

Review on Microstructure of Concrete

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Abstract- Microstructural analysis is unique and advanced method of finding properties of concrete. X-Ray Diffraction Analysis (XRD), Scanning Electron Microscope (SEM) and Energy Dispersive Spectroscopy (EDS) are the general technique used to visualize the micro structural behavior of concrete during hydration process. From the microstructure of concrete we can able to find the physical and chemical properties of concrete which is difficult in normal testing methods. The microstructure shows ITZ state in concrete and hydration process which is the major process happening inside the concrete and it makes concrete strengthen. The hydration process in concrete will leads to formation of C-S-H gel, Ca (OH)₂ crystals, and other mineral compounds which influences the individual properties of concrete.

Keywords- Microstructure, Cement, Concrete, hardened cement paste, XRD, SEM, EDS.

I. INTRODUCTION

Concrete is a composite material that consists of a binding medium, aggregate particles and different admixtures. It can be formed in various proportions. Concrete to consist of three phases: a Cement paste, aggregate, and Interfacial Transition Zone (ITZ) between them. In addition to Ordinary Portland Cement, the essential components of the base of concrete are aggregates and water. Microstructure is the very small scale structure of a material, defined as the structure of a prepared surface of material as revealed by a microscope above 25× magnification. The microstructure of a material (such as metals, polymers, ceramics or composites) can strongly influence physical properties such as strength, toughness, ductility, hardness, corrosion resistance, high/low temperature behavior or wear resistance. These properties in turn govern the application of these materials in industrial practice. Microstructure at scales smaller than can be viewed with optical microscopies often called nanostructure, while the structure in which individual atoms are arranged is known as crystal structure. Basically, two types of measurements of microstructures are made. The first group includes measurements of depths (i.e., depth of decarburization, depth of surface hardening, or coating thicknesses). These measurements are made at a specific location (the surface) and may be subject to considerable variation. The second group of measurements belongs to the field referred to as stereology.

Stereology is the three-dimensional interpretation of two-dimensional cross sections of materials or tissues. It provides practical techniques for extracting quantitative information about a three-dimensional material from measurements made on two-dimensional planar sections of the material. To obtain reproducible data, these surface conditions must be measured at a number of positions on a given specimen, and on several specimens if the material being sampled is rather large.

II. LITERATURE REVIEW

Gai-Fei Peng and Zhi-Shan Huang (2008) carried out the study on the change in microstructure of hardened cement paste due to elevated temperatures. X-ray diffraction (XRD) and Scanning Electron Microscope (SEM) analysis was used to find the Microstructure of Concrete. The pore distribution of hardened cement paste in concretes NSC-40, HPC-70 and HPC-110 was tested by using Mercury Intrusion Porosimetry (MIP). The rate of decomposition C-S-H gel was increased with temperature above 600 c and it was formed from SEM and XRD. It was the most important cause for the loss in strength of HPC.

Tao Ji et al., (2005) conducted a study on water permeability and microstructure on concrete with nano silica. Two types of concrete was used. Normal concrete and nano silica concrete was prepared by using same superplasticizers (TW-7). For nano silica concrete, cement was replaced with nano silica. Basic tests like fresh and hardened test are followed for both concrete by means slump test, compressive strength, permeability test at age of 28. ESEM was used to analyse the microstructural properties of both concrete. The results of ESEM was favour for nano silica concrete as it has good homogeneous and dense microstructure when compared with normal concrete. The voids in C-S-H gel was filled by nano particles in nano silica concrete and bond became tight which leads to good strength.

Mahmoud Khashaa Mohammed, Andrew Robert Dawson and Nicholas Howard Thom (2013) investigated production, microstructure and hydration characteristics of sustainable SCC using limestone powder and fly ash. 33% of cement was replaced by both fly ash and limestone powder. The properties of concrete are fresh and hardened properties was tested. The changes in microstructure of concrete with fly

ash and limestone were determined by using XRD analysis, SEM analysis and EDS analysis. Microstructure of scc having limestone doesn't had dense when compared with microstructure of scc having fly ash. A high amount of Ca (OH)₂ and CaCO₃ was found on ssc with limestone and its due to dehydroxylation of CH. From this research it was found that fly ash is better as replacement of cement in scc.

Nicolas Burliona*, Dominique Bernardb, Da Chena. (2009) carried out Microstructure during leaching process in concrete and leaching process is determined. The porosity leads to many disadvantages in the properties of concrete. For 24 hours and 61 hours leaching was found by using 3D microstructure. The microstructure was determined using XCMT and it allows us to determined 2D microstructure and 3D reconstruction of the microstructure of mortar. By using ammonium nitrate solution microstructure of mortar were found by XCMT. For the study of durability of mortar or concrete structure this technique is very helpful.

ZhaohuiXie and Yunping Xi (2001) conducted an experimental study on the hardening mechanism of class F fly ash activated by water-glass and NaOH. The paste prepared for two compositions and by using SEM, XRD and EDS the mineralogical properties of the two samples were found. The minerals such quartz, hematite, mullite and magnetite presence are analysed by XRD in hardened paste containing fly ash. Due to the reaction between class F fly ash and water-glass, pasted formed into gelatinous matrix and its analysed using SEM micrograph. By using XRD pattern the low-ordered crystalline structure of dissolved fly ash and water-glass. The formation of huge quantity of crystalline structure was revealed from the mixture of class F fly ash, water-glass and NaOH and it was determined by means of SEM and EDS. The improvement in the strength of the materials is due to reduction in modulus of water-glass from 1.65 to 1.0, the crystalline sodium silicate in the past results in the compact structure.

AmeerA.Hilal. (2016) shows the detail study of microstructure of high performance concrete. The properties like strength, durability and hydration process was studied in this research. Mercury intrusion porosimetry (MIP), Optical microscopy (OM), Scanning electron microscopy (SEM), Micro-computed tomography (μ CT), these are the methods used in this project. The microstructure of normal concrete and high performance concrete was studied. Due to the physical and chemical contribution of the additives the microstructure of HPC is more homogenous than NC. The strength and durability was improved by using additives and also it results in the reduction of porosity. By adding mineral admixtures weak area (ITZ) was strengthen and less porous.

Finally durability of concrete is increased because of mineral admixtures added in concrete.

Salim Barbhuiya, PengLoy Chow and ShazimMemon (2015) studied the microstructure, hydration and nanomechanical properties of concrete with metakaolin. The metakaolin was added as 0%, 5%, 15% and 20% by mass of cement. XRD, MIP, Nanoidentation, sorptivity and compression tests were carried out to find mechanical and microstructural properties of concrete. At 10% of replacement of cement with metakaolin the compressive strength of concrete got increased. At 15% of replacement of cement the depth of carbonation was increased. Addition of metakaolin in concrete results in reduction of porosity in concrete. The change of portlandite into C-S-H gel were analysed by XRD which leads to modification relative properties.

Divya Chopra, Rafat Siddique, Kunal, (2015) carried out with RCPT Rapid chloride permeability test, SEM Scanning electron microscope, XRD X-Ray diffraction, EDS Energy dispersive spectroscopy. XRD and SEM analyses revealed the increased formation of a CSH gel for all mixes which helps explain the increased compressive strength for 15% RHA concrete. Replacement of cement by RHA as a supplementary cementitious material, has positive effect on all properties of self-compacting concrete. Fresh properties results showed that with increase in amount of RHA workability decreased. Lowest workability was obtained by the mix containing 20% RHA. Increases of about 25% strength at 7 d, 33% strength at 28 d and 36% strength at 56 d were observed with increases in RHA content from control mix to 15% cement replacement. Increasing RHA content increased the compressive strength of the concrete up to 15% RHA content whereas above this value there was a decrease in strength due to reduced hydration reaction and lower cement content. A similar trend was shown as for compressive strength. The split tensile strength increased up to 15% replacement of cement by RHA. SCC mixes develop split tensile strength ranging from 2.0 to 2.8 MPa, from 2.5e3.7 MPa, from 2.8 to 4.0 MPa at 7, 28 and 56 d, respectively. SCC mixes made with RHA reduced the chloride ion penetrability. Increase in replacement decreased the charge passed. Very low permeability was achieved by the 15% RHA replacement to cement and moderate permeability was recorded for the control mix. Including RHA into the matrix reduced the pores for all mixes. Porosity decreased with increase in curing time due to increased rate of hydration with time. The lowest porosity was obtained by the mix containing 15% RHA. XRD and SEM analyses revealed the increased formation of a CSH gel for all mixes which helps explain the increased compressive strength for 15% RHA concrete. Pores and cracking were at maximum for the control mix. The densest

structure was observed for 15% replacement with RHA which resulted in the highest compressive strength for the mix.

Yogesh Aggarwal and Rafat Siddique (2014) investigated the influence of waste foundry sand and bottom ash. Both of samples were taken in different percentages (0%-60%). These samples were added as replacement of sand in concrete and it doesn't affect the strength of concrete until it reaches level of 60% replacement of sand with these two samples waste foundry and bottom ash. The proper and clear fibrous pattern of C-S-H gel was analyzed by SEM micrograph and from this its found that it makes concrete more against aggressive situation which is observed from chloride penetration tests.

Hulya Kus and Thomas Carlsson (2003) experimentally demonstrated the chemical degradation of autoclaved aerated concrete (AAC) through natural and artificial weathering conditions. The prepared samples are exposed to natural and artificial weathering conditions. XRD, SEM are used to analyze the microstructural changes in the samples after exposing to climate and transformation of tobermorite to calcium carbonate was found. Calcium and gypsum were formed due to weathering and its found by using SEM and EDS. From the above results its concluded that there is no changes in microstructural and ageing in both natural and artificial weathering.

Hong-Sam Kim, Sang-Ho Lee and Han- Young Moon (2007) conducted an experimental study on the strength and durability of high strength concrete using metakaolin as a cementitious material. The silica fume and metakaolin were prepared with 5%, 10%, 15% and 20% of nine different mixes. The strength (compressive, tensile and flexural strength) and durability (Rapid chloride penetration test, Acid attack test, Freezing and thawing test and Accelerated carbonation test) of concrete samples were tested. At 10% and 15% of replacement of metakaolin showed increase in the strength of concrete. The chemical attack can be avoided by addition of silica fume and metakaolin. For both silica fume and metakaolin replacement of cement are similar. From the above results its concluded that metakaolin is better alternative for silica fume.

LIU Shuhua1, YAN Peiyu2. (2010) carried out research on Effect of limestone powder on microstructure of concrete. The objective of this study was to obtain the microstructure of the concrete containing limestone powder and to confirm its filling effect and hydration characteristics. The effect of limestone powder on microstructure of concrete was studied by using mercury intrusion porosimetry (MIP), scanning electron microscopy (SEM) and X-ray diffraction

(XRD) techniques. The compressive strength of concrete containing 100 kg/m³ limestone powder can meet the strength requirement. The filling effect of limestone powder can make the paste matrix and the interfacial transition zone between matrix and aggregate denser, which can improve the performance of concrete. The mix proportion design was based on the strength grade of concrete, C30, C40, C50 and C60. MIP was used to determine the pore size distribution of concrete. Porosity, pore size and pore shape are significantly influenced by mix proportion. As expected, the total pore volume in the four concretes is much lower than that in traditional concrete. And with the increase of the strength grade, the total porosity is decreasing, especially for C60 sample. The critical pore diameters, defined as the peaks in the curves, giving the rate of mercury intrusion per change in pressure (differential curves), do not show a significant change. The size of most pores in the four concretes is lower than 50 nm, which does little or no harm to performance of concrete. Scanning electron microscopy was used to determine the microstructure and the phase distribution of the samples. In order to get a high contrast images for image analysis, an acceleration voltage around 12.5 kV was used. The magnification factor of region in each image was 5000. With the help of image analysis techniques, the grey-scale histogram of original image was used to distinguish the different phases. Within the grey-scale from 0 (black) to 256 (white), the pores, C-S-H gel, CH, limestone powder and anhydrous cement are identified respectively. XRD measurements were performed on a Philips X'Pert diffractometer equipped with a graphite monochromator using Cu K α radiation and operating at 40 kV and 20 mA. Step scanning was used with a scan speed of 2°/min and sampling interval of 0.02°(2 θ). XRD was used to identify the hydrates in the cement paste containing limestone powder. Fragments of specimens broken off and washed with acetone were examined by SEM equipped with energy dispersed X-ray spectroscopy (EDS). This analysis was specially carried out to identify weather limestone powder can hydrate with hydration products of cement. The compressive strength of concrete containing 100 kg/m³ limestone powder can meet the strength requirement. The filling effect of limestone powder can make the paste matrix and the interfacial transition zone between matrix and aggregate denser, which can improve the performance of concrete. Limestone powder does not have pozzolanic properties, but it is still unhydrated at the age of 28 d.

Romualdas mačiulaitis, marija vaičienė, ramunė žurauskienė (2010) done a research is to analyse the properties of raw materials, used in the production of expanded-clay lightweight concrete with the catalyst waste, and estimate their influence on the properties of the mixture of lightweight

concrete. In order to utilise various waste materials as additives in the production of lightweight concretes efficiently, it is necessary to carry out a detailed analysis, to determine their mineralogical, chemical, granulometric composition. Microstructure of the analysed Portland cement and catalyst utilised in the reactor of catalytic cracking, X-ray structural analysis of waste material is provided in the research. Additionally, the analysis of the crush resistance of coarse aggregate (expanded-clay) carried out, is described in the research. The granulometric composition of the sand was determined, as well as the particles' size of the catalyst and cement was analysed. The hardened expanded clay – lightweight concrete was analysed during the research. Microstructure analysis of the expanded-clay lightweight concrete was carried out. Grains of the fine-grained catalyst waste are larger than the particles of Portland cement, and their shape is spherical (contrary to Portland cement). Size of 80 % of the particles in the used catalyst varies from 30 μm to 112 μm . Catalyst waste analysed belongs to the group of zeolites. After the X-ray structural analysis it was identified that it is faujasite. The size of Portland cement particles varies from 0.3 to 140 μm , 50% of Portland cement articles have size of 7 μm . When particles of used catalyst are compared with the particles of binding materials, we can notice that catalyst particles are similar to the particles of binding material. They are of the same shape, but the diameter of the latter ones is 4 times smaller. The amount of sand particles with the size from 1 to 0.5 mm in the mixture is equal to 46.73 %, from 4 to 2 mm – 30.2 % and from 0.25 to 0.125 mm – 20.16%. The size of the particles of coarse aggregate is 2–4 mm. considering the results of microstructure and X-ray analysis of used catalyst, it can be concluded that this material can replace a part of binding materials and behave in the formation mass as an active mineral additive. The contact in the expanded-clay lightweight concrete between the catalyst waste, granule of expanded clay and cement stone is strong. During the compression of the samples, coarse aggregates and cement past start to break after 28 days of hardening.

Vitoldas Vaitkevicius, Evaldas Šerelis and Harald Hilbig(2014) conducted an investigational study that reports the micro structural variations of Ultra High Performance concrete (UHPC) with inclusion of glass powder. Glass powder composition was prepared in four different composition. The mechanical property and microstructural property was calculated for four different composition of ultra high performance concrete. The hydration process was found by microstructural analysis. The strength and performance got increased due to addition of silica fume.

C.S. Poon, z.h. shui, l. lam . (2004) discussed about effect of ITZ(Interfacial Transition Zone) on compressive

strength. The narrow region around the aggregate particles with fewer cement particles, and thus more water. This is called the Interfacial Transition Zone, abbreviated ITZ. The ITZ is a region with a higher w/c, and thus a higher porosity, than the bulk paste. At 90 days, the HPC recycled aggregates concrete achieved similar strength values to the natural aggregate concrete. The results are explained by the differences in porosity and pore structure of the two types of aggregates, and possible interactions between the aggregates and the cement paste. The high-performance concrete and normal-strength concrete recycled aggregates induced different interfacial transition zone microstructures in the recycled aggregate concrete. A relatively dense interfacial zone was present in the high-performance recycled aggregate concrete whereas a loose and porous product layer filled the normal-strength concrete interfacial transition zone. The interfacial transition zone formation is related to moisture movement and chemical reactions in the recycled aggregate concrete. The porous interfacial transition zone microstructure in the normal-strength concrete can be attributed to the higher porosity and absorption capacity of the recycled aggregate. The interfacial transition zone microstructure in concrete with recycled aggregates appeared to be an important factor in governing strength development of the recycled aggregate concrete. It is expected that the mechanical properties of recycled aggregate concrete can be improved by modifying the surface properties and the pore structure of the recycled aggregates.

K AMRAN M. NEMATI. (1997) undergone research on Fracture Analysis of Concrete Using Scanning Electron Microscopy. A special experimental technique has been developed that enables the preservation of the compressive stress induced micro cracks in concrete as they exist under applied loads. This technique involves injecting a molten-metal alloy into the induced cracks and solidifying it before unloading. Scanning electron microscopy was employed to capture images from the cross sections of the concrete specimens. These images were then used to study the generation and interaction of the compressive stress-induced micro cracks and the effect of confinement on micro crack behaviour. A special experimental technique was developed which enabled the preservation of the compressive stress-induced micro cracks in concrete as they exist under applied loads. This technique involved injecting a molten-metal alloy into the induced cracks and solidifying it before unloading. The use of SEM to extract images from the concrete specimens for different testing conditions is explained. The images rendered were analyzed using an image analyzer. The image analyzer identifies Wood's metal, which represents the crack network in concrete specimens. The SEM identifies the

geometric aspects of features in the microstructure, such as microcracks in concrete represented by Wood's metal.

III. CONCLUSION

Concrete property is unique in nature and as improvement in technology various types concrete came. In those types each concrete has different in their property. The microstructure reveals all properties of concrete and from the above reviews its clearly shows that the property of concrete can be changed by adding additives or admixtures. By adding any additives the microstructure of concrete will change and the microstructure of concrete shows strength, durability, crack etc., and results avoiding defects too can be found from microstructural analysis.

From microstructure analysis carried in above researches clearly shows that replacement with cement or sand or coarse aggregate results in increase in strength, reduction in porosity etc.. And replacement by cheaper materials good in both economic and physical property.

REFERENCES

- [1] Divya Chopra, Rafat Siddique and kunal,” Strength, permeability and microstructure of Self-compacting concrete containing rice husk”, BIOSYSTEMS ENGINEERING 130 (2015) 72-80.
- [2] Salim Barbhuiya , PengLoy Chow and ShazimMemon,” Microstructure , hydration and nanomechanical properties of concrete containing metakaolin”, Construction and Building Materials 95 (2015) 696–702.
- [3] Vitoldas Vaitkevicius, EvaldasŠerelis and Harald Hilbig,” The effect of glass powder on the microstructure of ultra high performance concrete”, Construction and Building Materials 68 (2014) 102– 109.
- [4] Tao Ji,” Preliminary study on the water permeability and microstructure of concrete incorporated nano- SiO₂” Cement and Concrete Research 35 (2005) 1943 – 1947.
- [5] Gai-Fei Peng and Zhi-Shan Huang “Change in microstructure of hardened cement paste subjected to elevated temperatures” Construction and Building Materials 22 (2008) 593–599.
- [6] Nicolas Burliona*, Dominique Bernardb, Da Chena. (2009) “x-ray micro tomography: application to microstructure analysis of a cementitious material during leaching process”Journal name: ELSEVIERRResearch 36 (2006) 346 – 357
- [7] AmeerA.Hilal. (2016) “Microstructure of Concrete” Department of Civil Engineering, Faculty of Engineering, University of Anbar, Anbar, Iraq, <http://dx.doi.org/10.5772/64574>
- [8] C.S. Poon, z.h. shui, l. lam . (2004), “Effect of microstructure of ITZ on compressive strength of concrete prepared with recycled aggregates” journal name: Elsevier 18 (2004) 461–468
- [9] Romualdas mačiulaitis, marija vaičienė, ramunė žurauskienė . (2010) “Microstructure analysis of the structure of materials used for the mixture of expanded – clay lightweight concrete with additives of raw material” <https://www.researchgate.net/publication/268431089>
- [10] LIU Shuhua1, YAN Peiyu2. (2010) “Effect of limestone powder on microstructure of concrete”, state key laboratory of water resource and hydropower engineering science, wuhan university, wuhan 430072, China DOI 10.1007/s11595-010-2328-5
- [11] K AMRAN M. NEMATL. (1997) “Fracture Analysis of Concrete Using Scanning Electron Microscopy” Department of Civil and Environmental Engineering, University of California at Berkeley, Berkeley, California, USA
- [12] Meena murmu and Suresh Prasad Singh” Hydration Products, Morphology and Microstructure of Activated Slag Cement” International Journal of Concrete Structures and Materials, Vol.8, No.1, pp.61–68, March 2014
- [13] B.Vidivelli* and M. Mageswari, (2010) “Study on flyash concrete using sem analysis” Journal of Environmental Research And Development
- [14] Rafat Siddique, “Effect of fine aggregate replacement with Class F fly ash on the mechanical properties of Concrete” Cement and Concrete Research 33 (2003) 539–547
- [15] Hu’lya Kus*, Thomas Carlsson ,” Microstructural Investigation of naturally and artificially autoclaved aerated concrete”, Cement and Concrete Research 33 (2003) 1423–1432
- [16] Prinya Chindaprasirt a, Chai Jaturapitakkul, Theerawat Sinsiri “Effect of fly ash fineness on microstructure of blended cement paste” Construction and Building Materials 21 (2007) 1534–154.
- [17] W.W.J. Chan, C.M.L. Wu “Durability of concrete with high cement replacement, Cement and Concrete Research 30 (2000) 865±879
- [18] P.Kumar Mehta and Paulo.J.M.Monteiro, “Concrete Microstructure Properties and Materials”3rd Edition,2006