Concrete Microstructure - A Review

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Abstract: Microstructural study on concrete is a unique technique to find out the morphological features of concrete. X-Ray Diffraction Analysis (XRD), Scanning Electron Microscope (SEM) and Energy Dispersive Spectroscopy (EDS) are the general techniques used to visualize the microstructural behavior of concrete during hydration process. The specific characteristics within the concrete can be visualized through these modern techniques. The mineral data obtained from the microstructural study will help to interpret the unique behavior of concrete and presence of minor compound inside the hardened cement paste of concrete. The hydration process in concrete will lead 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.

1. Introduction

In general, the bond between the microstructure and the property of concrete are at the heart of recent material science. Concrete is very dense and heterogeneous microstructure in nature. Therefore, it is extremely complicated to comprise realistic models of its microstructure from which the performance of the material can be predicted.

The microstructure of concrete governs the physical properties of concrete such as Compressive strength, Tensile strength and Flexural strength, these properties can be customized by making appropriate alterations in the composition of materials in the concrete mix.

This paper discusses the microstructural study on concrete and the influence of replacement concrete ingredients on the physical properties of concrete.

2. Literature Review

Divya Chopra, Rafat Siddique and Kunal (2015) studied the effect of replacement of cement with rush husk ash (RHA) as additional cementitious materials and experimented its effect on strength, durability, and microstructure through their unique properties. The flow properties (Slump flow test, V-Funnel, U-Box and L-Box tests), mechanical properties (compressive strength and split tensile strength) and durability (porosity and RPCT) were tested. From the outcome, the fresh properties of self compacting concrete with RHA revealed that increase in RHA decreases the workability. The compressive strength of self compacting concrete with RHA increases up to 15% replacement of cement with RHA. The micro structural analysis (XRD and SEM) showed the huge formations of C-S-H gel and steady state of hydration process at 15% replacement of cement by RHA which was the main reason for the improvement in the strength and durability of self compacting concrete with RHA.

Salim Barbhuiya, Peng Loy Chow and ShazimMemon (2015) studied the microstructure, hydration and nanomechanical properties of concrete with metakaolin. The incorporation of metakaolin in concrete was 0%, 5%, 10% and 15% by mass of cement. The properties of concrete with metakaolin were tested by means of compressive strength test, sorptivity, accelerated carbonation, X-Ray Diffraction (XRD) analysis, mercury intrusion porosimetry (MIP) and Nanoindentation. From the test results, the compressive strength of concrete as increased with increase in the inclusion of metakaolin up to 10% of the mass of cement. At the low water-binder ratio, the sorptivity of concrete decreased with the inclusion of metakaolin. The carbonation depth of concrete was increased with 15% replacement of cement with metakaolin. The porosity of concrete reduces with increase in incorporation of metakaolin. The inclusion of metakaolin in concrete reduces the porosity and develops hydration process. The XRD pattern showed that the inclusion of metakaolin in concrete changes portlandite into C-S-H gel which leads to modification relative properties of concrete.

Malkit Singh and Rafat Siddique (2014) studied the possibility of utilization of coal bottom ash as substitute for fine aggregate in concrete. The test result showed that the compressive strength of concrete with bottom ash at the curing age of 28 days was not affected badly. However, after 90 days of curing age, compressive strength of bottom ash concrete surpassed that of conventional concrete. SEM and XRD studies indicated that the C–S–H gel structure was somewhat less monolithic than that of conventional concrete and total intensity of
(ettringite) hydrous calcium aluminum sulfate was not changed with the inclusion of coal bottom ash in concrete.

Yogesh Aggarwal and Rafat Siddique (2014) investigated the influence of waste foundry sand and bottom ash in equal quantities as partial replacement of fine aggregates in various percentages (0% to 60%), on concrete properties. The inclusion of waste foundry sand and bottom ash as fine aggregate does not affect the strength properties negatively as the strength remains within limits except for 60% replacement. The micrograph from SEM analysis showed the proper and clear fibrous pattern of C-S-H gel which makes the concrete more opposed to aggressive situation as observed from chloride penetration test values.

Vitoldas Vaitekevicius, Evaldas Šerelis and Harald Hilbig (2014) conducted an investigative study that reports the microstructural variations of Ultra High Performance concrete (UHPC) with inclusion of glass powder. Four compositions are prepared with different quantity of glass powder. The four compositions of UHPC with glass powder were tested for its mechanical and microstructural properties. From the microstructural analysis, the hydration process was accelerated because of increased in dissolution rate of Portland cement. This leads to formation and development of opaque microstructure of UHPC. The composition with blend of silica fume and glass powder showed good performance and increase in compressive 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. The rate of replacement of both fly ash and limestone was 33% of cement content. The fresh and hardened properties of concrete samples were tested. X-ray Diffraction Analysis (XRD), Scanning Electron Microscope (SEM) and Energy-dispersive Spectroscopy (EDS) analysis were performed to examine the microstructural changes in concrete with fly ash and limestone. The microstructural study showed that SCC containing fly ash had dense microstructure than SCC containing limestone powder. Due to dehydroxylation of CH, high amount of Ca (OH)₂ and CaCO₃ was visualized in SCC with limestone powder. As a result, fly ash was suitable for creation of sustainable SCC.

Rafat Siddique, Yogesh Aggarwal, Paratibha Aggarwal, El-Hadi Kadri and Rachid Bennacer (2011) explored the strength durability and microstructural properties of concrete partially replaced with used foundry sand. The proportion of fine aggregates in the mix was replaced with range of 0% to 60%. Strength and durability properties of concrete with used foundry sand and ordinary concrete were examined. From the outcomes of the tests performed, the optimal usage of foundry sand as partial replacement for fine aggregate was found out to be 30% and not more than 50%. The XRD and SEM analysis showed the dense microstructure with reduced voids which causes the increase in strength and resistance to violent environment. The development of C-S-H gel, micro cracks on the concrete were visualized through micrographs of concrete and interrelated with the mechanical uniqueness of the concrete.

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. The microstructure of hardened cement paste was tested using X-ray diffraction (XRD) and Scanning Electron Microscope (SEM) analysis. Mercury Intrusion Porosimetry (MIP) test was performed to determine the pore size distribution of hardened cement paste in concretes NSC-40, HPC-70 and HPC-110. Due to coarsening effect of pore structure in hardened cement paste, the mechanical properties of HPC is affected. From XRD and SEM analysis, the rate of decomposition of C-S-H gel was increased with temperature above 600ºC which was the most important cause for the loss in strength of HPC.

Prinya Chindaprasirt, Chai Jaturapatkul and Theerawat Sinsiri (2007) investigated the changes on microstructure and pore structure of blended cement paste with fly ash. Type I Portland cement was replaced with original fly ash (OFA) and classified fly ash (CFA). Portland cement were replaced with fly ashes at 0%, 20% and 40%. Pore size distribution (Mercury Intrusion Porosimetry) and microstructure (XRD and SEM) of blended cement paste with two different fly ashes were tested for 7, 28 and 90 days. The pore size of cement paste incorporated with original fly ash (OFA) was lower than the cement paste with classified Fly ash (CFA). From the XRD pattern, it was noticed that Ca(OH)₂ decreased with increase in fly ash which leads to faster pozzolanic reaction and hydration process in blended cement paste with fly ash (40% replacement). The SEM observations showed the dense and homogeneous microstructure of cement paste with finer fly ash which was mainly due to enhancement pozzolanic, hydration reaction and nucleation effect.

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. Nine different mixes containing different proportions
of metakaolin and silica fume were prepared with 5%, 10%, 15% and 20% weight of the binder. 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. The Strength tests showed the best possible strength characteristics were obtained for 10% and 15% replacement of metakaolin. The fine powder of metakaolin and silica fume showed improves the resistance against the chemical attack which was mainly due filler effect of the two binders. It was confirmed that mechanical and durability characteristics of concrete with metakaolin and concrete with silica fume are considerably similar. From the overall experimental analysis, the metakaolin exhibits as a capable alternative for silica fume.

Tao Ji et al., (2005) conducted a study on water permeability and microstructure on concrete with nano silica. Normal concrete (NC) and nano silica concrete (SC) are the two mix proportions used along with the superplasticizers (TW-7). In nano silica concrete (SC), a part of cement was replaced with nano silica. Fresh and hardened properties of normal concrete and nano concrete were tested by means of slump flow test, compressive strength, permeability test at the age of 28 days. The microstructural analysis was performed by means of ESEM techniques. The study revealed that nano silica concrete has enhanced water penetration resistance than the normal concrete. The ESEM test revealed the microstructure of nano silica concrete which has homogeneous and dense microstructure than normal concrete. The nano silica particles filled the voids of C-S-H gel and form a tight bond with the particles of C-S-H which makes binding paste matrix more compact and improves durability and mechanical properties of concrete.

Hui Li, Hui-gang Xiao, Jie Yuan and Jinping Ou (2004) carry out an investigational study on the microstructural behavior on cement mortar with nano-particles such as nano-Fe$_2$O$_3$ and nano-SiO$_2$. The mechanical and microstructural properties of the cement mortar with nanoparticles were experimentally studied by means of compressive strength, flexural strength and SEM analysis. From the experimental study, the strength properties of the cement mortars with nano-SiO$_2$ and with nano-Fe$_2$O$_3$ were improved much higher than the ordinary cement mortar mixture with same water binder ratio (0.5). The micrograph obtained from SEM analysis showed the microstructure of the cement paste with nanoparticles have a uniform and compact microstructure. The nanoparticles not only acts as a filler but also acts as an activator to endorse the hydration and improve the microstructure of cement paste if the nanoparticles are evenly dispersed.

Hulya Kus and Thomas Carlsson (2003) experimentally demonstrated the chemical degradation of autoclaved aerated concrete (AAC) through natural and artificial weathering conditions. The samples are prepared and exposed to natural and artificial weathering conditions. The microstructural changes and ageing of samples were examined through light optical microscopy, X-ray Diffraction technique (XRD) and Scanning Electron Microscope (SEM). The transformation of tobermorite into calcium carbonate was observed during the exposure time. Due to weathering, calcite and gypsum were developed as noticed in the SEM and EDS Analysis. Hence, the microstructure and ageing in both naturally and artificially weathered are similar based on the mineralogical data obtained from the microstructural analysis.

Zhaohui Xie and Yunping Xi (2001) conducted an experimental study on the hardening mechanism of class F fly ash activated by water-glass and NaOH. Two different compositions of pastes are prepared. The mineralogical details of the two samples are identified and analyzed using XRD, SEM and EDS. SEM micrograph of hardened paste made of class F fly ash with water-glass showed that the paste formed into a sphere-shaped gelatinous matrix due to the reaction between class F fly ash and water-glass. The XRD pattern also showed the presence of quartz, hematite, mullite and magnetite in the hardened paste containing fly ash. The dissolved fly ash and water-glass in a state of the low-ordered crystalline structure were observed through XRD pattern. The mixture of class F fly ash, water-glass and NaOH revealed the formation of huge quantity crystalline structures were observed using SEM and EDS. Due to reduction in modulus of water-glass from 1.65 to 1.0, the crystalline sodium silicate in the past results in compact structure which improve the strength of the material.

W.W.J. Chan and C.M.L. Wu (2000) studied the durability properties of concrete by replacing cement with silt and clay. Carbon black particles, Crushed granite, silts and clay with size under 150µm and finely crushed granite sizes between 75µm and 150µm were used for high cement replacement in concrete. These non-reactive waste materials replace the cement content with different mix proportions. The physical characteristics such as compressive strength, sorptivity and water permeability of concrete were tested on 8 different trial mixes. Additionally, the variations in microstructure of concrete due to hydration process were also studied through XRD, SEM and EDS Analysis. As a result, the increase in strength of...
Concrete would decrease the sorptivity and permeability of water in the given concrete mixes. The XRD, SEM and EDS results showed that the non-reactive materials used as a replacement for cement could also give micro filler effect and nucleation site for hydration on cement.

3. Conclusion

From this review of articles, it was observed that the change in the microstructure of concrete reflects the changes in properties of concrete. Thus, the microstructural change in concrete is mainly due to surrounding conditions such as time, temperature, chemical degradation due to acid attack etc., the microstructure of concrete varies with respect to the proportion of concrete ingredients such as cement, aggregates, and water content.

The concrete microstructure can also be modified through replacement of concrete ingredients with other waste and cheap byproducts which will be more useful to produce sustainable high performance concrete.

4. References

[10] Hong-Sam Kim, Sang-Ho Lee and Han-Young Moon “Strength properties and durability aspects of high strength concrete using Korean metakaolin.”