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RESEARCH ARTICLE OPEN ACCESS

Microstructural Analysis of M30 Grade Concrete Using Scanning Electron Microscopy (SEM) Method

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ABSTRACT
The influence of the ingredients on the mechanical performance is assessed by determining the compressive strength of the samples. The analysis of the microstructure of the concrete specimen is done through various methods like X-ray diffraction method (XRD), Scanning Electron Microscope (SEM), Energy Dispersive Spectroscopy (EDS).

The microstructure analysis comprises the findings of particular parameters such as size of grains and internal pore structure to complete portrayal of multi-component systems. The microstructure analysis is carried out to recognize the performance of concrete in fresh and hardened state, response of concrete to the surrounding environment and various failure mechanisms. It also services to identify the unidentified ingredients in the cement mortar.

Keywords – Concrete, EDS, Microstructure analysis, SEM, XRD,

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I. INTRODUCTION

The type, size, shape, quantity and dissemination of various phases present in concrete constitute its structure. The structure is dynamic in nature, starting from nanometer scale, to Micrometer scale, to millimeter scale. The components of the structure above the millimeter scale can easily be seen by the naked eye, however the elements below the millimeter scale typically have to be determined with the help of microscopes, Scanning Electron Microscopy (SEM), Atomic Force Microscopy (AFM) and Transmit Electron Microscopy (TEM). The traditional term macrostructure is usually used for the structure, noticeable to the human naked eye. The boundary of resolution of the naked human eye is around one-fifth of a millimeter (200 µm). The traditional term microstructure is adopted for the microscopically magnified part of a macrostructure. The magnification capacity of modern electron optical microscopes is of the range of 105 to 106 times; thus, the use of transmission and scanning electron optical microscopy techniques has made it imaginable to identify the assembly of materials to a very minute fraction.

Currently, the fast progress of innovative experimental mediums such as the Transmission Electron Microscope (TEM) has made it imaginable to witness the microstructure of the products of hydration of concrete at a very small nanometer scale.

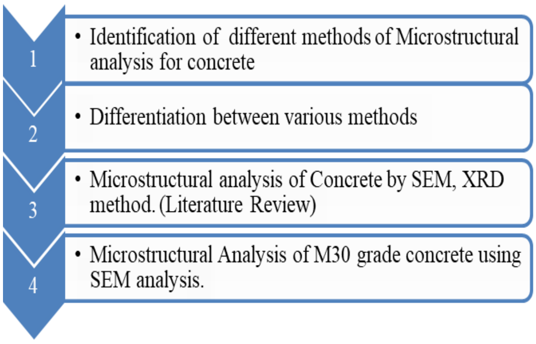
Microstructure analysis stresses on the art and science of making, understanding. It is helpful in investigating the microstructures in materials to improve the understanding of the performance of the materials.

II. OBJECTIVES OF THE PRESENT WORK

Objectives of the Research work are given as follows:

- To identify different methods of Microstructural analysis of concrete.
- To differentiate between various methods of analysis.
- To evaluate the results of different methods like XRD, SEM etc.
- To identify the Microstructural Analysis of M30 grade concrete using SEM analysis

III. METHODOLOGY



IV. METHOD FOR PREPARATION OF SPECIMEN

Preparation of specimen has developed a long recognized art in the optical microscopy of sample material, predominantly in the areas of metallurgy and mineralogy. The specimens thus prepared ideally are representative of the structure of interest and are free from damage and contamination.

Figure 1 illustrates s the general procedure for making the specimens.

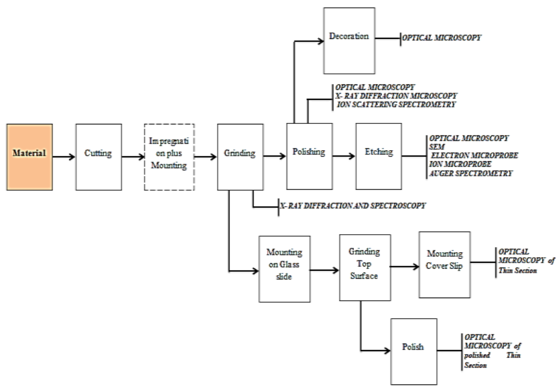


Figure 1: General method for preparation of specimen

The first step is the preparation of a fresh surface which will positively comprise the characteristics of interest. Once the preferred plane has been nominated, a section of the material constrained on one side by this plane is detached using hacksaw or automatic abrasive cut-off machine.

The following step in the specimen preparation procedure is impregnation and mounting. This step is presented in a dotted box in Figure 1 to designate that it may not be essential for every sample. If a material is porous, it normally needs some form of impregnation to lessen pickup of grinding and polishing abrasives which end in scratching and disproportionate relief. In this procedure, an organic polymer is embedded into the porosity of the specimen by vacuum impregnation. This process may be augmented by succeeding over pressuring to additional force the polymer into the specimen.

The next step in the preparation of the sample, as shown in Figure I, is grinding. The sample of each grinding step is to take away the broken surface layer causing from the instantaneously earlier step.

The subsequent step in the specimen preparation system is that of polishing; this is usually done by mechanical means.

However, chemical or electrolytic polishing either independently or in grouping with mechanical polishing is finding increased application.

The last step in this preparational system is etching. The various conventional methods existing are chemical, electrolytic and thermal etching.

V. METHODS OF MICROSTRUCTURAL ANALYSIS

Microstructure incorporates a widespread range of structural levels, from the minute scale to that of the engineering constituent, and comprises all discontinuities inside and among the phases such as dislodgments grain borders, phase interfaces, pores, and fissures.

In microstructural terms, concrete is an outstandingly composite organization of solid phases, minute opening and water, with a great degree of heterogeneity. This heterogeneity can be considered on several levels. At the simplest level, concrete consists of aggregates particles, distributed in a cement paste. On a more detailed level, the paste itself is a combination of unreacted cement, hydration products, cavities and water and at still finer level these segments themselves possesses a very complex microstructures.

There are many techniques which have been used to study microstructure; they can be separated largely into two classifications. Indirect or bulk techniques provide info on average characteristics of the whole microstructure. Examples of indirect techniques are thermogravimetry (TG) and X-ray diffraction (XRD), which can be applied to find the amounts of certain phases in the sample. Techniques used to acquire additional information about the pore-size distribution, such as mercury intrusion porosimetry (MIP) and methanol absorption, are also indirect techniques as they give no information as to how the pores are organized in space. The other group of techniques is direct or microscopical techniques which provide information about the way in which the component phases are arranged in the microstructure.

The advantage of the indirect or bulk methods is that they make available the information in a measureable form so that various trials can be matched objectively. In other way, the information resulting from direct methods typically takes the form of figures or images. Images are particularly very valuable in assigning an intense impression of the microstructure nevertheless comparisons between various samples are additionally subjective or informative and depend on the experience, knowledge and understanding of the researcher or observer.

Study of Micro structural of concrete is a distinctive method to identify the morphological characteristics of concrete. X-Ray Diffraction Analysis (XRD) and Scanning Electron Microscope (SEM) are the universally adopted technique or method used to portray the micro structural comportment of concrete through hydration course. The precise features in the interior of the concrete can be envisioned via these present techniques. The data attained from the micro structural study will assists to understand the unique actions of concrete and existence of minor compound within the hardened paste of cement concrete.

Following are different methods of Microstructural Analysis:

1. X-Ray Diffraction Analysis (XRD)
2. Scanning Electron Microscope (SEM)

1. X-Ray Diffraction Analysis (XRD):

The X-ray diffraction (XRD) method compromises of a suitable method to find the mineralogical investigation of crystalline solids. If a crystal-like mineral is brought in contact to X-rays of a specific wavelength then covers of atoms deflect the rays and create a array of peaks, which is representation of the mineral. The horizontal scale (diffraction angle) of a usual XRD pattern provides the crystal lattice spacing, and the vertical scale (peak height) provides the intensity of the diffracted ray. When the sample being X-rayed comprises of more than single mineral, the concentration of specific peaks from the specific minerals are proportionate to their amount.

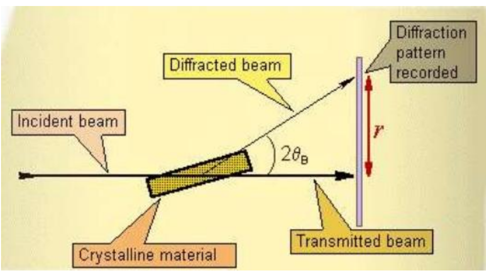


Figure 2: Schematic Diagram of X-Ray Diffraction

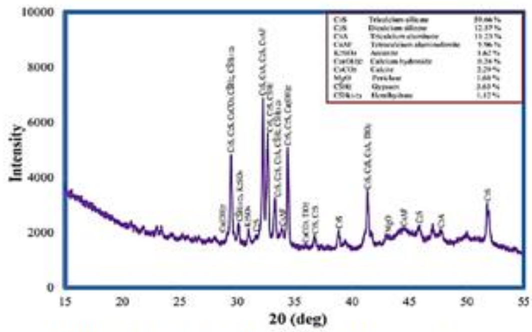


Figure 3: X-ray diffraction of concrete

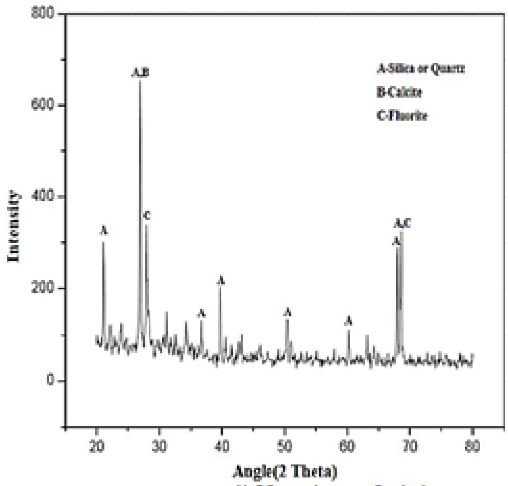


Figure 4: X-ray diffraction of plain concrete

The XRD analysis is normally carried out at room temperature by means of powder X – Ray diffraction with filtered 0.154 nm Cu, Kα radiation. Samples are scanned in a continuous mode from 10θ-80θ with a scanning rate of 2θ per minute.

The X-Ray powder diffraction provides the graph between the X – Ray light intensity, which is distributed on the sample and angle variance of the deflected X – Rays.

The XRD method can be adopted to recognize the single crystals, and to make known the single crystal structure. It can be also adopted to recognize various crystals which are available in a mix, e.g. minerals in a stone. For minerals having inconstant formulations and structures (clays), XRD is the best technique for recognising the formulations and finding their percentage in a sample.

2. Scanning Electron Microscope (SEM):

A scanning electron microscope is that kind of electron microscope which prepares images of a sample by scanning the top surface with a focused beam of electrons.

A scanning electron microscope (SEM) scans a focused electron beam over a surface to create an image. The electrons in the beam interact with the sample, producing various signals that can be used to obtain information about the surface topography and composition.

This is influential method, mostly when the microscope is fitted with a microprobe analyser. It comprises techniques similar to X-ray fluorescence to identify the chemical composition of hydrates. The higher resolution of the SEM allows the microstructure of the hydrated cement paste in concrete or mortar to be identified and studied. But, attention must be implemented when understanding the images as sample preparation and the vacuum needed by most of the microscopes can produce specifications, which are not available in the wet paste.

Working of SEM:

- ➢ The SEM uses electrons instead of light to form an image.
- ➢ A beam of electrons is produced at the top of the microscope by heating of a metallic filament.
- ➢ The electron beam follows a vertical path through the column of the microscope. It makes its way through electromagnetic lenses which focus and direct the beam down towards the sample.
- ➢ Once it hits the sample, other electrons (backscattered or secondary) are expelled from the sample.
- ➢ Detectors collect the secondary or backscattered electrons, and convert them to a signal that is sent to a viewing screen similar to the one in an ordinary television, producing an image.

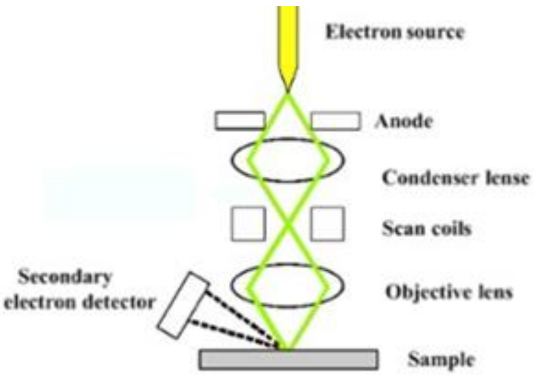


Figure 5: Schematic Representation of SEM components

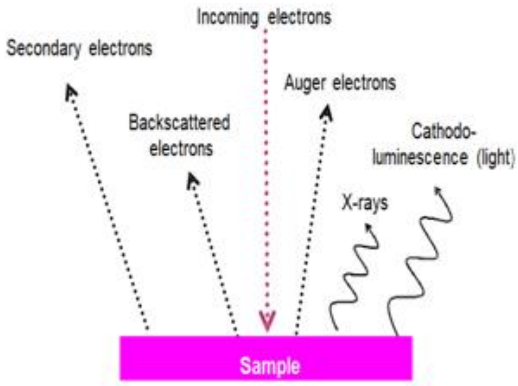


Figure 6: Working of Electrons

The advantages of a scanning electron microscope include its wide-array of applications, the detailed three-dimensional and topographical imaging and the versatile information garnered from different detectors. SEMs are also easy to operate with the proper training and advances in computer technology and associated software make operation user-friendly.

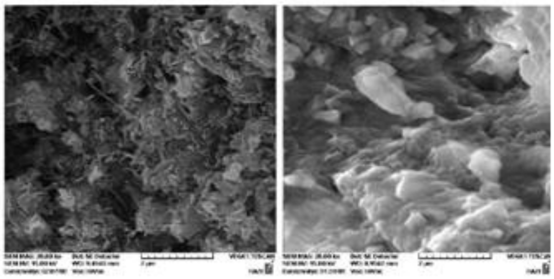


FIGURE 7: SEM OF OPC CONCRETE & SEM OF NANO SILICA CONCRETE

This instrument works fast, often completing SEI, BSE and EDS analyses in less than five minutes. In addition, the technological advances in modern SEMs allow for the generation of data in digital form. Although all samples must be prepared before placed in the vacuum chamber, most SEM samples require minimal preparation actions.

<u>Difference between SEM and XRD Method</u>

Method	General Operating Environment	Data Output	Advantages	Limitations
SEM	20 kV approx. Vacuum chamber Low M.A. current	3-D views of secondary Electron images. X-ray spectra for elements with $Z>10$.	Pore geometry; grain morphology; diagenetic sequences; micro-textures. Ties to some log response and φ-k analyses.	Sample size limited. High organic content frequently causes short filament life. Carbon or Au/ Pd coating generally needed. Shielding required.
XRD	10–40kV X-ray wavelength fixed. Low M.A. current	Diffraction patterns or digital files for comparison to "standards" files.	Mineralogy on semi quantitative scale. Best method for determining clay mineralogy. Does not require microscope.	Sample prep. Powder. Abundances based on measured intensities and areas.

VI. LITERATURE REVIEW

Strength properties and microstructural analysis of self-compacting concrete incorporating waste foundry sand - Ravinder Kaur Sandhu, Rafat Siddique, *Construction and Building Materials 225 (2019) 371–383*

This paper shows the outcomes of an experimental analysis on the effect of waste foundry sand (WFS) as a fine aggregate replacement on the fresh, strength and micro-structural properties of self-compacting concrete (SCC). Characteristics strength and microstructural examinations were carried out up to the age of 365 days. Tests outcomes shows that all concrete mixes displayed the characteristics of fresh state Self-Compacting Concrete. The 28 day compressive strength of Self-Compacting Concrete was 58 MPa. There was 6.38–18.76% decrease in the strength of SCC with the use of about 5–30% WFS at 28 days. Micro-structural outcomes of XRD and SEM authenticate the experimental results. Hence, WFS could be properly used in the manufacturing of SCC.

Concrete Microstructure - A Review- A.S.Adithya Saran1 & P.Magudeaswaran, *Imperial Journal of Interdisciplinary Research 1670-1673 (2016)*

From this review of articles, it was detected that the variation in the microstructure of concrete replicates the variations in properties of concrete. Therefore the microstructural alteration in concrete is mostly due to neighbouring conditions such as time, temperature; chemical changes due to acid attack etc., the microstructure of concrete differs with respect to the quantity of concrete constituents such as cement, aggregates and water.

The concrete microstructure can be improved by the replacement of concrete constituents with other discarded and inexpensive by-products which will be more valuable to prepare high performance concrete which is sustainable.

Microstructural analysis of interfacial transition zone (ITZ) and its impact on the compressive strength of lightweight concretes- P. Vargas, Oscar Restrepo-Baena, Jorge I. Tobón, *Construction and Building Materials 137 (2017) 381–389*

This study aims at researching the influence of physical characteristics such as density, porosity and lightweight aggregate morphology such as pumice and expanded clays, on the microstructure and thickness of ITZ, and identifies the influence that these parameters have on the mechanical properties such as compressive strength of lightweight concretes (LWC). Lightweight aggregates were studied by X-ray Diffraction (XRD), Scanning Electron Microscopy (SEM) and X-ray Fluorescence (XRF), to determine their mineralogical, morphological and chemical characteristics.

Microstructural analysis of self-compacting concrete modified with the addition of nanoparticles - Paweł Niewiadomskia, Damian Stefaniuk, Jerzy Hoła, *Procedia Engineering 172 (2017) 776 – 783*

Based on study researchers have concluded that the addition of nanoparticles progresses the microstructure of self-compacting concrete (SCC). Study of the porosity results showed that concretes improved with the adding of nanoparticles are characterized by lower porosity in relative to pore size of up to 0.015mm in evaluation to concrete which does not comprise nanoparticles in its structure. The positive effect of the adding of nano-powders on the microstructure of the established concrete is also established by the results of the total porosity of the samples. In addition, the showed studies have shown that nano-powders can certainly influence both the hardness and elastic modulus of the cement matrix of concrete. The attained results allow the hypothesis that the suitable usage of nano-materials as an additive for the manufacture of self-compacting concrete (SCC) can increase its largely understood microstructure, and therefore result in its improved durability.

Strength, permeability and microstructure of Self-compacting concrete containing rice husk - Divya Chopra, Rafat Siddique and kunal, *Biosystems Engineering 130 (2015) 72-80*.

Researchers has studied the effect of replacement of cement with rush husk ash (RHA) as supplementary cementitious materials and investigated its effect on strength, durability, and microstructure through their exclusive properties. The micro structural analysis (XRD and SEM) exhibited the enormous formations of C-S-H gel and stable state of hydration procedure at 15% replacement of cement by RHA which was the key reason for the development in the strength and durability of self-compacting concrete with RHA.

VII. MICROSTRUCTURAL ANALYSIS OF M30 GRADE CONCRETE USING SEM ANALYSIS

A typical Mix design for M-30 Concrete is tabulated as below:

Table 1: Mix Design proportion of M30 Concrete

Cement (kg/m^3)	w/c	Water Content (kg/m^3)	Fine Aggregate (kg/m^3)	Coarse Aggregate (kg/m^3)	
				20mm	10mm
430	0.46	211.64	592.74	686.34	454.08

Figure 8: SEM of Concrete at Age of 28 Days

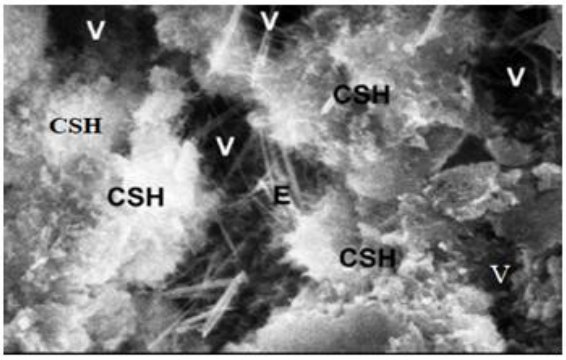


Figure 9: SEM of Concrete higher magnification at Age of 28 Days

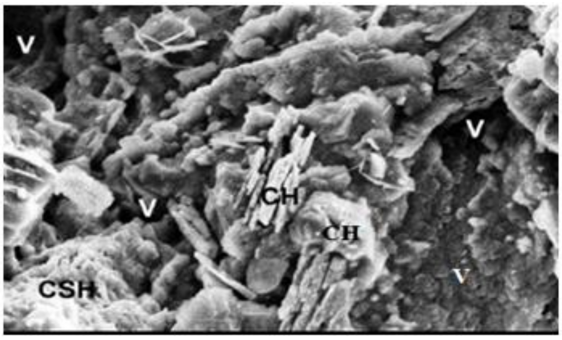


Figure 10: SEM of Concrete at Age of 90 days

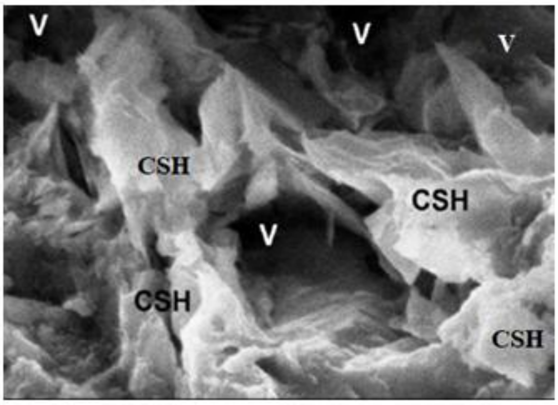


Figure 11: SEM of Concrete higher magnification at Age of 90 Days

VIII. CONCLUSION:

- ❖ For years, scanning electron microscopy (SEM) has assisted to widespread beyond the limitations of understanding of the globe for scientists and non-scientists of all ages. Display of magnified or zoomed images or pictures of materials and these objects aids take the entire context to the world which we all live in.
- ❖ Analysis of microstructure of concrete is the current methodology to scrutinize the mineral alignment in the concrete.
- ❖ Suitable method for specimen or sample preparation is very much necessary while using the SEM for the study of cementitious materials and hardened concrete. Rough surfaces, such as those prepared using only fracture, saw cut, or rough-lapped preparations, are not suitable.
- ❖ The much higher resolving power of the SEM, together with the elemental analysis capabilities of the EDX microprobe, creates a tool that greatly expands our capabilities to study the microstructure of concrete, including the interaction between the various cementitious materials being used today and the microstructural effects of deterioration mechanisms.

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