

SEM Analysis on Sustainable High Performance Concrete

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ABSTRACT: This research reports on the experimental study on Sustainable High Performance Concrete and its microstructural behaviour. The major concrete constituents were replaced with alternative materials. Cement was partially replaced with Fly ash (30% and 35%) and Silica fume (7.5 % and 10%) and fine aggregate was entirely replaced with manufactured sand. Coarse aggregate was partially replaced with recycled aggregate by 30%, 40% and 50% .Seven concrete mixes were prepared and casted into cubes. The concrete cubes was tested for compressive strength after 28 days curing and a considerable value of compressive strength was observed in Mix 4 (7.5 % S.F+ 30% F.A+ 40% RCA).From SEM micrograph of Mix 4, The chemical reaction of silica fume and fly ash with Portlandite ($\text{Ca}(\text{OH})_2$) leads to production of additional C-S-H gel which fairly improves the strength of Mix-4. From the overall study, the substitution of cost-effective materials in the place of normal concrete ingredients considerably improves the strength and microstructural behaviour of concrete

KEYWORDS: Concrete, Recycled Aggregate, Hydrated cement paste, Microstructure, SEM.

I. INTRODUCTION

Concrete usually has unique and complicated microstructure which makes it hard to observe and explore the presence of mineral. Replacement of concrete ingredients alters the concrete microstructure and mechanical properties of concrete. However, there may be some imperfection and failure due to replacement of major concrete ingredients. Analysis of microstructure of concrete is the modern approach to examine the mineral composition in the concrete. X-ray Diffraction Analysis, Scanning Electron Microscope and Energy Dispersive Spectroscopy analysis are some of the contemporary techniques used for phase identification, micrograph and chemical characterisation of the unknown elements in the hydrated cement paste of concrete. The outcome from the micro structural study of concrete would give a clear idea about the development and distribution of hydration products in the hydrated cement paste obtained from the concrete sample. In this research paper, the sustainability of concrete was studied and analysed through Scanning Electron Microscope. The comparison between the compressive strength of the mixes were analysed and correlated with the microstructure of the concrete mixes. The microstructure of the concrete mixes were analysed using Scanning Electron Microscope (SEM) which practically helps to visualize the microstructure of the hydrated cement paste. The objective of this study is to experimentally analyse and study the strength and microstructural behaviour of sustainable High performance concrete by replacing the concrete elements with alternative cost effective by-products.

II. EXPERIMENTAL PROGRAM

A. MATERIALS

1.1 Cement, Fly ash and Silica Fume

OPC (53 grade) conforming IS 1489-1991 was used in this research. Specific gravity of cement was 3.15 and has the standard consistency value as per the standards. . In this study, Cement was partially replaced with Silica Fume (7.5%

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and 10%) and Class F Fly ash (30 and 35%). The replacement of cement content was different for each mix. The Chemical Composition of Cement and Fly ash is shown below in **Table.1**.

1.2 Fine Aggregate

In this experimental study, manufactured sand was used as fine aggregate for concrete mix. The physical properties of manufactured sand was tested and compared with the properties of natural sand in the following **Table.2**. The physical properties of the coarse aggregates were tested as per IS 2386 (Part 1-6)

Table.1
Physical Properties of Natural Sand and Manufactured Sand

Properties	Natural Sand	M-Sand
Bulk Density	1670 kg/m ³	1728kg/m ³
Specific Gravity	2.74	2.70
Water Absorption	1.29	0.982%
Fineness modulus	2.42	2.83

1.3 Coarse Aggregate

In this experimental study, Natural Coarse Aggregates (NCA) and Recycled Coarse Aggregates (RCA) were used. The recycled aggregates were collected by crushing the waste concrete cubes. Coarse aggregate with size of 12.5mm was used in this study. The comparison between the properties of Natural and Recycled Coarse Aggregate is illustrated in the **Table.3**. The physical properties of the coarse aggregates was tested as per IS 2386 (Part 1-6)

Table.2
Physical Properties of Natural and Recycled Coarse Aggregate

Properties	NCA	RCA
Specific Gravity	2.649	2.67
Bulk Density	1670	1728
Water Absorption	0.78%	0.88%
Impact Value	24.3 %	22.75%
Fineness modulus	6.5	6.01

Note: The values shown in the Table.2&3 are obtained by means of standard test process as per IS 2386

B. MIX PROPORTION

In the present study, M60 grade of concrete was adopted and designed based on ACI method. Seven combinations of concrete mixes were prepared with the water-cement ratio of 0.31. The major concrete constituents was replaced with alternative by-products with certain Cement was replaced with Fly ash (30% and 35%) and Silica fume (7.5 % and 10%) and fine aggregate was entirely replaced with manufactured sand. Coarse aggregate was partially replaced with recycled aggregate by 30%, 40% and 50%. The mix proportion designed for normal mix is given below in the **Table.4**.

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Seven mixes were prepared with different ranges of replacement of concrete ingredients. Cement was replaced with Silica Fume and Fly ash. Natural Sand was completely replaced with Manufactured Sand. Natural Coarse Aggregate was replaced with recycled aggregate collected from the concrete cube. Seven mix proportions and its percentage of replacement of concrete ingredients are illustrated in the **Table.5**

Table.3
Mix Proportion

S.No	Description	
1	Grade of Concrete	M60 grade
2	Proportion of Mix	1 : 0.31 : 1.05 : 1.96
3	W/C ratio	0.31
4	Cement	579 kg/m ³
5	Water	179 kg/m ³
6	Fine Aggregate	608 kg/m ³
7	Coarse Aggregate	1136 kg/m ³
8	Admixture	1.79 litre

Table.4
Percentages of Replacement

Mix	Mixes (%)		RCA (%)	Cement			Water	Coarse Aggregate		Fine Aggregate (M-Sand)	Admixture (Conplast SP 430)
	S.F	F.A		Cement	Silica Fume	Fly ash		NCA	RCA		
1	0%	0%	0%	579.19	0.00	0.00	179.55	1135.6	0.0	608	1.79
2	7.5%	30%	30%	361.99	43.44	173.76		794.9	340.7		
3	10%	35%	30%	318.55	57.92	202.72		794.9	340.7		
4	7.5%	30%	40%	361.99	43.44	173.76		681.4	454.2		
5	10%	35%	40%	318.55	57.92	202.72		681.4	454.2		
6	7.5%	30%	50%	361.99	43.44	173.76		567.8	567.8		
7	10%	35%	50%	318.55	57.92	202.72		567.8	567.8		

C. TEST METHODS

1.1 Compressive Strength

The compressive strength was conducted as per IS 516 (1959). The concrete was mixed and casted in 100 x 100 x 100 mm³ cube mould. The casted cube mould was compacted by vibration process for 1 minute. Then, the casted cubes are allowed to set for 24 hrs under room temperature.

After 24 hours, the casted cube moulds are removed and submerged in to the water bath as shown in the **Fig.1**. After 28 days curing, the cube samples are taken out from the water bath and tested form compressive strength in Compression Testing Machine

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Fig.1 Casting, Curing and Testing of Concrete

1.2 Scanning Electron Microscope Analysis

In this current study, the hydrated cement paste obtained from the seven powder samples are subjected to SEM analysis. The range of scale used in SEM analysis was 5 μm with the resolution of x5000. The detailed process of sample preparations for SEM analysis is described below.

After Compressive testing was finished, the cube samples are crushed and the hydrated cement was collected from the innermost core of the concrete cube sample. The collected samples are sieved through 300 μ sieve.

The sample preparation was done by cone and quartering method for reducing the sample size. The sample was dispensed on flat surface so that it takes on a conical shape. The top of the conical shape was flattened. The cone is divided into quarters. Two opposite quarters was discarded; the other two are combined. The process was repeated until the suitable sample size was reached the sample preparation process for micro structural analysis is pictured below **Fig.2**



Fig.2 Cone and Quartering Method for SEM Sample Preparation

III. RESULTS AND DISCUSSIONS

A. COMPRESSIVE STRENGTH

The compressive strength of seven concrete mixes was determined at the age of 28 days and their comparison details were pictured in the **Fig 3**. The concrete cubes were tested for compressive strength after 28 days curing and a fair value of compressive strength was obtained in Mix 4 (7.5 % S.F+ 30% F.A+ 40% RCA) which was

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slight lesser than the normal concrete mix. Even though Mix-4 didn't reached the target strength, it reached to an adequate strength which can also be used as a alternate for the normal concrete mix in certain concreting works

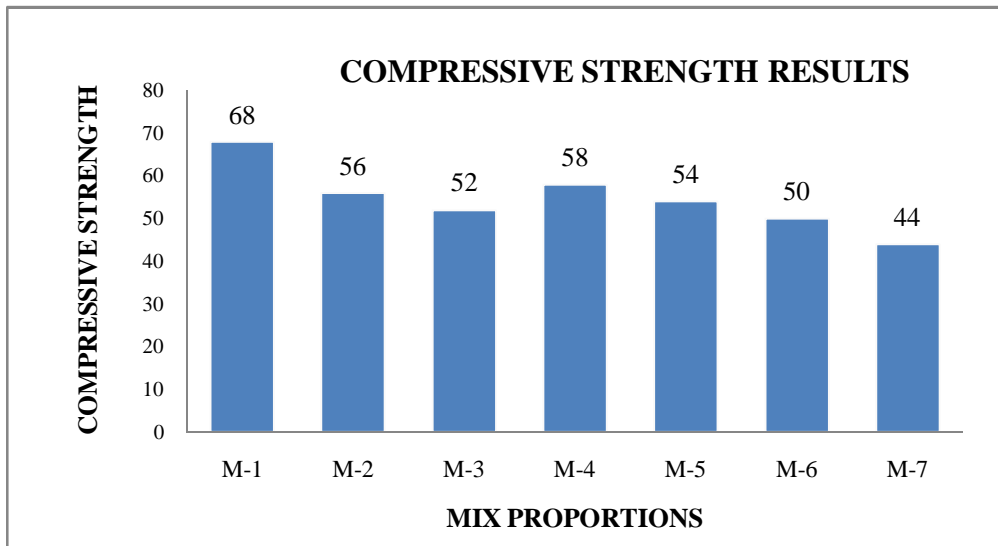


Fig.3 Compressive Strength of Concrete Mixes After 28 Days Curing

B. SEM ANALYSIS

The formation and distribution of hydration products of hydrated cement paste of seven different mix proportions are pictured below. The microstructure of the seven mixes were examined and compared with the nominal mix. The microstructure and strength properties of all the seven mixes were correlated based on the hydration products formed after 28 days. The reason behind the strength of the concrete was analysed and explained based on the growth of hydration products in the microstructure of concrete mixes.

Mix -1 Normal Concrete

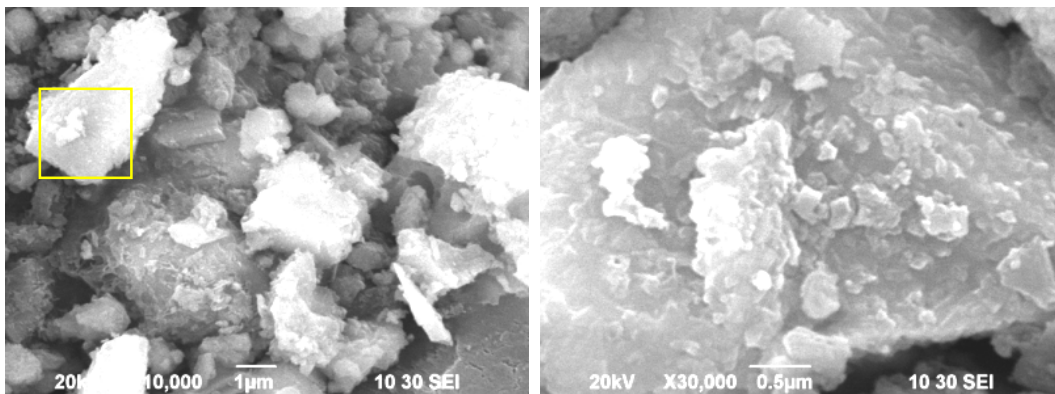


Fig 4 SEM Micrograph of Mix 1(Normal Concrete)

From the SEM Micrograph shown above in Fig 4, it was observed that C-S-H gel was vastly spread on the mixture of hydrated cement paste which was the main cause for the effective strength. Formation of Portlandite Ca (OH)

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$\text{Ca}(\text{OH})_2$ and Calcite (CaCO_3) was visualized all over the exterior surface of hydrated cement paste. These expansion and distribution of mineral elements was one of the reasons for the effective strength of the mix. Replacement of fine aggregate with manufactured sand did not exposed any flaw in strength but, the range of distribution of minerals was changed due to replacement of fine aggregate with manufactured sand.

Mix -2 (7.5 % S.F+ 30% F.A+ 30% RCA)

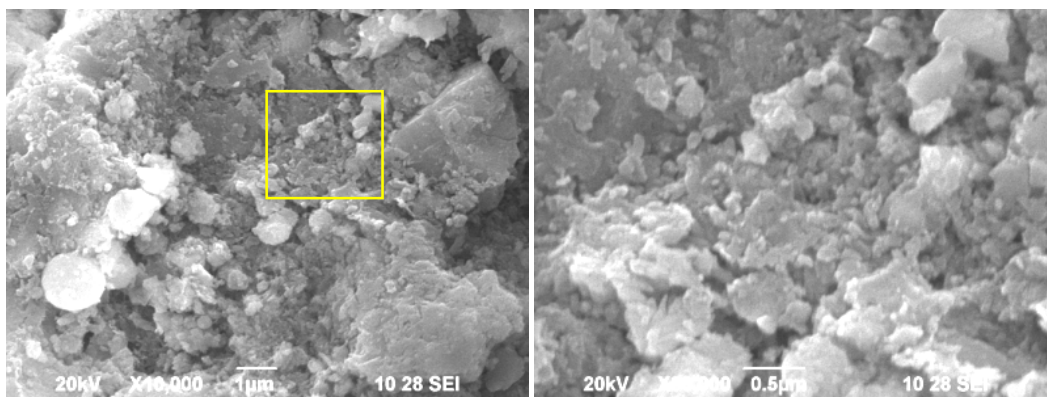


Fig 5 SEM Micrograph of Mix 2(7.5 % S.F+ 30% F.A+ 30% RCA)

In **Fig.5**, The SEM image of Mix-2 was pictured in which the microstructure of hydrated cement paste was visualized. From the SEM micrograph of the hardened cement paste, the distribution of C-S-H was nearly decrease at stage of 28 days due to replacement of cement with fly ash and silica fume. In this mix, the range of development of C-S-H was less which may be due to unreacted particles present in the mix. The accumulation of other major mineral elements such as Portlandite $\text{Ca}(\text{OH})_2$ and Calcite (CaCO_3) crystals are minimal in the microstructure of the mix. The main reason for the decrease in strength was the lack of hydration of particles present in the hardened cement matrix.

Mix-3 (10% S.F+ 3. 5% F.A+ 30% RCA)

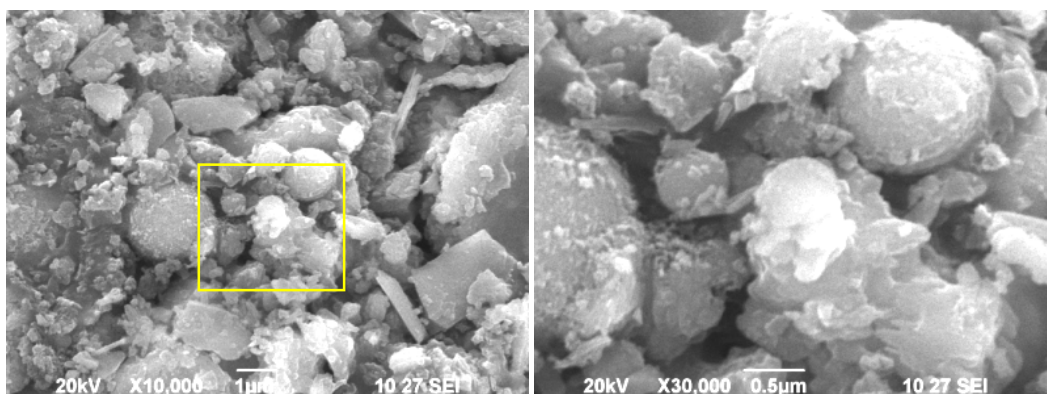


Fig 6 SEM Micrograph of Mix 3 (10% S.F+ 35% F.A+ 30% RCA)

In **Fig 6**, SEM morphology of hydrated cement matrix of Mix-3 was pictured. The Formation of minimum amounts of calcite crystals was found on the surfaces of the hydrated cement paste particles. The non-reacted particles present in the hydrated cement paste leads to development of indistinct structure where the formation of calcite was quiet reduced. The utilization of supplementary materials such as fly ash and silica fume in the mix became unsuccessful because of the lack of chemical reaction between the grain particles. In this mix, Due to the non-reacted particles in the hardened cement paste, the strength of concrete mix was affected.

Mix-4 (7.5 % S.F+ 30% F.A+ 40% RCA)

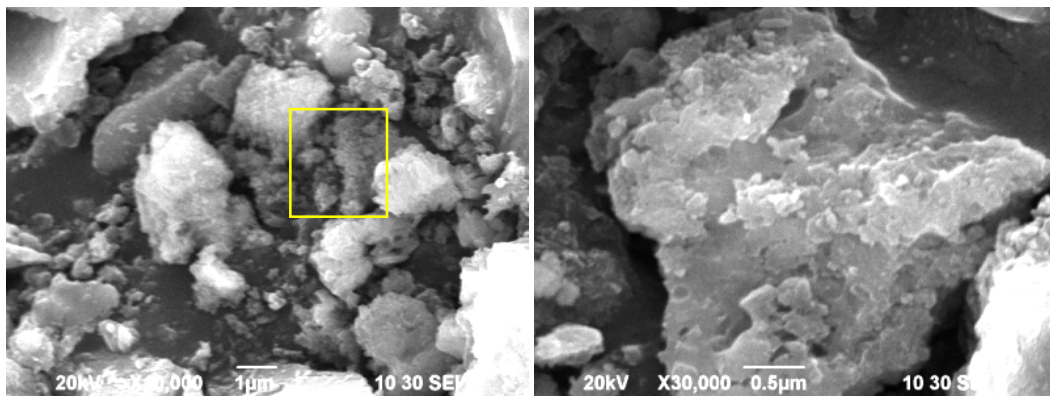


Fig 7 SEM Micrograph of Mix 4(7.5 % S.F+ 30% F.A+ 40% RCA)

The microstructure of hydrated cement paste of Mix-4 was shown in the **Fig 7**. The considerable strength obtained in this mix was due to the pozzolanic activity of silica fume and fly ash. The chemical reaction of silica fume and fly ash with the Portlandite $\text{Ca}(\text{OH})_2$ leads to production of additional C-S-H gel which was the main reason for the strength of this mix. The rate of hydration process in Mix-4 was similar to normal concrete mix but, the existence of minerals elements was quiet different from Mix-1 which manipulates the strength of concrete mix. Even though the strength of concrete mix was not up the anticipated level, it reached to a considerable strength which can also be used as a replacement for the normal concrete mix in certain concreting works

Mix-5 (10% S.F+ 35% F.A+ 40% RCA)

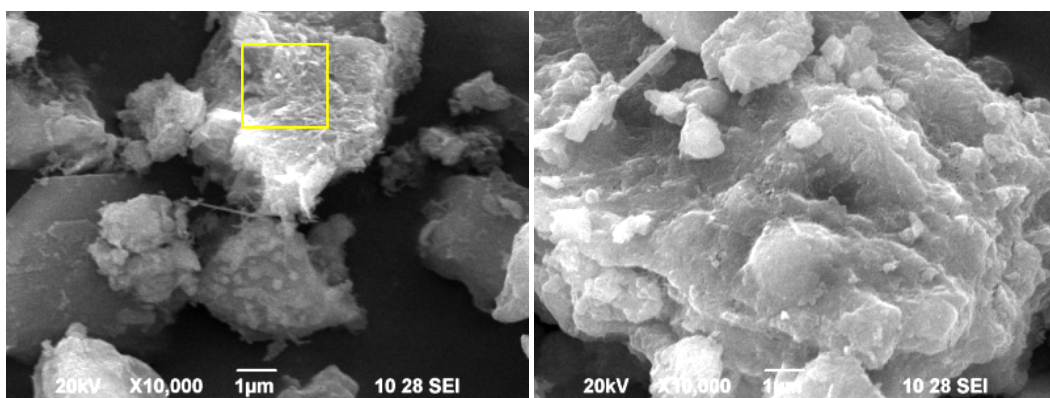


Fig 8 SEM Micrograph of Mix 5 (10 % S.F+ 35% F.A+ 40% RCA)

The SEM micrograph pictures in the **Fig 8** illustrates the microstructural variation during hydration process and production of hydration products in Mix 5, the rate of hydration was insufficient to achieve the strength of concrete due to the unreacted grains present in the hydrated cement paste. Excessive addition of Recycled aggregates in the mix makes the mix vulnerable. C-S-H formation was incomplete and lack of chemical reaction between the Portlandite ($\text{Ca}(\text{OH})_2$) and silica in the silica fume and fly ash results in the inadequate strength in concrete. In the SEM micrograph, the presence of ettringite was clearly evidenced in the micrograph which leads to reduction of development and distribution of C-S-H gel in the hardened cement paste.

Mix-6 (7.5 % S.F+ 30% F.A+ 50% RCA)

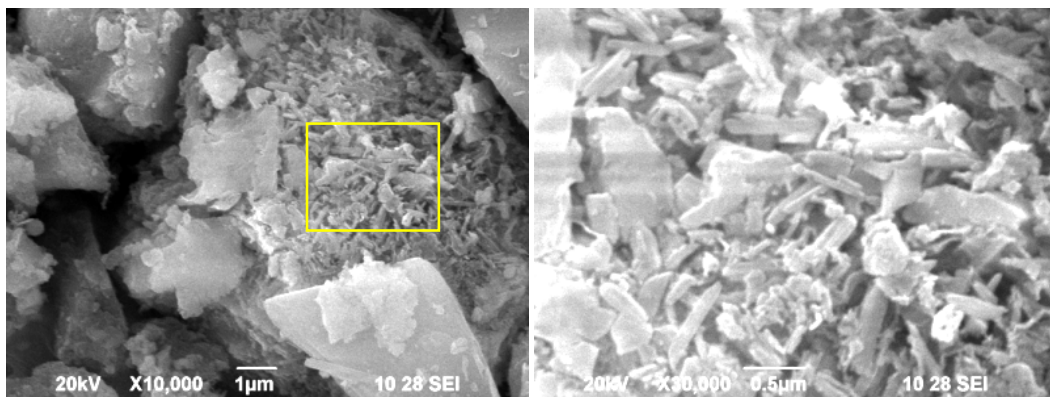


Fig 9 SEM Micrograph of Mix 6 (7.5 % S.F+ 30% F.A+ 50% RCA)

In **Fig.9**, the development and distribution of C-S-H was much lesser due to the unreacted particles of the supplementary material. The existence of C-S-H gel in the mix was inappropriate which affects of strength of the mix. The growth of Calcite crystals was quite high in quantity due to excessive of decomposition of C-S-H gel. These microstructural conditions affected the strength of Mix-5. The reaction between the Portlandite and Silica content in the mix was stopped after 28 days which was also responsible for the low strength of the mix.

Mix-7(10 % S.F+ 35% F.A+ 50% RCA)

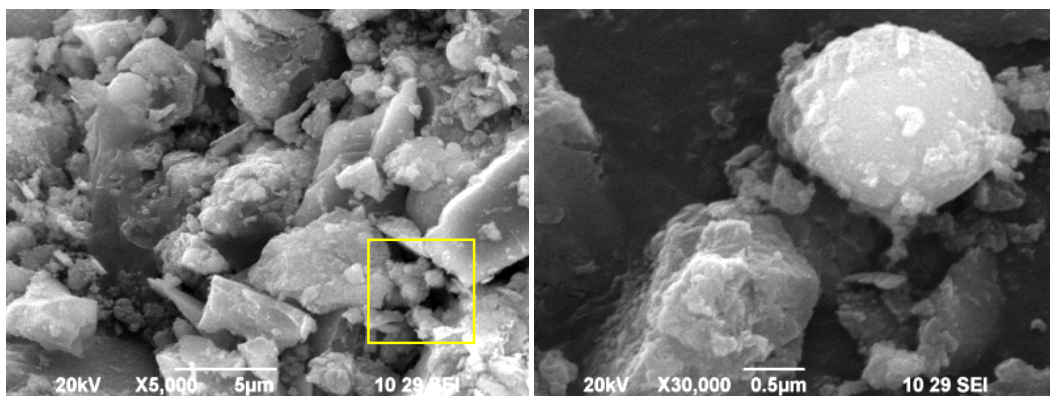


Fig 10 SEM Micrograph of Mix 7 (10 % S.F+ 35% F.A+ 50% RCA)

SEM micrograph shown in the **Fig.10** exposes the microstructural behaviour of Mix-7. The blended cement matrix was covered with the hydrated products such as Portlandite and Calcite. The decomposition process of C-S-H was interrupted which may be due to lack of calcium content in the mixture. Similar to Mix-5 & 6, the reaction between the hydrated products was stopped after 28 days which was due to lack of formation of C-S-H gel in the mix. The inappropriate development of mineral elements in the mix caused the deflection in strength of the mix.

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C. DISCUSSION

From overall study, it is clear that the microstructural behaviour of concrete influences the strength characteristics of the mix. The addition of cost-effective materials such as silica fume, fly ash, Recycled coarse aggregate and M-sand changed the behaviour of microstructure of concrete and also influences the compressive strength of concrete mixes. From the test results of compressive strength, it was observed that replacement of concrete ingredients fairly improves on the strength of concrete mixes. The strength of Mix-4 was not up to the expected level but, the mix is possibly suitable for normal concreting works.

In SEM Observations, the existence of mineral elements and their reactions with the supplementary materials are studied which gives an initiative to understand the microstructure of the concrete mixes. Based on the comparison of the microstructure of concrete mixes, it is clear that the hydration process in the mixes with supplementary materials was different from conventional concrete mix. In Mix-4, the hydration process was quiet similar to the normal concrete mix but, the development of C-S-H gel in the mix was quiet lesser compared to the normal concrete mix (Mix-1).

IV. CONCLUSION

The following conclusion are drawn from the obtained experimental study

1. The concrete cubes were tested for compressive strength after 28 days curing and a fair value of compressive strength was obtained in Mix 4 (7.5 % S.F+ 30% F.A+ 40% RCA) which was slight lesser than the normal concrete mix.
2. Even though Mix-4 didn't reached the target strength, it reached to an adequate strength which can also be used as an alternate for the normal concrete mix in certain concreting works.
3. From the SEM Analysis, Inclusion of M-sand, Recycled Coarse Aggregate (RCA), silica fume and fly ash in the mix leads to production of additional C-S-H gel which helps to achieve the fair strength in Mix-4. The rate of hydration process in Mix-4 was similar to normal concrete mix but, the existence of minerals elements was quiet different from Mix-1 which manipulates the strength of concrete mix
4. From the overall study, the substitution of cost-effective materials in the place of concrete ingredients nearly improves the strength and microstructural behaviour of concrete.

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