.55	0.60	0.65	0.70		
A16-B16	3.65	2.90	2.71	2.40	1.70		
B16	3.21	2.56	2.37	2.30	1.93		
B16-C16	2.80	2.52	2.36	2.29	1.68		

Table 6.1 The brittleness indexes related to the changes of the water/cement ratios

By using the results obtained from the tests, in the formulations A(k.w + a) + B, A(c/k.w + a) + B, A(c/k.w + a) + B(c/k.w + a) + C and A(c + w + a) + B(c/k.w + a) + C(m) + D, the correlation coefficients of the properties of the hardened concrete are calculated for every formulations. In Table 6.2, the correlation coefficients of some properties of the concrete obtained from the formulation A(k.w + a) + B are seen.

Property	ki*	(SD1)i	R(ki*)	R(ko)	(SD2)i
Cylinder Compressive Strength-MPa	1,04	0,301	0,9715	0,9321	1,04
Dry unit weight-kg/cm <sup>3</sup>	3,46	1,000	0,8924	0,8924	1,00
Splitting Strength-MPa	0,99	0,286	0,8150	0,7758	1,05
Brittleness Index	1,38	0,399	0,9347	0,9192	1,02
Elasticity Modulus-MPa	1,35	0,390	0,9322	0,9154	1,02

Table 6.2 ki<sup>\*</sup>,

(SD1)i, R(ki<sup>\*</sup>), R(ko) and (SD2)i of the properties according to the formulation A(k.w + a) + B

#### 7.CONCLUSIONS

With the increasing water/cement ratio, the dry unit weight, the splitting and the compressive strengths, the brittleness index and the elasticity modulus decrease. The granulumetric composition of the aggregate does not pratically effect the unit weight and the brittleness index. However, the compressive and the splitting strengths and the elasticity modulus are a little smaller than the unit weight and the brittleness index for the granulumetry between B16-C16 because of the more air voids in the granulumetry between B16-C16. When the compressive strength increases, the brittleness index and the ratio between the compressive strength and the splitting strength rises. The fact reveals that when the strength increases, the concrete becomes brittle. The correlations between the properties are strong because the sensitivity of the properties to the pore structure of the cement paste is similar.

## 8.REFERENCES

[1] Brandt, A.M., 1999, Cement and Other Binders, Cement Based Composites, Materials Mechanical Properties and Performance, pp 47-52, London.

[2] Diamond, S., 1995, Microstructure of Cement Based Materials/Bonding and Interfaces in Cementitious Materials, Materials Research Center, Vol:370.

[3] Zimbelmann, R., 1989, A Contribution to The Problem of Cement-Aggregate Bond, Cement and Concrete Research, 15, pp 801-808.

[4] Leo,F.M.,1970, The Chemistry of Cement and Concrete,third Edition,Edward Arnold Publishers Ltd.,London

[5] Hansen, C.T., Radjy, F. and Sellevold, E.V., 1973, Cement Paste and Concrete, Annual Review of Materials, Vol 3, pp:233-268, Denmark.

[6] Sereda,P.J.,Feldman,R.F. and Romachnadran,V.S.,1980, Structure Formation and Development in Hardened Cement Pastes,Proc.7<sup>th</sup> International Congress on The Chemistry of Cement,Vol 1,Paris.

[7] Okpala,D.C.,1999, Pore Structure of Hardened Cement Paste and Mortar, The International Journal of Cement Composites and Lightweight Concrete, Vol 11, No:4, USA.

[8] Mehta,P.K. qnd Aitcin,P.C., 1990, Microstructural Basis of Selection of Materials and Mix Proportions For High Strength Concret,2<sup>nd</sup> International Symposium onHigh Strength Concrete,pp 265-285,Berkeley,California.

[9] Oktar,O.N., Moral,H. and Tasdemir,M.A.,1984, Sensitivity of Concrete Properties to The Pore Structure of Hardened Cement Paste,Cement and Concrete Research,Vol26,No:11,1619-1627,Istanbul [10] Oktar,O.N., Moral,H, and Tasdemir,M.A.,1984,Factors Determining The Correlations Between Concrete Research,Vol 26 No:11,16191627,Istanbul.