

**STRENGTH AND DURABILITY PROPERTIES OF
SELF-COMPACTING CONCRETE INCORPORATING
RICE HUSK ASH AND METAKAOLIN**

*A Thesis submitted In
partial fulfillment of the requirement for the
award of the degree of*

**DOCTOR OF PHILOSOPHY
IN
CIVIL ENGINEERING**

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CERTIFICATE

This is to certify that thesis entitled, “**Strength and durability properties of self-compacting concrete incorporating rice husk ash and metakaolin**”, submitted by Mr. Anhad Singh Gill in partial fulfillment of requirements, for the award of degree of Doctor of Philosophy in Civil Engineering, submitted in the Department of Civil Engineering, Thapar Institute of Engineering & Technology, Patiala is a record of the candidate’s original research work carried out by him under my guidance and supervision.

The matter embodied in this thesis has not been submitted into any other University or Institute for the award of any degree.



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DECLARATION

I hereby certify that the work which is being presented in the thesis entitled “**Strength and durability properties of self-compacting concrete incorporating rice husk ash and metakaolin**” being in partial fulfillment of the requirements for the award of the degree of Doctor of Philosophy in Civil Engineering submitted in Department of Civil Engineering, Thapar Institute of Engineering & Technology, Patiala is an authentic record of my own work carried out by me under the supervision of Dr. Rafat Siddique and refers other research’s work duly listed in the reference section.

The matter presented in this thesis has not been submitted in part or full to any other University of Institute for the award of any degree in India or abroad.



(Anhad Singh Gill)

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ABSTRACT

This study is about design and development of self-compacting concrete (SCC) using supplementary cement materials (SCMs). These SCMs has wide applications in concrete which contribute in enhancement of strength and durability. In this study, two types of SCMs have been used; Metakaolin (MK) and Rice Husk Ash (RHA). MK is a SCM which results from the heating of kaolinite clay in span of specified temperature, while RHA is a by-product obtained from the paddy fields. Rice husk when burnt in range of specified temperatures it results in RHA.

To carry out the investigations, firstly, a control mix (M1), without any of the SCM (MK & RHA) was designed. Then, three mixes (M2, M3, M4) were prepared with partial replacement of cement with MK in proportions of 5, 10 and 15%. Three other mixes (M5, M6, M7) were made with the partial replacement of fine aggregates with RHA in the varying proportions of 10, 20 and 30%. Then, on the basis of 28-day compressive strength, ten additional mixes (M8 – M16), with combination of both (MK & RHA) were made. In mixes M8 – M10, MK content was kept constant at 5% & RHA content was varied between 10 to 30%. Similarly, in mixes M11 – M13 & mixes M14 – M16 MK content was kept constant at 10% & 15% respectively, and RHA content was varied between 10 to 30%, similar to mixes M8 – M10. Mixes made with MK and RHA were tested for fresh properties, strength properties (compressive strength and splitting tensile strength), durability properties (water absorption, sulphate resistance, RCPT) and micro-structural properties (SEM and XRD) up to age of 365 days. Statical analysis of the results was also undertaken.

All the mixes fulfilled the EFNARC criteria for the fresh properties. Use of MK and RHA increased the water demand but that can be managed by increasing the slight dose of super-plasticizer. The inclusion of MK and RHA positively affected the compressive strength at all ages. Mix M11 (10MK10RHA) showed the highest strength upgradation. It showed 26%, 27%, 42% and 48% increase in strength than the control mix at 7, 28, 90 and 365 days respectively. Also, all other mixes exhibited more strength than the control mix. Similar trends were observed for splitting tensile strength results. The highest strength was achieved by the mix M11 (10MK10RHA), which showed the maximum strength of 2.74, 4.43, 5.12 and 5.96 MPa at 7, 28, 90 and 365 days respectively. It was observed that strength increased up to 10% RHA and after that it starts decreasing, while in case of MK it increases up to 10% and slightly decreases at 15%.

The water absorption and porosity for mixes containing MK+RHA was lowered as against the control mix. Inclusion of MK and RHA positively affected water absorption and porosity but at the same time level of water absorption and porosity rises with the increased percentage of MK and RHA. Inclusion of MK and RHA has positive impact on sulphate resistance and RCPT of SCC mixes. All the mixes made with the use MK and RHA had better resistance to sulphate attack as against the control mix. With 15%MK and 10%RHA there was 44%, 74% and 77% decrease in chloride permeability at 28, 90 and 365 days respectively

SEM images showed lesser voids and more homogenous structure along with formation of C-S-H gel for the mixes made with MK and RHA. XRD images showed maximum amount of unused Silica for the mix made without MK and RHA, while mixes with MK and RHA had lesser amounts of unused silica. This is the reason for better performances of these mixes.

All the test results of SCC mixes were statically analysed and comparatively studied using 'ANNOVA' test. The mean values of mixes made with MK and RHA was higher than the control mix for all strength and durability properties. Also, lower bound and upper bound interval was higher for all mixes made with MK and RHA as compared to control mix. The correlation analysis depicted strong relationship among the various results.

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LIST OF UNITS

Units	Word(s)
Kg	Kilogram
g	Gram
mg	Milligram
°C	Degree Celsius
%	Percent
m	Meter
cm	Centimetre
mm	Millimetre
µm	Micrometre
N	Newton
KN	Kilo Newton
MPa	Mega Pascal
GPa	Giga Pascal
Kg/m ³	Kilogram per cubic meter
N/mm ²	Newton per square millimetre
θ	Theta

ABBREVIATIONS

Abbreviations	Word(s)
ACI	American Concrete Institute
ASTM	American Society for Testing Materials
BIS	Bureau of Indian Standards
CM	Control Mix
CH	Calcium Hydroxide
CS	Calcium Silicate
CSH	Calcium Silicate Hydrates
EDS	Energy Dispersive X-ray spectroscopy
GGBFS	Granulated Ground Blast Furnace Slag
HRWR	High Range Water Reducer Admixture
MK	Metakaolin
OPC	Ordinary Portland Cement
RHA	Rice Husk Ash
RCPT	Rapid Chloride Permeability Test
SCC	Self-compacting Concrete
SCMs	Supplementary Cement Materials
SF	Silica Fume
SEM	Scanning Electron Microscopy
WA	Wood Ash
XRD	X-ray Diffraction

Chapter 1

Introduction

The chapter gives basic introduction of the topic and its various constituents. A brief overview is given about self-compacting concrete (SCC), its definitions as stated by research of various authors, various advantages and limitations, motive behind its development, basic difference between normal vibrated concrete and SCC, then introduction about various SCMs. Then significance of the research is discussed along with the gap in the research area, research objectives and methodology used.

1.1 Self-Compacting Concrete

Construction industry is among the oldest industries, running across the decades and it had played a very crucial role in the modernization. The country's development depends on its construction industry's strength to great extent. So, it is very important that a country should have strong construction industry. Construction industry these days is using many construction materials, but concrete is one material which is used from past so many years. It is one of the oldest construction materials used in construction. The evolution of concrete had completely changed the outlook of construction industry. It made construction easier to great extent, at the same time increasing its durability also. There is no hesitation in saying that its significance will increase with the time.

Modernization is the other thing which comes with the time. So, construction industry is no different case. A lot of changes have occurred in construction industry with time. So, to meet these modern-day construction standards and to meet the challenges of other modern construction materials in order to sustain in the construction market, timely modifications are necessary. One of such modification in the concrete is the evolution of SCC.

1.1.1 Description of SCC

SCC is a class of concrete which can move by itself and no external vibrations are required. SCC evolution took place in Japan in year 1988 and Professor Ozawa (1989) was behind this evolution. But before this the idea of SCC was purposed by Hajime Okamura (1997) in year 1986. The main reason behind this development was the durability. Also, at that point Japan was facing shortage of professional labour and the increase in reinforcement volumes had added another problem. So, the proper compaction of concrete was becoming laborious job. This all was affecting the durability of the concrete. Evolution of SCC proves to be very

advantageous for the construction industry specially to deal with the issues regarding cast-in-place concrete operations (Okamura and Ouchi, 1999). There is no afflict of workers expertise on self-compacting concrete, neither the quantity nor shape of reinforcement in the formwork has much effect on SCC and as SCC has fluidity on higher side at the same time remaining homogenous, makes SCC suitable for use in any kind of environment (Bartos, 2000).

The concept of SCC was not all together new as there have been quite number of examples in the past where concrete operations had been done without external vibrations. Under water concreting, mass concreting and shaft concreting are the common examples. But these concreting operations are not much reliable from strength point of view and neither from quality. SCC's evolution was primarily directed towards achieving the elevated quality in terms of strength and durability and that too on consistent basis. Earlier, SCC's use to count on cement content and generally it was kept on higher sides. That makes concretes vulnerable to shrinkage as well as segregation. So proper care was required for placing the concrete. After the arrival of super-plasticizers, they were used in SCCs but that affected the overall costs of the concrete.

The main reason behind the evolution of contemporary SCC was to achieve a superior quality concrete. The process started in Japan in 1980s and the root of this evolution was below par performances of concrete structures, which was mainly due to ununiform and uncomplete compaction (Dehn et al, 2000). It was discovered that there is no means which can assure the uniform and complete compaction of concrete, so the centre of interest shifted towards different type of concrete which do not require any external vibrations. This open on to the evolution of SCC by Professors Okamura and Ozawa in year 1986 in Japan. This concept of SCC was swiftly taken by large Japanese contractors. They also used their techniques to develop SCC and tested it by their own testing methods and techniques (Bartos, 2000).

In the starting of 1990s, a very restricted public knowledge was available regarding SCC, which was mainly in Japanese language. The big contractor houses which developed their SCC kept the basic concept as secret to get the monetary benefits. They sold the SCC under various commercial names such as non-vibrated concrete, super quality concrete etc. On the same time when Japanese started using SCC, research was going on in another parts of world such as Scotland, Canada etc on mix designs and quality aspects of concrete. The main motive was to develop a concrete matching the standards of Japanese SCC, for which they were using new admixtures and other methods (Ferraris, 1999).

1.1.2 Definition of SCC

There are number of definitions as stated by various authors, but the common among all was that SCC is the concrete which moves by itself under own weight and no external vibrations are required as in case of the normal vibrated concrete.

EFNARC (2005) stated that “Self-compacting concrete is an innovative concrete that does not require vibration for placing and compaction. It is able to flow under its own weight, completely filling form work and achieving full compaction, even in the presence of congested reinforcement.”

The British Standard (BS EN 206-9, 2010) defined SCC as the “concrete that is able to flow and compact under its own weight; fill the form work with its reinforcement, ducts, box outs etc, whilst maintaining homogeneity.”

1.1.3 Motive for development of SCC

SCC is a kind of superior quality concrete which can move itself without any external vibrations. It can even attain consolidation by filling the form work in case of limited space due to congested reinforcement arrangement (Zhu et al., 2001). So, this makes SCC useful under the conditions which does not fully or partly permit the external compaction of concrete. Conditions like concreting under the water, concreting in pile foundations or where there is congested setup of reinforcement bars makes a tough case for the external compaction of the concrete. So, SCC is the most suited kind of concrete under such circumstances (Khayat et al., 2004).

As discussed earlier, the main motive behind the discovery of SCC was inability to achieve absolute compaction of concrete, which was resulting in poor performances of concrete structures. The quality and consistency were suffering. Also, in cases of congested reinforcement setup, professional workers are required. This was the major problem in Japan around year 1983. Suddenly there was scarcity of experienced, qualified and professional workers around 1983 in Japan. As a result, there was ununiform and uncomplete compaction in fresh state, which was reflecting in the quality of the structures.

So, in the late 1980s, Japan started work on the idea of eliminating the need of external compaction for the concrete. As cent percent compaction was not easy to achieve as well as it requires professional workers, so this idea can solve the dual purpose. Work started in the University of Tokyo on this concept and by the 1990s a new kind of concrete was developed

that does not require external vibrations, i.e. self-compacting concrete. SCC could pass through tight openings in the formwork and could fill its every corner (Bartos, 2000).

By 1990s, the preliminary version of SCC was ready and was pushed for first kind of a real test. The results were positive and it performed adequately. The preliminary version was given name as “High Performance Concrete.” Concurrently, Professor Aitcin defined “High performance concrete” as concrete with superior durability as of its low water-cement ratio (Ouch et al., 1996). After that Okamura (1997) gave the new name to this concrete as “Self-compacting high-performance concrete.”

1.1.4 Advantages of SCC

SCC can be looked upon as "the most comprehensive evolution". Coming into existence due to lack of professional workers in Japan, SCC is now used worldwide specially in the European countries. The reason behind its growing popularity is the number of advantages it offers in construction as well as in quality. Some of the advantages are given below (Krieg, 2003 and ENFARC, 2002).

- Speedy construction,
- Decreased professional workers,
- Easy process of placing,
- Absolute and uniform compaction,
- Quality surface finish,
- Enhanced durability,
- Elevation in bond strength,
- Reduction in noise pollution, as vibrators are eliminated, and
- Safe working environment.

As external vibrations are eliminated it results in number of advantages. This also results in more even colour of concrete as due to absence of vibrations which minimise the segregation between solid particles and liquid. This results in superior finish and quality.

In case of normal concrete structures, the quality of structures can be elevated by using greater volumes of reinforcement, restricting cracks by providing smaller diameter reinforcement bars and using the proper form work. But on the same time all these factors decrease the probability of uniform and complete compaction (Okamura et al., 2003). Self-compacting concrete can use

the above-mentioned factors to improve the quality along with proper compaction. This also leads in increased efficiency by decreasing the time taken for construction as well as cost incurred on labour.

The main disadvantage of SCC is its overall cost. As SCC have powder content on higher side along with the dosage of admixtures, which normal vibrated concrete do not have. So due to this SCC becomes 20 to 60% costlier than normal vibrated concrete (Nehdi et al., 2004). But in cases of large-scale projects this dis-advantage is subsided by savings in the cost of labour as well as in time.

1.1.5 Difference between SCC and NVC

The key property of SCC is its ability to remain homogenous while it is transported and then placed in the formwork. So, it should have increased resistance to segregation between mortar and aggregates while it flows between the spaces in the formwork (Okamura et al.,2003). So, it is very important property for SCC as it directly effects its performance. Now this homogeneity i.e. increased resistance to segregation and ability to flow freely can be acquired by:

1. Providing higher quantities of fine sized particles,
2. keeping the w/p ratio on the lower side,
3. Superplasticizer usage (Okamura et al., 2003).

As quantity of fine particles is on the higher side, the SCC shows similarity with the structure of “High-performance concrete” which have no flaws in placing and compaction, self-compaction abilities in the fresh state, no shortcomings in early age and strong defence against the external factors in the hardening stage. On the same time as content of coarse aggregates is limited, it can cause the negative effect on some properties like modulus of elasticity, creep and shrinkage. These properties in turn can affect the deformation capability, prestress loss and reflection characteristics (Mata, 2004).

SCC basically have cement, coarse aggregates, fine aggregates, filler material like fly-ash, silica fume etc., water and super-plasticizer. Cement and aggregates used for normal concrete can be used in SCC. So, there is not much difference between the composition of both except the increased quantity of fine particles to attain the self- compacting abilities. So, this is basically done by adding SCMs like fly-Ash, silica fume, limestone powder etc. along with the addition of super-plasticizers.

There are basically three key characteristics of SCC i.e. high deformability, controlled flowability and segregation resistance (Khayat et al., 2004). The first property i.e. high deformability can be explained as ability to flow freely so that it can be able fill all spaces in the form work. This property is governed by the size and amount of aggregates and also the amount of friction solid particles has among themselves. This can be controlled by addition of HRWRs and by the quantity and size of the aggregates in the mix. The second property i.e. controlled flowability can be explained as ability of concrete to pass through and around the obstacles in the form work. It mainly depends upon the geometry and shape of the form work. The third property i.e. segregation resistance can be explained as its ability to stay homogenous while transportation and placing. It is connected to the cohesiveness of concrete in its fresh state. This can be controlled by using a VMA in the mix, or by keeping the paste volume on higher sides or by taking the free water content on lower side.

So, the process of making SCC involves design of suitable mix keeping the above-mentioned factors in mind. The designed SCC mix should be checked for its fresh stage properties and should possess all fresh stage properties. In fact, it should have three above mentioned key properties in its fresh stage. If SCC have these abilities in its fresh stage then it can significantly boost the quality of concrete structures.

1.1.6 Applications of self-compacting concrete

Since its development in 1980's (Okamura, 1997) SCC had travelled a long way in terms of its applications. Developed due to shortage of skilled labour in Japan (Okamar and Ouchi, 1993) SCC is now looked as one of the finest inventions in concrete industry due to its enhanced strength and durability and also the ease with which it can be laid in the framework. In the past decade SCC had witnessed a rapid increase in its use all over the world. It's increased strength and durability had landed it on the hot seat. SCC has been widely used for the construction work in harsh environmental conditions. Also, SCC is finding its application in construction of containment vessels, chimneys, nuclear reactors, water tanks, crude oil storage tanks, highways, tunnels, under water construction, bridges and many other structures.

The Sodra Lanken (1997) project in Sweden is finest example of uses of SCC in construction. The four-lane highway consist of number of rock tunnels which includes concrete lining. Use of SCC for this project was perfect as it has large volumes of reinforcement and rough surfaces of rock masses.

Burj Khalifa (2010) constructed in Dubai is another example of use of SCC in modern day construction industry. The building is 828 meters tall and consists of 166 stories. It is the tallest structure in the history.

Dragon Bridge (2012) in Seville, Spain is another significant project made with the use of SCC. The magnificent bridge, divided into four spans has a unique shape of dragon.

Ritto Bridge in Japan is example of fine construction work with the use of SCC. The bridge is situated on New Meishin Expressway. The bridge consists of number of piers as high as 65 meters. The high strength concrete and reinforcement was used to make it earthquake resistant. So, positioning of heavy reinforcement was done and hence SCC was the perfect type of concrete to be used under the given circumstances.

Kaiga project is example of use of SCC in India. The project constructed basically to boost the quality, so the use of SCC was ideal for the project, as its ability to enhance the quality of the structure.

1.2 Supplementary Cement Materials

SCMs can be the waste products of some industry productions and can be the side products of certain productions also. The production of industrial by-products in present times is quite on the higher side. Sometimes the disposal of these by-products causes a big problem. Also, their disposal can cause an environmental problem (Smarzewski and Hunek, 2016). So, it is very needful that disposal of these by-products should be given due importance and should be done in such a way that can reduce the burden on environment. Also, the use of aggregates in concrete is depleting the natural resources (Milicevic et al., 2016). So, it becomes very important to preserve these natural resources and to find a certain alternate to these natural resources.

Use of these industrial by-products in concrete is one of the mind-blowing solutions to the above-mentioned problems (Tittareli and Morconi, 2010). This can fulfil the dual purpose, as their use in concrete can reduce their disposal problem and also using these products in place of natural aggregates will help in preserving the natural aggregates, ultimately reducing the burden on the environment. There are number of industrial by-products such as marble dust, glass waste, foundry sand, coal bottom ash, waste plastic etc. which has been used as replacement for aggregates in the past. The use of these by-products in place of natural aggregates depends upon their properties (physical and chemical). Their use in concrete

industry is very beneficial and government should encourage this practise (Regev et al., 2014). There is quite some research available on the use of these by-products as replacement of fine/coarse aggregates and cement in concrete. Some of the SCMs are discussed below.

1.2.1 Metakaolin

Metakaolin (MK) is a SCM resulting from the kaolinite clay. MK is basically generated from burning of kaolinite clay in the temperature spans of 650 – 800° C. On heating kaolin losses up to 14% hydroxyl water and converts to MK (Caldarone et al., 1994). Mk's purity level can be managed by controlling its production and ensuring the quality control of superiority. MK has high contents of alumina and silica. This makes MK a pozzolanic material as this silica and alumina reacts with calcium hydroxide (CH) that is product of hydration reaction. This in turn makes calcium hydro-silicate (C-S-H) and calcium hydro-alumino-silicate (gehlenite – C₂ASH₈). The past literature has solidarity that MK helps in enhancing the mechanical (at early ages) and durability properties. The reason stated for this enhancement is the action of C-S-H gel. It refines the capillary structure of the concrete, which in turn enhances the strength and durability properties.

As MK is produced from kaolinic clays, it is a kind of eco-friendly product. The reason is the non-production of CO₂ in its making, yet it finds very infrequent use in concrete. The reason behind this infrequent use is basically the price factor of MK (Guneyisi and Gosoglu, 2008). Mk price is oh higher sides as compared to cement prices. The reason can be it's less production and can also be due to the strategies of producers. So, the only reason the MK find its use in concrete is its ability to enhance strength and durability rather than economical or environmental (Vejmelkova et al., 2010).

SCC has been produced using SCMs like fly-ash, silica fume etc. in the past (Khayat, 1999). But the use of MK in SCC was not done much in the past and is comparatively new approach (Hubertova and Hela, 2007). The use of MK in SCC results in good fresh state properties as compared to silica fume (SF) addition and that too with the small dosage of HRWRs. The reason is MK's particle, which is fine than cement but bigger than SF (Caldarone et al., 1994). MK offers other handful of advantages also such as its ability to bleed lesser amounts of water, creamer texture and its ability to provide quality finish to concrete surface (Caldarone et al., 1994).

At present, there is not much literature available on the consequences of MK addition on the various properties of SCC. But the past research has also established the fact that MK is an acceptable addition in concrete along with hydraulic lime binder (Cachim et al., 2010). Some literature even suggested that MK is more effectual than SF (Kapoor et al., 2016). Also, addition of MK results in good workability as compared to SF and at the lower costs (Hassan et al., 2012). So, it will very interesting thing to probe the detailed role of MK in the properties of SCC, specially from the durability aspect.

Physical and Chemical Properties of MK

MK's particle is 3 μm in size (average). Its properties should be in accordance with the guidelines set by ASTM C 618. These requirements are given in Table 1.1 and it's physical and chemical compositions are given in Table 1.2 and 1.3.

Table 1.1: Specifications for MK (ASTM C 618)

Modified specification requirement limit	Limit
Item	
Silicon dioxide (SiO_2) + Aluminum oxide (Al_2O_3) + Iron oxide (Fe_2O_3)	Min 85 %
Alkalis	Max 1.0 %
Loss on Ignition	Max 3.0 %
Fineness: Amount retained when wet – sieved on 45 μm sieve	Max 1.0 %
Strength activity index at 7 days (% of control)	85
Increase of drying shrinkage of mortar bars at 28 days	Max 0.03 %

Table 1.2: Physical composition of MK

Property	Value				
	Guneyisi et al. (2010)	Siddique (2011)	Madandoust& Mousavi (2012)	Sfikas et al. (2014)	Badogiannis et al. (2015)
Specific gravity	2.5	2.60	2.6	2.5	-
Bulk density (g/cm ³)	-	0.3 – 0.4	-	-	1.41
Physical form	Powder	Powder	Powder	Powder	Powder
Color	Off White	Off White	Off White	Off White	Off White
Specific surface area (m ² /g)	-	-	2.54	1.41	1.41

Table 1.3: Chemical composition of MK

Constituents	Percent				
	Guneyisi et al. (2010)	Madandoust& Mousavi (2012)	Karahan et. al. (2012)	Aghabaglou et. al. (2014)	Badogiannis et al. (2015)
CaO	0.78	0.2	0.40	0.29	0.03
SiO ₂	52.68	52.1	63.0	63.53	47.85
Al ₂ O ₃	36.34	42.8	31.0	32.36	38.20
Fe ₂ O ₃	2.14	1.6	1.10	0.54	1.29
MgO	0.16	0.21	0.30	0.18	0.04
SO ₃	-	0.00	0.05	0.01	-
K ₂ O	0.62	0.32	-	1.08	-
Na ₂ O	0.26	0.11	-	0.33	-
LOI	0.98	-	0.95	1.00	12.30

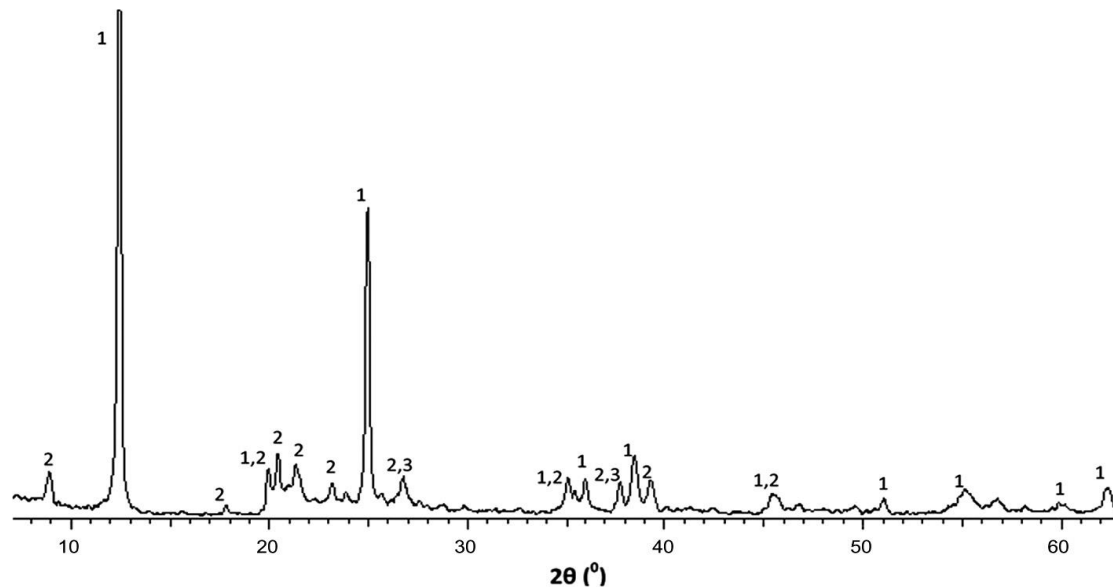


Fig. 1.1: XRD pattern of kaolin: (1) kaolinite, (2) illite and (3) quartz (Sfikas et al., 2014)

As metakaolin is derived from kaolin, some researchers tried to identify the properties of kaolin. Sfikas et al. (2014) performed XRD on kaolin and tried to estimate the minerals present on the basis of XRD peaks. It can be observed from Fig.1.1 that the kaolin contains kaolinite and certain amounts of illite and quartz. The amount of kaolinite is quite high in kaolin as compared to other two compounds.

Advantages

Use of MK in concrete has number of advantages. Number of researchers had tried to investigate the role of MK in concrete and outlined the various advantages of MK. Siddique (2011) in his publication stated the various benefits of MK addition in concrete. These are given below:

- Enhancement in compressive and flexural strengths
- Increased resistance to permeability
- Enhanced resistance to chemical attack
- Enhanced durability
- Reduction in alkali-silica reactivity (ASR)
- Resistance to shrinkage
- Improved workability and quality finishing

1.2.2 Rice husk ash

RHA is a by-product obtained from paddy fields. India is one of the largest sources of rice production and rice cultivation is done on large scales. Once the rice season is complete, and rice has been processed from the field, lot of rice husk was left for disposal. Even all over the world huge quantity of rice husk (120 million metric tons) was left behind. The disposal of such large quantities is problem in itself and needs some innovative solution. Now if this rice husk is flawlessly burnt at temperatures beneath 700°C , it gives rise to RHA. This RHA is a rich source of amorphous silica (Chindaprasirtet al.,2007; Khani et al., 2009). The main constituents of rice husk are cellulose, lignin and silica. Now if this rice husk is burnt at prescribed temperature range, it results in elimination of cellulose and lignin. The only thing left after burning is silica, which becomes the main constituent of RHA. The quality of RHA can be managed by controlling the burning process of rice husk and can result in higher quality of RHA particle with desired particle size and surface area.

Also, RHA can be of two types i.e. crystalline and non-crystalline. The burning conditions of rice husk decides the type of RHA produced among the above-mentioned types. If the burning is done uncontrolled and at higher temperatures above 800°C , the produced RHA will be crystalline in nature. It will have below par pozzolanic properties (Nagataki, 1993). If the burning takes place at controlled temperatures i.e. $500 - 800^{\circ}\text{C}$, the produced RHA will be non-crystalline in nature. It will have silica content on higher side and will have excellent pozzolanic properties (Nagataki, 1993).

RHA is a pozzolanic material as of high silica composition. Its particle size is very fine and quality can also be controlled. So, it's no wonder that an addition of RHA in concrete can result in a number of benefits (Nehdi et al., 2003). These benefits include enhanced quality, overall cost cutting as RHA is just a waste product and also it can help in effective disposal of RHA. This can help in reducing environmental burden as well as carbon dioxide emission. RHA's ability to provide above mentioned advantages depends upon its silica content as well as its surface area (Cook, 1984; Mehta, 1992). Its reactivity can also be increased by increasing its fineness.

The past research on RHA's application in concrete is affirmative of the fact that RHA can successfully blended in concrete. Some literature suggested that up to 20% RHA can provide positive results in concrete (Chao-Lung et al., 2011). RHA's addition in SCC had resulted in decreased unit weight, water absorption, porosity, compressive strength, UPV and overall cost

(Safiuddin et al., 2010). So that means RHA can be blended in SCC and it affects most of the properties positively and some of them negatively.

Physical and Chemical compositions of RHA

The particle of RHA is quite fine when looked in comparison with cement. It ranges between 5 – 10 μm and its colour is grey to black. So RHA particle is finer than cement but bigger than metakaolin and silica fume. RHA's silica content is on higher side. Normally its content lies between 80 to 85%. Also, silicon dioxide (SiO_2), aluminium oxide (Al_2O_3) and iron oxide's (Fe_2O_3) minimum amalgamate percentage should be 70 as per ASTM standards. The minimum value for LOI for RHA is 12 %. Some of key chemical and physical properties are given below in Table 1.4. and 1.5.

Table 1.4: Chemical composition of RHA.

Constituents	Percentage (%)		
	Mehta (1992)	Zhang et al. (1996)	Bui et al. (2005)
Silica (SiO_2)	87.2	87.3	86.98
Alumina (Al_2O_3)	0.15	0.15	0.84
Iron Oxide (Fe_2O_3)	0.55	0.55	1.40
Magnesium Oxide (MgO)	0.35	0.35	0.57
Sodium Oxide (Na_2O)	1.12	1.12	0.11
Potassium Oxide (K_2O)	3.68	3.68	2.46
Sulfur Oxide (SO_3)	0.24	0.24	-
Loss on ignition (LOI)	8.55	8.55	5.14

The surface area of RHA particle is quite on higher side and its structure is cellular in shape (Zhang and Malohtra, 1996). This property of RHA particle allows it to act as a VMA (Memon et al., 2011). As RHA is used in SCC to replace cement or fine aggregates, resulting in more available surface area. The increased surface area helps in water retaining which further results in modified viscosity. The enhanced viscosity boosts the segregation resistance of SCC mixes. Simultaneously, the increase in viscosity also leads in declined filling and passing ability. For this HRWR such as poly-carboxylatecopolymer is required to be added to the mix.

Table 1.5: Physical composition of RHA

Property	Value			
	Mehta (1992)	Zhang et al. (1996)	Feng et al. (2004)	Bui et al. (2005)
Mean Particle Size (μm)	-	-	7.4	5
Specific Gravity	2.06	2.06	2.10	2.10
Fineness: Passing 45 μm (%)	99	99	-	-

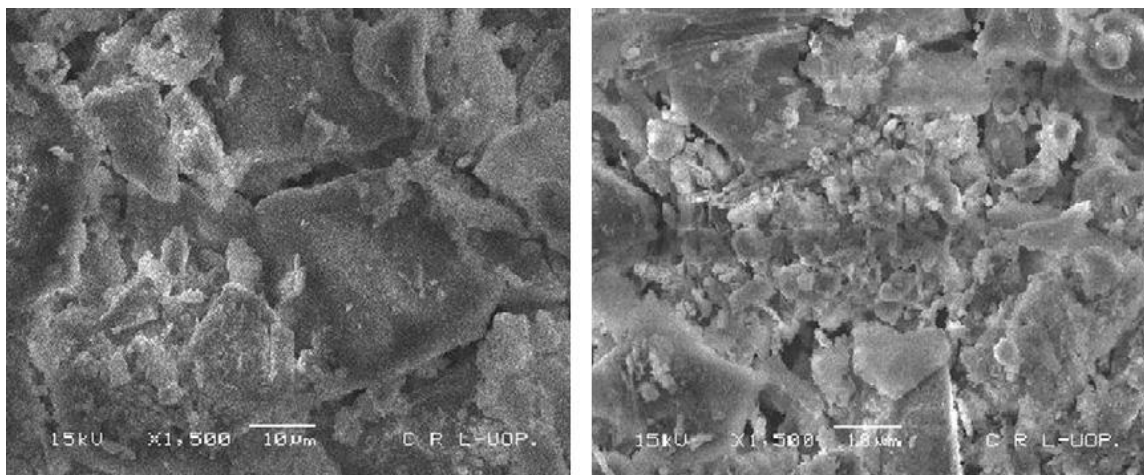


Fig. 1.2: Raw RHA without sieving and RHA with 150 μm sieving (Memon et al., 2011)

Memon et al., 2011 gave the above SEM image for the RHA used in their research work. They observed that RHA had a highly irregular particle shape, while sieving of RHA lead to the reduced size of RHA particles. Also sieving resulted in decreased un-burnt carbon particles, which helped in upgraded quality. So, it can be clearly observed that RHA particle is irregular in shape and have large surface area.

Khan et al. (2012) also gave the SEM images for the RHA used in their work, shown in Fig. 1.3 and 1.4. They observed that the RHA particles are of irregular shape and there was presence of micro pores also. They stated that this irregular shape is the main reason behind the significant alteration of properties of the mixes. So basically, RHA particle is of an irregular shape.

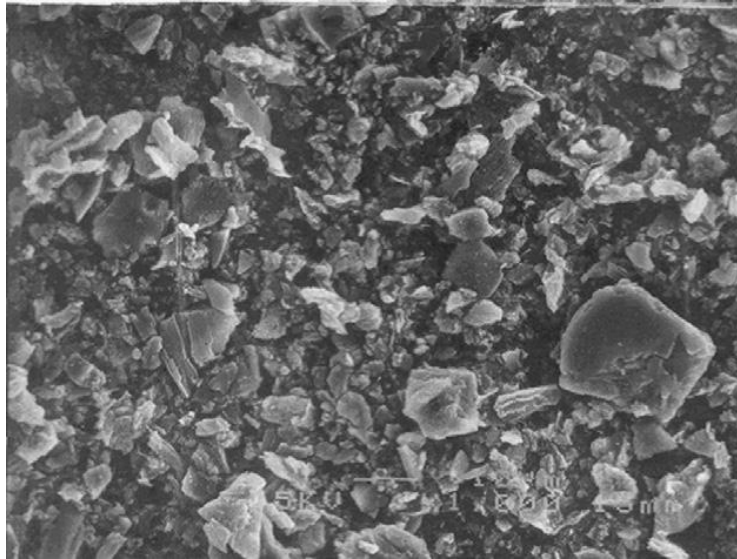


Fig. 1.3: SEM image of RHA (Khan et al., 2012)

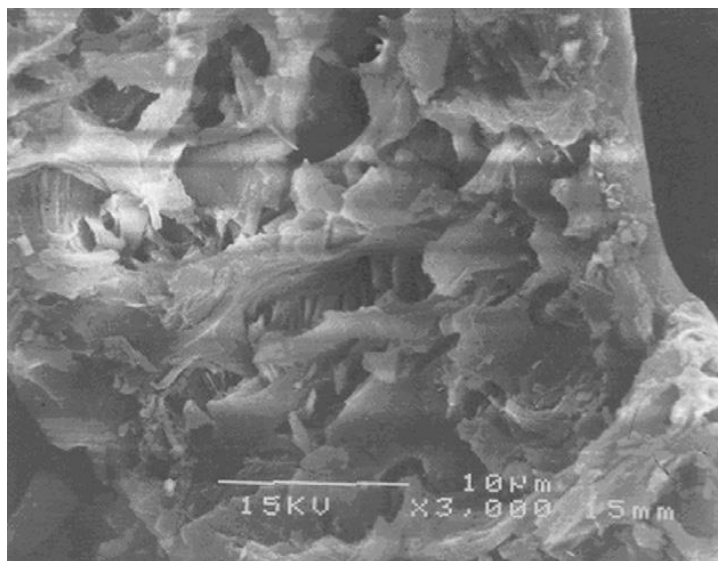


Fig. 1.4: Magnified SEM image of RHA (Khan et al., 2012)

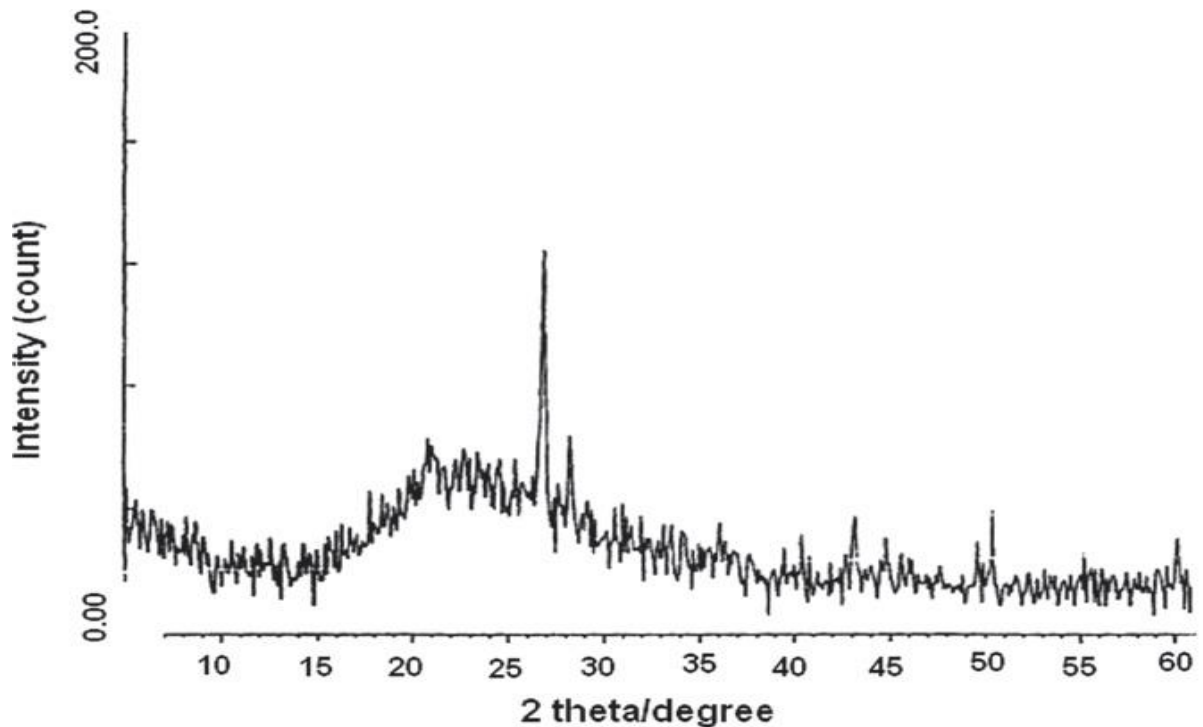


Fig. 1.5: XRD pattern of RHA (Khan et al., 2012)

Khan et al., (2012) also gave x-ray diffraction analysis of RHA in a powder form, shown in Fig. 1.5. The XRD pattern of RHA confirmed its rich silica content. The peaks of SiO_2 clearly indicates towards its pozzolanic nature as well as non-crystalline type. The major reflection or peak of crystalline SiO_2 occurs at Bragg 2-theta angle of 26.64. It has been stated in the past literature also that RHA has silica content on higher sides. The burning process should be controlled and flawless which results in non-crystalline RHA, rich in silica and makes it ideal for use in concrete.

1.2.3 Silica fume

Silica fume (SF) is basically by-product of silicon alloys manufacturing. Production of silicon alloys involves minimization of quartz constituent by using coal and iron. The whole process is done in electric furnaces and at high temperatures around 2000°C (St John, 1998). Now, during the process silicon gas makes its way to the furnace top. Once it reaches the top, it comes in air contact which results in its condensation. As a result, it takes shapes of amorphous silica particles of size ranging between $0.1 - 1\ \mu\text{m}$. These particles are called SF. That means SF particle is extremely fine as compared to cement particle. Surface area of SF particle is also on the higher sides. Chemically SF is rich in silica content, with silica making 85 – 96 % of its composition (Kosmatka et al., 2002).

So, the composition of SF makes it perfectly suited for playing a positive role as a constituent of concrete. This is well defined by number of researchers. As SF has high silica content so when SF is added, pozzolanic action takes place. This silica combines with calcium hydroxide (C-H) from cement ending up in of C-S-H gel (Mindless et al., 2003). This C-S-H gel acts as the source of increased strength and durability of the concrete. Also, C-S-H helps in pore structure refinement of the mix. As SF has very fine size this also helps in betterment of pore structure of the mix. These fine particles tend to fills the voids and pores thus resulting in enhanced properties of the concrete (St John, 1998). But on the other hand, addition of SF leads in elevated water requirement of the mix. The high surface area and fine size of SF leads in high quantities of retained water, thus resulting in viscous mix. So, to overcome this, superplasticizer is required to be added in the mix. SF if used in controlled proportions it can lead in enhancement of strength at early age along with some durability properties (Ramachandran, 1984). The typical physical and chemical composition of silica fume are given in Table 1.6 and 1.7.

Table 1.6: Physical composition of SF (khayat,and Aitcin, 1987)

Color	white or pale-grey to a dark grey
Specific Gravity	2.2 - 2.55
Specific Surface Area	About 20000 m ² /kg
Particle Size	Mostly fine spheres with a mean diameter of .1 micron
Bulk Loose Density	230-300kg/m ³

Table 1.7: Chemical composition of SF (Khayat, and Aitcin, 1987)

Constituents	Percent
SiO ₂	90-96
Al ₂ O ₃	0.5-0.8
MgO	0.5-1.5
Fe ₂ O ₃	0.2-0.8
CaO	0.1-0.5
Na ₂ O	0.2-0.7
K ₂ O	0.4-1.0
C	0.5-1.4
S	0.1-0.4

1.2.4 Ground Granulated Blast Furnace Slag (GGBFS)

GGBFS is basically secondary product of iron production. The manufacturing of iron basically involves simultaneously heating of three products namely iron ore, limestone and Coke. This heating is done in blast furnace at temperature range of 1500° C. This results in formation of molten iron and molten slag. Molten iron is heavier in weight and settles down while molten slag comes at the top. The main constituents of this slag are silica and alumina, which comes from iron ore, while it also has presence of oxides which it takes from limestone. This molten slag is cooled down with the help of jets of water at larger pressures. When molten slag comes in contact of the water, it starts converting into the particles of granular structure. These particles have size not larger than 5 mm. The reason for their smaller sizes is the rapid cooling by water jets. Now, this granulated slag is further grinded to powder of very fine size. The grinding operation involves the use of rotating ball mill. This fine size powder is called GGBFS. Also, there are different methods of cooling the slag and the method used defines the resulting product. Some of these resulting products are air-cooled blast furnace slag (ACBFS), expanded or foamed slag, palletized slag, and GGBFS.

GGBFS is normally white in appearance. Addition of GGBFS in concrete gives a bright and lighter look to the concrete as compared to greyish look provided by the cement addition. Also,

the GGBFS addition results in quality finishing of surface. Typical physical and chemical compositions of GGBFS are given in Table 1.8.

Table 1.8: Physical composition of GGBFS (Tasong et al., 1999)

Property	Value
Physical form	Off white powder
Bulk density (Kg/m ³)	1200
Specific gravity	2.9
Specific surface (m ² /kg)	425-470

GGBFS main chemical constituents are CaO, SiO₂, Al₂O₃, MgO. So basically, it consists of silica, alumina and some oxides. The presence of these constituents makes GGBFS suitable for use in the concretes. So, depending upon its chemical composition GGBFS offers number of advantages when used in concrete. Typical chemical composition of GGBS as reported by Tasong et al. (1999) is given in Table 1.9.

Table 1.9: Chemical properties of GGBS (Tasong et al., 1999)

Composition	Percentage (%)
SiO ₂	35.34
Al ₂ O ₃	11.59
Fe ₂ O ₃	0.35
CaO	41.99
MgO	8.04
MnO	0.45
S ₂	1.18
S ₀₃	0.23

1.2.5 Wood ash

Wood ash (WA) is a leftover product of wood combustion. When wood or products of wood such as bark, saw dust etc. are burnt it results in formation of inorganic and organic residue, which is generally termed as WA. The use of WA in concrete depends upon its chemical and physical properties, which in turn depends upon the type of wood, species to which it belongs and the method used for its burning. So, it becomes very important to identify the species and types of wood which can result in useful WA production. Then it becomes necessary to utilise correct techniques for combustion of these wood as combustion techniques depends upon number of factors such as temperature at which it is burnt, type of boiler used for burning, fuel used for burning etc. The effects of these factors should be carefully examined as these can have reverse affect on WA production. Like the production of ash decreases with the increased burning temperatures (Etiegni and Campbell, 1991).

Similarly, the type of wood also has saying in quality and quantity of WA produced. Like more quantities of WA are produced if the wood used for burning is of hard type, while the quantity of WA decreases with the use of soft type of wood. Then barks and leaves results in more quantities of WA production as compared to other parts of the trees. Then other factors such as type of combustion, temperatures at which burning is done, type of fuel used for burning, the type of wood and the area from where wood is collected etc have important saying in the quality and quantity of WA produced. So, there can be high degree of variability in quality of WA depending upon above mentioned factors. Therefore, it becomes significant to test the WA before using it in concrete.

WA normally consist of carbon in span of 6 – 30 %. Some of the other main constituents of WA are calcium, which is in the span of 8 – 33 %, potassium in span of 3 – 4 %, magnesium in span of 1 – 2 %, phosphorus in span of 0.3 – 1.4 % and sodium in span of 0.2 – 0.5 % (Campbell, 1990). The percentages of these constituents effect the quality of WA in one or the other way. The typical chemical composition of wood ash is given in Table 1.10.

Table 1.10: Chemical properties of Wood Ash (Abdullahi, 2006)

Constituent	Percentage (%)
SiO ₂	31.8
Al ₂ O ₃	28
Fe ₂ O ₃	2.34
CaO	10.53
MgO	9.32
K ₂ O	10.38
NaO	6.5
LOI	27

1.3 Significance in the Research

India is a country with wide range of industry sector. There are large number of industries which have production on large scales. So undoubtedly, the production of any of the useful product will also results in generation of by-products. As India has wide spectrum of industry units so the waste production is also on the higher sides. So, it is need of hour that these waste products should be managed in efficient way, so that they do not become burden on environment. One of the innovative ways of management is their utilization in concrete. This serves the dual purpose as it will help in cutting the overall cost of concrete along with efficient disposal of these by-products.

One of such by product is RHA. Once the rice season is complete, and rice has been processed from the field, lot of rice husk was left for disposal. Now if this rice husk is flawlessly burnt at temperatures beneath 700⁰C, it ends up in RHA. This RHA is a rich source of amorphous silica. Constituents of SCC makes it costlier as compared to normal concrete. There is intensive research going on to minimize the cost of SCC and many researchers have tried to achieve SCC using RHA. Being a by-product, RHA can be helpful in cost cutting of SCC. The limited research that has done, provides positive results and indicates towards the more use of RHA in SCC.

MK is basically a pozzolanic material which generates from the burning of kaolinite clay in the temperature span of 650 – 800° C. MK has high contents of alumina and silica. This makes MK a pozzolanic material as this silica and alumina reacts with C-H from cement. The past literature has solidarity that MK helps in enhancing the mechanical (at early ages) and durability properties of concrete along with environmental benefits (Vejmelkova et al., 2010).

1.4 Gap in the Research Area

Concrete has the widest application in the field of civil Engineering and its demand is increasing day by day. There were many advancements in the concrete technology depending on the day to day demands and requirements. SCC is the step forward in the advancement of the concrete industry. For self-compaction it is very clear that it should have good flowability, which requires addition of super-plasticizers and supplementing materials. This makes SCC costlier.

There is intensive research going on and many researchers have tried to achieve SCC using RHA as it does not hamper the flowability and strength of SCC. So, it can be used as fine aggregate in different proportions, which can help in cutting the total cost of SCC without hampering the strength properties of SCC. MK is basically a pozzolanic material and there is not much research on use of metakaolin in SCC. A little research that is available gives us idea that metakaolin being rich in silica content enhances the strength and durability of concrete. Also, here is very less research on durability aspect of SCC using metakaolin, specially resistance to sulfate attack, which is a vital property for SCC. Neither there is any attempt made to use these two products together.

So, the principal goal of the research is to use these two products i.e. rice husk ash and metakaolin in combination. RHA will be used as replacement for fine aggregates, and the goal of using RHA is to cut the overall cost. While metakaolin will be used as replacement of cement, and the objective of using metakaolin is to enhance strength of concrete and to check whether it can improve its durability also.

1.5 Research Objectives

The objectives of the proposed study are as under: -

1. Development and characterization of SCC containing RHA and MK as partial replacements of fine aggregates and cement, respectively.

2. To study the strength properties of SCC containing RHA and MK as partial replacements of fine aggregates and cement, respectively.
3. To study the durability properties of SCC mixes containing RHA and MK as partial replacements of fine aggregates and cement, respectively.
4. SEM & XRD characterization of SCC mixes containing RHA and MK.

1.6 Methodology

SCC will be designed using the appropriate mix design and incorporating RHA and MK to have compressive strength in span of 30 to 40 MPa at the age of 28 – days. RHA will be used to replace fine aggregates in three proportions between 10 to 30%. While metakaolin will be used to in place of cement in three proportions between 5 to 15%. Tests would be carried out to determine various properties of cement, fine aggregates, coarse aggregates, rice husk ash and metakaolin.

Properties to be investigated

The following test would be performed for: -

Fresh concrete properties (EFNARC 2005)

- Slump flow
- U-box
- L-box
- V-funnel

Strength properties

- **Compressive strength:** Compressive strength of SCC containing RHA and metakaolin will be carried out as per IS: 516-1959. On an average 3 specimen would be cast for each series.
- **Splitting tensile strength:** Splitting tensile strength of SCC containing RHA and metakaolin will be carried out as per IS: 5816-1999. On an average 3 specimen would be cast for each series.

These properties would be determined at the age of 7, 28, 90 and 365 days.

Durability properties

- **Sulfate resistance:** Sulfate resistance of SCC containing RHA and metakaolin will be carried in accordance with ASTM C 1012. On an average 3 specimen would be cast for each series.
- **Rapid chloride permeability test:** RCPT of SCC containing RHA and metakaolin will be carried in accordance with ASTM C 1202. On an average 3 specimen would be cast for each series.
- **Water absorption:** Water absorption of SCC containing RHA and metakaolin will be carried out as per ASTM C 642-2002. On an average 3 specimen would be cast for each series.
- **SEM & XRD:** SEM & XRD test would be conducted for concrete specimens.

These properties would be determined at the age of 28, 90 and 365 days

1.7 Organization of Thesis

Thesis is organised in five chapters as detailed below:

Chapter 1- Introduction: This chapter gives introduction about self-compacting concrete, its various applications and advantages, various SCMs- metakaolin, rice husk ash, silica fume, GGBFS, wood ash, then research objectives and methodology.

Chapter 2 – Literature review: It deals with the work done by the various researchers in the past on SCC using various SCMs.

Chapter 3 – Experimental programme: It provides the details of experimental programme for the present research, materials used, techniques adopted for casting and testing.

Chapter 4 – Results and discussions: It gives detailed results of the various tests conducted in the present research and their discussion.

Chapter 5 – Conclusions: It deals with conclusions of the study.

The list of **references** used for the present research is also given after the end of chapter 5.

CHAPTER 2

Literature Review

This chapter gives details about the published literature on self-compacting concrete and use of industrial by-products and supplementary cement materials in SCC.

2.1 Properties of SCC made with Metakaolin

Qian and Li (2001) probed strength properties of concrete using metakaolin (MK) partially in place of cement. MK was used in three percentages i.e. 5,10 and 15 %. MK used had particle size of 2 microns. They performed splitting tensile strength test. They observed that the MK enhanced the tensile strength and it continues to increase with MK percentages. There was growth of around 7 % strength with 5 % MK addition, 15 % with 10 % MK and 28 % with 15 % MK addition. So, results stated that MK can be successfully used in concrete and can be used to replace cement.

Karoline and Arnaldo (2010) looked into the role of fine size of MK in self-consolidating concrete. As self-consolidating concrete should possess ability of free flow and to pass through the congested reinforcement setup in the form work at same time it should remain homogeneous. So, to fulfil this SCC requires quantity of fine particles on higher sides along with addition of super-plasticizers. The quantity of fines can be increased by addition of mineral admixtures such as MK. So, to probe the role of fineness, authors used MK of three different fineness in place of cement in span of 5 – 35%. They observed from the fresh properties results that as the fineness increases, it leads in more consumption of super-plasticizer. But at the same time the dosage of super-plasticizer can be decreased by adjusting the paste volume. Paste contents of 0.45 and 0.50 showed better results and required less dosage of super-plasticizer. But in terms of strength properties results were poor at the above-mentioned paste volumes.

Vejmelkova et al. (2011) used metakaolin and blast furnace slag in SCC and explored its various fresh and hardened stage properties. They observed that the mixes made with MK are less viscous as compared to the mixes made with blast furnace slag. Also, the mixes with MK showed notable yield stresses. In terms of strength properties, again mixes made with MK had edge over mixed made with blast furnace slag. Strength of mixes having MK shows significant improvement at both early and later stages. It was more than the mixes made with blast furnace slag. Same was the case with durability properties. Mixes with MK showed decline in water

absorption and water penetration. There was insignificant mass loss for these mixes after 56 cycles of freeze and thaw.

Guneyisi et al. (2012) used combinations of various mineral admixtures such as fly ash (FA), ground granulated blast furnace slag (GGBFS), silica fume (SF) and metakaolin (MK) in their study of SCC. They designed total of 65 mixes and to make sure that their mixes passes the fresh properties tests, they utilized super-plasticizer also. They used FA and GGBFS in percentages of 20, 40 and 60 %, while SF and MK were used in percentages of 5, 10 and 15%. It was observed that FA has negative effect on compressive strength with its increased percentage, while the addition of GGBFS doesn't affected the strength to much extent. On the other hand, both MK and SF affected the strength positively. Their addition leads to enhancement of strength as compared to the mix made without their addition. When these were used in ternary combinations, PC + SF + MK combination gave the best results in terms of compressive strength. In terms of drying shrinkage FA, GGBFS and MK gave the positive results, while SF leads in increase in drying shrinkage of the mix.

Hassan et al., (2012) probed the role of metakaolin (MK) and silica fume (SF) use in SCC. They replaced cement with MK and SF in different proportions. MK was used in span of 3 – 25 %, while SF was used in span of 3 – 11 %. They ensured that all mixes pass the slump flow test so for that they adjusted the dosage of HRWR accordingly. The test results indicated that MK and SF had constructive effect on the strength of the mixes. In case of SF, best results were obtained at 8% substitution which resulted in 14% strength enhancement. Same results were obtained for MK, but in case of MK strength continues to increase with increasing percentage of MK. In case of drying shrinkage, SF performed better than MK after 400 days. The mixes containing MK performed better in water absorption and ultrasonic pulse velocity. Mix having 20% MK showed minimum of weight gain and pulse velocity, which mean it had lesser pores and voids. The chloride permeability was also positively affected by MK inclusion. There was almost around 90% reduction in chloride permeability with 20% addition of MK.

Karahan et al. (2012) scrutinize the consequences of metakaolin (MK) presence on properties of self-consolidating light weight concrete (SCLC). They developed SCLC mixes containing different MK contents. They used MK to replace fly ash in varying percentages of 0, 20, 40 and 60%. The results for fresh properties suggested that inclusion of MK decreased the filling and passing ability, which worsened with the increasing percentages of MK. They also observed that MK inclusion doesn't affected the strength of SCLC in any positive way. But

MK inclusion certainly affected the durability of SCLC in positive way. There was up to 10% decrease in water absorption and porosity at 60% MK content. Both initial and secondary sorptivity decreased for MK mixes when compared to the mix without MK. MK addition had notable effect on chloride ion penetration, which showed noteworthy improvement with MK.

Madandoust and Mousavi (2012) scrutinize metakaolin (MK) effect on properties of SCC. They replaced cement with MK in span of 0 – 20% and also used three w/b ratios of 0.32, 0.38 and 0.45. The SCC with MK in its fresh state passed all the tests and gave adequate performance, even without use of and super-plasticizer. In hardened state the results were quite encouraging. In terms of compressive strength, there was around 27% increase with MK addition at early age. Same was the case in tensile strength and electrical resistivity of mixes having MK. There was increase of 11.1% and 26% in tensile strength and electrical resistivity. A low water absorption was also observed with the MK inclusion. On the whole they concluded that 10% MK can be considered as optimum replacement percentage.

Madandoust and Mousavi (2012) made use of MK for their research on SCC. They used four different proportions of MK to replace cement. MK was used in span of 5 – 20%. The strength properties had notable improvement with MK addition specially at the early age. There was around 27% enhancement in compressive strength of the mix made with MK when related to mix without MK. From durability point of view, they observed that use of MK resulted in low water absorption. On the whole they concluded that 10% MK can be considered as optimum replacement percentage.

Perlot et al. (2013) observed the effect of combination of metakaolin (MK) and limestone on various properties of SCC. They used MK in form of powder as well as slurry. MK has positive effect on properties of concrete and reasons behind this are the pozzolanic nature of MK and its fine size. Also, utilization of MK in concrete can reduce the environmental pollution as it reduces CO₂ emission. They designed different SCC mixes having varying content and form of MK. They also included limestone to observe the combined effect. They stated that MK can be used in slurry form along with limestone filler. They had positive effect on the strength and durability properties of SCC. It also offered some other benefits such as short mixing time, increased workability and enhanced strength at early ages. The use of MK in slurry form is also suitable for SCC to be used for precast usage.

Ioannis et al. (2014) examined the role of metakaolin (MK) on characteristics of self-compacting concrete. For their study they replaced cement or limestone powder with metakaolin. They prepared total of nine mixes which included one control mix and rest of the mixes were prepared by addition of MK. The fresh state tests indicated that higher mk/c or mk/p levels negatively affects the workability. To compensate this the dosage of superplasticizer is required to be increased. They stated that this negative effect is due to the high surface area of MK particles. They also observed from their results that the inclusion of MK makes the mix more viscous. Also, MK inclusion resulted in minor blocking issues, specially at the higher percentages of MK. At the same time MK inclusion increased the segregation resistance of the mixes. In mechanical properties they observed that there was increase in compressive strength and splitting tensile strength with higher metakaolin replacement levels and this was observed for both replaced materials, i.e. cement and limestone powder.

Efstratios et al. (2015) looked into the effects of incorporation of metakaolin (MK) on durability properties of Self-Compacting Concrete. They used MK partially in place of cement and limestone powder at different levels. They prepared total of nine mixes, one control mix, four mixes by replacing cement with metakaolin and four other mixes by replacing limestone powder with metakaolin. They detected that as MK is added in the mixes, it affected its open porosity. The values of porosities decreased with higher levels of MK. Along with this there was decrease in gas permeabilities also with the addition of MK. The co-efficient for gas permeability showed decrease in its value with increase in MK levels. It was also observed that there was exponential decrease in chloride migration co-efficient, which means addition of MK results in enhancement of chloride penetration resistance of SCC mixes. All the mixes having MK got upgraded by at least two classes in terms of chloride penetration resistance.

Gill and Siddique (2015) probed the strength properties and sulphate resistance of self-compacting concrete incorporating metakaolin (MK). For this purpose, they used MK in span of 5 – 15% in place of cement. Three different percentages of MK were used in the above-mentioned span along with w/b ratio of 0.48. Results stated that SCC mixes having MK passed all the tests in its plastic state. In hardened state tests again MK inclusion effected the results. There was roughly a 25% increase in 28 - day strength, when MK percentage raised to 15% from 5%. Splitting tensile strengths were in spans of 1.6 to 2.2 N/mm², from 2.35 to 3.0 N/mm² and 2.55 to 3.3 N/mm² at 7, 28 and 56 days respectively with metakaolin content. At age of 28 days there is 18 to 40% increase, when compared to control mix at same age. MK inclusion

enhanced the sulphate resistance. When measured in terms of compressive strength loss, there is loss of .8, .7 and .6% for replacement level of 5, 10 and 15% respectively.

Singh and Singh (2016) tried to examine the effect of recycled coarse aggregates (RCA) on certain properties of SCC. They also used metakaolin (MK) in their study. They used RCA in percentages of 0, 25, 50, 75 and 100% in place of natural coarse aggregates (NA). The designed SCC mixes were tested for carbonation and electrical resistance. Test results indicated toward the negative performance of mixes made with 100% RCA in relative to mix made with 0% RCA. But at the same time addition of 10% MK along with RCA helps in neutralising the negative effect of use of RCA. So they concluded that RCA can be used to make SCC, which can help in preserving the natural aggregates, but in addition to that MK should also be used along with RCA to compensate the negative effect of RCA on properties of SCC.

Gill and Siddique (2018) investigated the durability and micro-structural properties of self-compacting concrete (SCC) made up of metakaolin (MK) and rice husk ash (RHA). For this purpose, metakaolin (MK) was used to replace cement by weight in three different proportions of 5, 10 and 15% and fine aggregates were replaced by rice husk ash (RHA) in proportion of 10%. A total of four mixes, including the control mix, were designed. Test results revealed that all the mixes passed all plastic stage tests of SCC. The addition of MK and RHA affected the workability but which can be improved by increasing the slight dose of water reducing admixture. Furthermore, hardened stage tests results were also positive. The use of MK and RHA positively affected the compressive strength, increasing it by 27%, 42% and 48% at 28, 90 and 365 days respectively in relation to the control mix. Durability properties showed significance improvement with the use of MK and RHA. Micro-structural analysis further confirmed the positive trend of results.

2.2 Properties of SCC made with Rice Husk Ash

Ahmadi et al. (2007) tried to use rice husk ash (RHA) partially in place of cement for designing SCC mixes. RHA was used in span of 0 – 20% and studied its effect on strength of SCC. Test results for their work revealed that mixes having RHA showed decline in compressive strength till age of 60 days in relative to mix without RHA. But after 60 days results start getting positive for mixes with RHA. These mixes showed enhancement in compressive strength after 60 days of curing. Among all the mixes, mix made with 20% RHA had the best of results in terms of

strength. So, from their work they draw conclusion that using RHA in SCC is a lucrative option as it can help in reduction in cost as well as improvement in quality.

Safiuddin et al. (2010) looked into the use of rice husk ash (RHA) in self-compacting high-performance concrete (SCHPC). They used RHA in span of 0 – 30% along with varying w/b ratios. Mixes in their fresh state showed sublime filling ability with slump values between 600 – 770 mm. In terms of compressive strength insertion of RHA leads in positivity of results. The best of the lot was mix having 30% RHA and made at 0.35 w/b ratio. They stated that this constructive effect is generally result of fine size and high silica content of RHA (Sensale, 2006). The ultrasonic pulse velocity results were in the span of 4.730 – 5.097 km/s. The values specify that mixes had superb internal conditions, as fine size of RHA tends to fill the voids. Test results also pointed out decreased water absorption for RHA mixes and it goes on decreasing with the increasing RHA content. Most of the mixes had water absorption less than 5%. Again, here the fine size of RHA plays vital role in enhancement of water absorption resistance.

Atan and Awang (2011) carried out their study on various characteristics of SCC incorporating ternary blends of portland cement, raw rice husk ash and, fine limestone powder, pulverized fuel ash or silica fume. A total number of six SCC mixes were designed incorporating various combinations of additives plus one control mix (CM) designed using similar mixture composition to that of the SCC mixes. Slump-flow tests reveal that TM3 and TM5, which incorporate FA/RHA and SF/RHA blends respectively, have the highest slump-flow of 640 mm. Subsequently all ternary SCC mixes which incorporate RHA are shown to exhibit high requirement for mixing water. The high demand for mixing water involving RHA addition could be attributed to the porosity and boat-like shape of its particles (Bouzouba and Lachemi, 2001). Compressive strength test was performed at age of 7, 14, 28, 60 and 90 days curing. Specimens were 100mm cubes. They concluded that SCC mix which incorporates ternary blends of powder material comprising of 70% ordinary Portland cement, 15% pulverized fuel ash and 15% raw rice husk ash obtains high compressive strength of 43.4 MPa and flexural strength of 6.2 MPa. UPV results provide indications that raw RHA particles exhibit compatible inter-particle interactions with LP and FA particles. The combinations of raw rice husk with either pulverized fuel ash or fine limestone powder could be successfully used in SCC production.

Memon et al. (2011) looked into the feasibility of using rice husk ash (RHA) for viscosity alteration of SCC. As, SCC needs addition of viscosity modifying agents, that results in increased costs of SCCs. So RHA being a waste product, this idea can help in reducing the cost. They used varying proportions of RHA along with super-plasticizer dosage and w/b ratios. Water – binder ratio is 0.4 for mixes without RHA and 0.38 for mixes with RHA. The results for the slump flow were in the span of 595 – 795 mm. In these results they observed the trend of increase in slump with increase in super-plasticizer dosage. In hardened state test results, there was different trend. Mix made with 5% RHA showed decline in strength with raise in super-plasticizer dose, similar to the mix without RHA. In case of mix with 10% RHA trend gets reversed. Strength follows the same pattern as dosage of super-plasticizer. The reason stated for this improvement was enhancement in workability and adequate self-compaction. RHA addition also upgraded the water absorption resistance of the mixes. From their studies they concluded that RHA can be used to develop SCC, cutting the total cost along with improvement in quality of concrete.

Sua-iam et al., (2011) carried out research on feasibility of designing SCC by using untreated rice husk ash partially in place of fine aggregates. They used silica sand as fine aggregates and crushed limestone rock as coarse aggregates. Silica sand was replaced by RHA in span of 10 – 60%. Hardened state properties were tested by ultrasonic pulse velocity test. They result values were in span of 0.7 – 4.8 km/s. These values clearly indicate the superb condition of interior of these mixes. They concluded that RHA can be used in place of fine aggregates in developing SCC.

Safiuddin et al. (2011) concentrated their studies on mortars worked out from various SCCs made with rice husk ash (RHA). The main aim of the study was to monitor the flowability of these mortars. The mortars have varying proportions of RHA along with w/b ratios. Cement was replaced by RHA in percentages of 10, 25, 20, 25 and 30%. Test results indicated that RHA inversely affected the flowability of mortars. The reason stated by the authors was the high surface area of RHA particles. Increased Surface area increases the water retaining capacity of RHA, which directly effects the workability. Mixing and handling capabilities of mortars also get inversely affected by RHA inclusion. RHA percentages above 15% causes notable reduction in flowability. This inverse affect can be nullified by adding super-plasticizer to the mortars.

Safiuddin et al. (2012) investigated the key properties SCC including rice husk ash (RHA). They used RHA in span of 0 – 30% along with w/b ratios between 0.30 – 0.40. RHA was used in proportions of 0, 5, 10, 15, 20, 25 and 30%. Test results indicated towards increase in its ability to pass and fill spaces in form work with increase in RHA content and decrease in w/b ratio. Reason for the same is reduced aggregate quantity and enlarged volume of paste. Majority of SCC mixes passed the eligibility criteria of passing and filling ability, leaving few mixes having RHA quantities more than 20%. The high percentages of RHA resulted in viscous mixes because of fine size of RHA particles and their high surface area.

Sua-iam and Makul (2013) studied the fresh properties such as T₅₀ slump flow, J-ring flow, V-funnel, hardened properties such as compressive strength and ultrasonic pulse velocity of SCC incorporating limestone and rice husk ash. RHA is used in place of fine aggregates in proportions of 0%, 10%, 20%, 40%, 60%, 80% or 100% by volume and also employed water reducing admixture. They observed from their results that addition of RHA inversely affected the fresh state properties. RHA being fine in size retains more water which makes the mixes viscous. Results also stated that mixes made with RHA + LS showed satisfactory fresh properties. In terms of compressive strength, RHA helps in hiking the strength at all the ages. But the mix made with 100% RHA as fine aggregates showed minimum of strength among the lot. This positive effect can be attributed to the fine size and rich silica content of RHA. Control mix had highest pulse velocity while mix with 100% RHA showed minimum of pulse velocity.

Gill and Siddique (2017) looked into strength and micro-structural properties of SCC containing Metakaolin (MK) and Rice Husk Ash (RHA). For this purpose, SCC mixes were prepared where cement was replaced by weight in three proportions of 5, 10 and 15% by metakaolin; and fine aggregates were replaced by RHA in percentage of 10, 20 and 30. In total sixteen mixes were prepared. The experimental results indicate that SCC mixes produced with MK, RHA & in combination of MK & RHA satisfies norms of EFNARC. The compressive strength and splitting tensile strength results were also positive. Mixes produced with MK and RHA showed gain in compressive strength at all the ages. Micro-structural analysis confirmed the strength development pattern of SCC mixes made with MK, RHA and in combination of both.

Sandhu and Siddique (2017) reviewed the past work and tried to figure out the role of rice husk ash (RHA) in modifying the properties of SCC. Due to increased industrialisation the production of waste products is on the rising and it becomes equally important that they should

be disposed in efficient manner. The production of these products is on large scale and if used in right way they can be boon for the construction industry. One of such by-products is RHA. When rice husk is flawlessly burnt at temperatures beneath 700°C , it gives rise to RHA. RHA is a pozzolanic material due to its rich silica content. Its particle size is very fine and quality can also be controlled. So, its no wonder that addition of RHA in concrete can results in number of benefits. These benefits include enhanced quality in terms of strength and durability, overall cost cutting as RHA is just a waste product and also it can help in effective disposal of RHA. This can help in reducing environmental burden as well as carbon dioxide emission.

Patel and Shah (2018) evaluated geopolymer based SCC having ground granulated blast furnace slag and rice husk ash. They used GGBFS as primary binder in their design of SCC, which was then substituted by RHA in span of 5 – 25%. They tried to investigate the fresh and hardened state properties of their designed SCC. Test results revealed that addition of RHA had inverse affect on workability. There was decrease in workability of mixes with addition of RHA. They stated that ideal replacement percentage for RHA is 5%, which decreased workability by 2.81% but simultaneously it enhanced the compressive strength by 3.02%. SEM analysis also showed dense microstructure for mix made with 5% RHA and are in contention with the strength properties results.

Raisi et al. (2018) observed the mechanical behaviour of SCC adding rice husk ash (RHA). They stated reason behind their research as the high workability of self-compacting concrete, for which reason it can fill the formwork by flowing of its own and no external vibrations are required. Total of 240 hardened concrete specimens were casted. They partially replaced cement with RHA in span of 0 – 20% along with w/b ratios of 0.38, 0.44, 0.50, 0.56, 0.62 and 0.68. Test results indicated towards inverse affect of RHA on the workability of SCC mixes. On contrary, the hardened state properties such as compressive strength, modulus of elasticity, and splitting tensile strength increased with use of RHA, but this positive effect was only till 5% RHA substitution.

2.3 Properties of SCC made with other SCMs

2.3.1 Silica fume

Dehwah (2012) probed into the certain characteristics of SCC using quarry dust powder (QDP), silica fume (SF) and fly ash (FA). Properties examined were corrosion resistance, chloride permeability and chloride diffusion. Test results indicated lower initiation time of

reinforcement corrosion for mixes made with SF and QDP as compared to mixes made with FA. There were no signs of corrosion in mixes made with SF and QDP even after 990 days. In case of chloride permeability mixes made with QDP + SF showed low permeability than mixes made with QDP or FA. Mix made with 8% QDP had lowest diffusion co-efficient among the lot.

Bingol and Tohumcu (2013) looked into the outcomes of different curing methods in the characteristics of SCC. They used fly ash and silica fume as additions to SCC. Curing methods used were air, water and steam curing. Silica fume was partially used in place of cement in span of 5 – 15%, while fly ash was used in span of 25 – 55%. Results indicated constructive effect of addition of above-mentioned by-products on fresh state properties. Taking about hardened state properties, mix made with 15% SF addition gave the highest compressive strength. Also, they stated that the effect of air curing was negative on the compressive strength of all mixes. Also, the strengths of concretes made with the mineral admixtures were higher than concrete mixes made without the use of admixtures at steam curing conditions.

Wongkeo et al. (2014) studied the various characteristics of SCC having high level fly ash (FA) and silica fume (SF) in binary and ternary combinations. Cement substitution was done with fly ash in span of 40 – 70% and silica fume 0 – 10%. They observed from their results that inclusion of fly ash had negative impact on compressive strength. Strength decreased at almost all ages. But when SF was added along FA, mix showed increased strength at 7 -day age. So, the addition of SF effected the strength in positive way. Maximum strength achieved by combine effect of FA and SF was in the range of 60 MPa. In terms of charges passed, FA addition had positive effect. There was reduction observed in total charges passed, but alongside this addition leads in increased water absorption. The combination of SF and FA further minimized the passage of charges through mix. Authors go on to state that combination of FA and SF can be successfully used in SCC, which helps in upgrading the strength and durability properties.

Benaicha et al. (2015) securitize the influence of silica fume (SF) and viscosity modifying agent (VMA) on the properties of SCC. The wraith of a vast range of industrial mineral powders like silica fume and limestone filler and organic products such as superplasticizers and viscosity modifying agent, increases significantly the range of concrete rheological performance as suggested by the past literature. In accordance to this, authors tried to figure out the percentages of SF and VMA that results in similar properties of SCC. For this they

conducted various tests in fresh and hardened state. Drawing conclusion from their results they stated that both VMA and SF can be used in place of one another.

Leung et al. (2016) presented the results of utilizing fly ash and silica fume in SCC. They conducted sorptivity test to assess the water absorption of the mixes. They used SF and FA in number of combinations to take place of cement in defined spans. Results signalled towards improved water absorption with addition of FA and SF. In case of FA addition only, reduction in water absorption occurred but significantly only when percentages of FA exceeded 20%. On the other hand, combination of SF and FA had more prominent affect on water absorption reduction. Reduction in water absorption was directly proportional to percentages of SF and FA used in the mix. They also observed elevated compressive strength at age of 28 – days with use of FA and SF. But they were unable to find any relation between compressive strength and sorptivity.

Ardalan et al. (2017) conducted the experimental study to inquire into the role of pumice powder on characteristics of SCC. Along with it they also made use of silica fume (SF) to enhance quality of SCC. In all the designed mixes pumice, fly ash and slag was used in place of cement in spans of 10 – 50%. Experimental results pointed out that when these materials are used in binary combinations along with cement they resulted in inverse effect on fresh and hardened state properties. Decline in these properties was observed. Subsequently when pumice and SF were used in ternary combination, it helped in enhancing the properties of SCC. So they summed up that SF can be used in SCC along with other binders to refine the various properties.

Satish et. Al (2017) looked into the consequences of partial replacement of pozzolanic materials like silica fume and fly ash by cement on properties of SCC. They replaced cement by silica fume in proportions of 2, 4, 6, 8 and 10%, while fly ash was used in four different proportions of 10, 20, 30 and 40%. To study the various properties, they casted 135 cube samples and 90 cylinders. The water to binder ratios for all mixes was maintained at 0.36 along with water reducing admixture and viscosity modifying agent (VMA). They observed that the spread for all mixes were within the specified range recommended by EFNARC (2002). Also, they observed that the spread diameter increases with the FA content, while in the case of silica fume it decreases. They attributed this to the fact that silica, in finely divided form, reacts with CH, which in turn makes secondary cementitious C-S-H which makes the concrete mix denser and more cohesive. Also, the combined use of both the pozzolanic material in SCC negated the

undesirable effects of each other on flowability characteristics. In case of strength properties, they increased gradually when only silica fume was used. They came to conclusion that mixes made with SF had much upgraded strength as compared to mixes with FA.

Aleem and Hassan (2018) studied the pros and cons of adding silica fume (SF) to the self-consolidating rubberized concrete (SCRC). Basically, they tried to use crumb rubber (CR) in place of fine aggregates with minimum strength loss. The test results indicated that use of SF leads in raised strength as well as admissible fresh properties of mixes made with 25% addition of CR. Addition of SF or MK had positive impact on characteristics of SCRC. But at the same time mixes with SF performed much better than mixes with SF in terms of plastic stage properties. SF mixes required less quantity of super-plasticizer. They also observed that the addition of steel fibres (SFs) to SLFSCRC mixtures had prominent affect on strength properties. The summed up saying that making SCRC is feasible with crumb rubber alongg with additions of SF and MK, which helped in refining the properties of mixes.

Mohan and Mini (2018) carried their studies in direction of cost cutting of self-compacting concrete (SCC). As SCC requires addition of super-plasticizers and viscosity modifying agents, which turns SCC into a costlier affair. Here authors tried to explore this issue and tried to cut down the overall cost. For this purpose, they used ground granulated blast furnace slag (GGBFS) and silica fume (SF) in SCC. Both materials were used in spans of 0 – 15%. They used fixed w/b ratio along with varying dosage of super-plasticizer. Their results indicated that addition of 10% SF provided the best of results in terms of mechanical properties. They ended up saying that both SF and GGBFS can be successfully used in SCC.

Khan and Ali (2019) probed into the concept to improve the concrete behaviour with fly-ash (FA), silica fume (SF) and coconut fibres (CF). They tried to develop fly ash silica fume based plain concrete (FA-SPC) and fly ash silica fume coconut fibre reinforced concrete (FA-SCFRC). For this they used SF in place of cement in proportion of 15% along with FA in span of 0 – 15%. Then CF were also added to cement in percentage of 2%. Test results indicated that FA-SCFRC had better upgraded properties as related to FA-SPC. Mix made with addition of 10% FA along with CF showed best of properties among the lot. Hence the ends up saying that FA and SF can be used in SCC along with CFs.

2.3.2 Ground granulated blast furnace slag and wood ash

Elinwa et al. (2008) investigated the fresh state characteristics of SCC made with sawdust ash (SD). Along with this they also utilised naphthalene sulphonate (NS) and melamine sulphonate (MS) as water reducing admixtures. Test results indicated that slump flow of the designed mixes was in span of 665 – 685 mm, which was within EFNARC limits. On the other hand, V-funnel results were in span of 8.2 – 8.4 seconds. Same was in case of V-funnel and L-box test values, which satisfied the criteria laden by EFNARC. So, they end up with finding that SD has successfully application in SCC as all fresh state properties of SCC were satisfied the criteria of EFNARC.

Boukendakdji et al. (2012) looked into the role of addition of granulated blast furnace slag (GGBFS) and different type of super-plasticizer in SCC. GGBFS was used in place of cement in span of 10 – 25% along with two varieties of super-plasticizers. Results pointed out better properties of SCC made with addition of polycarboxylate type super-plasticizer. These mixes showed enhanced performances in fresh as well as hardened state. Talking about GGBFS, they stated that its addition had positive impact on fresh state characteristics of SCC. Workability of mixes increased when up to 20% GGBFS was used in place of cement. On the contrary, compressive strength showed inverse trend with GGBFS addition. So, they concluded that GGBFS can be helpful in upgrading the fresh properties of SCC but at the same time strength of SCC is also compromised.

Dinkar et al. (2013) made an attempt to design SCC with help of ground granulated blast furnace slag (GGBFS). They study of past literature purposes the use of GGBFS in concrete as it is highly pozzolanic in nature which can result in upgraded quality of concrete. Here authors tried to make use of GGBFS on efficiency as main target. So, for that they outlined for mix designs to have strength of 30, 60, 90 and 100 MPa. They observed in case of grade 30 concrete; the mix made with 80% addition of GGBFS had similar rate of strength gain as of simple mix. Their 28-day strength was in par with the normal concrete and at 90-days they exhibited upgraded strength as compared to normal concretes. So, they wind-up that GGBFS can be used to produce SCC with upgraded quality.

Zhao et al. (2015) looked in direction of producing SCC using fly ash (FA) and ground granulated blast furnace slag (GGBFS). They used FA and GGBFS in proportions of 20%, 30% and 40% and the fresh, mechanical and durability SCC were evaluated. The results bespeak of increase in slump flow with use of FA and GGBFS. Along with it, this addition also helped in

increasing the setting time and decreasing the wet density. On the contrary, this addition inversely affected the hardened state properties. There was reduction observed in strength of mixes as well as increase in water absorption. Carbonation depths were also on higher sides. At the same time this addition helped in enhancing the chloride permeability resistance of the mixes.

Dadsetan and Bai (2017) looked into the properties of SCC mingled with metakaolin (MK), ground granulated blast furnace slag (GGBFS) and fly ash (FA). They used these materials partially in place of cement. They tested their designed mixes for strength properties as well as for micro-structural properties by SEM & XRD. Results pointed out in upgraded properties of mixes made with above mentioned materials. They ended up concluding that among both, MK provided the superior results in terms of strength as well as micro-structure.

Patel and Shah (2018) evaluated geopolymer based SCC having ground granulated blast furnace slag and rice husk ash. They used GGBFS as primary binder in their design of SCC, which was then substituted by RHA in span of 5 – 25%. They tried to investigate the fresh and hardened state properties of their designed SCC. Test results revealed that addition of RHA had inverse affect on workability. There was decrease in workability of mixes with addition of RHA. They stated that ideal replacement percentage for RHA is 5%, which decreased workability by 2.81% but simultaneously it enhanced the compressive strength by 3.02%. SEM analysis also showed dense microstructure for mix made with 5% RHA and are in contention with the strength properties results.

Usman et al. (2018) did a feasibility study based on use of wood waste in place of cement for making an eco-congenial self-compacting cement paste. They made use of two different varieties of saw dust i.e. coarse SD (CSD) and fine SD (FSD) to replace portland cement in various proportions by weight. They used saw dust ash in proportions of 2%, 5%, and 7% to replace cement and evaluated the fresh and hardened state properties. They noticed that addition of saw dust causes strength reduction in span of 11 – 34% but simultaneously it minimises the shrinkage strain, mainly due to increased water absorption of saw dust. There was almost reduction of 45 – 80% in shrinkage with the use of SD as compared to the mix made without SD. Authors concluded that the SD's potential to absorb higher quantities of water and then at later stage it's potential to release the same water, causes more moisture for cement reaction. So, they end up concluding that use of SD can lead in upgraded durability of the concrete.

Chapter 3

Experimental Program

This chapter gives the particulars about the experimental programme used for the measurement of fresh properties (slump flow, L – box, U – box, V – funnel), strength properties (compressive strength, splitting tensile strength) durability properties (water absorption, porosity, sulphate resistance, rapid chloride permeability) and micro-structural properties (SEM and XRD) of SCC prepared with MK and RHA.

3.1 Materials Used

3.1.1 Cement

The OPC, conforming to the Indian standards was used in this work. Cement was obtained from Patiala (India). The cement does not have any knots and of grey with light greenish shade. Various tests were conducted as per IS: 8112 (1989) and outcomes are tabulated in Table 3.1. Silt content was found to be 4 percent. Chemical compositions of the cement, analyzed by EDS, are tabulated in Table 3.2 and EDS image for cement is shown in Fig. 3.1.

Table 3.1: Physical properties of cement

Property	Results
Color	Grey with a light greenish shade
Consistency (%)	34
Initial setting time (minutes)	38
Final setting time (minutes)	550
Specific gravity	3.2
Expansion (mm) (Le – Chatler’s) (mm)	6
Silt Content (%)	4

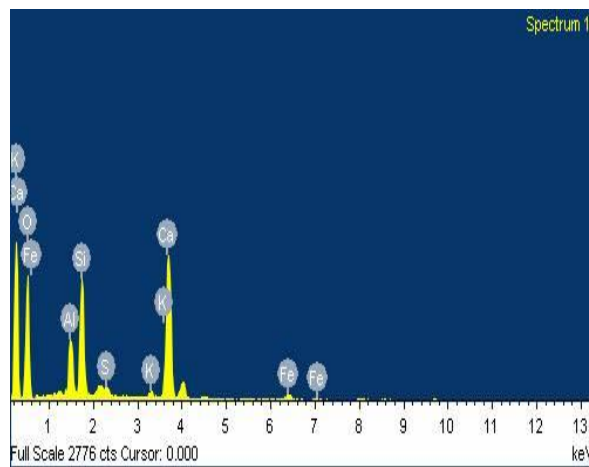
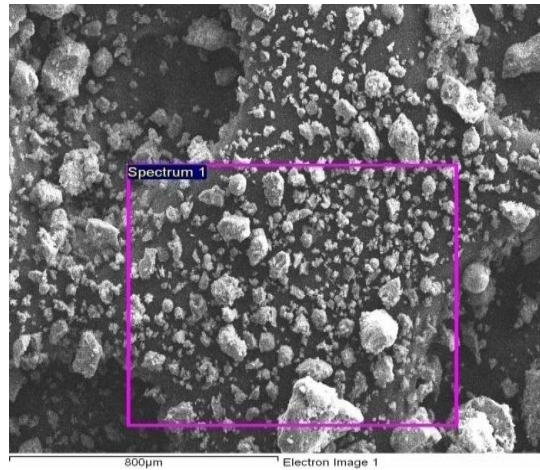


Fig. 3.1: EDS image of cement

3.1.2 Metakaolin

Metakaolin, used in this work, was of off-white color. It was obtained from Faridabad (India). EDS analysis of metakaolin was conducted and results are given in Table 3.2. SEM analysis of metakaolin was done at 20000x magnification. It shows that metakaolin particle is quite small as compared to cement and RHA particle. It is shown in Fig. 3.2. Specific gravity of metakaolin was 2.65 and its particle size was less than 1 micron. EDS image for metakaolin is shown in Fig. 3.3. The image shows the higher percentages of elements Al, Si and O which further results in formation of SiO_2 (51.10 %) and Al_2O_3 (43.80 %), the two main components of MK. As per ASTM standards the combined percentages of these two compounds should be more than 90%, which is correct for the MK used in this work. This results in better quality of MK.

Table 3.2: Chemical properties of cement, metakaolin and rice husk ash

Composition (%)	Cement	Metakaolin	Rice husk ash
Silicon dioxide (SiO ₂)	21.96	51.10	93.50
Aluminum Oxide (Al ₂ O ₃)	5.05	43.80	0.55
Ferric oxide (Fe ₂ O ₃)	3.96	1.60	0.23
Magnesium oxide (MgO)	1.56	0.30	0.31
Calcium oxide (CaO)	63.45	0.20	1.11
Sodium oxide (Na ₂ O)	0.36	0.10	0.10
Potassium oxide (K ₂ O)	0.64	0.20	1.40
Sulphur trioxide (SO ₃)	1.62	0.05	0.07

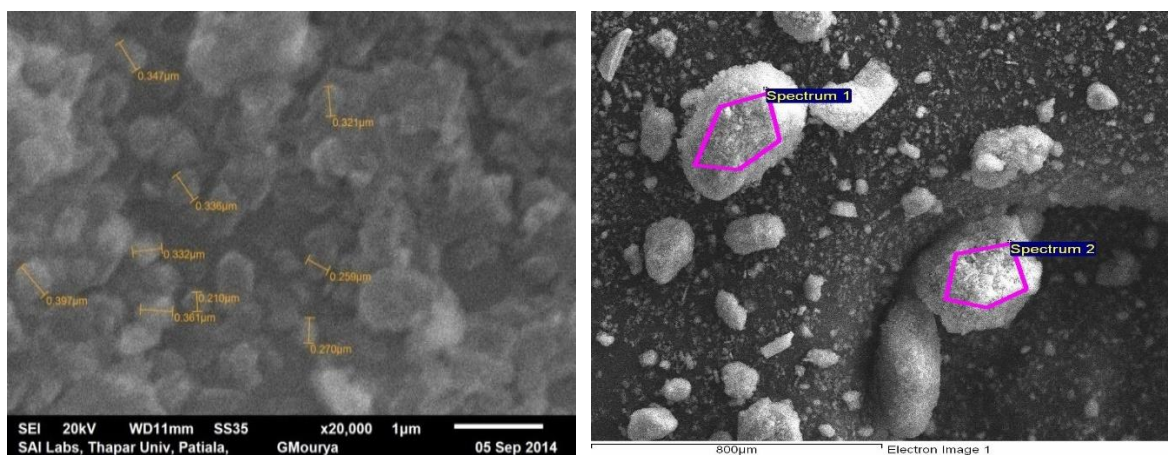


Fig. 3.2: SEM image of metakaolin

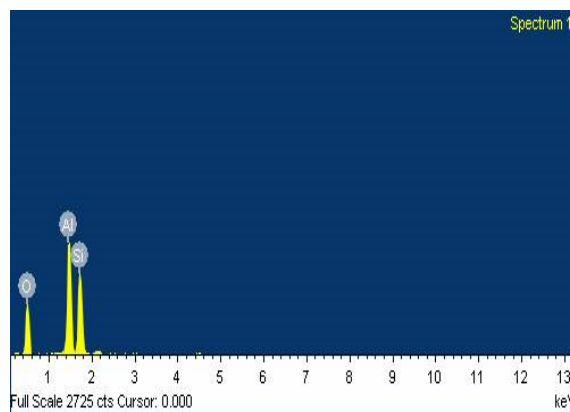


Fig 3.3: EDS image for metakaolin

3.1.3 Rice husk ash (RHA)

RHA, used for this work, was of black color. It was obtained from Ludhiana (India). Rice husk ash was obtained at the cost of Rs 1 per kg. EDS analysis was used to identify the chemical properties and results are tabulated in Table 3.2. SEM analysis of RHA, done at 1000x magnification, revealed that RHA particle was of irregular shape. The size of RHA particle was quite small as compared to cement particle. It is quite evident from Fig. 3.4. The particle size of RHA was less than 20 microns.

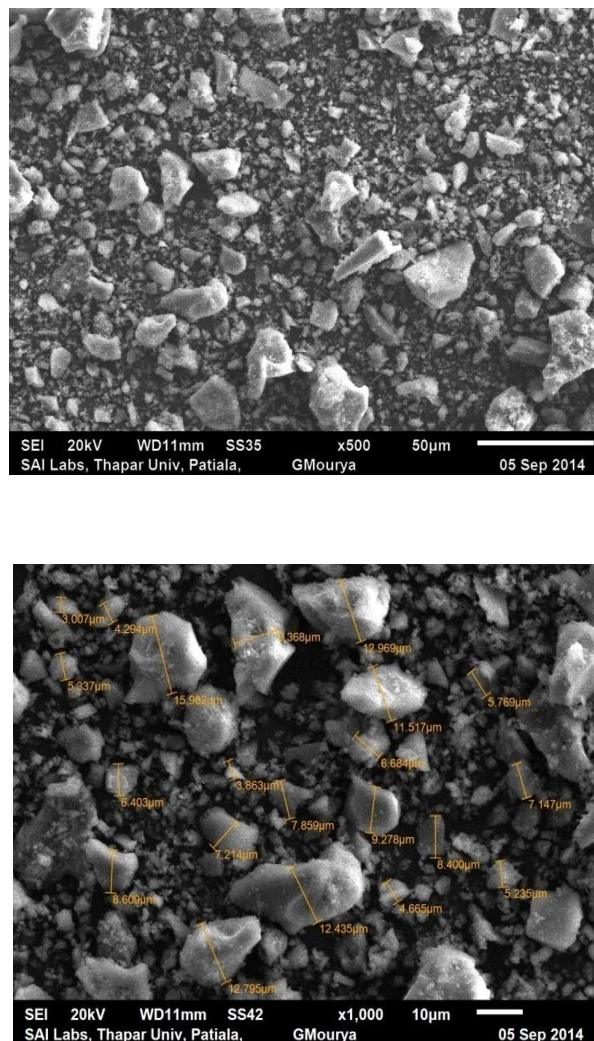


Fig. 3.4: SEM images of RHA

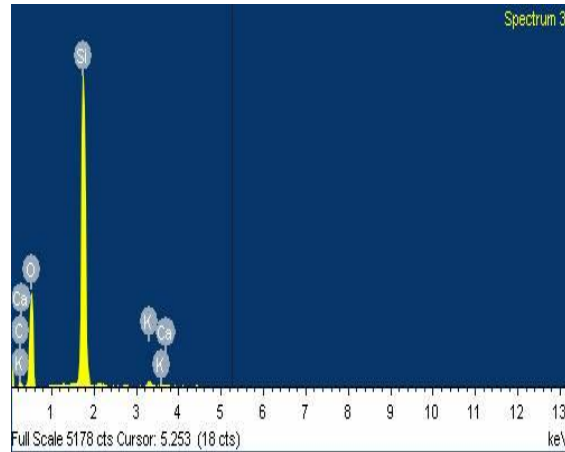


Fig. 3.5: EDS image for RHA

3.1.4 Fine and coarse aggregates

The sand and coarse aggregates used for this work were obtained from Patiala (Punjab). Tests were performed for specific gravity, sieve analysis/grading curve, and water absorption as per Indian standard specifications (IS: 383-2016). The results are tabulated in Table 3.3. The fineness modulus for fine and coarse aggregates was determined from sieve analysis. The results are tabulated in Table 3.4 and 3.5. The fineness modulus of fine aggregates was 2.65 and they belong to zone II. When fine aggregates were replaced by 10% RHA, the value of fineness modulus was 2.45, at 20% RHA replacement level, the value of fineness modulus come to be 2.08 and at 30% RHA replacement level, fineness modulus was 1.81. Particle size distribution curve is shown in Fig. 3.6.

Table 3.3: Physical properties of fine and coarse aggregates

Property	Fine aggregates	Coarse aggregates
Specific gravity	2.56	2.68
Fineness modulus	2.65	6.40
Water absorption (%)	0.90	0.56
Size	<4.75 mm	<10mm
Bulk Density (Kg/m ³)	1665	1715

Table 3.4: Sieve analysis results for fine aggregates

I.S. Sieve Size	Weight retained (gm)	Percentage weight retained (gm)	Cumulative percentage of weight retained	Percentage passing	IS: 383-2016 Requirements for zone II
10 mm	00	00	00	100	100
4.75 mm	12	1.2	1.2	98.8	90 – 100
2.36 mm	51	5.1	6.3	93.7	75 – 100
1.18 mm	228	22.8	29.1	70.9	55 – 90
600 µm	204	20.4	49.5	50.5	35 – 59
300 µm	334	33.4	82.9	17.1	8 – 30
150 µm	131	13.1	96	4.0	0 – 10
Pan	40	4.0	100	00	-

Weight taken = 1000 gm

Fineness modulus of fine aggregates = 2.65

Table 3.5: Sieve analysis of coarse aggregates

I.S. Sieve Size	Weight retained (gm)	Percentage weight retained (gm)	Cumulative percentage of weight retained	Percentage passing	IS: 383-2016 Requirements for zone II
40 mm	00	00	00	100	-
20 mm	70	3.5	3.5	96.5	90 – 100
10 mm	690	34.5	38	62	40 – 85
4.75 mm	1210	60.5	98.5	1.5	0 – 10
Pan	30	1.5	100	00	-

Weight taken = 2000 gm

Fineness modulus of coarse aggregates = 6.40

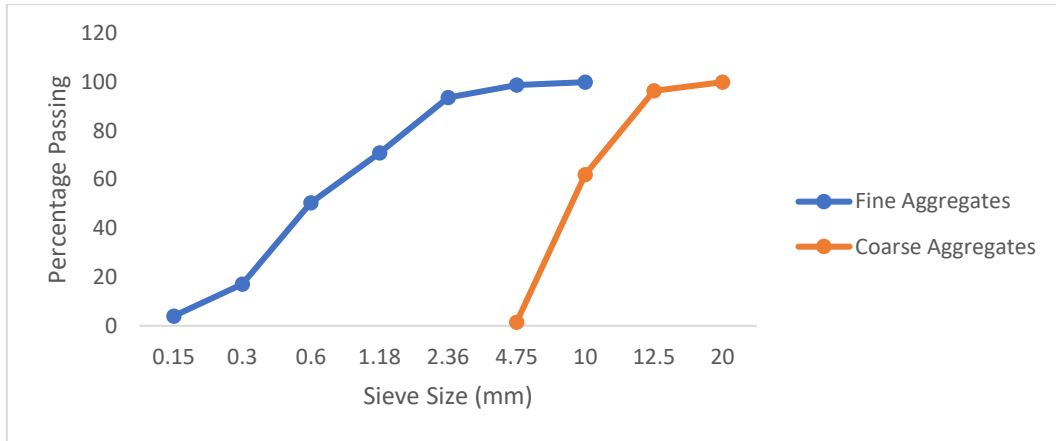


Fig. 3.6: Particle size distribution curve for fine and coarse aggregates

3.1.5 Admixture

Admixture used in this work was Auramix 400, which was of brown color. Admixture was obtained from Chandigarh (India). Fig 3.7 shows the used admixture. The liquid state of the admixture was used, which helped in reducing the water demand. It satisfies the IS: 9103 (1999) specifications. It is based on Sulphonated Naphthalene Polymers. Its specific gravity ranges from 1.205 to 1.215; and pH at 27°C remains between 7.0 and 8.0.



Fig 3.7: Super-plasticizer

It has appearance of brown color, with volumetric mass of 1.09 Kg/litre at 20° C. The chloride content of Auramix 400 is generally nil and alkali content is typically less than 1.5 g Na₂O equivalent/litre of admixture. It helps in cement dispersion. Auramix 400 combines the properties of water reduction and workability retention.

3.1.6 Magnesium sulphate

Magnesium sulphate was used in the sulphate resistance test for concrete. It is used in the form of solution of strength 5%. It was procured in powder form from Patiala (Punjab); and a solution of strength 5% was prepared by adding it into the water. The powder was white in color. Fig 3.8 shows the powder form of used Magnesium sulphate.



Fig 3.8: Magnesium Sulphate

3.1.7 Water

Tap water available in lab was used for casting and curing of concrete specimens. It was conforming to the standards of IS: 456 (2000).

3.2 Mix Design

Since the concept of SCC came to limelight in 1986, the SCC found number of applications worldwide. The reason for its fame was not just its properties but also the possibility of usage in its composition of local materials. Also, the usage of waste products in the production of SCC made it an attractive product. The design concepts of SCC and NVC are quite un-identical. One major difference is that the SCC accepts more amalgamations of materials and reaches the same properties, yet these combinations often times contain several variables.

3.2.1 The European guide for SCC [EFNARC, 2005]

The guidelines were developed under a European project for promoting new types of concrete. This project was conducted in 1998 and included representatives of major concrete organizations of Europe. This guide does not provide an actual sizing method for SCC mixtures rather presenting minimum and maximum values for the materials used (Table 3.6). However, the guidelines contain certain steps to be followed in order to achieve a quality SCC. The values contained therein are not limitative but informative, with the possibility of also producing SCC

mixtures by using quantities and percentages not falling exactly within the values provided by the guide.

Table 3.6: Optimum SCC quantities (EFNARC, 2005)

Constituents	Mass (Kg/m ³)	Volume (litres/m ³)
Fine part (addition)	360 – 600	-
Paste content	-	300 – 380
Water	150 – 210	150 – 210
Coarse aggregates	750 – 1000	270 – 360
Fine aggregates	The fine aggregate content depends on other materials, usually using 48 – 55 % of the total aggregate weight.	
Water/fine part ratio	-	0.85– 1.0

- Selection of particularized required performances.
- Selection of required materials– assessing correct amount of water required for reformed paste slump and stability; - assessing the correct amount of aggregates and other additives, so the concrete can fulfil the required criteria.
- Mixture production –a concrete mix prepared can be checked for small variations in the quantities of constituents used; - testing of fresh and hardened concrete properties.
- Authenticating and fine-tuning the performances obtained in the laboratory. Distinguish the beneficiary needs and if these needs are met then only a certain concrete verification are done on site.
- If these beneficiary needs are not met, then the basic redesign of mix must be tried.
- Depending on the problem occurred, one of the following measures may be taken into considerations:
 - Trying the different cement/powder ratio and the water/binder ratio;
 - Trying with different type of addition than used earlier;
 - Trying the other proportions of fine aggregates and the super-plasticizer dosage;
 - Viscosity modifying admixtures can be used to reduce mixture sensitivity;
 - Changing the quality of coarse aggregate used.

3.2.2 Mixture proportions

SCC mixes were designed in accordance to the EFNARC (2005) guidelines. Sixteen concrete mixes were prepared in all. First was control mix (M1), without any of the SCM (MK & RHA). Three mixes (M2, M3, M4) were prepared with partial replacement of cement with MK in proportions of 5, 10 and 15%. Three other mixes (M5, M6, M7) were made with the partial replacement of fine aggregates with RHA in the varying proportions of 10, 20 and 30%. The volume of coarse aggregates was fixed at 670 Kg/m³. W/b ratio of 0.44 was also maintained. An admixture, 1.5 to 2% by weight of total powder content was used. For control mix, cement used was 480 kg/m³; and fine aggregates used were 900 kg/m³. These were suitably replaced for other mixes.

Then, on the basis of 28-day compressive strength, ten additional mixes (M8 – M16), with combination of both (MK & RHA) were made. In mixes M8 – M10, MK content was kept constant at 5% & RHA content was varied between 10 to 30%. Similarly, in mixes M11 – M13 & mixes M14 – M16 MK content was kept constant at 10% & 15% respectively, and RHA content was varied between 10 to 30%, similar to mixes M8 – M10. Mix proportions are tabulated in Table 3.7.

For the mixes with combine use of MK and RHA, a total of 45 samples, consisting of 30 cubes of size 150 mm X 150 mm, for compressive strength (7,28, 90 and 365 days), sulphate resistance (28, 90 and 365 days), and water absorption and porosity test (28, 90 and 365 days), 12 cylinders of dimensions 150mm diameter and 300mm length, for splitting tensile strength test (7,28, 90 and 365 days), and 3 cylinders of size 100mm X 200 mm, which were further cut into size of 100mm X 50mm, for RCPT test (28, 90 and 365 days) were casted. For mixes with individual use of MK and RHA, six samples were casted for each mix, which consisted of cubes of dimensions 150mm X 150 mm, for compressive strength test (7 and 28 days).

Table 3.7: Mixture Proportions

Mix ID	Type of mix	Cement (Kg/m ³)	Fine aggregates (Kg/m ³)	Coarse aggregates (Kg/m ³)	Metakaolin (Kg/m ³)	Rice husk ash (Kg/m ³)	W/b ratio	S.P. (%)
M1	Control Mix	480	900	670	-	-	0.44	1.5
M2	5MK	456	900	670	24	-	0.44	1.8
M3	10MK	432	900	670	48	-	0.44	1.8
M4	15MK	408	900	670	72	-	0.44	1.8
M5	10RHA	480	810	670	-	90	0.44	1.5
M6	20RHA	480	720	670	-	180	0.44	1.5
M7	30RHA	480	630	670	-	270	0.44	1.5
M8	5MK10RHA	456	810	670	24	90	0.44	2
M9	5MK20RHA	456	720	670	24	180	0.44	2
M10	5MK30RHA	456	630	670	24	270	0.44	2
M11	10MK10RHA	432	810	670	48	90	0.44	2
M12	10MK20RHA	432	720	670	48	180	0.44	2
M13	10MK30RHA	432	630	670	48	270	0.44	2
M14	15MK10RHA	408	810	670	72	90	0.44	2
M15	15MK20RHA	408	720	670	72	180	0.44	2
M16	15MK30RHA	408	630	670	72	270	0.44	2

3.3 Casting of Test Specimens

The testing moulds were cleansed and oiled fitly before casting. The mixes were prepared in the concrete mixer. After preparation of mix, it was first tested for various fresh properties and then attentively laid into the moulds. It was allowed to be in moulds for 24 hours and then carefully taken out of moulds to keep them in the water tank in the hardened state. All these specimens were then tested accordingly for the various properties as described in Table 3.8.

Table 3.8: Various properties with size of specimen and age of testing

Property	Reference Code	Size of specimen	Age (Days)
Properties in fresh state			
Slump flow	EFNARC, 2005	-	-
L – box	EFNARC, 2005		
U – box	EFNARC, 2005		
V – funnel	EFNARC, 2005		
Strength properties			
Compressive strength	BIS:516:1959	150 X 150 mm cube	7,28,90 and 365
Splitting tensile strength	BIS:5816:1999	150 X 300 mm cylinders	
Durability Properties			
Water absorption	ASTM C 642-06	150 X 150 mm cube	28,90 and 365
Porosity	ASTM C 642-06	150 X 150 mm cube	
Sulphate resistance	ASTM C 1012-10	150 X 150 mm cube	
RCPT	ASTM C 1202	100 X 50 mm cylinders	



Fig 3.9: Curing of SCC specimens

3.4 Fresh State Properties

3.4.1 Slump flow test

This is the basic test generally come in use to measure the flow of SCC in horizontal direction without presence of any hurdles. Slump cone of standard dimensions is used with only difference that SCC is not rodded as done in standard test (Ferraris, 1999). Once cone is lifted, SCC starts spreading. The diameter of the concrete is measured in horizontal direction. This test gives idea about the consistency, filling ability and workability. According to EFNARC criteria SCC should have diameter in the span of 650 – 800 mm.



Fig 3.10: Slump flow test for SCC

This is the commonly used method as of its relatively simple procedure and equipment. The equipment used for the slump flow test should comply with EN 12350-2. It should have base bed with the minimum size of 900 x 900 mm and minimum thickness of 2mm. It should be made of waterproof and rigid material. Then Abrams cone with standard dimensions is used in accordance to ISO 4190.

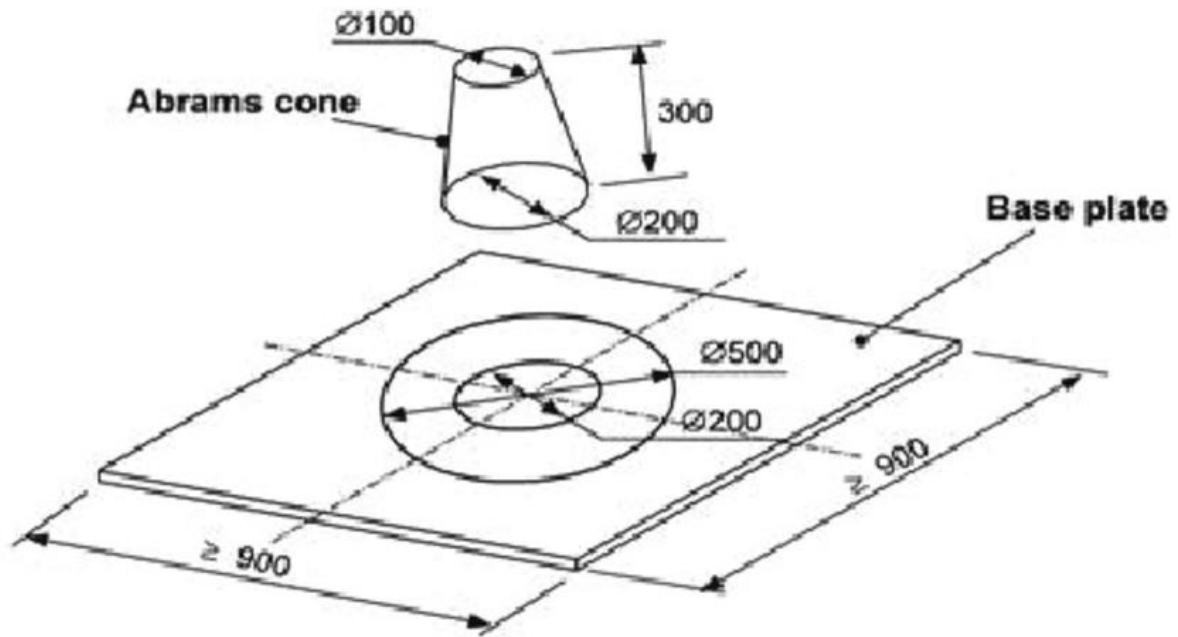


Fig. 3.11: Slump flow test apparatus (Ouchi et al., 2000)

3.4.2 L-box test

This was initially used to determine characteristics of concrete about to cast under water. But this test is also applicable to the concretes with high flow rate values such as SCC. This test determines properties such as flow rate, ability to pass through tight spaces. The equipment is made up of two chambers perpendicular to each other. Both chambers are portioned by three reinforcing bars of standard size (Dietz et al., 2000). SCC is filled in the vertical chamber and concrete is allowed to flow through these bars. Once it comes to rest the height of concrete in both chambers is noted i.e. H1 and H2.

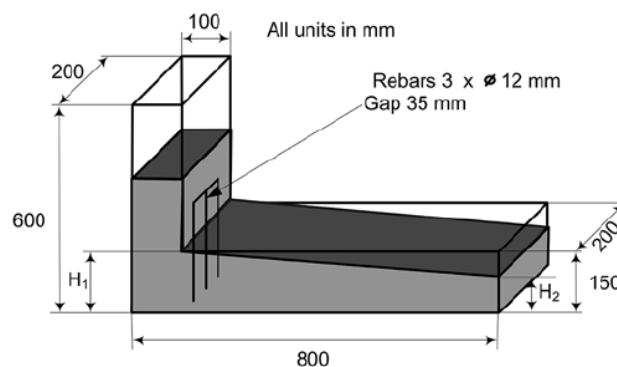


Fig. 3.12: Schematic of L-box (Ouchi et al., 2000)



Fig. 3.13: L-box test apparatus

3.4.3 U- box test

The test equipment consists of two vertical chambers having shape of U letter. Both chambers are portioned by three reinforcing bars at gap of 50 mm. SCC is filled in first chamber and then the gate between the two chambers is opened and concrete is allowed to flow through reinforcing bars through the gate. As concrete comes to halt, its height in both the chambers is measured i.e. H1 and H2 and the difference in both the heights is calculated. Time for the whole procedure should not exceed 5 minutes. Now, the less is the difference between both heights more positive is the result. So, if $H1 - H2$ value is near to zero, concrete had excellent filling and passing ability (EFNARC, 2005).

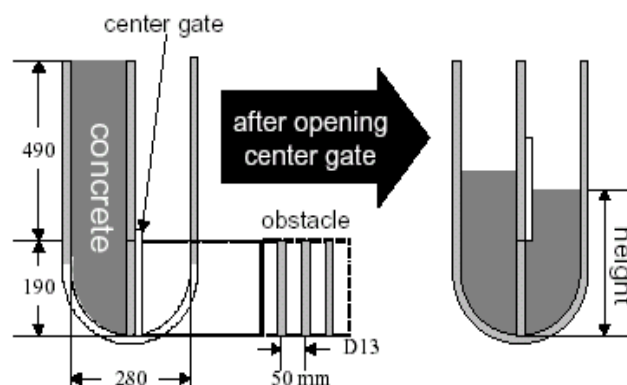


Fig. 3.14: U-box test (Ouchi et al., 2000)

3.4.4 V- funnel test

It tells about the viscosity and filling ability of SCC. It also gives information about static segregation resistance by increasing the waiting time between filling the container with concrete and the actual commencement of the test. The equipment has the shape of V letter with specified dimensions (Fig. 3.15). The basic idea of the test is to make concrete pass through the orifice in certain specified time (Dietz et al., 2000). Concrete is filled in the V-shape chamber (around 12 litres). Once chamber is filled, the bottom lid of the orifice is opened and time taken to pass the concrete through orifice is noted.

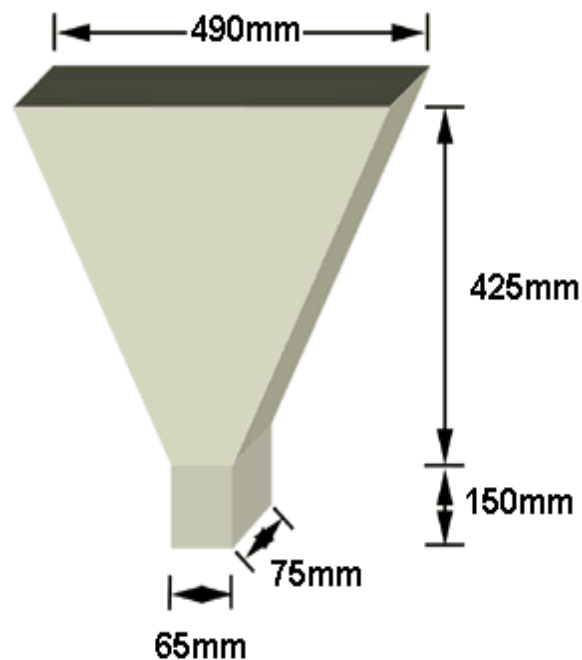


Fig 3.15: V-funnel test apparatus

3.5 Hardened State Properties

3.5.1. Strength properties

3.5.1.1 Compressive strength test

This is the basic test to assess the hardened strength of concrete. Samples are in the form of cubes and testing can be done at various ages.

Procedure

Universal compressive testing machine or such kind of machine which can apply compressive load at rate of 5.148 KN/sec, as per specifications can be used for this test. Cured cube specimens are kept between the two steel platens of the machine and load is applied at specified rate. Specimen is kept in the centre of the platens so that load applied is uniform. The point at which specimen breaks or cracks, that load is noted down. Strength can be found out by dividing this load with the area of sample. Digital machines these days directly gives the strength of the given sample.





Fig 3.16: Compressive strength testing machine and cube moulds

Testing age

Testing can be done at the curing ages as per Indian standards. Most preferably this test is performed at the age of 7 and 28 days. Test at age of 56 days can also be performed. In case of later ages strength, it can also be performed at the ages of 90 and 365 days.

Number of samples

Minimum of three samples should be tested for one mix and average strength of three samples should be taken as final strength at that specified age.

3.5.1.2 Splitting tensile strength test (BIS: 5816 - 1999)

The samples taken are generally in shape of cylinders of 150 mm diameter and 300 mm length. Strength can be assessed at various ages. Universal compression testing machine can be used for performing this test. The machine consists of two steel platens of sufficient size to accommodate the testing sample. As this test is generally performed on cylindrical shaped samples, so plywood strips are also used. Two plywood strips of length generally longer than specimen length are used on both the faces. The reason behind their use is to apply load in uniform way. Load is applied at a constant rate of 1.4 KN/min.

Procedure

- Firstly, the ends of the specimen are aligned in same axial plane by drawing lines along the diameter at both the ends.

- Then place the one plywood strip at the bottom platen/ bearing block, matching their centres.
- After aligning, place the cylindrical specimen on the plywood strip. The specimen should be placed in such a way that it is exactly in the centre of plywood strip.
- Place the other plywood strip on the top of specimen. It should be placed lengthwise on specimen and should be in the centre.
- Once placement and alignment are done, load is applied at the specified rate as per Indian standards.
- Load is applied until the specimen breaks or cracks.
- Once specimen breaks or cracks, that load is used in given formula,

$$f_{st}' = \frac{2P}{\pi ld}$$

Where,

P = Load at which specimen breaks or cracks

l = Length of specimen

d = Diameter of specimen

3.5.2 Durability properties

3.5.2.1 Water absorption

It is a very significant property to get the measure of the durability of concrete. Test was conducted in conformity with ASTM C 642-06. The procedure states that first measure the mass of each specimen and then oven dry the specimens at 105°C for 24 hours. The value of mass recorded first and the mass recorded after oven drying should not exceed 0.5 % and if this is the case then we can consider sample as dry. If the difference exceeds this value then the specimen is considered as wet. Then keep the specimen in oven for another 24 hours and again measure the mass. Then the difference between the value of mass recorded before keeping in oven second time and after oven drying should not be more than 0.5 %. This procedure is repeated until this mass values difference stops exceeding 0.5 %. Once it is less than 0.5 %, note down that value as A.

Now immerse the specimen in water for at least 48 hours. After that clean the specimen with a cloth so that all surface moisture is removed, called surface drying of specimen. Measure its mass after surface drying. Designate surface dry mass as B. Then keep the specimen in boiling

water for minimum time period of 5 hours. After that allow the specimen to cool for minimum time period of 14 hours. Then suspend the specimen in the bucket at a constant water level and measure the apparent mass. Designate this apparent mass as D. After that take out the specimen from water and clean it with damp cloth. After cleaning measure its mass and mark this mass as C. After recording these masses respectively, water absorption is calculated based on the given formulas.

$$\text{Absorption after immersion (\%)} = [(B - A) / A] \times 100$$

$$\text{Absorption after immersion and boiling (\%)} = [(C - A) / A] \times 100$$

$$\text{Bulk density, dry} = [A / (C - D)] \cdot p = g_1$$

$$\text{Bulk density after immersion} = [B / (C - D)] \cdot p$$

$$\text{Bulk density after immersion and boiling} = [C / (C - D)] \cdot p$$

$$\text{Apparent density} = [A / (A - D)] \cdot p = g_2$$

Where;

A = Mass of oven dried specimen

B = Mass of surface dry specimen after immersion

C = Mass of surface dry specimen after immersion and boiling

D = Apparent mass of specimen in water after immersion and boiling

g 1 = Bulk density, dry

g 2 = Apparent density

ρ = Density of water



Fig 3.17: Water absorption test in progress

3.5.2.2 Sulphate resistance

Test was conducted in conformity with ASTM C 1012 – 10. Solution of magnesium sulphate of strength 5 % was used for this test. This solution is used to submerge the SCC specimens and then these are tested at their respective ages. They are tested in universal compression testing machine and their compressive strength was compared with the compressive strengths of the specimen cured in simple water at same ages. The difference in the compressive strengths was noted down and compared as follow:

$$\text{Loss in compressive strength (\%)} = [(S1 - S2) / S1] \times 100$$

Where,

S1 = Compressive strength of specimen cured in water

S2 = Compressive strength of specimen immersed in magnesium sulphate solution at same age.



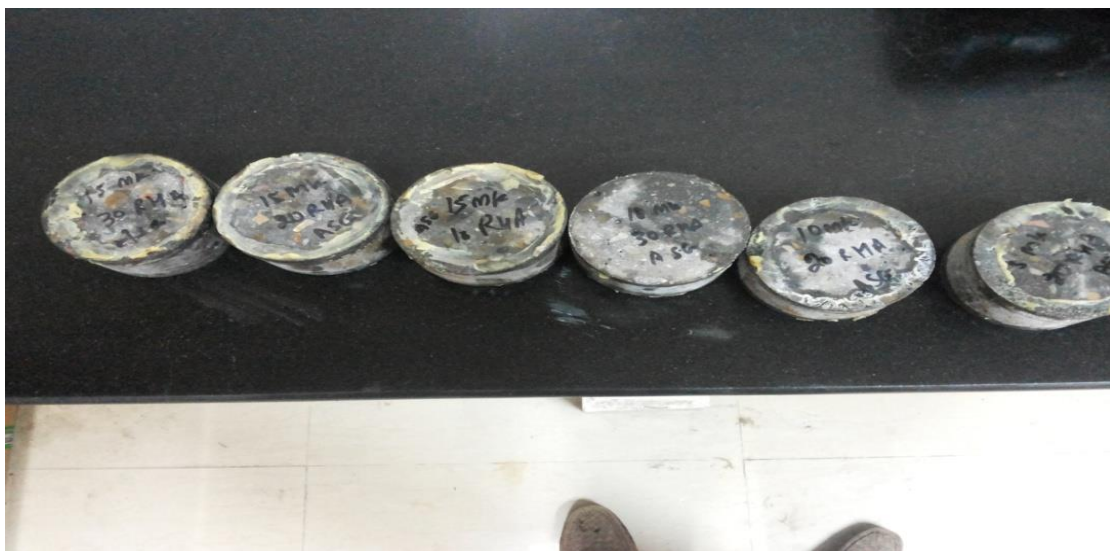
Fig 3.18: (a) Magnesium sulphate in powder form, (b) specimens immersed in magnesium sulphate solution, (c) specimen after 90 days of immersion in magnesium sulphate solution, (d) specimen after tested for compressive strength.

3.5.2.3 Rapid chloride permeability

The test was conducted in conformity with ASTM C 1202 – 10. For this specimens are taken in shape of cylinders of 50 mm length and diameter of 100 mm. These specimens are fitted in testing apparatus and electric charge is made to pass through these specimens for span of 6 hours. While testing 60V DC difference was kept between the ends of specimen. Also sodium chloride solution is used at one end while sodium hydroxide solution at other end. More the charge passes through the specimen, less is it's resistance to chloride ion penetration. Sample is designated as per ACTM C 1202-10 depending on charge passed through it.

Table 3.9: Chloride ion penetration based on charge passed (ASTM C 1202-10)

Charge passed (Coulombs)	Chloride ion penetration
>4000	High
2000 - 4000	Moderate
1000 - 2000	Low
100 – 1000	Very low
<100	Negligible



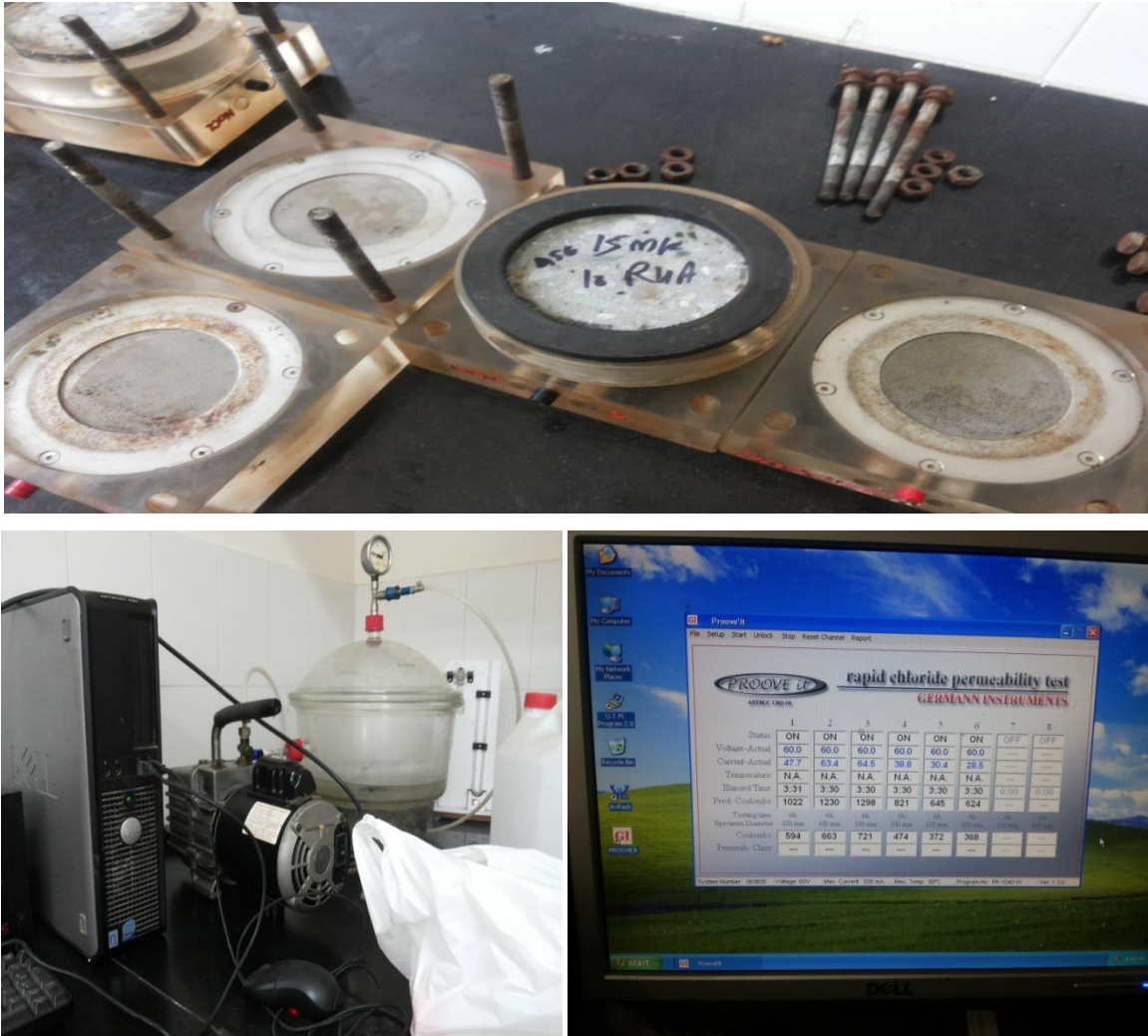


Fig: 3.19: (a) Specimens for RCPT, (b) specimens being fitted in test moulds, (c) RCPT set-up, (d) RCPT results

3.5.3 Microscopy (Scanning electron microscopy – SEM)

SEM is a very important test which gives information regarding the particle morphology, texture, porosity and agglomeration of sample. The sample for SEM analysis is mounted on a conductive substrate such as aluminium stub and gold plated in vacuum. The coated samples are kept at the sample stage of the microscope. The samples can be advanced along all three axis, tilted as well as rotated. Further, the data signal is released due to interactivity among electrons and atoms of the specimens. These data signal arise due to elastic and inelastic interactivity of beam electrons and atoms of the specimens. The elastic (electron nucleus) collisions produce back scattered electrons (BSE) and inelastic (electron – electron) produce secondary electrons. The back scattered electrons provide topographic as well as compositional

information about the specimens. In this research samples were taken in form of broken pieces from the concrete specimens. The concrete specimens used for various tests in study were crushed into small pieces and used as samples for SEM.

3.5.4 X-Ray Diffraction (XRD)

XRD is a very significant test used for finding crystalline phases and compositions. Diffraction peaks of sample under observation are studied in terms of their position and intensity. Then these are compared with known crystalline phases, which can help in identifying the target material. In the present study XRD test was conducted at SAI labs, Patiala. It was done using a Panalytical X'Pert pro, with Cu K α radiations. The X'Pert high score plus software was used to identify the phases.



Fig 3.20: (a) Sample being prepared for SEM & XRD analysis (b) Samples used for SEM and XRD analysis

Chapter 4

Result and Discussions

The chapter gives the results of various tests conducted in the present study. Starting from the fresh state to the hardened state, numbers of tests were used to analyse the various properties of SCC and effects of RHA and MK on these properties. MK was used to replace cement in proportions from 5% to 15%, while RHA was used to replace fine aggregates in proportions from 10% to 30%. In fresh state, slump flow, L – box, U – box and V- funnel tests were used to evaluate the characteristics of SCC. To measure the strength properties, compressive strength and splitting tensile strength tests were conducted. For durability properties, water absorption, porosity, sulphate resistance and rapid chloride permeability tests were conducted. Then SEM and XRD analysis were used to assess the microstructure and phase identification of SCC.

4.1 Fresh Properties

4.1.1 Slump flow

This test gives idea about the filling ability of the concrete. Varied trials were made with different volumes of cement, fine and coarse aggregates having different w/p ratios and percentage of super plasticizer. The water-powder ratio of 0.44 and super plasticizer percentage of 1.5 to 2 % provided the optimum results. The results for the slump flow test are given in Table 4.1.

On the basis of results of various trial mixes, the w/b ratio for control mix was fixed to 1.5, while for mixes made with incorporation of MK was fixed to 1.8, mixes made with RHA w/b ratio was 1.5 and mixes prepared with combine use of MK and RHA w/b ratio was fixed to 2. The slump flow test results for all the mixes were within the EFNARC range. It can be observed from the Table 4.1, when MK was added slump flow first increases and then it starts decreasing as compared to the slump of control mix. Similar results were obtained for the use of RHA also. While when MK and RHA were used in combination, slump flow decreased with the increasing percentages of MK and RHA. The use of both MK and RHA affected the workability, but not to a great extent. A little more water was required to be added with the rise in MK and RHA use. For this, the dosage of super plasticizer was increased to 1.8% for MK mixes, and 2% for combined MK and RHA mixes. However, mix M8 (5MK10RHA) exhibited the highest slump flow.

Table 4.1: Slump flow test results

Mix ID	Mix Type	Slump flow (mm)
		EFNARC (2005) Range: 650 - 800 mm
M1	Control Mix	680
M2	5MK	695
M3	10MK	670
M4	15MK	665
M5	10RHA	690
M6	20RHA	675
M7	30RHA	650
M8	5MK10RHA	755
M9	5MK20RHA	735
M10	5MK30RHA	720
M11	10MK10RHA	730
M12	10MK20RHA	705
M13	10MK30RHA	680
M14	15MK10RHA	695
M15	15MK20RHA	680
M16	15MK30RHA	670

It can be clearly distinguished from Fig. 4.2 that there is increase in the slump flow with the use of MK & RHA as compared to the control mix. But the slump flow starts decreasing with the increased percentages of MK & RHA. This can be attributed to the round shape and fine size of RHA particles as they replaced the fine aggregates. But as the RHA replacement level was raised, there was a reduction in the slump flow values because of the decrease in the

fineness modulus of fine aggregates containing RHA. Chopra et al. (2015), Memon et al. (2008) and Kannan and Ganesan (2014) reported that slump flow and other fresh properties of concrete decreased with increased percentage of replacement of RHA and MK. The greater surface area of MK and RHA particles can be the reason behind this.

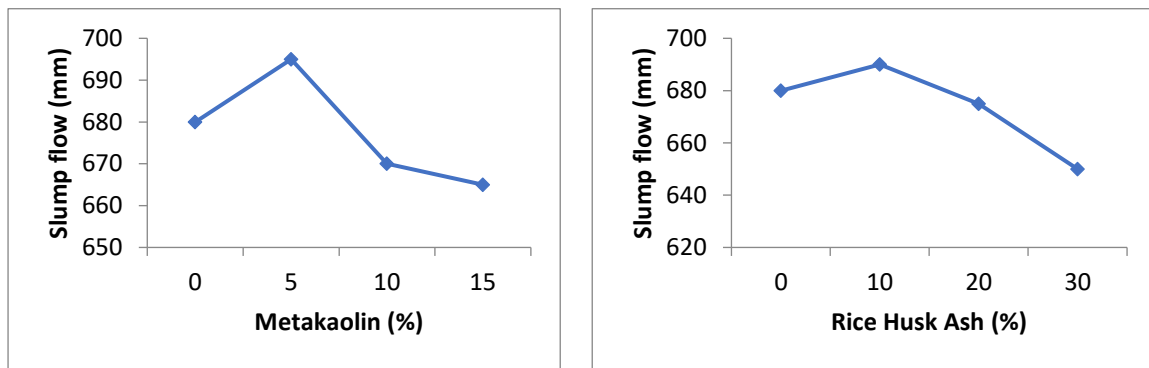


Fig 4.1: (a) Effect of addition of MK on slump flows (b) Effect of addition of RHA on slump flows

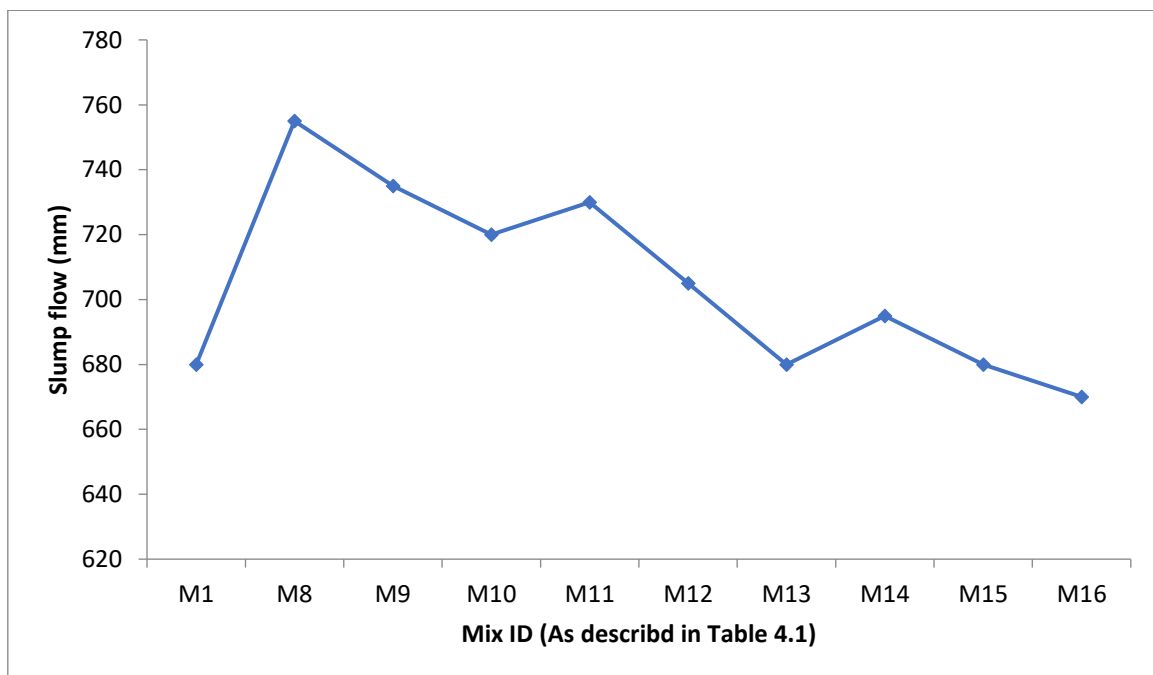


Fig 4.2: Comparison of slump flow values of SCC mixes made with combination of MK and RHA

4.1.2 L – box test

L – box values were in the range of 0.6 to 0.9. All the mixes satisfied the EFNARC criteria for L – box test, except mix M7. But Felekoglu et al. (2007) concluded that a blocking ratio from 0.6 to 1 is adequate to obtain assuasive filling ability. Mix M7 satisfied EFNARC (2005) criteria for slump flow, V-funnel and U-box tests. Thus, all the mixes have good filling ability. The L – box test results are given in Table 4.2. Decrease in the blocking ratio was observed with the use of MK and RHA.

Table 4.2: L – Box test results of all SCC mixes

Mix ID	Mix Type	L – box (H2/H1)
		EFNARC (2005) Range: 0.8 - 1
M1	Control Mix	0.9
M2	5MK	0.9
M3	10MK	0.8
M4	15MK	0.8
M5	10RHA	0.8
M6	20RHA	0.8
M7	30RHA	0.6
M8	5MK10RHA	0.9
M9	5MK20RHA	0.9
M10	5MK30RHA	0.9
M11	10MK10RHA	0.8
M12	10MK20RHA	0.8
M13	10MK30RHA	0.8
M14	15MK10RHA	0.8
M15	15MK20RHA	0.8
M16	15MK30RHA	0.8

4.1.3. V – funnel test

The V- funnel time for all the mixes ranges from 8 to 12 seconds, which were within the span specified by EFNARC. So, all mixes satisfied the criteria laid by EFNARC thus indicating good flow ability. The results are given in Table 4.3. As it can be observed from Table 4.3, the V – funnel time for SCC mixes decreased with the use of MK and RHA when compared to mix made without MK and RHA. But at the same time V – funnel times also increased with the increase in the percentages of MK and RHA, the pattern observed for the slump flow results.

Table 4.3: V – funnel results of all SCC mixes

Mix ID	Mix Type	V – funnel (sec)
		EFNARC (2002) range: 6 – 12 sec
M1	Control Mix	11
M2	5MK	10
M3	10MK	10
M4	15MK	12
M5	10RHA	10
M6	20RHA	11
M7	30RHA	12
M8	5MK10RHA	8
M9	5MK20RHA	9
M10	5MK30RHA	10
M11	10MK10RHA	8
M12	10MK20RHA	10
M13	10MK30RHA	10
M14	15MK10RHA	10
M15	15MK20RHA	11
M16	15MK30RHA	12

Memon et al. (2008), Chopra et. al. (2015) and Kannan and Ganesan (2014) reported that slump flow and other fresh properties of concrete decreased with increased addition of RHA and MK. They attributed this to the higher surface area provided by RHA and MK particles. Karahan et al. (2012) found interrelationship between the MK and T_{500} slump flow and V-funnel flow times. They stated that rise in MK content increases both T_{500} slump flow and V-funnel flow times. So, the present results were found to be in similar to their observations.

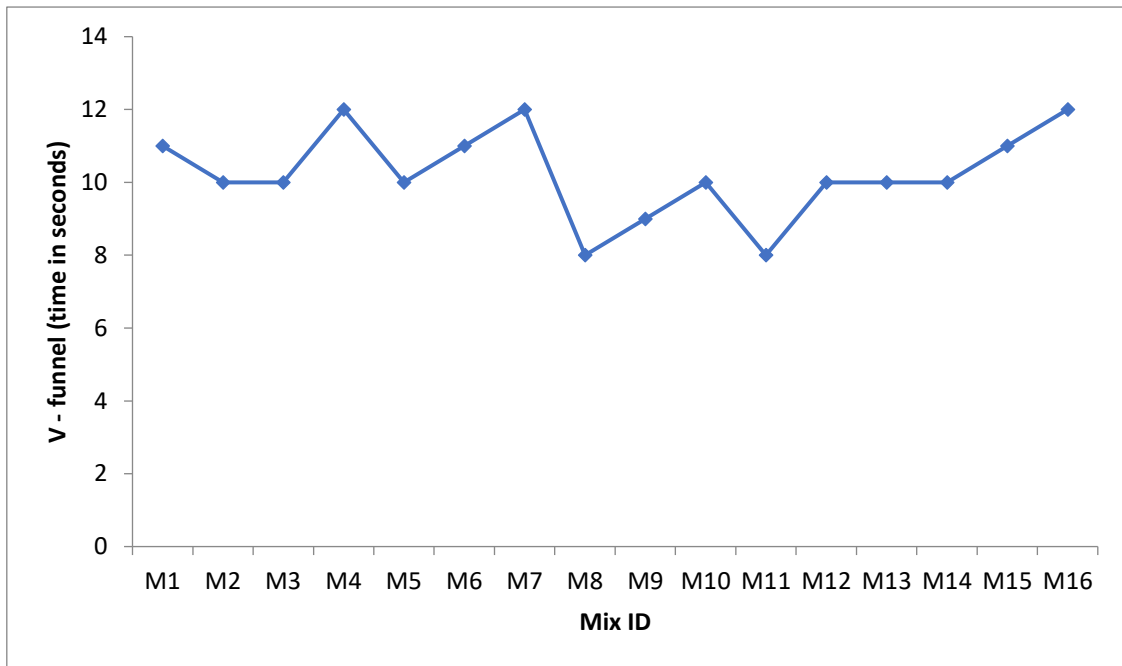


Fig 4.3: Comparison of V – funnel time for all SCC mixes



Fig 4.4: V – funnel test

4.1.4 U – box test

U – Box test is a very important test which gives idea about the filling ability of self-compacting concrete. The test result values were in span of 8 – 30 mm, which were in accordance with EFNARC criteria. The U – box results are tabulated in Table 4.4 and are compared in Fig. 4.5.

Table 4.4: U – box results of all SCC mixes

Mix ID	Mix Type	U – box (H2-H1)
		EFNARC (2002) Range: 0 – 30
M1	Control Mix	12
M2	5MK	19
M3	10MK	24
M4	15MK	29
M5	10RHA	18
M6	20RHA	24
M7	30RHA	30
M8	5MK10RHA	8
M9	5MK20RHA	11
M10	5MK30RHA	16
M11	10MK10RHA	10
M12	10MK20RHA	15
M13	10MK30RHA	19
M14	15MK10RHA	12
M15	15MK20RHA	17
M16	15MK30RHA	22

The U – box value for the control mix (M1) was 12. When MK was used in the mix this value increased to 19, 24 and 29 for 5, 10 and 15% replacement level of MK respectively. Similarly,

with the use of RHA, this value comes to be 18, 24 and 30 for 10, 20 and 30% replacement level of RHA respectively. So clearly the use of MK and RHA increased the U – box values of the mix. Similarly, when MK and RHA were used in combination, the U – box values comes in the range of 8 to 22. The mix M8, made with 5% MK and 10% RHA had the best U – box value of 8. According to EFNARC the lesser the difference in the level of concrete in two compartments, the better will be the filling ability of the mix. But all the mixes had U- box value within the EFNARC limits. The addition of MK and RHA leads in the increase of water demand, which can be the reason behind the increased U – box values of the mixes made with MK and RHA. This can be attributed to the higher surface areas of MK and RHA particles. Mix M8 (5MK10RHA) had the lowest U – box difference of 8. The above results of the present study were found in similar to Vejmelkova et al. (2011), who also reported rise in water demand with the addition of MK.



Fig 4.5: Comparison of U – box values of all SCC mixes

4.2. Strength Properties

4.2.1 Compressive strength

SCC mixes were first designed with the individual use of MK and RHA. MK was used to replace cement in 5, 10 & 15 %, while RHA was used in place of fine aggregates in percentages of 10, 20 & 30 %. On the basis of 28–day compressive strength, ten additional mixes (M8 – M16), with combination of both (MK & RHA) were made. In mixes M8 – M10, MK content was kept constant at 5% & RHA content was varied between 10 to 30%. Similarly, in mixes

M11 – M13 & mixes M14 – M16 MK content was kept constant at 10% & 15% respectively, and RHA content was varied between 10 to 30%, similar to mixes M8 – M10.

4.2.1.1 Compressive strength of mixes prepared with metakaolin

The results for the compressive strength tests for mixes using MK are given in Table 4.5. The 28 – day compressive strength of control mix was 41.4 N/mm², while compressive strengths of mixes made with 5, 10 and 15% MK were 45.5, 47.9 and 51.7 N/mm² respectively. The compressive strength results for mixes prepared with MK bespeaks of 24% increase in strength at 28 days when correlated to control mix. The reason for this significant improvement in compressive strength can be associated with the filling effect of MK particles, and the pozzolanic reaction of MK with calcium hydroxide. The results were found to be similar to those reported by Khatib and Hibbert (2005), Hassan et al. (2012), Madandoust and Mousavi (2012) and Kannan and Ganesan (2014). Madandoust and Mousavi (2012) found that the compressive strength for mixes ranged between 13.1 and 46.7 MPa. MK leads in the rise of compressive strength. They also added that the optimum results for compressive strength were achieved with 10–15% MK. The present work also provides similar findings.

4.2.1.2 Compressive strength of mixes prepared with rice husk ash

The compressive strength results with RHA are tabulated in Table 4.5. For mixes prepared with replacement of fine aggregates with rice husk ash, mix M5 (with 10% RHA) achieved the 7-day and 28-day strength as 28.3 and 43.4 MPa respectively, which is 2.8% and 4.8% higher than control mix. Control mix gained strength of 27.53 and 41.4 N/mm² at 7 and 28 days respectively. When percentage of RHA was increased to 20% and 30%, strength decreased at both 7 and 28 days. Mix made with 20% replacement of fine aggregates with RHA gained strength of 24.2 and 36.2 N/mm² at 7 and 28 days respectively. While the mix made with 30% replacement showed compressive strength of 19.3 and 30.3 N/mm² at 7 and 28 days respectively. The results of the present study were in par with Chik et al. (2011). They reported that mix containing 15RHA exhibited the maximum compressive strength at 28 days, but as the replacement level is increased from 15% strength starts decreasing.

Table 4.5: Compressive strength results of SCC mixes prepared with MK and RHA

Mix ID	Mix type	Compressive strength (MPa)	
		7-Day	28-Day
M1	Control Mix	27.53	41.4
M2	5MK	32.6	45.5
M3	10MK	35.8	47.9
M4	15MK	38.1	51.7
M5	10RHA	28.3	43.4
M6	20RHA	24.2	36.2
M7	30RHA	19.3	30.3

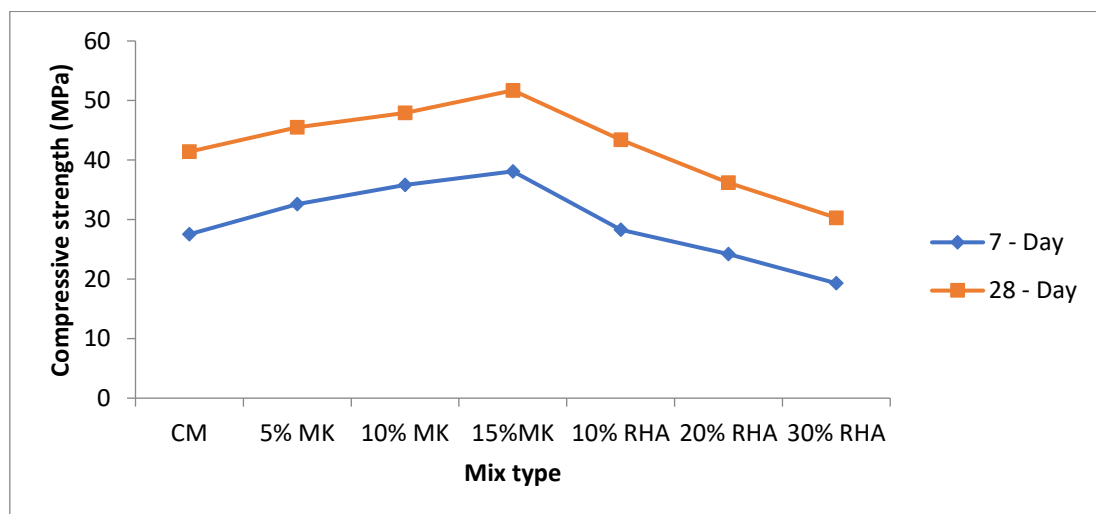


Fig. 4.6: Comparison of compressive strengths of SCC mixes made with individual use of MK and RHA

4.2.1.3 Compressive strength of mixes prepared with combine use of MK and RHA

Based on the results of compressive strength with MK and RHA separately, it was ascertained that compressive strength of mixes incorporating MK and RHA improved when correlated to the control mix. However, for RHA mixes, strength started to decrease after 10% replacement level. Thus, both MK and RHA were used to check the combined effect. The results are

tabulated in Table 4.6 and depicted in Figs 4.7 – 4.8. These Mixes include one control mix (M1), three mixes (M8,9,10) having 5% MK and 10 – 30% RHA, three mixes (M11,12,13) having 10% MK and 10 – 30% RHA and last three mixes (M14,15,16) having 15% MK and 10 – 30% RHA.

28-day compressive strength of the mixes gained from the combination of MK and RHA was more when correlated to control mix (M1). It was also higher than the mixes prepared with either MK and RHA. The mix M11 (10MK10RHA) showed maximum compressive strength of 34.70, 52.40, 64.86 and 71.45 MPa at the age of 7, 28, 90 and 365 days respectively, which was 26%, 27%, 42% and 48% more than the control mix at 7, 28, 90 and 365 days respectively. Similar trends were observed for other mixes also. The compressive strength results are depicted through various figures below.

Table 4.6: Compressive strength results for SCC mixes made with combination of MK and RHA

Mix ID	Mix type	Compressive strength (MPa)			
		7-Day	28-Day	90-Day	365-Day
M1	Control Mix	27.53	41.40	45.46	48.35
M8	5MK10RHA	28.76	49.90	57.63	62.10
M9	5MK20RHA	26.50	47.10	56.40	60.15
M10	5MK30RHA	29.30	44.26	56.46	59.40
M11	10MK10RHA	34.70	52.40	64.86	71.45
M12	10MK20RHA	28.23	47.60	56.56	63.50
M13	10MK30RHA	24.33	46.63	55.53	61.65
M14	15MK10RHA	38.10	52.03	62.30	69.05
M15	15MK20RHA	34.10	49.50	57.36	64.45
M16	15MK30RHA	32.13	46.63	52.60	59.95

The highest 28, 90 and 365 days compressive strength for mix M11 is due to combined effect of MK and RHA together. The mix M12 (15MK10RHA) showed a slight decrease in strength,

which could have been due to excessive chunk of silica available. The produced C-H was probably insufficient to react with the available silica. As a result, a small amount of silica was left without any chemical reaction (Rahman et. al., 2014). These results were found to be similar to those reported by Kannan and Ganesan (2014), who used MK and RHA separately and in combination to replace cement. They observed that compressive strength of mixes having combination of MK and RHA was more at all ages than mixes prepared with MK and RHA separately. Further they added that the compressive strength increased up to 15% with RHA, 20% with MK and 30% with the use of MK and RHA. In the present work, compressive strength increased up to 5% with RHA, 24% with MK, and 27% with the use of a combination of MK and RHA.

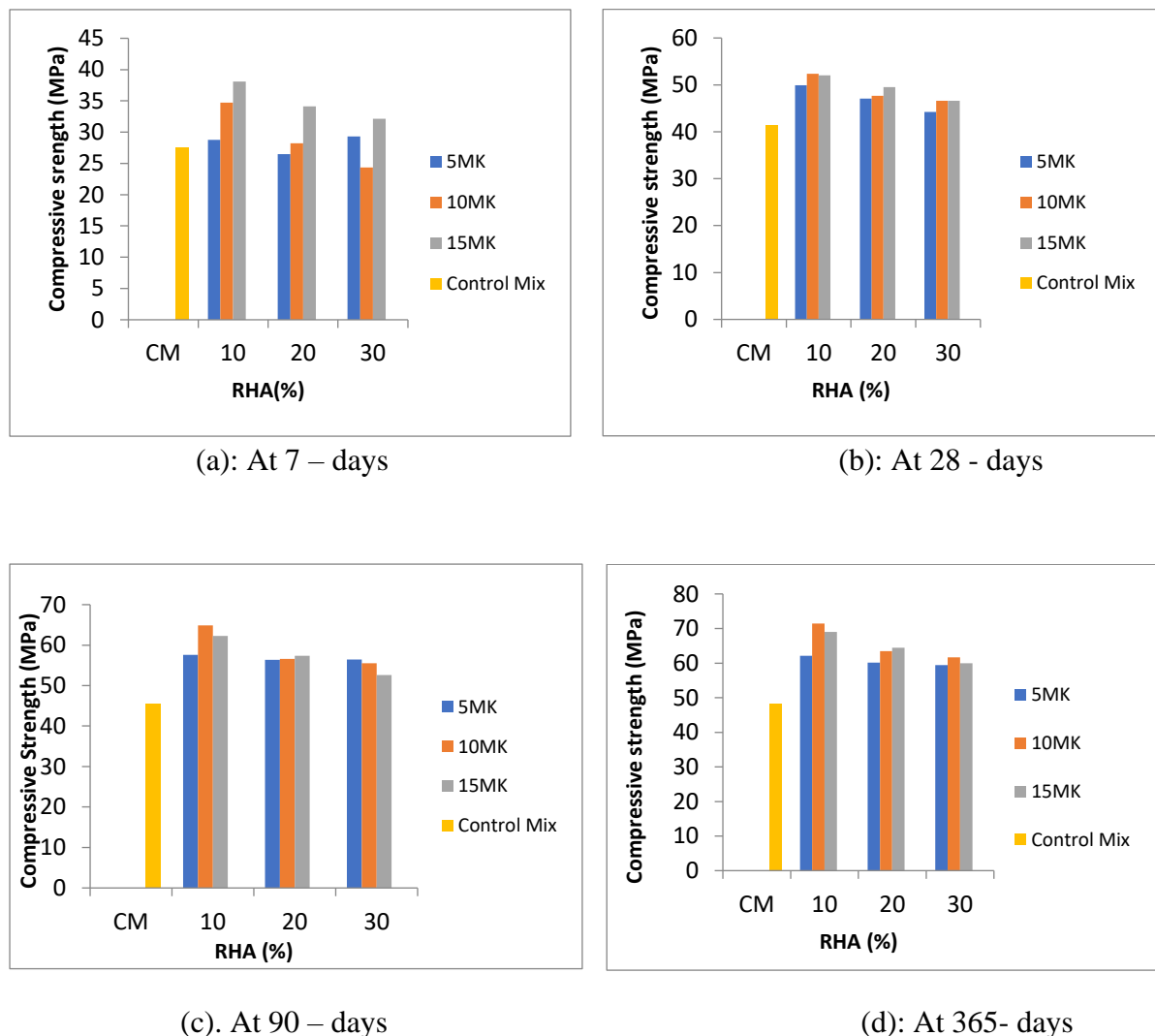
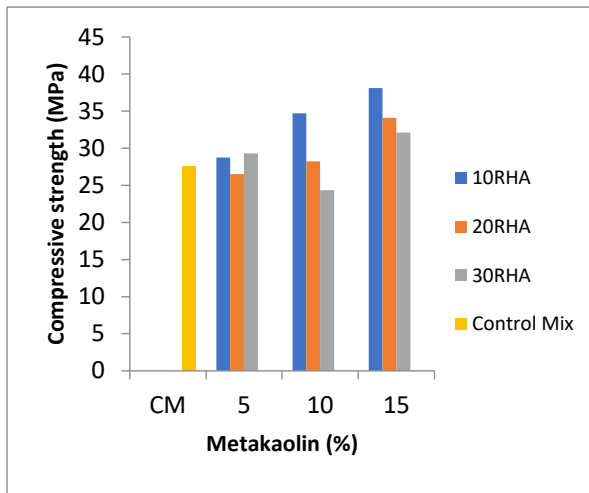
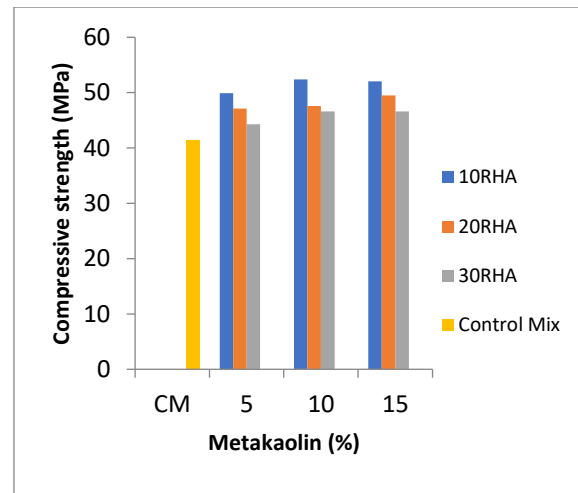


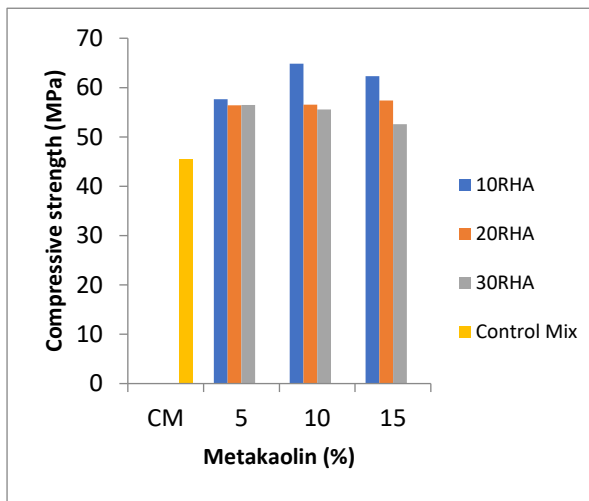
Fig. 4.7: Compressive strength of SCC mixes with RHA



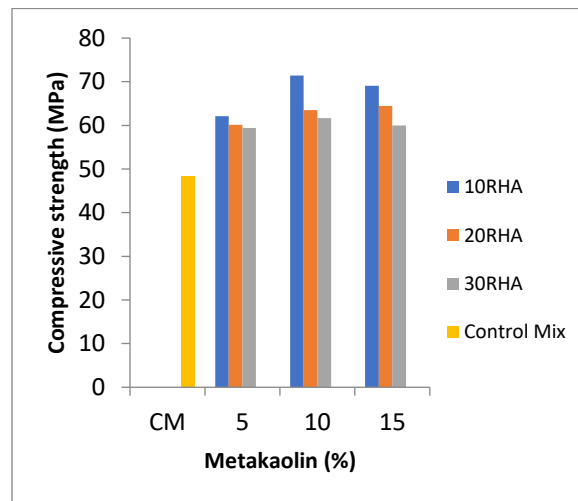
(a): At 7 – days



(b): At 28 - days



(c). At 90 – days



(d). At 365 - days

Fig. 4.8: Compressive strength of SCC mixes with MK

The Figs. 4.7(a – d) shows the effect of varying proportions of MK, with constant percentage of RHA at various ages. When percentage of RHA was kept at 10%, the compressive strength increases at 5 and 10% MK, while 15% MK causes a modest decrease of strength. Similar trends were achieved when percentage of RHA was kept at 30%. While at 20% RHA there were different trends observed. There was decrease in strength at 10% MK, while strength increases at 15% MK level. There were similar trends for similar percentages of MK and RHA at different ages.

The Figs. 4.8 (a -d) shows the effect of varying proportions of RHA, with constant percentage of MK at various ages. When percentage of MK was kept at 5%, there was increase in strength with increased percentage (upto 30%) of RHA at age of 7 days. There was 5, 26 and 38 % increase in strength at 10, 20 and 30 % RHA when compared to control mix. But at the age of 28 and 90 days, strength decreased at 30% RHA. Then there were similar trends when 10 and 15 % MK was used to replace cement. Strength increased at 10% RHA level, and then starts decreasing if RHA is used beyond that percentage. These trends were almost similar at all the ages.

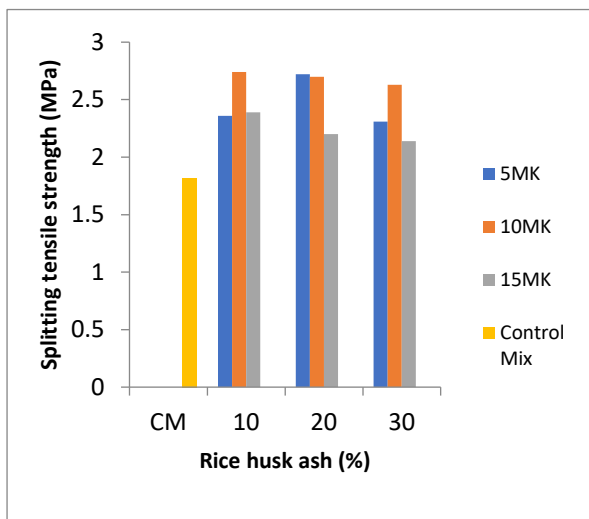
4.2.2 Splitting tensile strength

Similar trends as of compressive strength were observed for splitting tensile strength. The results are shown in Figs.4.9 – 4.10 and are tabulated in Table 4.7. Mixes prepared with MK showed more strength when correlated to mix M1. There was increase in strength of mixes made with all the percentages of MK, i.e. 5,10 and 15%. But the mixes prepared with RHA showed decline in strength with the increase in percentage of RHA. Mix made with 10% RHA showed increased strength as compared to control mix but when RHA was used at 20 and 30%, strength starts declining. Mixes prepared from the combination of MK and RHA displayed more strength at all ages when correlated to mix M1. The mix M11 (10MK10RHA) showed the maximum strength of 2.74, 4.43, 5.12 and 5.96 MPa at 7, 28, 90 and 365 days respectively. The studies carried out by Hassan et al. (2012), Madandoust and Mousavi (2012), Kannan and Ganesan (2014) and Chik et al. (2011) also provided the same results. Madandoust and Mousavi (2012) reported that there was approximately 9.8% increase in tensile strength of concrete at 28 days when 10% MK was used. This can be compared to 19.2 % increase in tensile strength as observed in this work. They also added that 10–15% MK substitution brings out finest results in splitting tensile strength, a finding similar to this work.

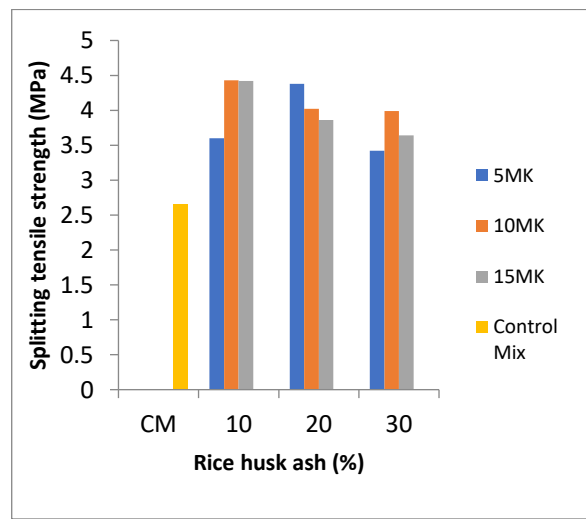
It can be observed from the Figs. 4.9 – 4.10 that strength with combined use of MK and RHA is more when correlated to the control mix. The substitution of RHA inversely affected the strength. Although strength of mixes made with MK and RHA were more when seen with respect to the control mix. The effect of RHA replacement on tensile strength can be observed from Figs. 4.9 (a – d). There was increase in strength up to 10% replacement level of RHA, but strength decreases at 20 and 30 % replacement levels. Similarly effect of MK replacement can be observed from Figs. 4.10 (a – d). There was increase in strength at 5 and 10 % replacement levels, but a slight decrease was observed in strength at 15 % replacement level.

Table 4.7: Splitting tensile strength results of SCC mixes with MK and RHA

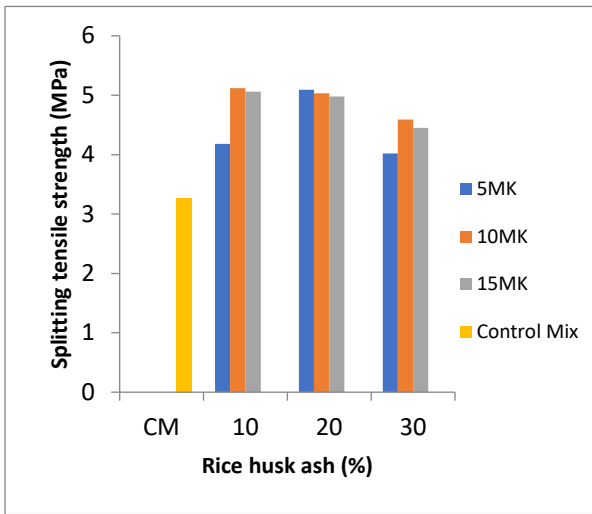
Mix ID	Mix type	Splitting tensile strength (MPa)			
		7-Day	28-Day	90-Day	365-Day
M1	Control mix	1.82	2.66	3.27	3.56
M8	5MK10RHA	2.36	3.60	4.18	4.83
M9	5MK20RHA	2.72	4.38	5.09	5.75
M10	5MK30 RHA	2.31	3.42	4.02	4.45
M11	10MK10RHA	2.74	4.43	5.12	5.96
M12	10MK20RHA	2.70	4.02	5.03	5.71
M13	10MK30RHA	2.63	3.99	4.59	5.01
M14	15MK10RHA	2.39	4.42	5.06	5.95
M15	15MK20RHA	2.20	3.86	4.98	5.75
M16	15MK30RHA	2.14	3.64	4.45	4.96



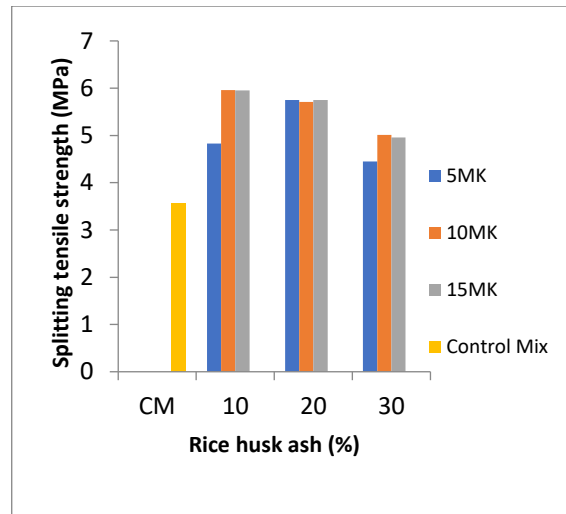
(a): At 7 – days



(b): At 28 - days

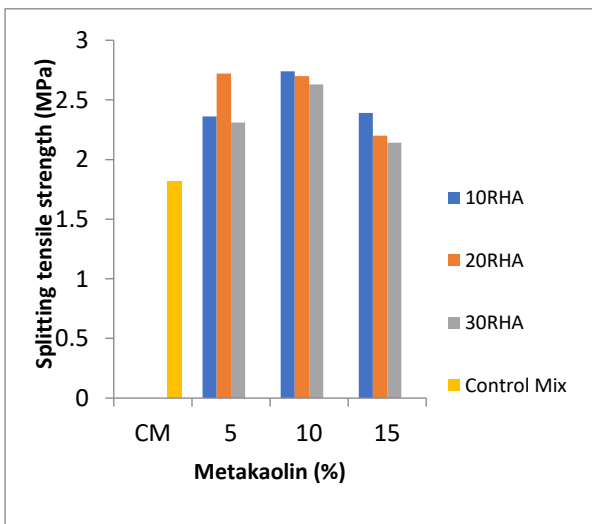


(c): At 90 – days

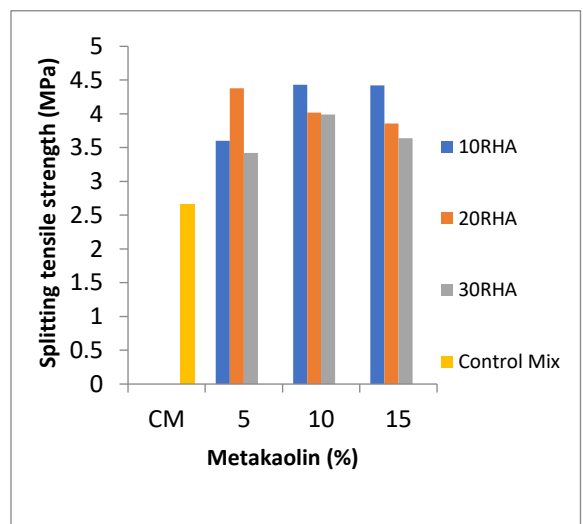


(d): At 365 – days

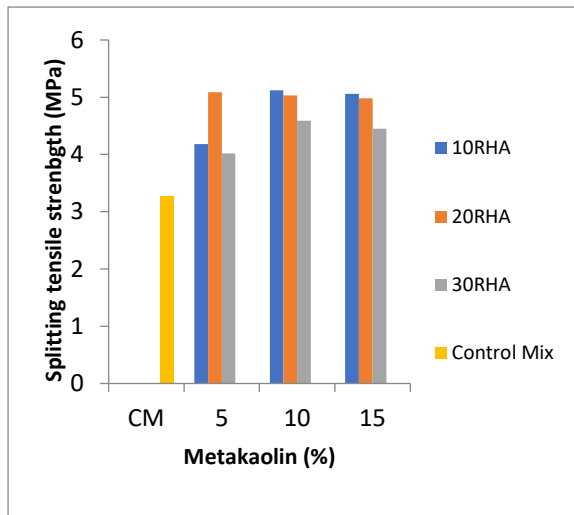
Fig 4.9: Splitting tensile strength of SCC mixes with RHA



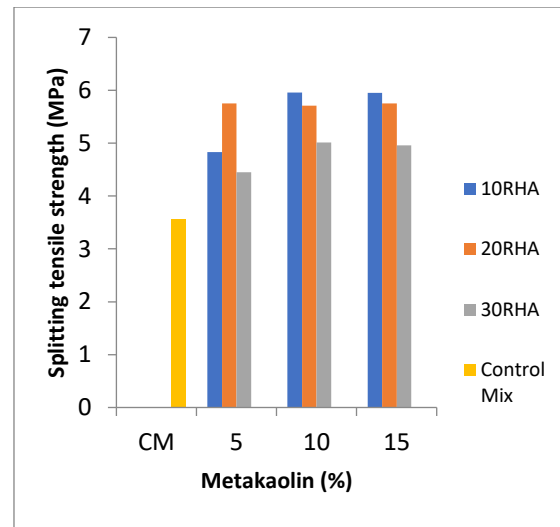
(a): At 7 – days



(b): At 28 – days



(c): At 90 – days



(d): At 365 - days

Fig 4.10: Splitting tensile strength of SCC mixes with MK

4.3 Durability Properties

4.3.1 Water absorption and porosity

Water absorption is a very important test to access the durability of concrete. Concrete should resist water absorption for longer life and greater durability. The results for water absorption and porosity are presented in Table 4.8 and Table 4.9. The water absorption and porosity for mixes containing MK+RHA was lowered as against the control mix (M1). Also, there was decrease in porosity as the curing period was increased. The water absorption (after immersion) of the control mix was 5.81%, 7.25% and 6.80% at 28, 90 and 365 days respectively, while the water absorption of mix M11 (10MK10RHA) was 3.93%, 4.90% and 4.10% at age of 28, 90 and 365 days. It can also be seen from table that water absorption is decreasing with the age of curing. For most of the mixes there was decrease in the values of water absorption and porosity between 90 days and 365 days. The pozzolanic reactions and the increased rate of hydrations can primarily be held for this change. The pozzolanic reaction between calcium hydroxide and silica results in production of C-S-H gel, which further helps in filling the voids resulting in dense concrete (Chopra et al. 2015). But it was also detected, that the level of water absorption and porosity rises with the increased percentage of MK.

This unfavourable effect can be associated with the MK and RHA's higher surface area, lower fineness modulus value, and subsequent water demand in the concrete mixing process. When fresh state SCC blended with MK and RHA, the workability gradually reduced. This diminution in the workability of MK and RHA-blended SCC creates voids in the concrete, and

may be withheld for poor resistance to water absorption (Kanan and Ganesan, 2014). The above results are match able with the results of Kannan and Ganesan (2014). They stated that when MK and RHA are used in SCC, water absorption was improved by 30 to 40%, and is lower than the mix without MK and RHA. They also observed that there is adverse effect on water absorption with the increase percentage of MK and RHA. This finding is in connate to findings in this work. There is 70% decrease in water absorption for mix made with combination of 5%MK and 10%RHA, whereas there is only 45% and 5% decrease in water absorption for mixes made with 10%MK10%RHA and 15%MK10%RHA respectively. When RHA content was increased there was 70% decrease in water absorption at 5%MK and 10% RHA, while there was decrease of 69% and 68% decrease at 5%MK20%RHA and 5%MK and 30%RHA respectively. So, there is adverse effect on water absorption with the increased percentages of MK and RHA. Rehman et al. (2014) and Ganesan et al. (2008) also observed similar trends in their research work with the use of MK and RHA. The results of water absorption and porosity are also compared through Figs. 4.11 - 4.12.

Table 4.8: Water absorption results of all SCC mixes

Mix ID	Mix Type	Absorption after immersion (%)			Absorption after immersion and boiling (%)		
		28 - Day	90 - Day	365 - Day	28 - Day	90 - Day	365 - Day
M1	Control Mix	5.81	7.25	6.8	5.92	7.84	7.16
M8	5MK10RHA	2.05	3.84	3.7	1.76	4.13	3.2
M9	5MK20RHA	2.11	4.15	4.2	1.82	5.01	5.1
M10	5MK30RHA	2.14	5.00	4.4	1.88	6.70	5.4
M11	10MK10RHA	3.93	4.90	4.1	3.25	6.90	5.7
M12	10MK20RHA	5.07	4.36	3.95	4.77	4.78	4.65
M13	10MK30RHA	4.58	4.94	5.2	3.77	5.90	5.98
M14	15MK10RHA	5.90	4.10	5.35	5.61	4.80	5.21
M15	15MK20RHA	5.25	6.20	6.41	4.65	7.35	7.23
M16	15MK30RHA	5.98	6.05	6.56	5.03	6.97	6.81

Table 4.9: Porosity results of all mixes

Mix ID	Mix Type	Porosity (%)		
		28 - Day	90 - Day	365 - Day
M1	Control Mix	7.15	10.65	10.4
M8	5MK10RHA	3.01	6.7	5.4
M9	5MK20RHA	6.38	11.14	9.7
M10	5MK30RHA	9.42	7.02	8.46
M11	10MK10RHA	3.27	7.4	8.1
M12	10MK20RHA	8.26	8.10	9.1
M13	10MK30RHA	7.82	11.55	11.91
M14	15MK10RHA	3.35	10.9	9.1
M15	15MK20RHA	6.88	11.24	11.35
M16	15MK30RHA	8.12	10.85	9.12

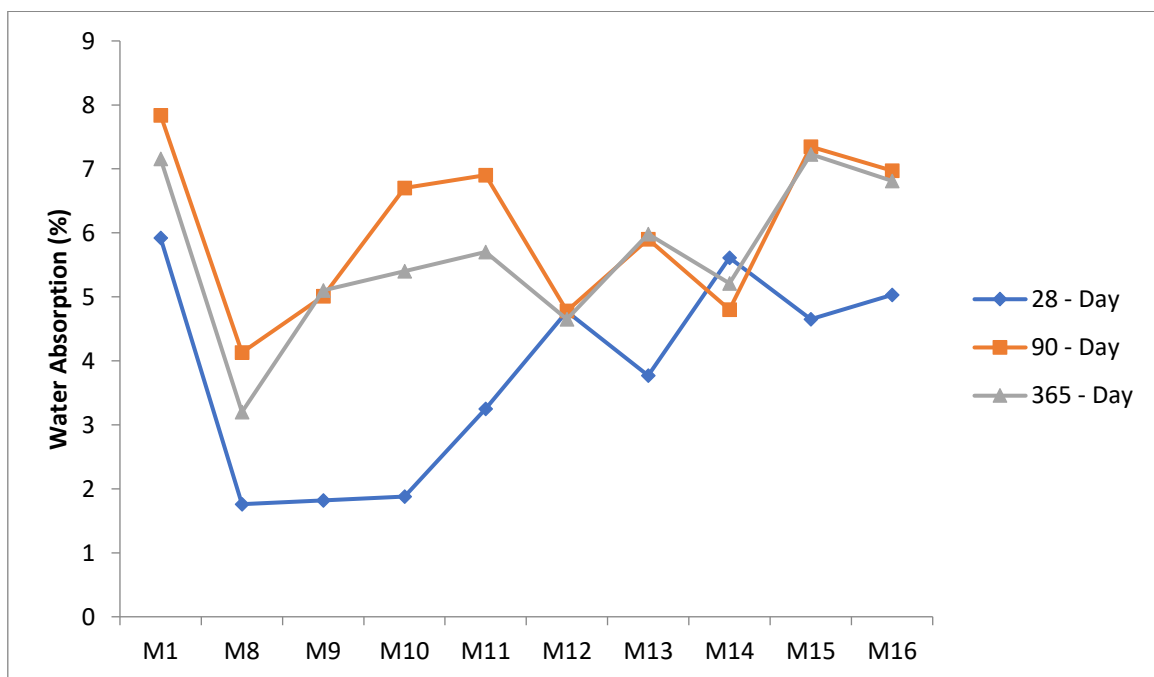


Fig. 4.11: Water absorption (after immersion and boiling) results of all SCC mixes

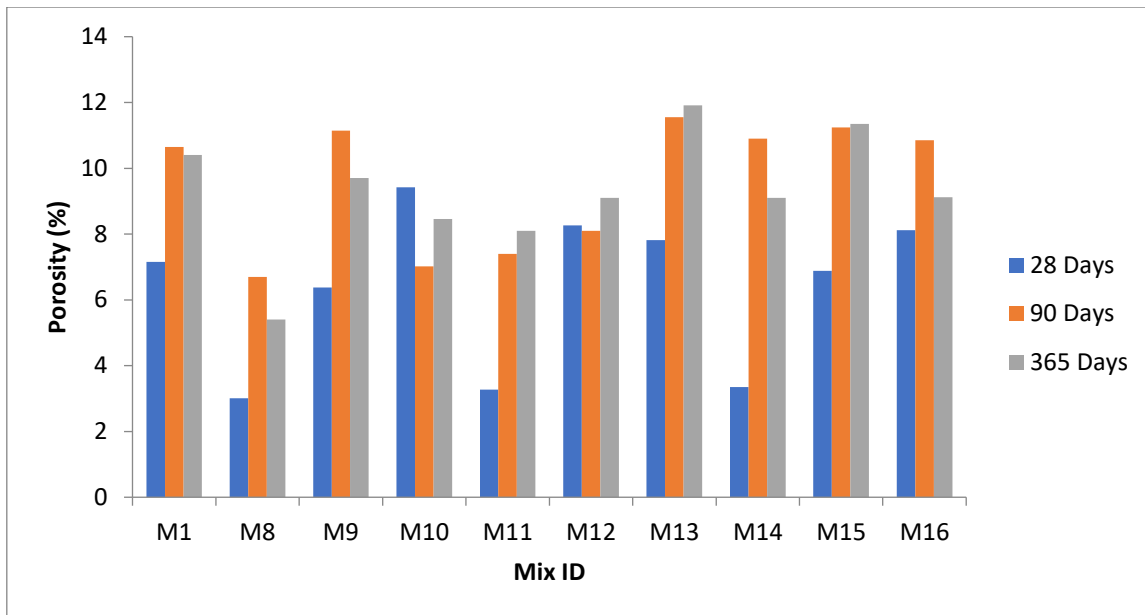


Fig. 4.12: Porosity results comparison of all SCC mixes

4.3.2 Sulphate resistance

The results for sulphate resistance test are tabulated in Table 4.10 and are compared in Figs 4.13 - 4.17. It was detected that the strength loss was maximum in control mix, while mix M11 had minimum of strength loss. All the mixes made with the use MK and RHA had better resistance to sulphate attack as against the control mix (M1). Previous studies have confirmed that the formation of calcium sulfoaluminate (ettringite) depends purely on the reaction between alumina (Al_2O_3) and sulfate (SO_4) and the ratio of the two compounds (Marco et al., 2008). The formation of ettringite leads to expansion in the concrete, which causes disruption of the set cement paste (Rawal, 2003). The combination of MK and RHA leads to lower alumina content with higher silica content (Kanan and Ganasen, 2014), which is the main reason for the better results for mixes made with MK and RHA.

Kannan and Ganesan (2014) stated the similar kind of findings as of this work. They exposed specimens to 5% solution of sulphuric acid, to check the sulphate resistance of specimens. They observed that SCC made with RHA & MK was more resistant against sulfuric acid attack than was SCC without MK and RHA, a finding similar to this work. In this work, there was 14%, 16% and 18% loss of compressive strength at 28, 90 and 365 days respectively for mix without MK and RHA, whereas there was only 3.1%, 4.5% and 4.8% loss of compressive strength at 28, 90 and 365 days respectively, when 10%MK and 10% RHA in combination are used in the mix. The results are also comparable to Said et al. (2010).

Table 4.10(a): Sulphate resistance results of all SCC mixes at age of 28 - days

Mix Id	Mix Type	Compressive Strength at 28 – days (MPa)		Loss in Compressive Strength (%)
		Water Curing	Sulphate Solution Curing	
M1	Control Mix	41.40	35.53	14 %
M8	5MK 10RHA	49.90	46.50	6.8 %
M9	5MK20RHA	47.10	43.5	7.6 %
M10	5MK30RHA	44.26	42.33	4.3 %
M11	10MK10RHA	52.40	50.76	3.1 %
M12	10MK20RHA	47.60	45.60	4.2 %
M13	10MK30RHA	46.63	45.63	2.1 %
M14	15MK10RHA	52.03	49.85	4.1 %
M15	15MK20RHA	49.50	44.86	9.4 %
M16	15MK30RHA	46.63	44.1	5.4 %

Table 4.10(b): Sulphate resistance results of all SCC mixes at age of 90 – days

Mix Id	Mix Type	Compressive Strength at 90 – days (MPa)		Loss in Compressive Strength (%)
		Water Curing	Sulphate Solution Curing	
M1	Control Mix	45.46	38.15	16%
M8	5MK10RHA	57.63	53.9	6.6%
M9	5MK20RHA	56.40	52.6	6.7%
M10	5MK30RHA	56.46	50.9	6.5%
M11	10MK10RHA	64.86	61.9	4.5%
M12	10MK20RHA	56.56	53.7	5%
M13	10MK30RHA	55.53	52.9	4.7%
M14	15MK10RHA	62.30	58.8	5.6%
M15	15MK20RHA	57.36	51.36	10.4%
M16	15MK30RHA	52.60	49.4	6.1%

Table 4.10(c): Sulphate resistance results of all SCC mixes at age of 365 – days

Mix Id	Mix Type	Compressive Strength at 365 – days (MPa)		Loss in Compressive Strength (%)
		Water Curing	Sulphate Solution Curing	
M1	Control Mix	48.35	39.65	17.9 %
M8	5MK 10RHA	62.10	57.75	7 %
M9	5MK20RHA	60.15	56.80	5.5 %
M10	5MK30RHA	59.4	56.35	5 %
M11	10MK10RHA	71.45	67.96	4.8 %
M12	10MK20RHA	63.50	59.88	5.7 %
M13	10MK30RHA	61.65	58.45	5.2 %
M14	15MK10RHA	69.05	65.10	5.2 %
M15	15MK20RHA	64.45	57.95	8.5 %
M16	15MK30RHA	59.95	56.25	5.9 %

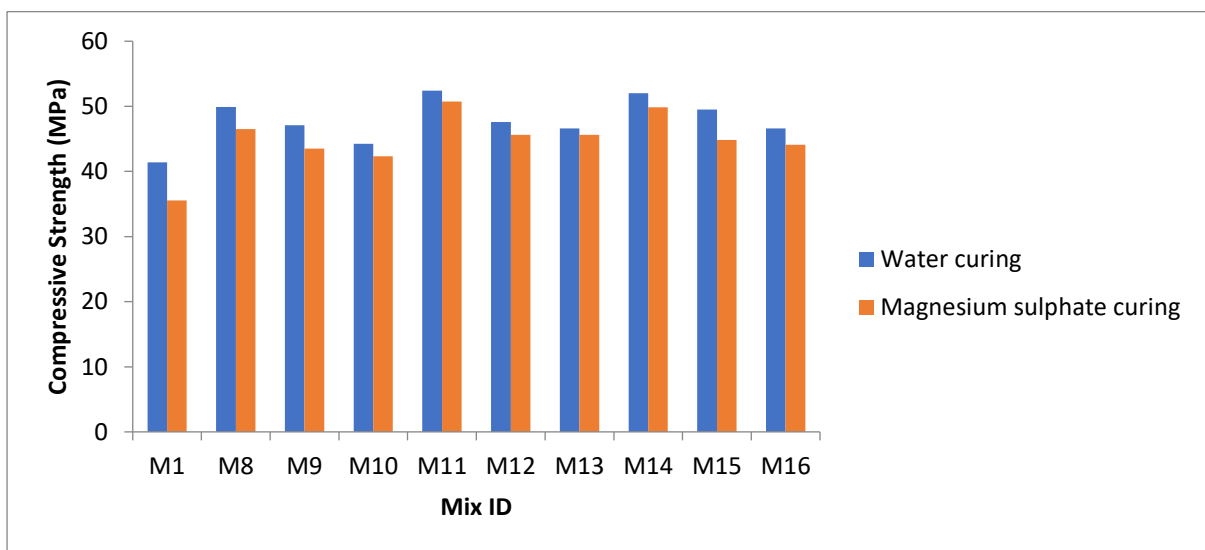


Fig 4.13: Sulphate resistance test results of all SCC mixes at 28 days

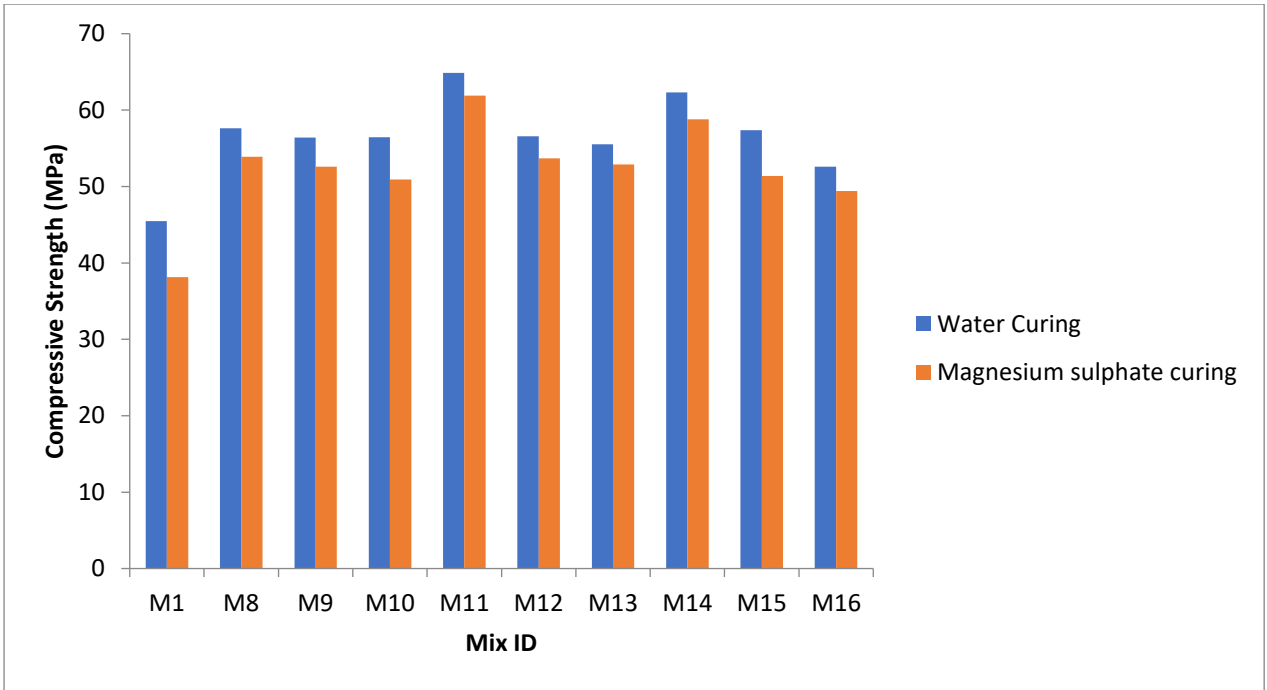


Fig 4.14: Sulphate resistance test results of all SCC mixes at 90 days

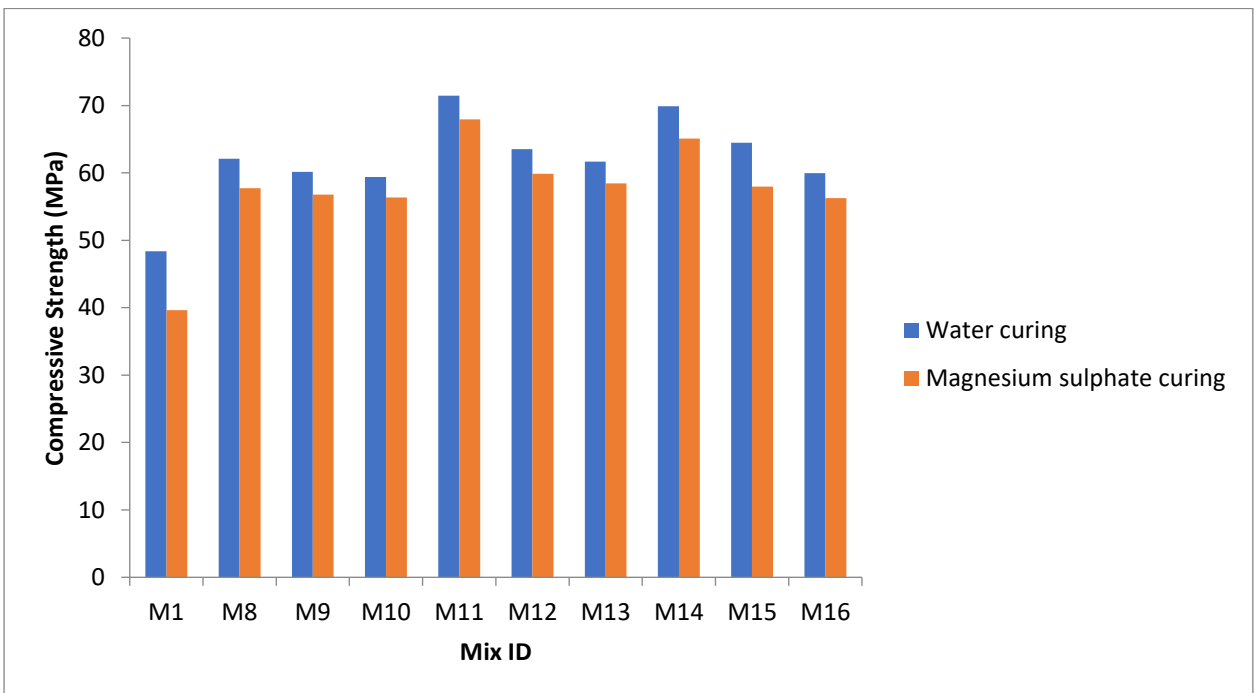


Fig 4.15: Sulphate resistance test results of all SCC mixes at 365 days

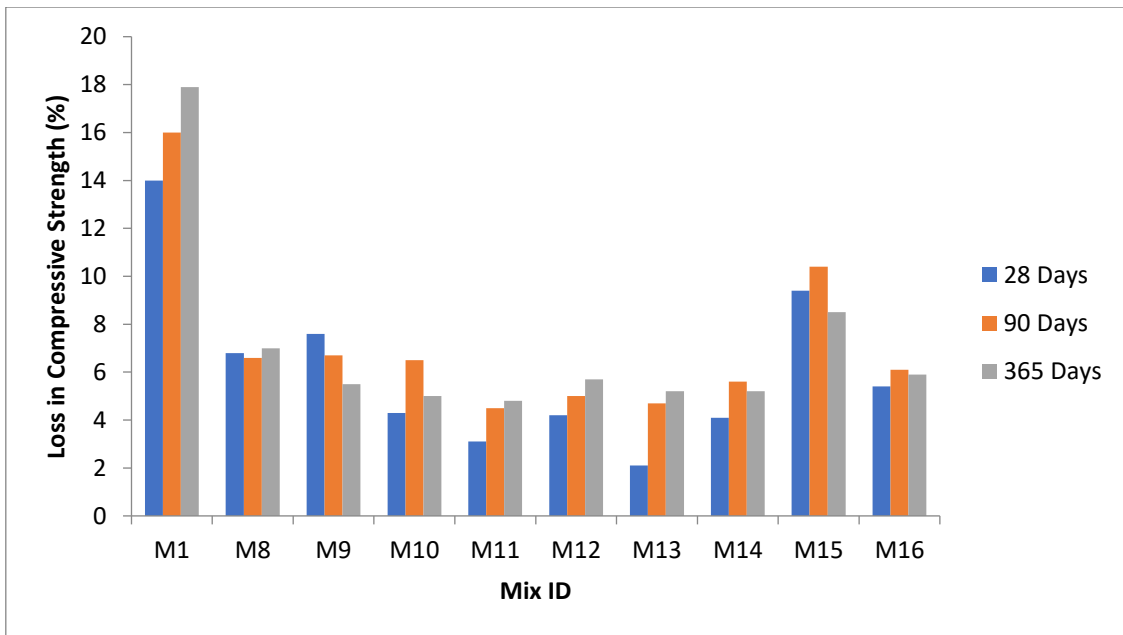


Fig 4.16: Percentage loss in compressive strength of all SCC mixes

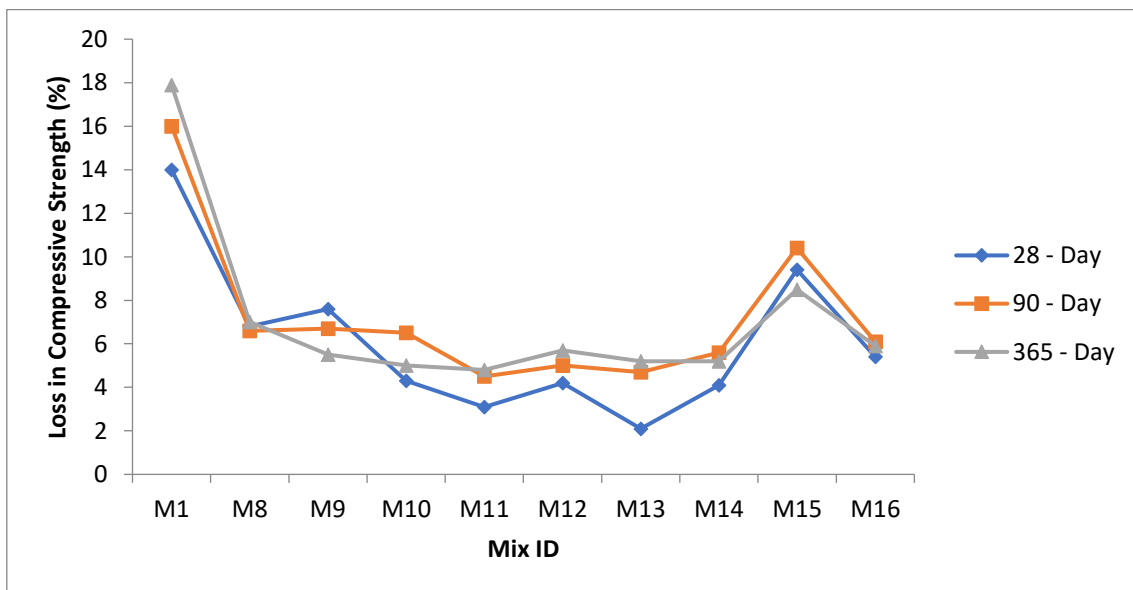


Fig 4.17: Percentage strength loss comparison of all SCC mixes

It can be noticed from the above figures that the strength loss was maximum for mix M1, made without MK and RHA. This strength loss increased with the increased age. The minimum strength loss at 28 days was observed for mix M13. There was 2.1% strength loss for this mix at 28 days as compared to 14% loss for the control mix (M1). So, there was 85% improvement in sulphate resistance with the use of MK and RHA. Talking about 90 and 365 days, minimum

strength loss was observed for mix M11. There was loss of 4.5% and 4.8% at 90 and 365 days respectively. So, there was 72% and 73% improvement in sulphate resistance at 90 and 365 days respectively with the use of MK and RHA. Talking about the percentages of MK and RHA, it was observed that sulphate resistance improved up to 10% replacement level of MK, while at 15 % there was more loss of compressive strength as compared to 5 and 10% replacement levels, but it was still less than the control mix. While in the case of RHA, 10% replacement level was found to be optimum level.

4.3.3 Rapid chloride permeability

Chloride penetration results for all concrete mixes in terms of the total charge passed are presented in Table 4.11 and Fig. 4.18. It was detected, that there was drop in chloride penetration with the use of MK and RHA. For mix M1, made without use of MK and RHA the permeability values were 1495, 175 and 171 coulombs at 28, 90 and 365 days respectively. When MK was used in percentages of 5, 10 and 15% with 10% constant percentage of RHA, permeability values decreases at all the ages. At 5%MK10%RHA the permeability values were 1273, 57 and 50 coulombs at 28, 90 and 365 days respectively. So, there was 15%, 67% and 70% improvement in chloride permeability with 5%MK and 10% RHA. At 10%MK and 10%RHA permeability values were 930, 47 and 38 coulombs at 28, 90 and 365 days respectively. So, there was 38%, 73% and 77% decrease in chloride permeability. With 15%MK and 10%RHA there was 44%, 74% and 77% decrease in chloride permeability at 28, 90 and 365 days respectively. The pozzolanic reaction of MK with $\text{Ca}(\text{OH})_2$ can be accredited for this low chloride penetration. Also, the fine size of MK particle helps in the discontinuity of the porosity network which further reduced the chloride penetration. The total charge passed for all blended mixes were in category of 'low' and 'very low' (As per ASTM C1202 standards).

Previous studies have established the fact that the chloride ion permeability of SCC with mineral admixtures is lower than that of SCC made without them (Zhu and Peter, 2003). Similar results were also obtained by Kannan and Ganeshan (2014), who observed that the chloride ion permeability of the SCC made with MK and RHA decreased up to 40%. Similar in this work, there is decrease of 77%, when MK and RHA was used in combination. So, the combined use of MK and RHA significantly improved the chloride ion permeability of SCC.

Table 4.11: Rapid chloride permeability test results of all SCC mixes

Mix ID	Mix Type	RCPT (Coulombs)		
		28 - Day	90 - Day	365 - Day
M1	Control Mix	1495	175	171
M8	5MK10RHA	1273	57	50
M9	5MK20RHA	1155	49	46
M10	5MK30RHA	1115	53	49
M11	10MK10RHA	930	47	38
M12	10MK20RHA	875	41	40
M13	10MK30RHA	925	44	42
M14	15MK10RHA	830	45	39
M15	15MK20RHA	790	39	38
M16	15MK30RHA	845	48	43

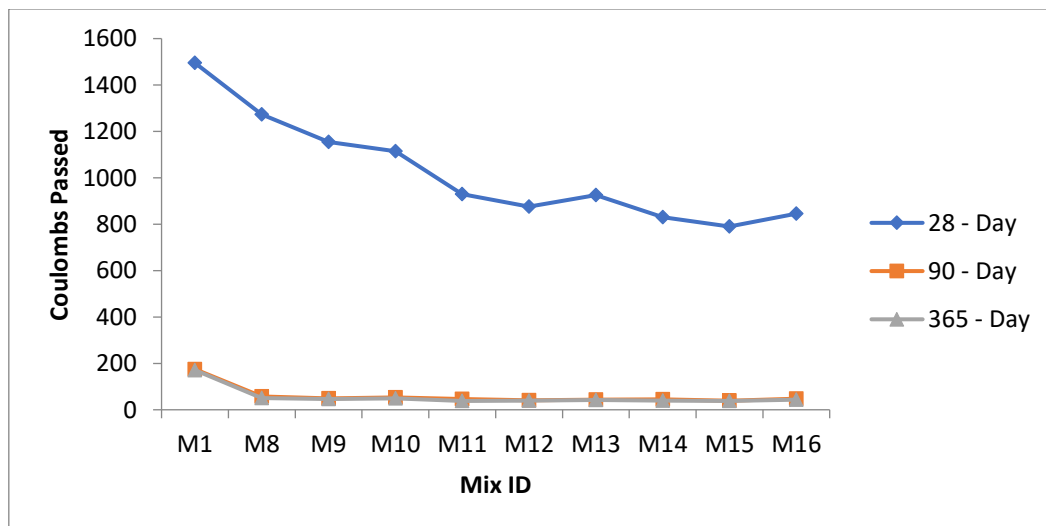


Fig 4.18: Rapid chloride permeability results comparison for all mixes

The above figure clearly indicates in the decrease of chloride permeability for all the mixes when compared to mix M1 (control mix). It can also be observed that permeability values keep on decreasing till mix M12, but there was a little rise for permeability values for mix M13, which was made with 10%MK and 30%RHA. Again, it can be observed that permeability

values take a little rise for mix M16, made with 15%MK and 30%RHA. These results can be correlated to the compressive strength results, where 10%RHA content gave the best results, while strength starts decreasing at 20% and 30% replacement levels. Here also the chloride permeability increases at 30% RHA replacement level as compared to 10% and 20% replacement levels. So, 10% RHA content was observed to be an optimum replacement content.

On the other hand, the effect of MK replacement can also be observed from the above figure. The replacement of cement with MK had the positive impact on chloride permeability. As observed from the figure, mix M1, made without addition of MK and RHA had the highest chloride permeability. At 28 days chloride permeability was 1475 coulombs for mix M1. The MK inclusion in all other mixes leads in decreased permeability values, lowest among all was 38 coulombs for mix M11(10MK10RHA). Now it can also be noticed from the Fig. 4.18 that permeability values take a little rise at 15%MK replacement level as compared to 5% and 10% replacement levels. Similar trends were also observed for compressive strength results where strength increased up to 10% replacement level of MK. So, it can be concluded that 10%MK and 10% RHA content can be considered as optimum replacement contents.

4.4 Micro-Structural Properties

4.4.1 Scanning electron microscopic (SEM) analysis

SEM images for mixes prepared with combination of MK and RHA are shown in Figs. 4.19 – 4.28. The SEM images for control mix at 28 and 90 days are shown in Fig. 4.19. The images clearly describe that the sample for mix without MK and RHA (M1) consists of voids and micro pores. This can be attributed to the poor performance of mix M1, when correlated to mixes containing MK and RHA. The voids can be clearly seen in Fig. 4.19. These voids and micro pores act as a passage for the water and other solutions. This is the reason for the poor performance of control mix in water absorption, sulphate resistance and rapid chloride permeability properties. Also, there is uneven spread of C-S-H gel in some parts along with the formation of ettringites. This leads to the lower strength values of the control mix.

However, in the case of the samples for mixes containing RHA and MK (Figs. 4.20 – 4.28) a more homogeneous structure can be seen. The fine size of the MK and RHA particles minimizes the pores to large extent. Also, the hydration process leads to the pore structure refinement. This reason has contributed towards the improved strength and durability properties of MK and RHA concrete. In mix M8, which is made with addition of 5% MK and

10% RHA, SEM image shows fewer voids as compared with SEM image of control mix. The SEM images of mix M9 and M10 shows formation of calcium silicate hydrate (C-S-H) gel, and the gel is widely spread. This formation of C-S-H gel further minimized the voids and also results in the improved strength and durability properties.

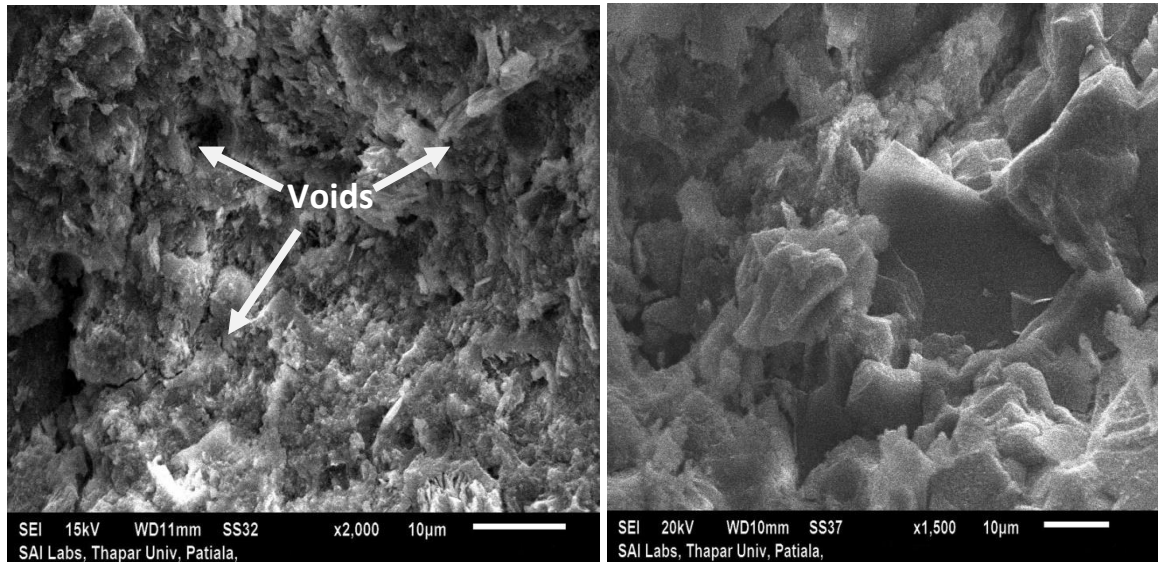


Fig 4.19: SEM image of mix M1 (Control mix) at 28 and 365 days

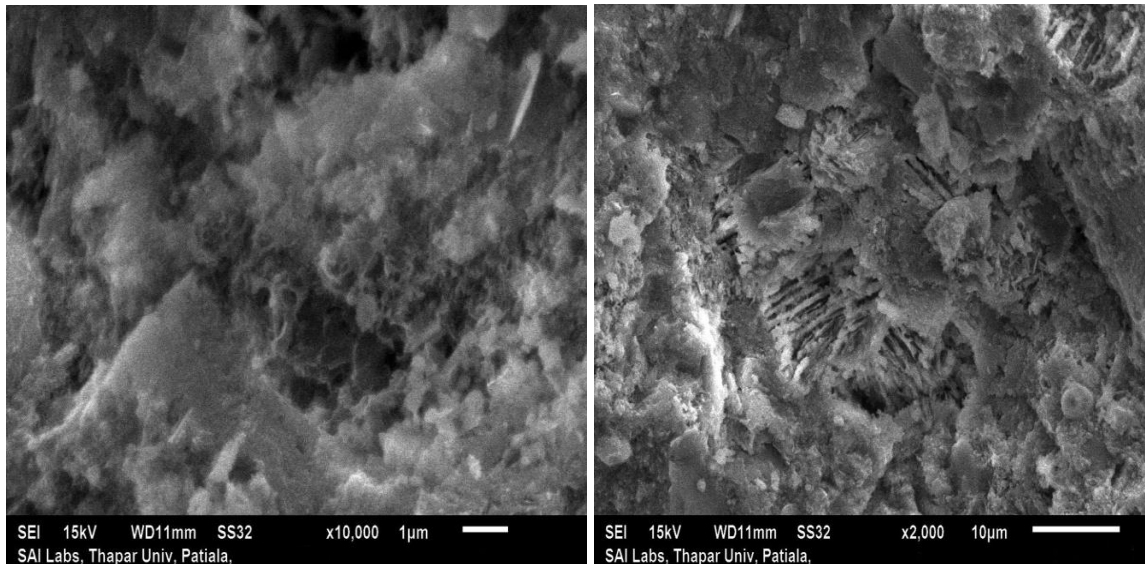


Fig 4.20: SEM image for mix M8 (5MK10RHA) at 28 & 365 days

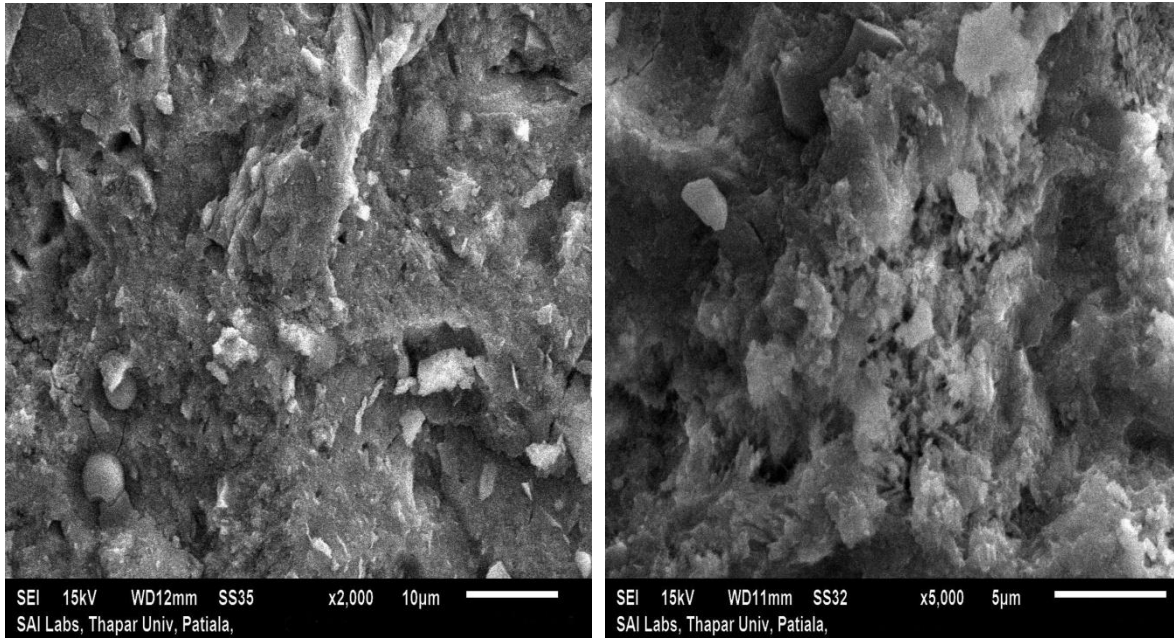


Fig 4.21: SEM image for mix M9 (5MK20RHA) at 28 & 365 days

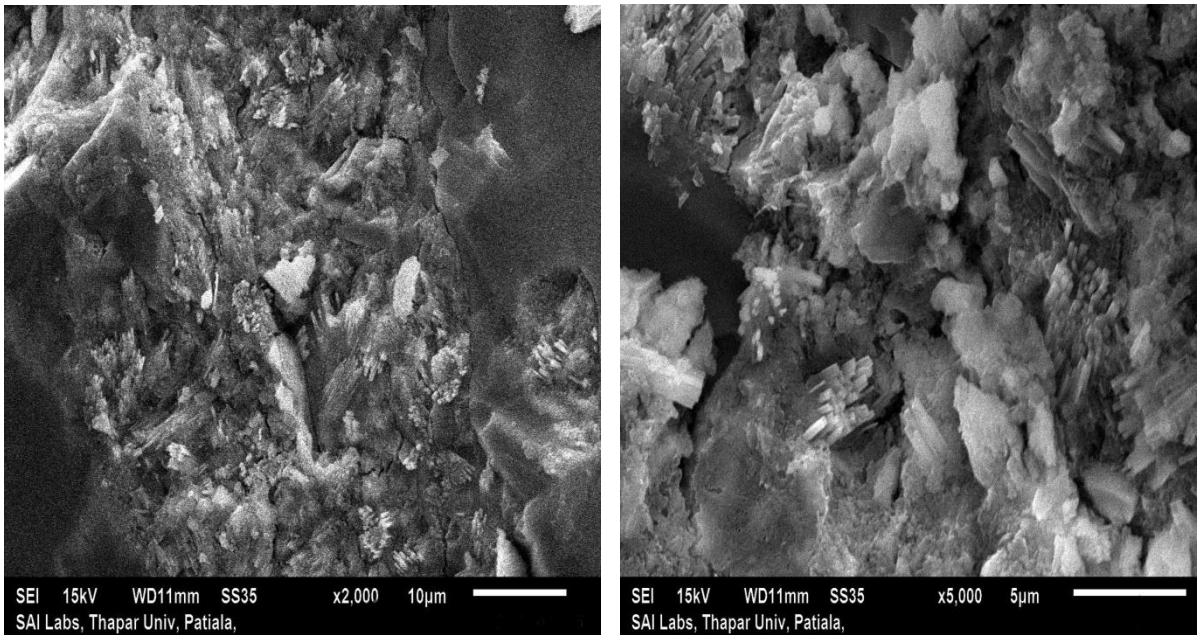


Fig 4.22: SEM image for mix M10 (5MK30RHA) at 28 & 365 days

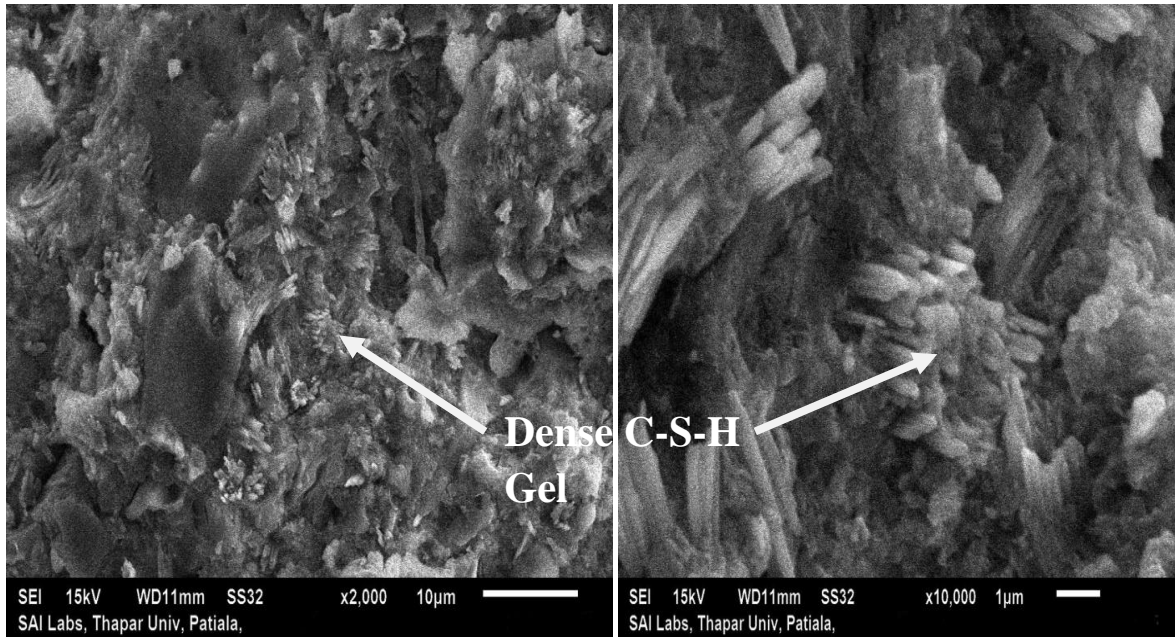


Fig 4.23: SEM image for mix M11 (10MK10RHA) at 28 and 365 days

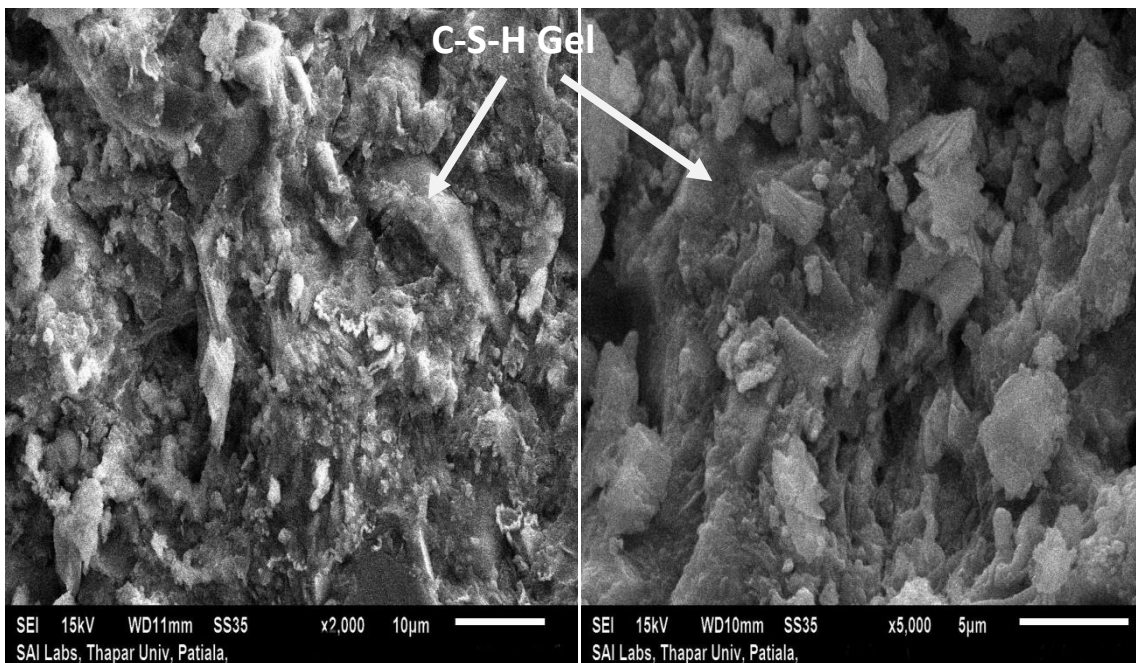


Fig 4.24: SEM image for mix M12 (10MK20RHA) at 28 and 365 days

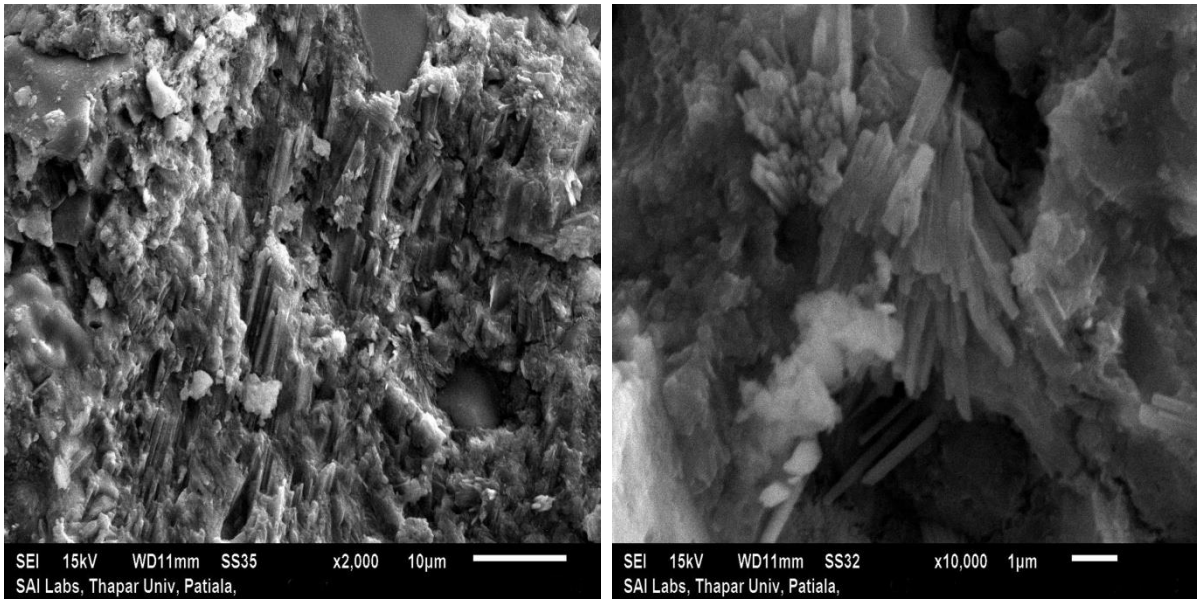


Fig 4.25: SEM image for mix M13 (10MK30RHA) at 28 and 365 days

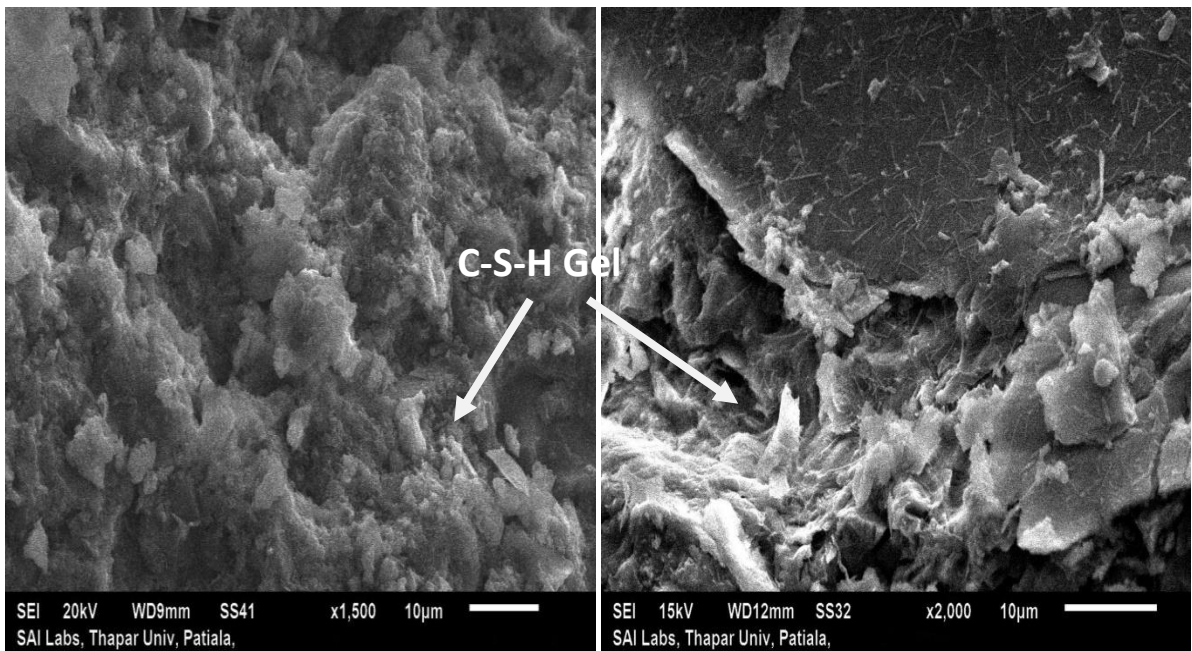


Fig 4.26: SEM image for mix M14 (15MK10RHA) at 28 and 365 days

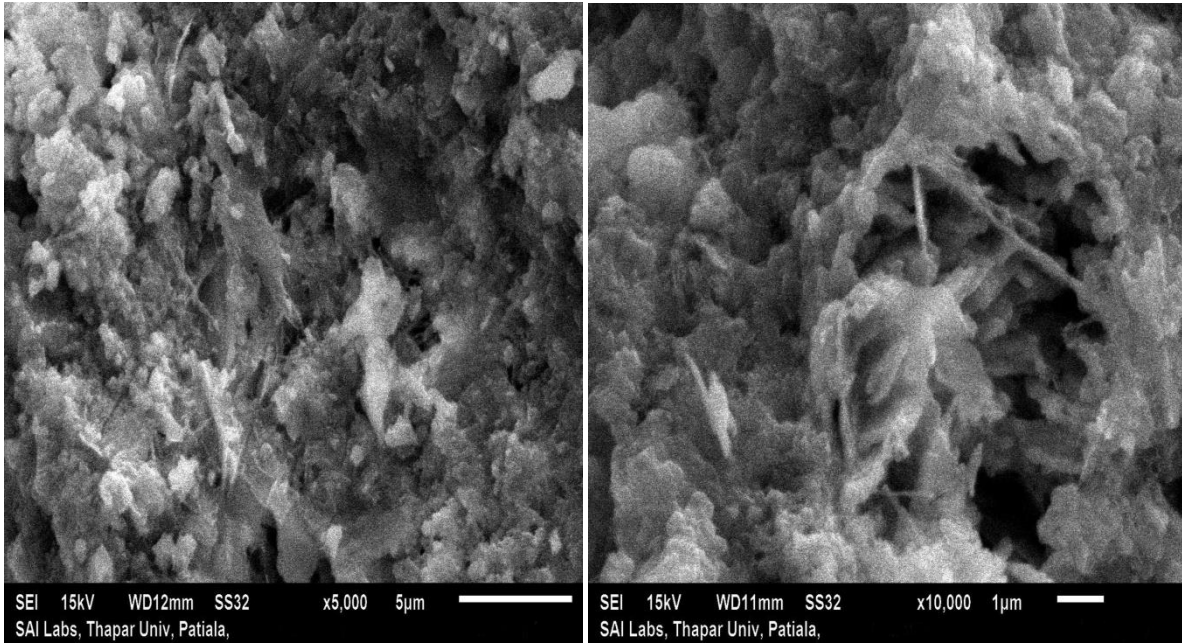


Fig 4.27: SEM image for mix M15 (15MK20RHA) at 28 and 365 days

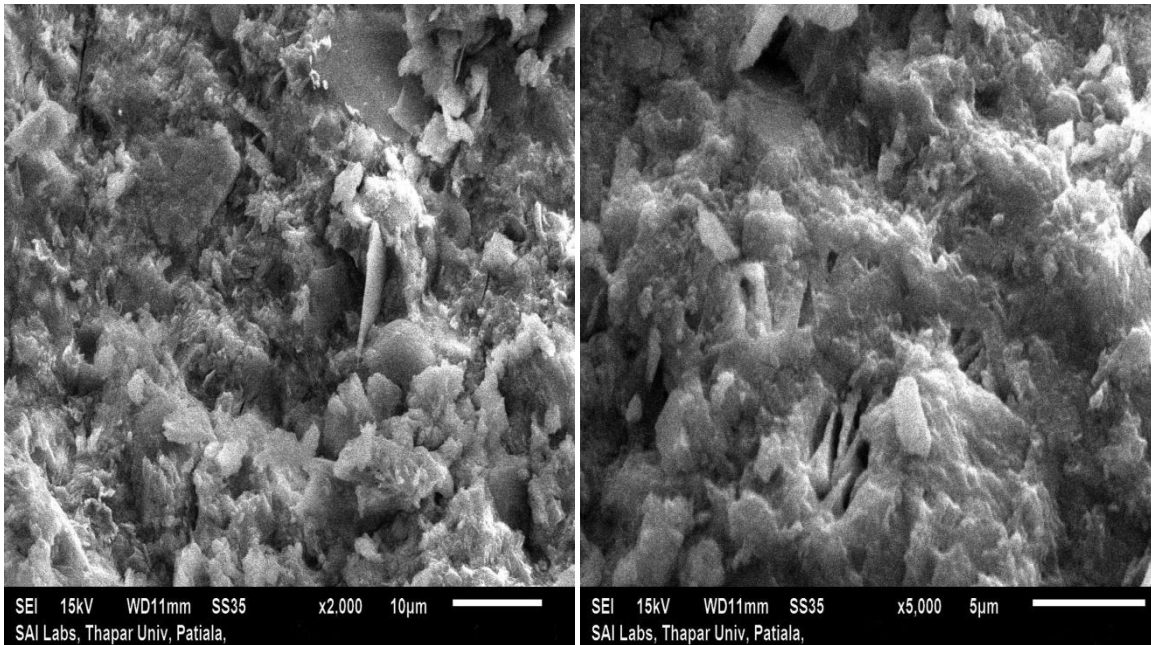


Fig 4.28: SEM image for mix M16 (15MK30RHA) at 28 and 365 days

As percentage of MK and RHA was increased, the structure becomes more uniform and denser. The pores were reduced to great extent. Fine particle size of MK and RHA can be accounted

for this. Fig. 4.23 shows the formation of dense and distinctly spread calcium silicate hydrate (C-S-H) gel and voids are minimized. Also, C-S-H gel can be observed in its short needle like form and fine bundles and ettringite needles. This is the reason why the mix M11 showed the best strength and durability properties. Hydration process can also be held for minimization of micro pores. It can be clearly seen that C-S-H gel is more widely spread for mixes containing MK and RHA, which leads to more strength.

In the cases when percentage of RHA is increased the mixes started disintegrating. The SEM images show inconsistent formation of C-S-H gel. This can be attributed for the reduced strength properties of these mixes. The slight decrease in the strength of these mixes could have been due to excessive chunk of silica left in the hydrated cement matrix. The produced C-H was probably insufficient to react with the available silica. Due to this a small amount of silica was left without any chemical reaction (Kim et al., 2007). SEM images clearly shows the dense matrix for mixes made with MK and RHA. With the rise in the percentage of MK and RHA, the matrix becomes denser and voids were eliminated. This is the reason for their better performance in strength properties.

4.4.2 XRD analysis

XRD images are shown in Figs. 4.29 - 4.38. It can be observed from the figures that the C_2S , C_3S , and C_4AF peaks were not visible, testifying the fact that they may be totally consumed. It can be seen from the Figs. 4.29 (a – b), that for control mix, the amount of unused silica is maximum, reaching up to 3500. At 28 days the amount of unused silica peak is even crossing mark of 3500. This can be directly linked to the performance of the control mix. For the mix M11 containing 10% MK and 10% RHA, the intensity of free silica is minimum. The SiO_2 peak for mix M11 reaches up to value of 2500 only at 365 days. There was more formation of C-S-H gel, the fact which testifies the strength and durability performance of this mix.

For mix M14 made with 15% MK and 10% RHA, the peak for SiO_2 reached up to 2900 and 2600 at 28 and 365 days respectively (Figs. 4.36 (a -b)), which is slightly more than mix M11. This can be linked to the slight decrease in strength of mix M14. It was found that with the increased metakaolin percentages in concrete mixes, utilization of silica in C-S-H gel increased. This was observed up to 10% MK level. At 15 % MK level, this trend differs. The presence of calcium hydroxide is very less in all concrete mix, which confirms the maximum consumption in the hydration reaction. This leads to the dense microstructure and additional development of C-S-H gel, leading to the improvement of microstructure of the concrete, which

has resulted in the improvement of the strength and durability properties. The highest peak was near 28° angle, so it is crystalline in nature.

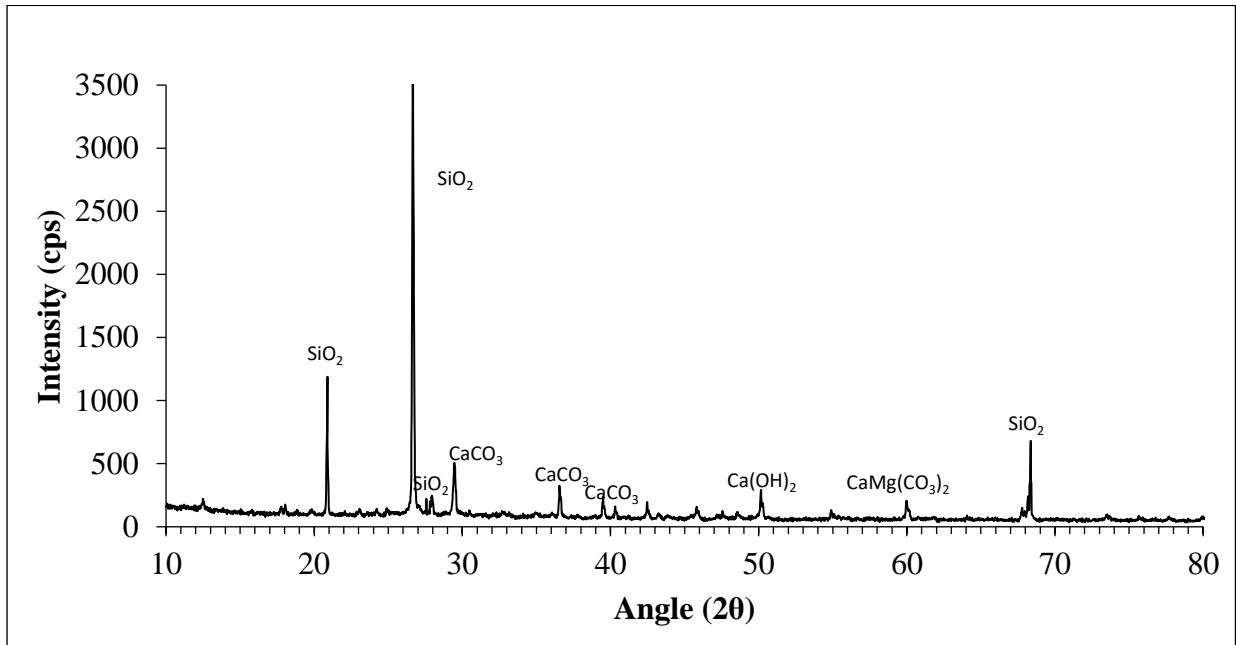


Fig. 4.29(a): XRD image for mix M1 (Control mix) at 28 days

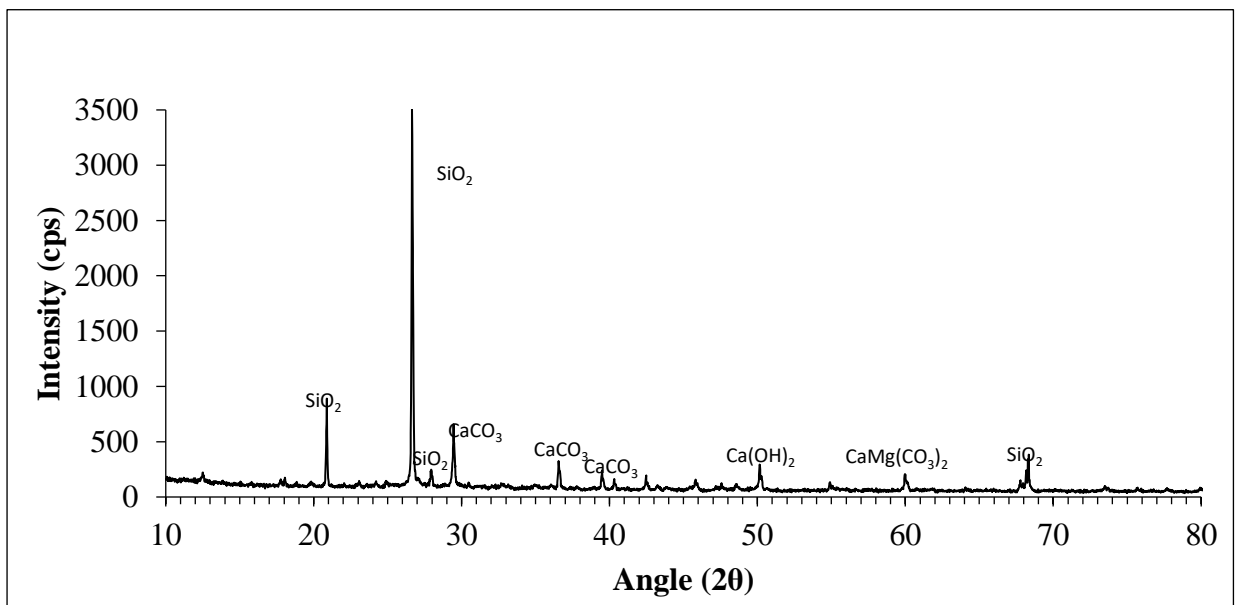


Fig. 4.29(b): XRD image for mix M1 (Control mix) at 365 days

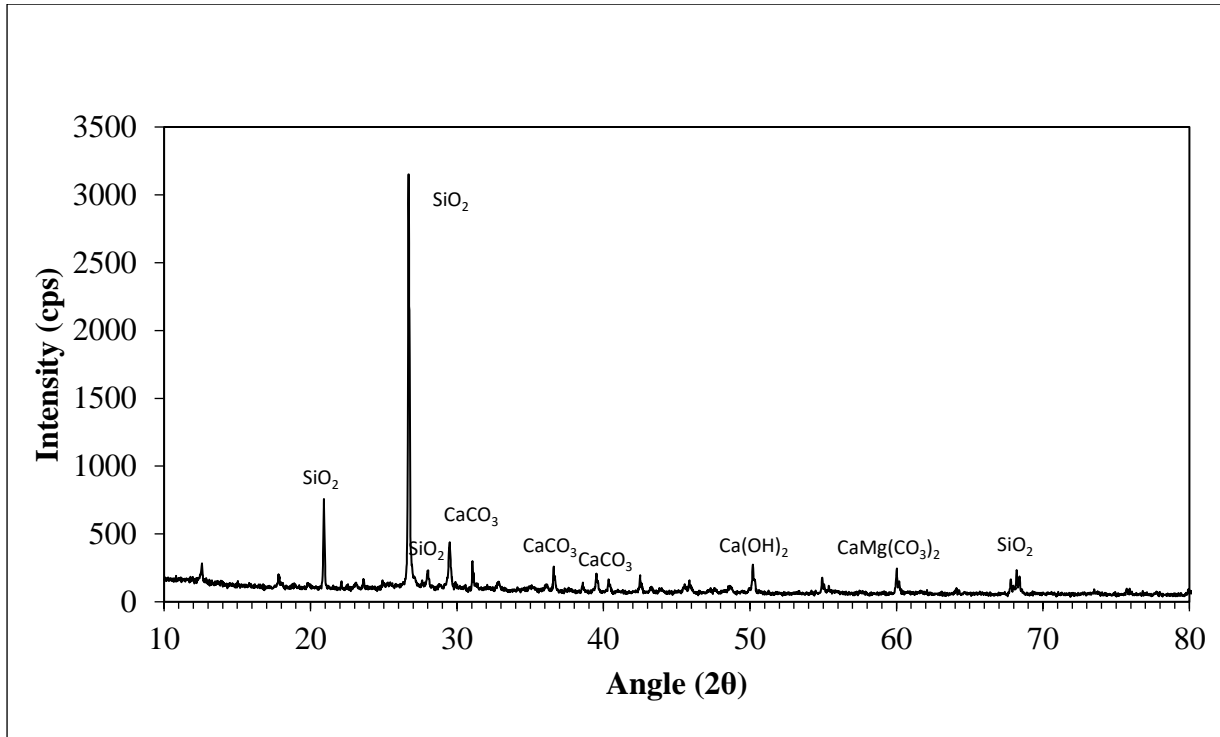


Fig. 4.30(a): XRD image for mix M8 (5MK10RHA) at 28 days

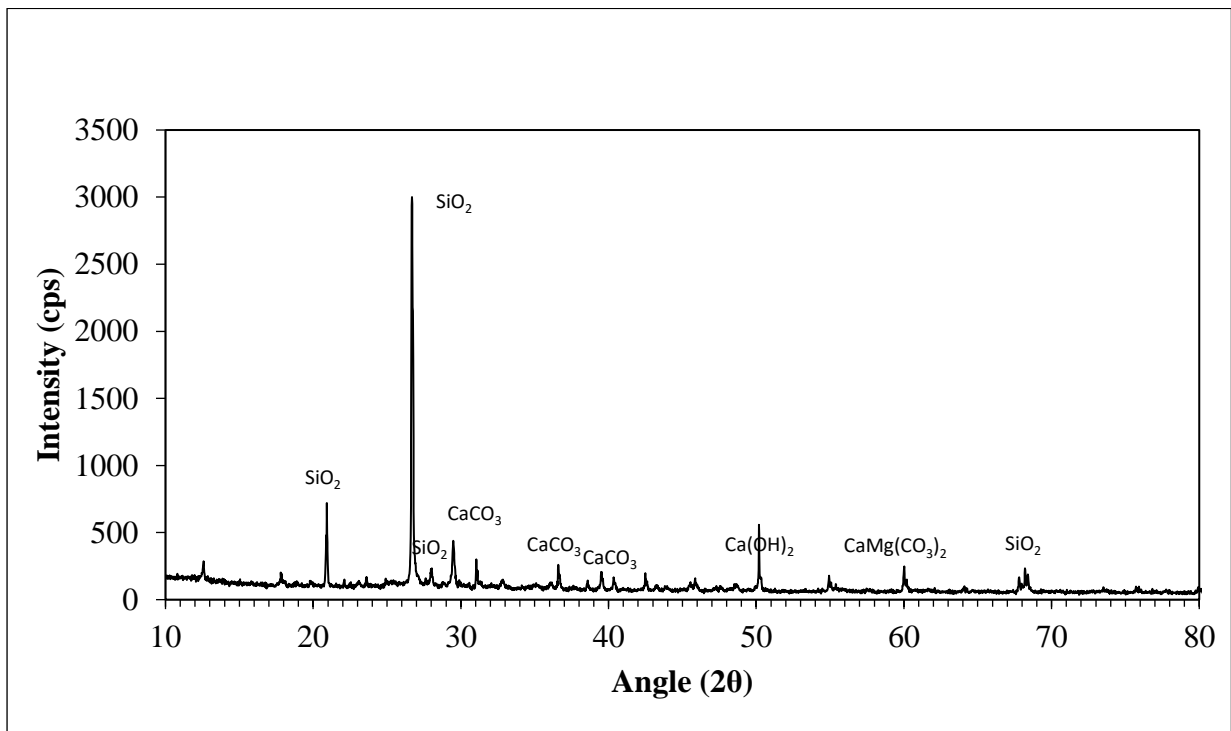


Fig. 4.30(b): XRD image for mix M8 (5MK10RHA) at 365 days

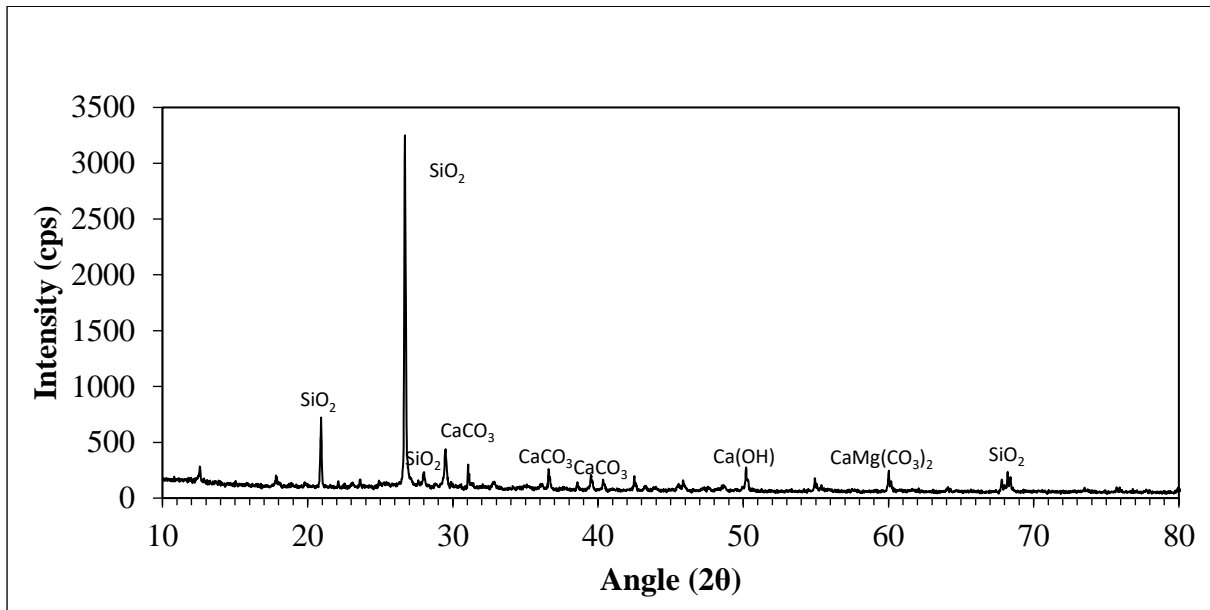


Fig. 4.31(a): XRD image for mix M9 (5MK20RHA) at 28 days

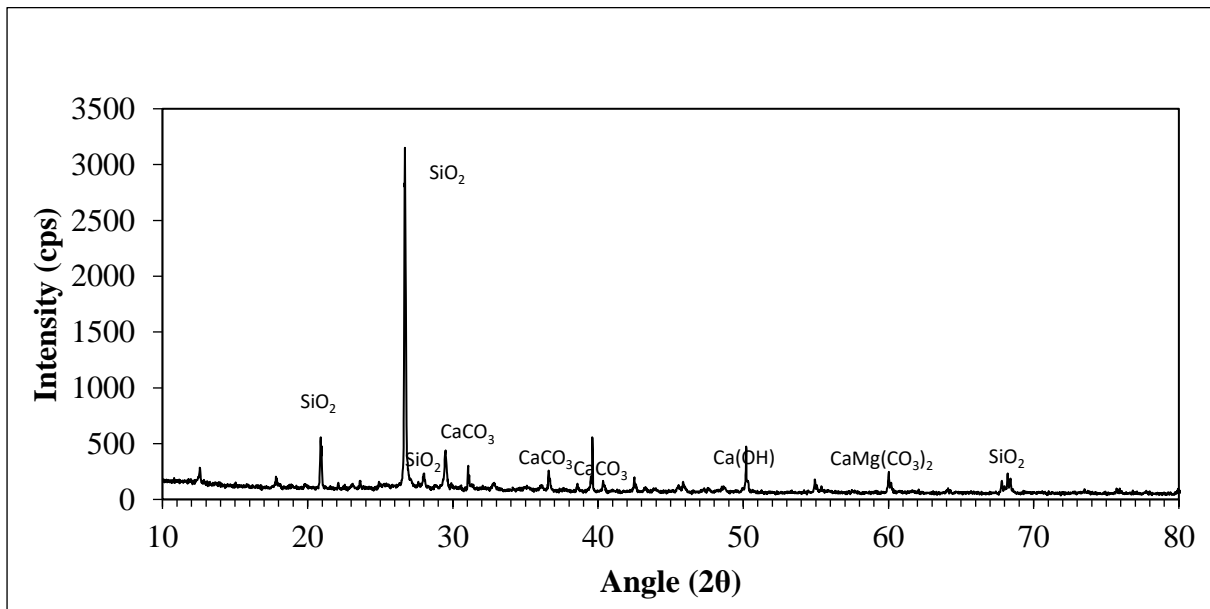


Fig. 4.31(b): XRD image for mix M9 (5MK20RHA) at 365 days

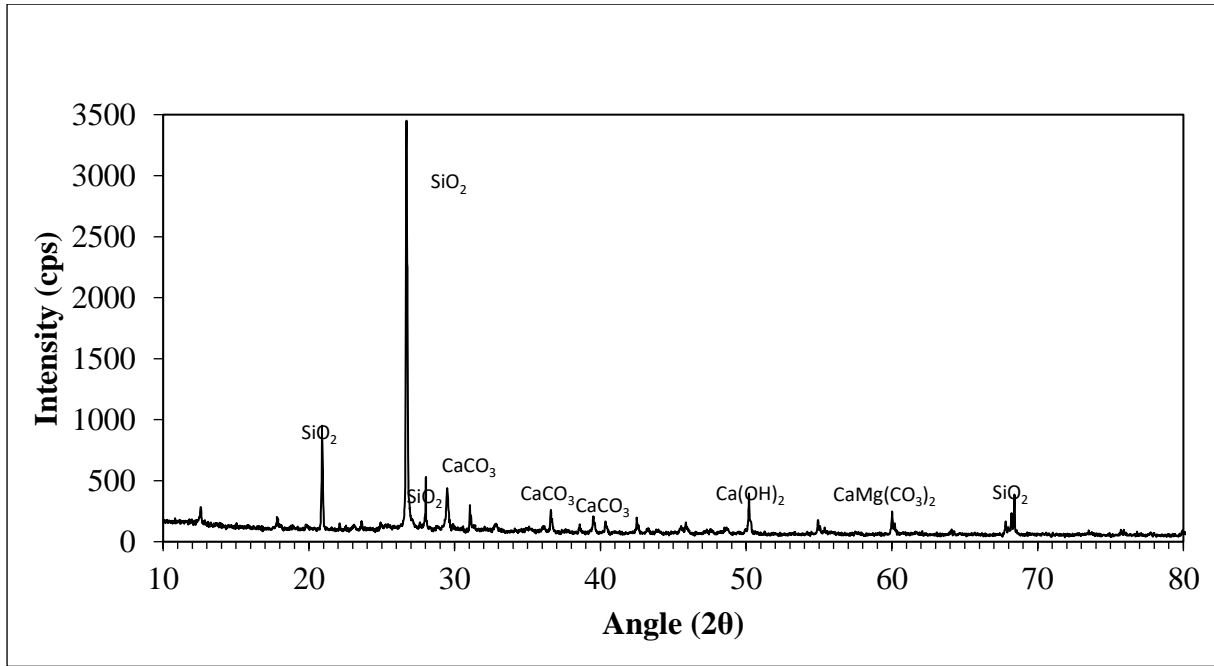


Fig. 4.32(a): XRD image for mix M10 (5MK30RHA) at 28 days

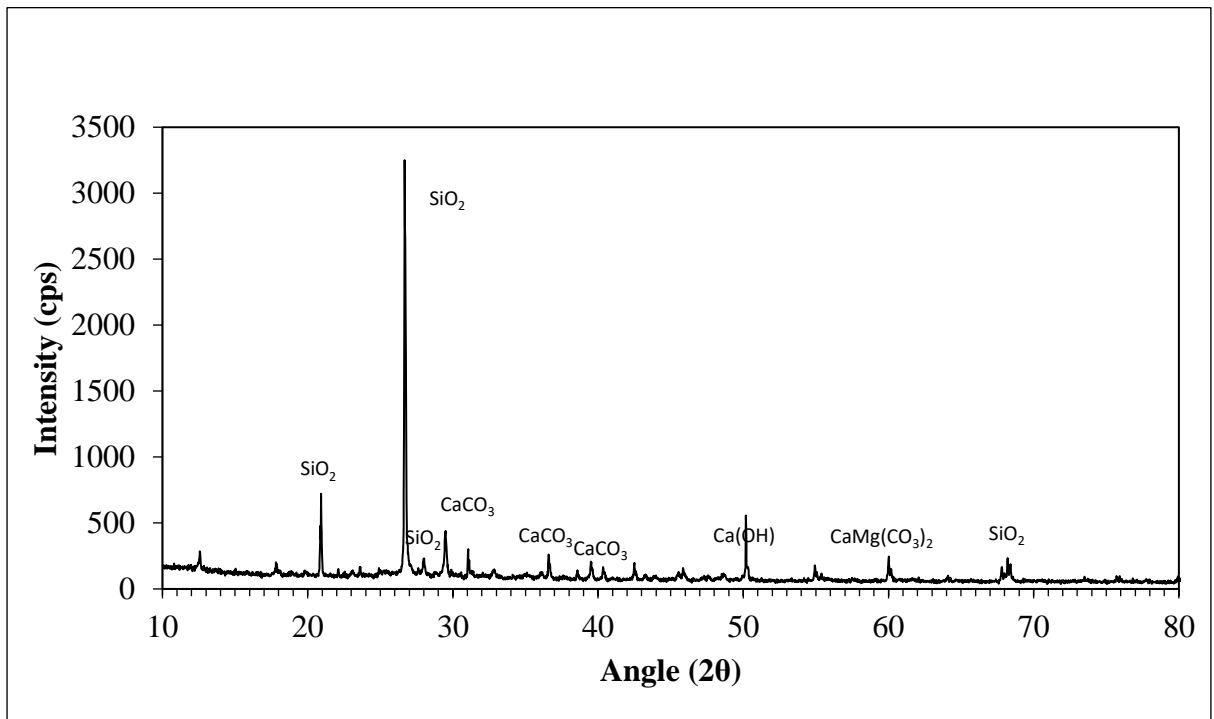


Fig. 4.32(b): XRD image for mix M10 (5MK30RHA) at 365 days

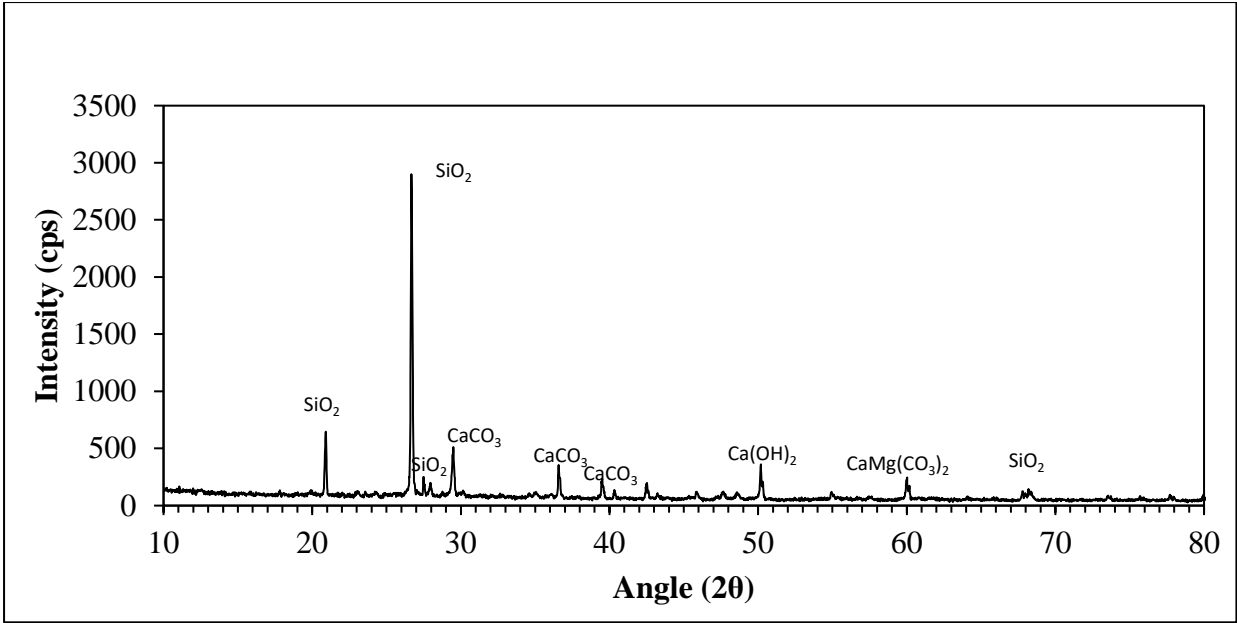


Fig 4.33(a): XRD image for mix M11 (10MK10RHA) at 28 days

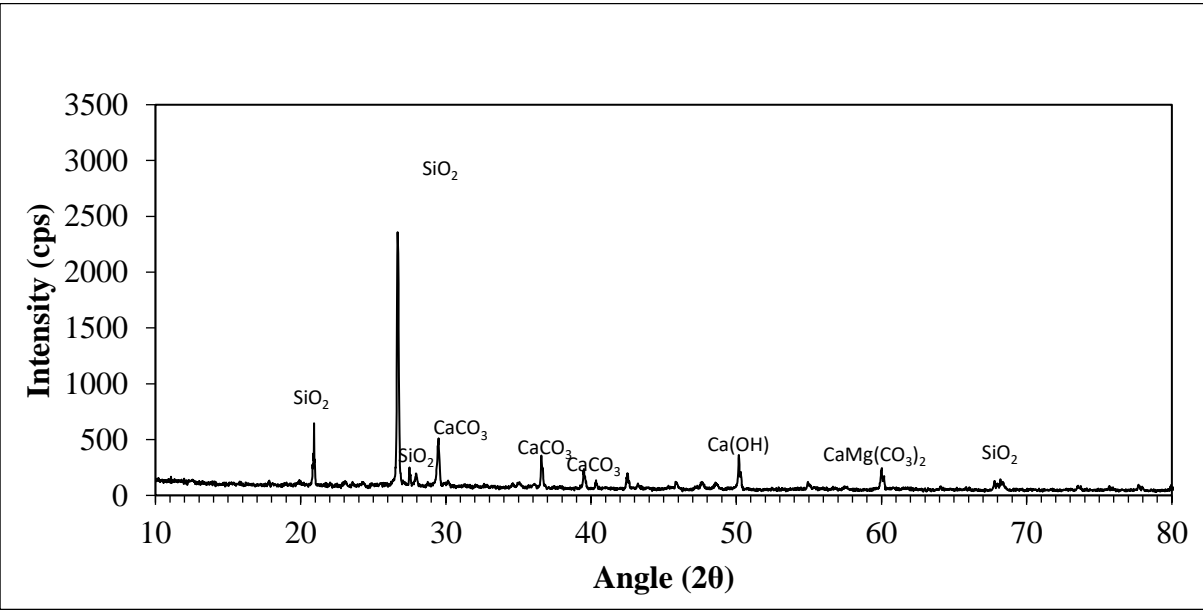


Fig 4.33(b): XRD image for mix M11 (10MK10RHA) at 365 days

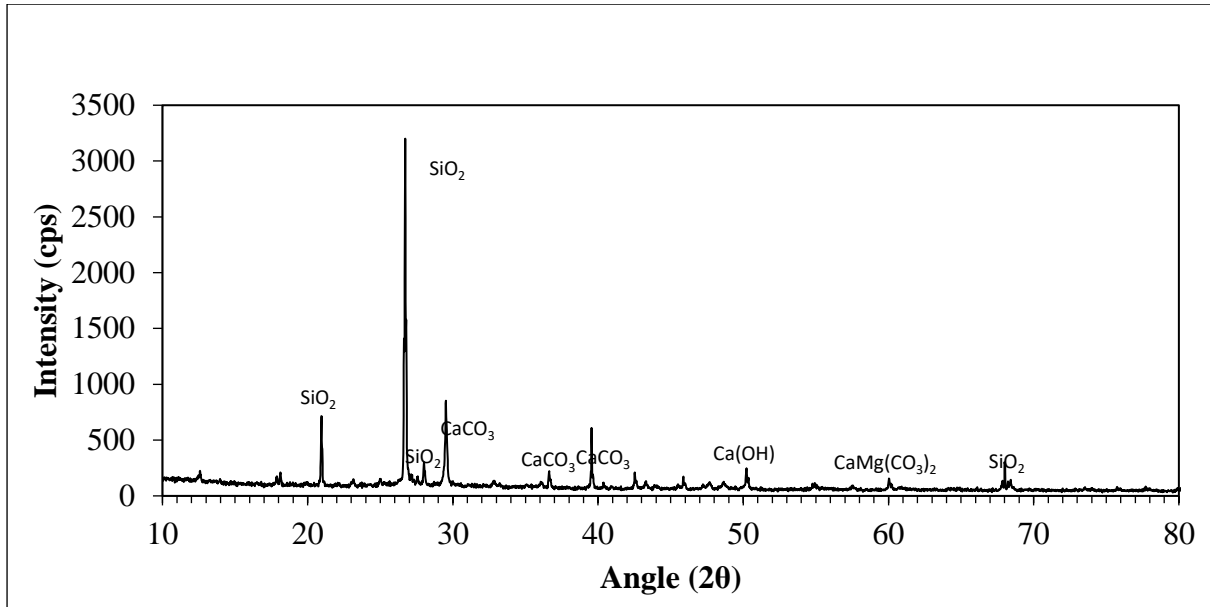


Fig 4.34(a): XRD image for mix M12 (10MK20RHA) at 28 days

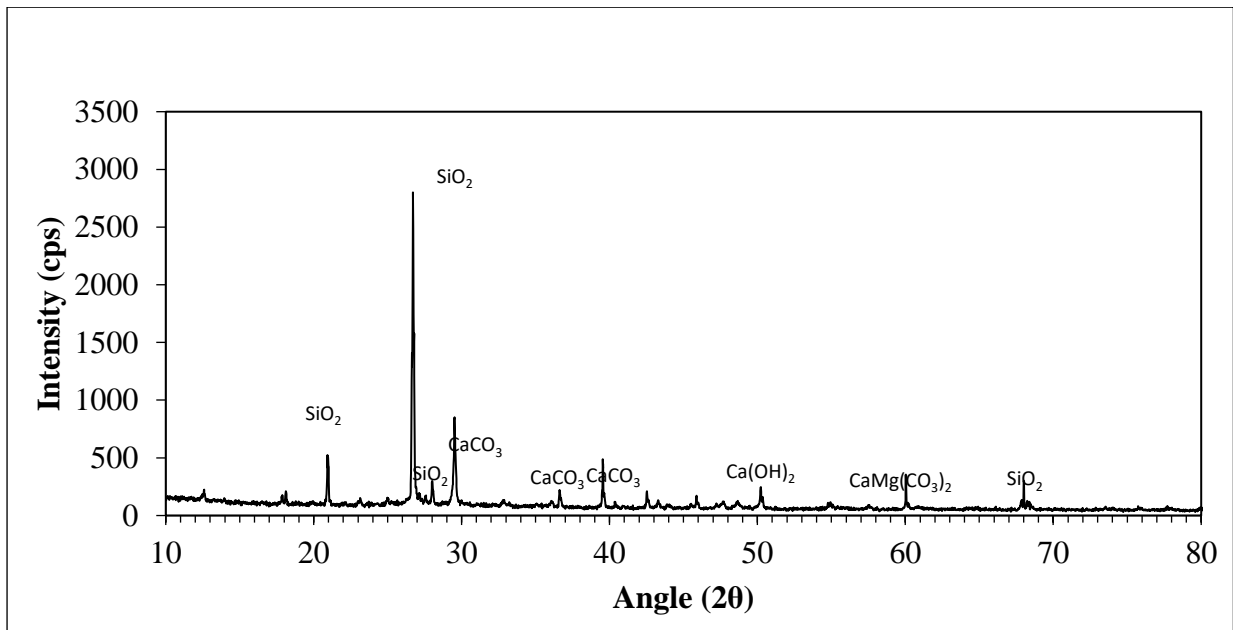


Fig 4.34(b): XRD image for mix M12 (10MK20RHA) at 365 days

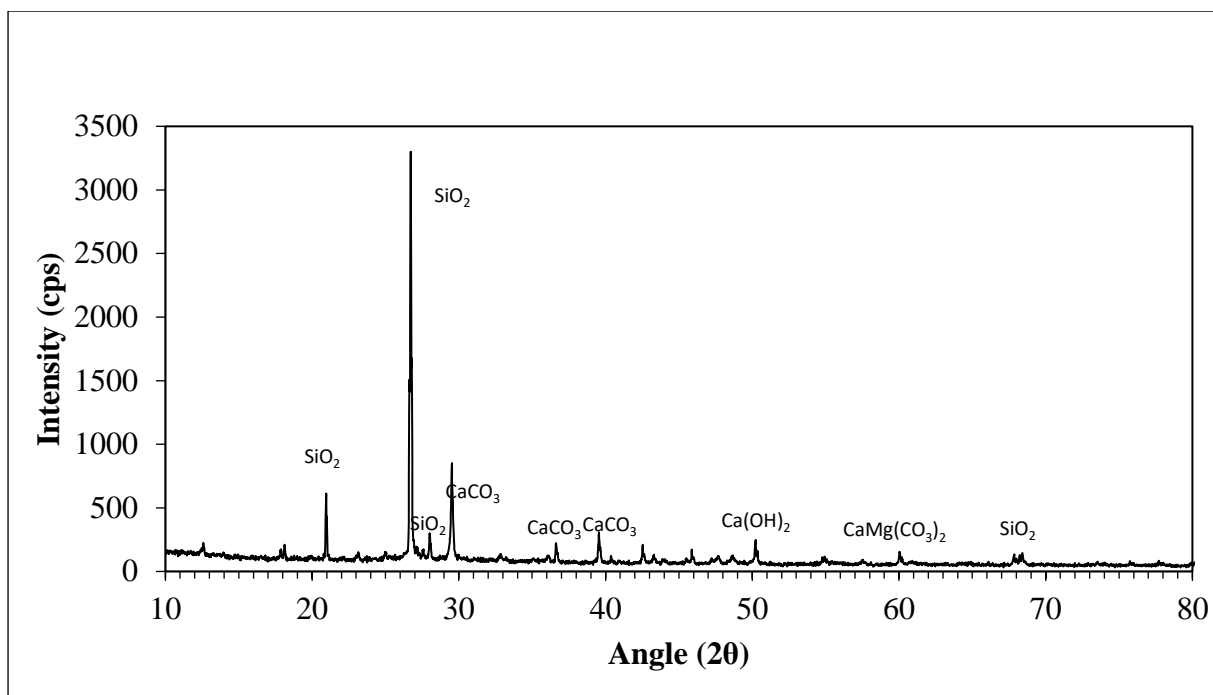


Fig 4.35(a): XRD image for mix M13 (10MK30RHA) at 28 days

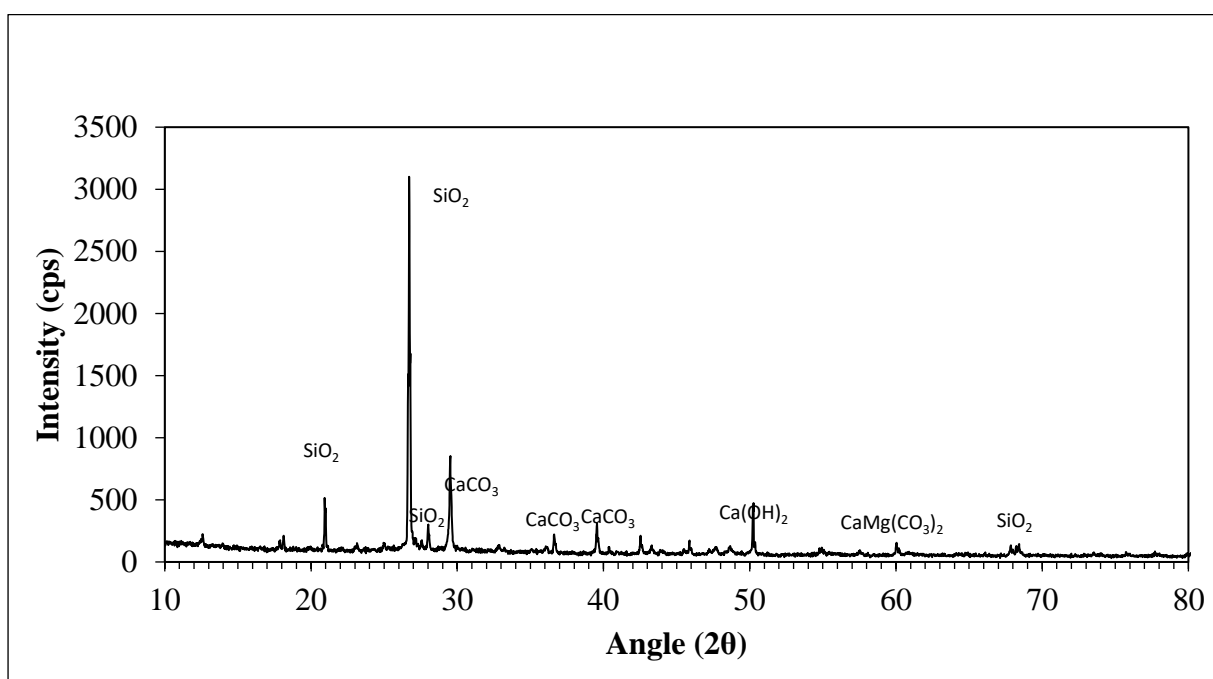


Fig 4.35(b): XRD image for mix M13 (10MK30RHA) at 365 days

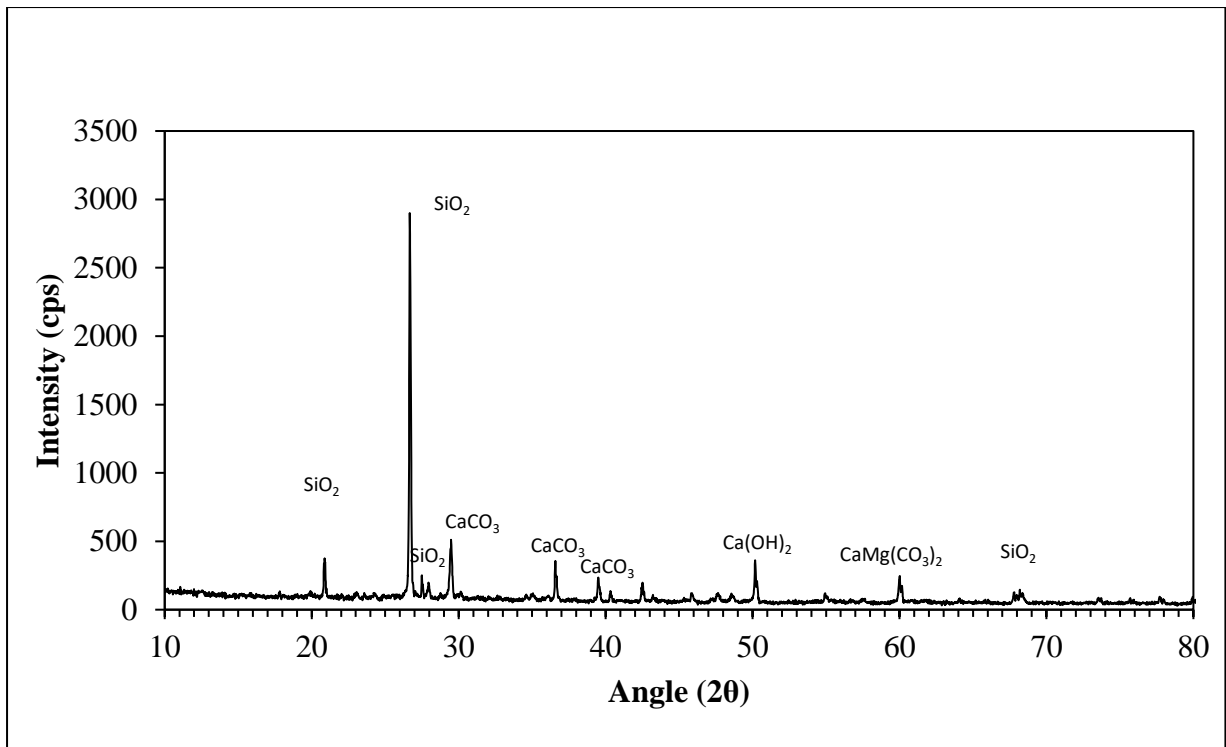


Fig 4.36(a): XRD image for mix M14 (15MK10RHA) at 28 days

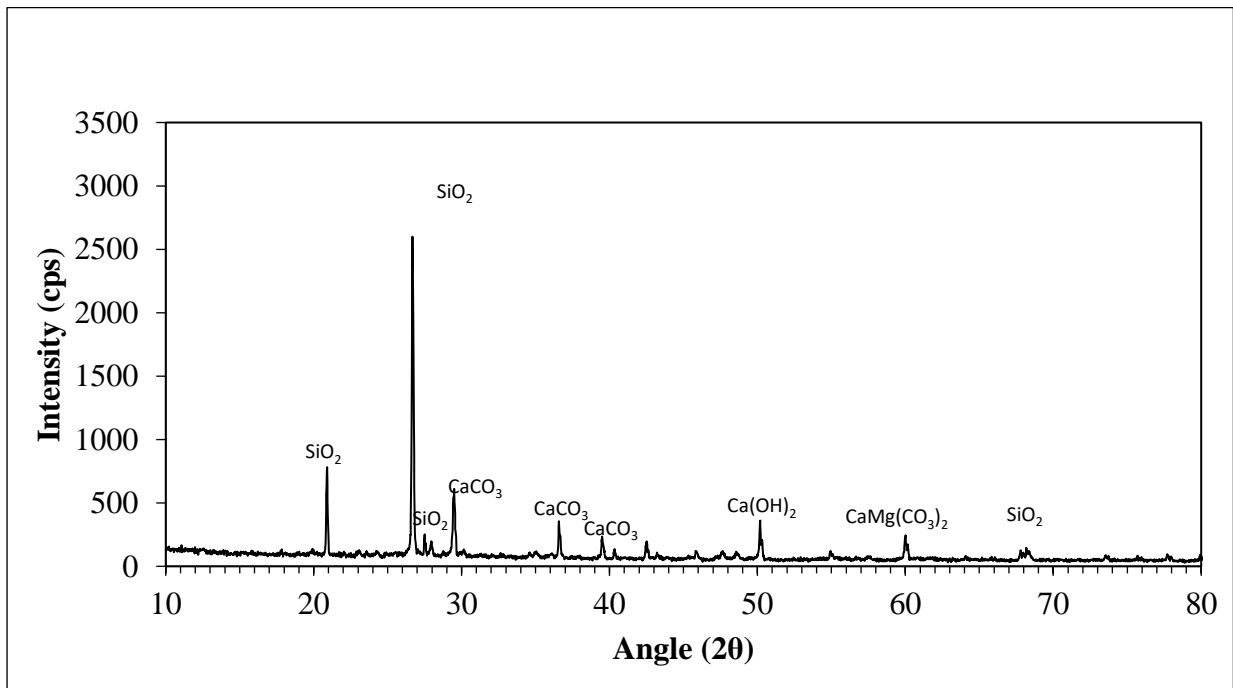


Fig 4.36(b): XRD image for mix M14 (15MK10RHA) at 365 days

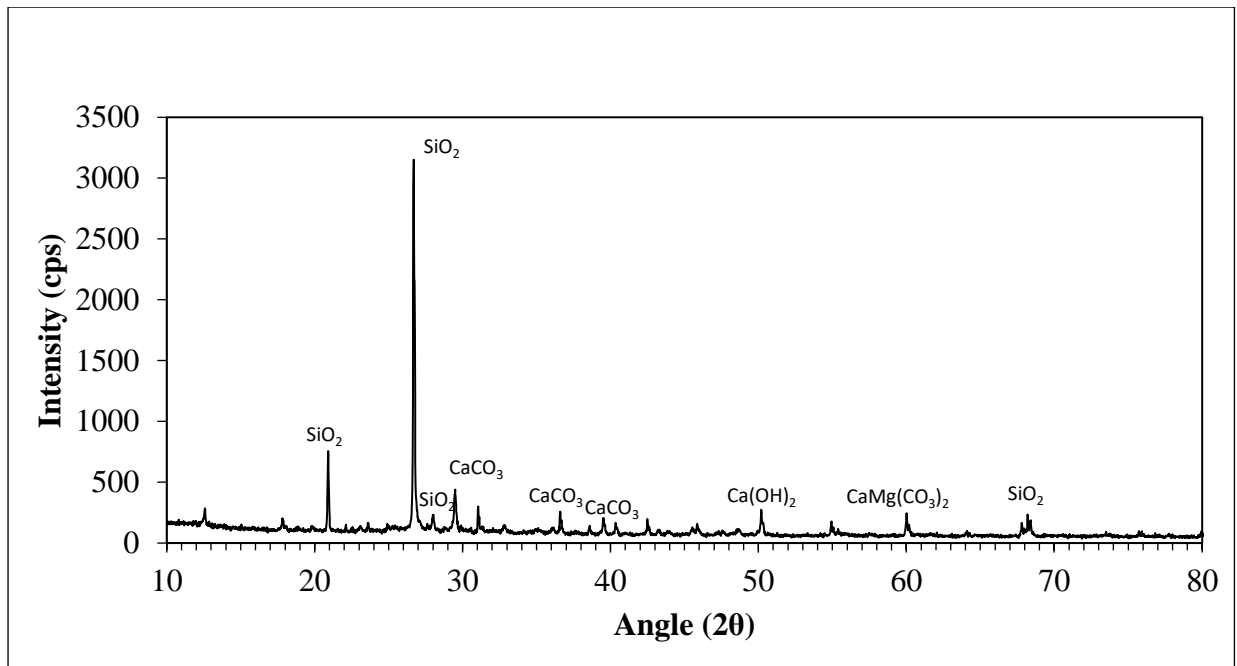


Fig 4.37(a): XRD image for mix M15 (15MK20RHA) at 28 days

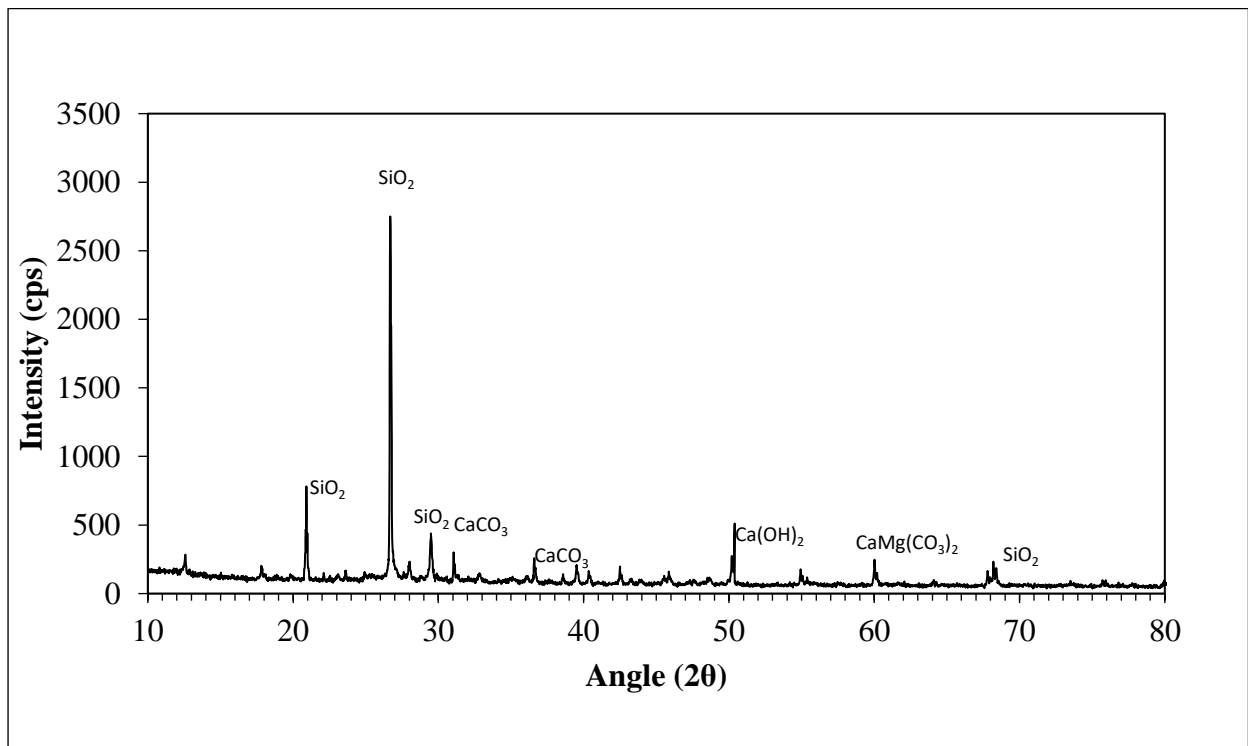


Fig 4.37(b): XRD image for mix M15 (15MK20RHA) at 365 days

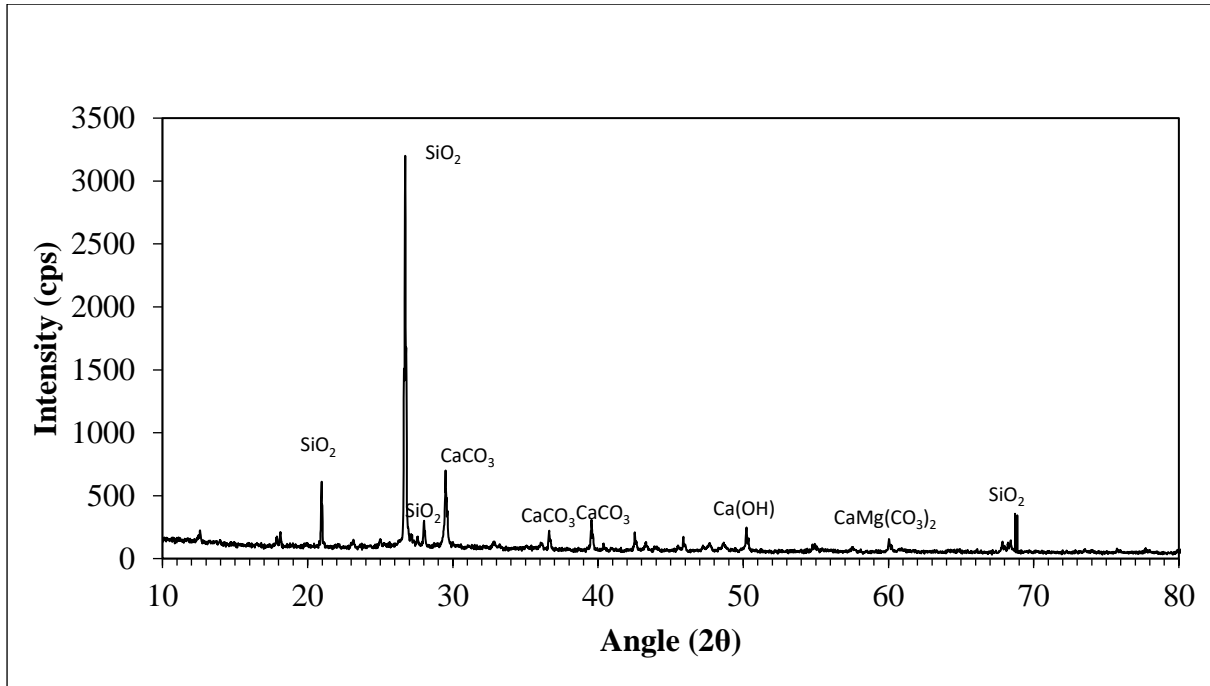


Fig 4.38(a): XRD image for mix M16 (15MK30RHA) at 28 days

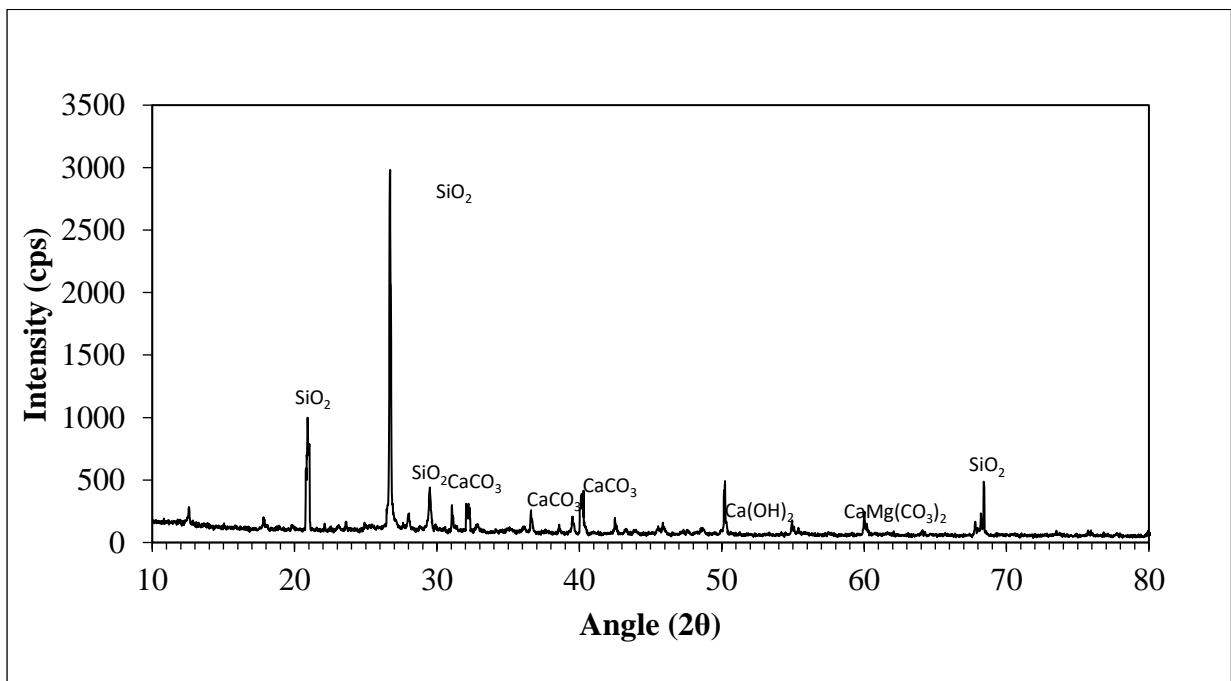


Fig 4.38(b): XRD image for mix M16 (15MK30RHA) at 365 days

As it can be seen from the XRD results, mixes made with use of MK and RHA showed better performances than the mix made without MK or RHA. It is evident from the fact that amount of unused silica for the control mix was more than 3500, which was the main reason behind poor performance of control mix. As MK and RHA were added, the amount of free silica starts decreasing, as it is used in formation of C-S-H gel which further helps in boosting the various properties of mixes. Mix M8 had quantity of unused silica in range of 3000, while mix M11 had 3250, which states that it is used in hydration reaction. But at the same point it can also be observed that increasing percentages of RHA has adverse effect. So it can be stated that use of MK and RHA improved the performance of SCC, but increased percentages of MK and RHA had adverse effect.

4.5 Statistical Analysis of Results

4.5.1 Comparative study of strength and durability properties of SCC mixes using 'ANNOVA' test

All the test results of SCC mixes were statistically analysed and comparatively studied using 'ANNOVA' test.

4.5.1.1 Compressive strength

Statistical analysis of compressive strength results of all ages of all mixes are given in Table 4.12 and 4.13.

Table 4.12: Statistical analysis of compressive strength results

Mix ID	N	Mean	Std. Deviation	Std. Error	95% confidence interval for mean		Minimum	Maximum
					Lower Bound	Upper Bound		
M1	4	40.6850	9.22168	4.61084	26.0113	55.3587	27.53	48.35
M8	4	49.5975	14.77753	7.38877	26.0831	73.1119	28.76	62.10
M9	4	47.5375	15.05974	7.52987	23.5741	71.5009	26.50	60.15
M10	4	47.3550	13.70578	6.85289	25.5460	69.1640	29.30	59.40
M11	4	57.3550	13.70578	6.75289	30.843	77.453	34.70	71.45
M12	4	48.9725	15.28347	7.64174	24.6531	73.2919	28.23	63.50
M13	4	47.0350	16.34467	8.17233	21.0270	73.0430	24.33	61.65
M14	4	55.3700	13.47313	6.73657	33.9312	76.8088	38.10	69.05
M15	4	51.3525	13.02197	6.51098	30.6316	72.0734	34.10	64.45
M16	4	47.8275	11.79798	5.89899	29.0543	66.6007	32.13	59.95
Total	40	48.3088	12.59350	1.99121	44.2812	52.3363	24.33	69.05

Number of samples for each mix were 4, as compressive strength at age of 7, 28, 90 and 365 days. Standard deviation and standard error are also shown in the above table. It can be seen that the mixes made with MK and RHA have higher mean value than the control mix. Also, the lower bound and upper bound limits for 95% confidence interval for mean were also higher for the mixes made with MK and RHA as compared to the control mix. This 95% interval for mean depicts the population forecasting mean. Table 4.13 represents the Ryan-Einot-Gabriel-Welsch range for compressive strength results. It can be seen that all mixes were lying in the same group. Also, control mix has lower value as compared to mixes made with MK and RHA.

Table 4.13: Ryan-Einot-Gabriel-Welsch Range for compressive strength results

Mix ID	N	Subset for alpha = 0.05
		1
Control Mix (M1)	4	40.6850
M13	4	47.0350
M10	4	47.3550
M11	4	47.3550
M9	4	47.5375
M16	4	47.8275
M12	4	48.9725
M8	4	49.5975
M15	4	51.3525
M14	4	55.3700
Sig.		.879

4.5.1.2 Splitting tensile strength

Statistical analysis for results of splitting tensile strength test of all SCC mixes is given in Table 4.14. Number of samples for each mix were 4, as splitting tensile strength at age of 7, 28, 90 and 365 days. The minimum and maximum value for the control mix comes to be 1.82 and 3.56, which is lower than all other mixes made with the use of MK and RHA. Mix M11 has minimum and maximum value of 2.74 and 5.96, which is higher than all other mixes. The lower bound and upper bound limits for 95% confidence interval for mean were also higher for the mixes made with MK and RHA as compared to the control mix. Table 4.15 represents the Ryan-Einot-Gabriel-Welsch Range for splitting tensile strength results. It can be seen that all mixes were lying in the same group. Also, control mix has lower value as compared to mixes made with MK and RHA.

Table 4.14: Statistical study of splitting tensile strength results of all SCC mixes

Mix ID	N	Mean	Std. Deviation	Std. Error	95% confidence interval for mean		Minimum	Maximum
					Lower Bound	Upper Bound		
M1	4	2.83	0.77	0.38	1.60	4.05	1.82	3.56
M8	4	3.74	1.05	0.52	2.07	5.41	2.36	4.83
M9	4	4.49	1.30	0.65	2.41	6.56	2.72	5.75
M10	4	3.55	0.93	0.46	2.07	5.03	2.31	4.45
M11	4	4.56	1.37	0.68	2.39	6.74	2.74	5.96
M12	4	4.37	1.31	0.65	2.28	6.45	2.70	5.71
M13	4	4.06	1.04	0.52	2.40	5.71	2.63	5.01
M14	4	4.46	1.51	0.76	2.05	6.86	2.39	5.95
M15	4	4.20	1.54	0.77	1.75	6.65	2.20	5.75
M16	4	3.80	1.23	0.62	1.84	5.76	2.14	4.96
Total	40	4.00	1.20	0.19	3.62	4.39	1.82	5.96

Table 4.15: Ryan-Einot-Gabriel-Welsch range for splitting tensile strength results

SAMPLE	N	Subset for alpha = 0.05
		1
Control Mix (M1)	4	2.8275
M10	4	3.5500
M8	4	3.7425
M16	4	3.7975
M13	4	4.0550
M15	4	4.1975
M12	4	4.3650
M14	4	4.4550
M9	4	4.4850
M11	4	4.5625
Sig.		.607

4.5.1.3 Water absorption

The results of statistical analysis of water absorption test results are given in Table 4.16 and Table 4.17. Number of samples for each mix were 3, as water absorption results at age of 28, 90 and 365 days. The mean for control mix comes to be highest among all the mixes, which indicates its poor performance. It has total mean value of 6.7967, which was greater than the mean values of mixes made with MK and RHA. Table 4.16 represents the Ryan-Einot-Gabriel-Welsch range for water absorption test results. It can be seen that mixes were lying in the different groups, which mean they are significantly different from each other in performance. Mixes are scattered in three groups. Also, the control mix has the highest value as compared to mixes made with MK and RHA.

Table 4.16: Ryan-Einot-Gabriel-Welsch Range

Sample	N	Subset			
		1	2	3	
Ryan-Einot-Gabriel-Welsch Range	M8	6	3.1133		
	M9	6	3.7317		
	M10	6	4.2533	4.2533	
	M12	6	4.5967	4.5967	4.5967
	M11	6	4.7967	4.7967	4.7967
	M13	6	5.0617	5.0617	5.0617
	M14	6	5.1617	5.1617	5.1617
	M15	6		6.1817	6.1817
	M16	6		6.2333	6.2333
	Control Mix	6			6.7967
Sig.			.091	.114	.053

Table 4.17: Statical analysis of water absorption test results

Sample		Mean	Std. Deviation	N
Control Mix	Absorption after immersion (%)	6.6200	.73668	3
	Absorption after immersion and boiling (%)	6.9733	.97352	3
	Total	6.7967	.79601	6
M8	Absorption after immersion (%)	3.1967	.99551	3
	Absorption after immersion and boiling (%)	3.0300	1.19411	3
	Total	3.1133	.98747	6
M9	Absorption after immersion (%)	3.4867	1.19249	3
	Absorption after immersion and boiling (%)	3.9767	1.86827	3
	Total	3.7317	1.42724	6

M10	Absorption after immersion (%)	3.8467	1.50816	3
	Absorption after immersion and boiling (%)	4.6600	2.49375	3
	Total	4.2533	1.89626	6
M11	Absorption after immersion (%)	4.3100	.51798	3
	Absorption after immersion and boiling (%)	5.2833	1.86033	3
	Total	4.7967	1.33262	6
M12	Absorption after immersion (%)	4.4600	.56666	3
	Absorption after immersion and boiling (%)	4.7333	.07234	3
	Total	4.5967	.39108	6
M13	Absorption after immersion (%)	4.9067	.31134	3
	Absorption after immersion and boiling (%)	5.2167	1.25349	3
	Total	5.0617	.83432	6
M14	Absorption after immersion (%)	5.1167	.92241	3
	Absorption after immersion and boiling (%)	5.2067	.40501	3
	Total	5.1617	.63904	6
M15	Absorption after immersion (%)	5.9533	.61809	3
	Absorption after immersion and boiling (%)	6.4100	1.52539	3
	Total	6.1817	1.07056	6
M16	Absorption after immersion (%)	6.1967	.31660	3
	Absorption after immersion and boiling (%)	6.2700	1.07685	3
	Total	6.2333	.71102	6
Total	Absorption after immersion (%)	4.8093	1.32992	30
	Absorption after immersion and boiling (%)	5.1760	1.64999	30
	Total	4.9927	1.49723	60

4.5.1.4 Sulphate resistance

The results of statistical analysis of sulphate resistance test results are given in Table 4.18 and Table 4.19. Number of samples for each mix were 3, as sulphate resistance results at age of 28, 90 and 365 days. The mean for control mix comes to be lowest among all the mixes, which indicates its poor performance. The 95% confidence interval for mean of control mix was also lowest in terms of both lower bound and upper bound. Also, mixes are lying in different groups which mean they are significantly different.

Table 4.18: Statistical analysis of sulphate resistance results

Mix ID	N	Mean	Std. Deviation	Std. Error	95% Confidence Interval for Mean		Minimum	Maximum
					Lower Bound	Upper Bound		
M1	3	37.7767	2.08522	1.20390	32.5967	42.9566	35.53	39.65
M8	3	52.7167	5.71759	3.30105	38.5134	66.9199	46.50	57.75
M9	3	50.9667	6.79877	3.92527	34.0776	67.8558	43.50	56.80
M10	3	49.8600	7.06762	4.08049	32.3031	67.4169	42.33	56.35
M11	3	60.2067	8.72414	5.03688	38.5347	81.8786	50.76	67.96
M12	3	53.0600	7.16148	4.13468	35.2699	70.8501	45.60	59.88
M13	3	52.3267	6.42920	3.71190	36.3556	68.2977	45.63	58.45
M14	3	57.9167	7.66328	4.42440	38.8800	76.9533	49.85	65.10
M15	3	51.3900	6.54505	3.77879	35.1312	67.6488	44.86	57.95
M16	3	49.9167	6.09146	3.51690	34.7847	65.0487	44.10	56.25
Total	30	51.6137	7.93734	1.44915	48.6498	54.5775	35.53	67.96

Table 4.19: Ryan-Einot-Gabriel-Welsch Range

Sample mix	N	Subset for alpha = 0.05	
		1	2
Control Mix	3	37.7767	
M10	3	49.8600	49.8600
M16	3	49.9167	49.9167
M9	3	50.9667	50.9667
M15	3	51.3900	51.3900
M13	3	52.3267	52.3267
M8	3	52.7167	52.7167
M12	3	53.0600	53.0600
M14	3		57.9167
M11	3		60.2067
Sig.		.178	.616

4.5.1.5 Rapid chloride permeability

The results of statistical analysis of RCPT results are given in Table 4.20 and Table 4.21. Number of samples for each mix were 3, as RCPT results at age of 28, 90 and 365 days. The mean for control mix comes to be highest among all the mixes, which indicates its poor performance. The 95% confidence interval for mean of control mix was also highest in terms of both lower bound and upper bound. This means control mix had lowest chloride hesitance among all the tested mixes. Also, mixes are lying in same group and control mix had the highest value.

Table 4.20: Statistical analysis of RCPT results

Mix ID	N	Mean	Std. Deviation	Std. Error	95% Confidence Interval for Mean		Minimum	Maximum
					Lower Bound	Upper Bound		
Control Mix	3	613.6667	763.25968	440.66818	-1282.38	2509.71	171.00	1495.00
M8	3	460.0000	704.08735	406.50502	-1289.05	2209.05	50.00	1273.00
M9	3	416.6667	639.41718	369.16768	-1171.73	2005.07	46.00	1155.00
M10	3	405.6667	614.30394	354.66855	-1120.35	1931.68	49.00	1115.00
M11	3	338.3333	512.41812	295.84474	-934.58	1611.25	38.00	930.00
M12	3	318.6667	481.79906	278.16682	-878.19	1515.52	40.00	875.00
M13	3	337.0000	509.22392	294.00057	-927.98	1601.98	42.00	925.00
M14	3	304.6667	454.96190	262.67238	-825.52	1434.85	39.00	830.00
M15	3	289.0000	433.87902	250.50017	-788.82	1366.82	38.00	790.00
M16	3	312.0000	461.59831	266.50391	-834.67	1458.67	43.00	845.00
Total	30	379.5667	481.39500	87.89030	199.81	559.32	38.00	1495.00

Table 4.21: Ryan-Einot-Gabriel-Welsch Range

Sample mix	N	Subset for alpha = 0.05
		1
M15	3	289.0000
M14	3	304.6667
M16	3	312.0000
M12	3	318.6667
M13	3	337.0000
M11	3	338.3333
M10	3	405.6667
M9	3	416.6667
M8	3	460.0000
Control Mix	3	613.6667
Sig.		.999

4.5.2 Correlation analysis of SCC properties

4.5.2.1 Relationship between compressive strength and splitting tensile strength

The relationship between compressive strength and splitting tensile strength of all the mixes at the age of 7, 28, 90 and 365 days is depicted in Fig. 4.39. The value of R^2 at 7 days come to be 0.0121, at 28 days 0.6439, at 90 days 0.6009 and at 365 days 0.7729. As there is increase in the age of specimens, the value of R^2 gets near to 1. The overall value of R^2 comes to be 0.8867, which depicts a strong relationship.

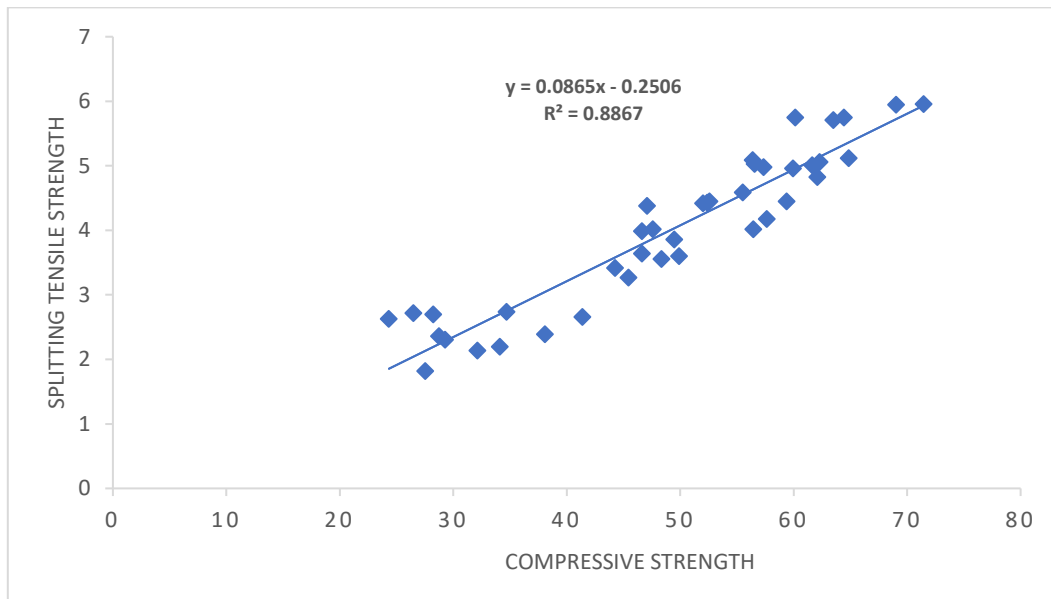


Fig. 3.39: Relation between compressive strength and splitting tensile strength of all SCC mixes at all ages

Table 4.22: Relationship between compressive strength and splitting tensile strength of all SCC mixes at respective ages

Age	R^2 value	Y value
7 days	0.0121	$-0.0078x + 2.6372$
28 days	0.6439	$0.1298x - 2.3561$
90 days	0.6009	$0.0909x - 0.5597$
365 days	0.7729	$0.1104x - 1.651$

4.5.2.2 Relationship between compressive strength and water absorption

The relationship between compressive strength and water absorption (after immersion) of all the mixes at the age of 28, 90 and 365 days is depicted in Fig. 4.40. The value of R^2 comes to be 0.0004 at 28 days, 0.4685 at 90 days and 0.1963 at 365 days. As there is increase in the age of specimens, the value of R^2 gets increases but again at 365 days value decreases. The overall value of R^2 comes to be 0.0003.

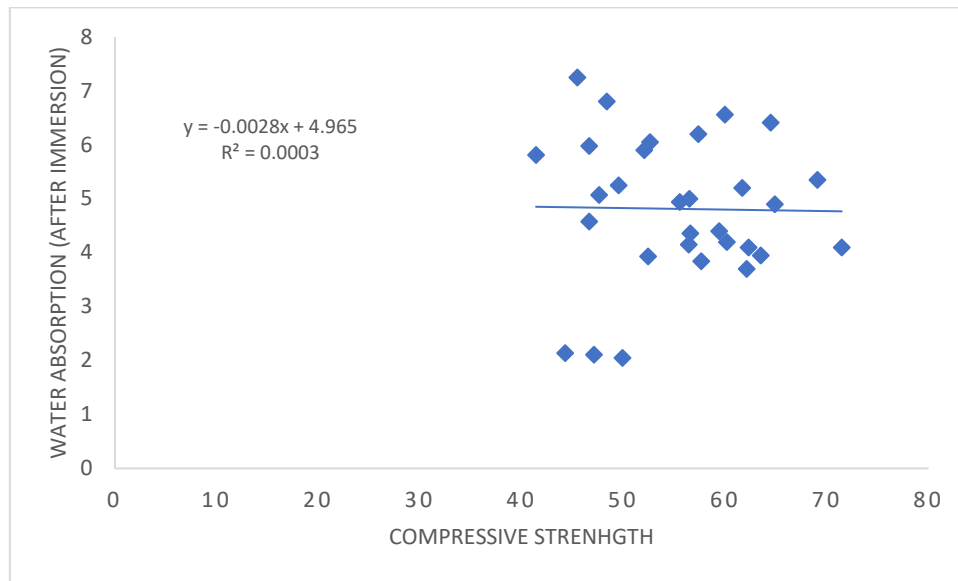


Fig. 4.40: Relation between compressive strength and water absorption (after immersion) of all SCC mixes at all ages

Table 4.23: Relation between compressive strength and water absorption (after immersion) of all SCC mixes at respective ages

Age	R^2 value	Y value
28 days	0.0004	-0.0096x + 4.7386
90 days	0.4685	-0.1444x + 13.241
365 days	0.1963	-0.0836x + 10.249

The relation between compressive strength and water absorption (after immersion and boiling) of all the mixes at the age of 28, 90 and 365 days is depicted in Fig. 4.41. The value of R^2 comes to be 0.0092 at 28 days, 0.1645 at 90 days and 0.1003 at 365 days. As there is increase in the age of specimens, the value of R^2 gets increases but again at 365 days value decreases. The overall value of R^2 comes to be 0.0611.

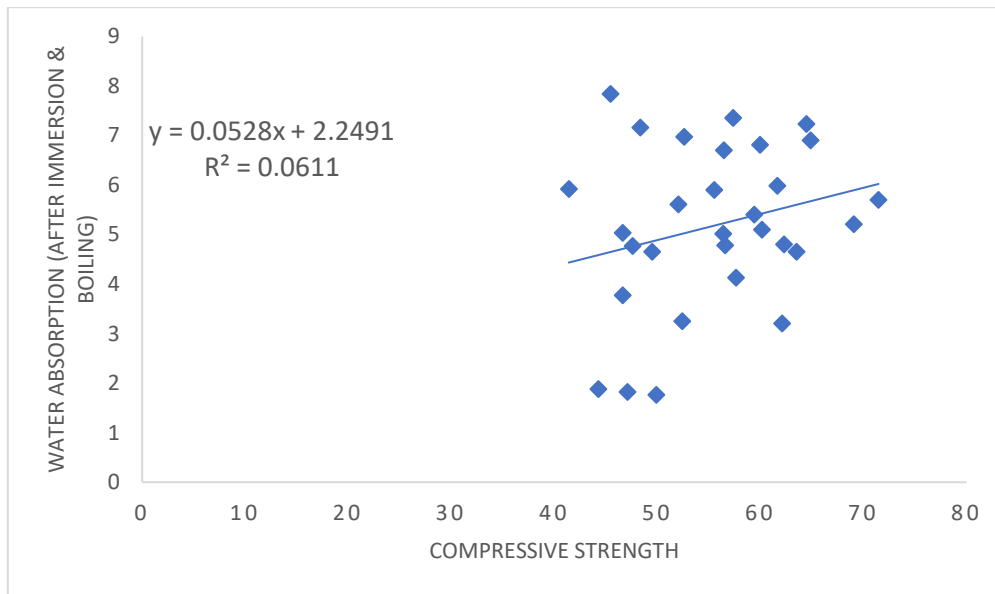


Fig. 4.41: Relation between compressive strength and water absorption (after immersion and boiling) of all SCC mixes at all ages

Table 4.24: Relation between compressive strength and water absorption (after immersion and boiling) of all SCC mixes at respective ages

Age	R^2 value	Y value
28 days	0.0092	$-0.0453x + 6.0093$
90 days	0.1645	$-0.1002x + 11.698$
365 days	0.1003	$-0.063x + 9.5522$

4.5.2.3 Relationship between compressive strength and porosity

The relationship between compressive strength and porosity of all the mixes at the age of 28, 90 and 365 days is depicted in Fig. 4.42. The value of R^2 comes to be 0.56 at 28 days, 0.1131 at 90 days and 0.0489 at 365 days. As there is increase in the age of specimens, the value of R^2 gets decreases. The overall value of R^2 comes to be 0.0463.

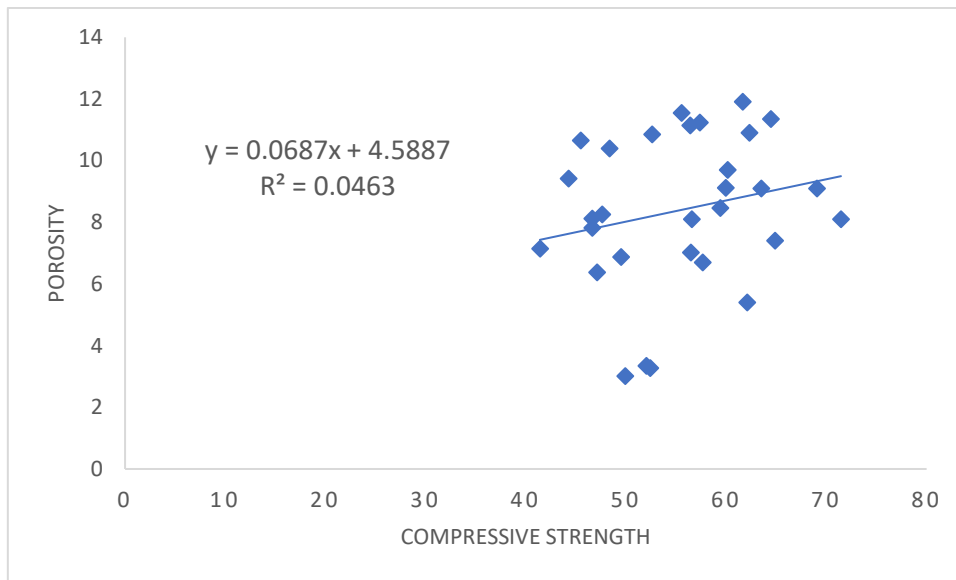


Fig. 4.42: Relationship between compressive strength and porosity of all SCC mixes at all ages

Table 4.25: Relationship between compressive strength and porosity of all SCC mixes at respective ages

Age	R^2 value	Y value
28 days	0.56	$-0.5155x + 30.981$
90 days	0.1131	$-0.1281x + 16.796$
365 days	0.0489	$-0.0647x + 13.277$

4.5.2.4 Relationship between compressive strength and sulphate resistance

The relationship between compressive strength and sulphate resistance of all the mixes at the age of 28, 90 and 365 days is depicted in Fig. 4.43. The value of R^2 comes to be 0.8932 at 28 days, 0.9572 at 90 days and 0.961 at 365 days. As there is increase in the age of specimens, the value of R^2 gets increases and is reaching near to 1. The overall value of R^2 comes to be 0.9534.

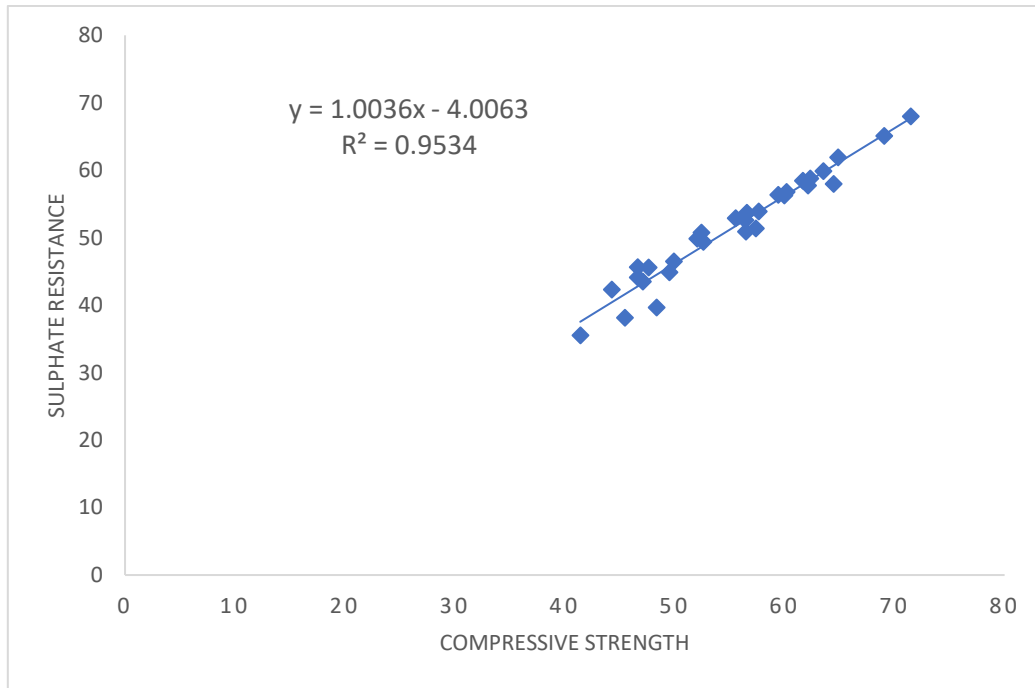


Fig. 4.43: Relationship between compressive strength and sulphate resistance of all SCC mixes at all ages

Table 4.26: Relationship between compressive strength and sulphate resistance of all SCC mixes at respective ages

Age	R^2 value	Y value
28 days	0.8932	$1.1753x - 11.25$
90 days	0.9572	$1.1741x - 13.994$
365 days	0.961	$1.17x - 14.934$

4.5.2.5 Relationship between compressive strength and RCPT

The relationship between compressive strength and RCPT of all the mixes at the age of 28, 90 and 365 days is depicted in Fig. 4.44. The value of R^2 comes to be 0.3609 at 28 days, 0.5551 at 90 days and 0.6642 at 365 days. As there is increase in the age of specimens, the value of R^2 gets increases and is approaching near to 1. This can be also be seen from RCPT results, where number of coulombs passed from all the SCC mixes are decreasing with the curing age. The overall value of R^2 comes to be 0.5747.

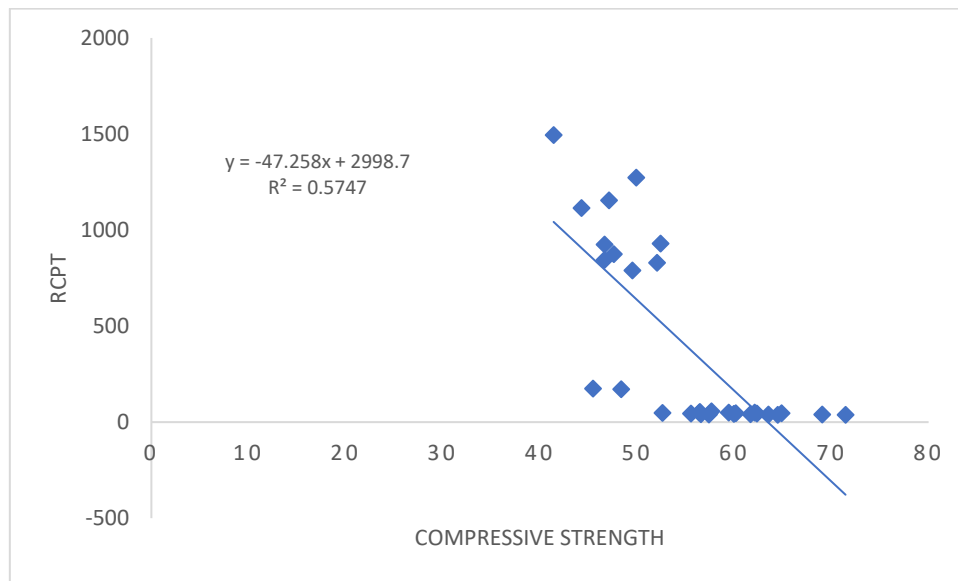


Fig. 4.44: Relationship between compressive strength and RCPT of all SCC mixes at all ages

Table 4.27: Relationship between compressive strength and RCPT of all SCC mixes at respective ages

Age	R^2 value	Y value
28 days	0.3609	$-40.759x + 2969.3$
90 days	0.5551	$-5.8464x + 390.22$
365 days	0.6642	$-5.3437x + 386.94$

4.5.2.6 Relationship between water absorption and porosity

The relationship between water absorption (after immersion and boiling) and porosity of all the mixes at the age of 28, 90 and 365 days is depicted in Fig. 4.45. The value of R^2 comes to be 0.0094 at 28 days, 0.0589 at 90 days and 0.5704 at 365 days. As there is increase in the age of specimens, the value of R^2 gets increases and is approaching near to 1. The overall value of R^2 comes to be 0.3053.

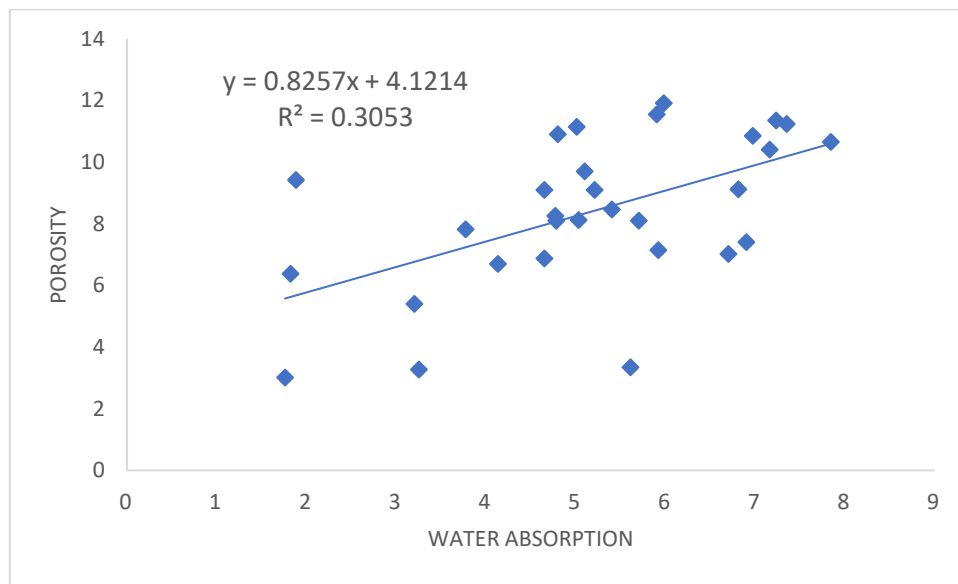


Fig. 4.45: Relationship between water absorption (after immersion and boiling) and porosity of all SCC mixes at all ages

Table 4.28: Relationship between water absorption (after immersion and boiling) and porosity of all SCC mixes at respective ages

Age	R^2 value	Y value
28 days	0.0094	$0.1413x + 5.8227$
90 days	0.0589	$0.3746x + 7.2931$
365 days	0.5704	$1.1109x + 2.9944$

4.5.2.7 Relationship between water absorption and sulphate resistance

The relationship between water absorption (after immersion and boiling) and sulphate resistance of all the mixes at the age of 28, 90 and 365 days is depicted in Fig. 4.45. The value of R^2 comes to be 0.0274 at 28 days, 0.2397 at 90 days and 0.1545 at 365 days. As there was increase in the age of specimens, the value of R^2 gets increases but at 365 days value again decreases. The overall value of R^2 comes to be 0.0149.

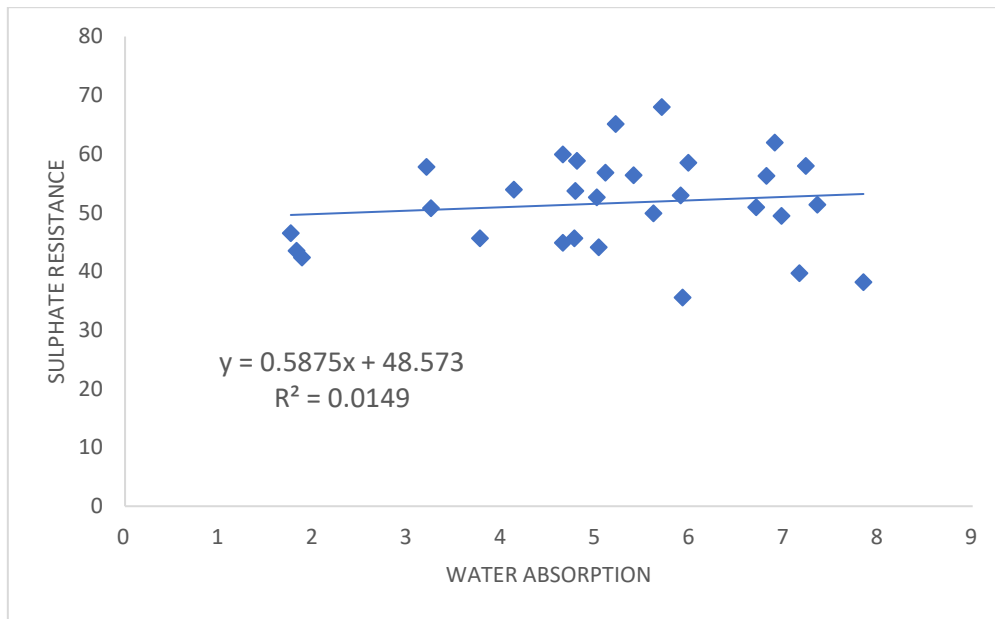


Fig. 4.46: Relationship between water absorption (after immersion and boiling) and sulphate resistance of all SCC mixes at all ages

Table 4.29: Relationship between water absorption (after immersion and boiling) and sulphate resistance of all SCC mixes at respective ages

Age	R^2 value	Y value
28 days	0.0274	$-0.4362x + 46.544$
90 days	0.2397	$-2.3797x + 66.73$
365 days	0.1545	$-2.357x + 70.917$

4.5.2.8 Relationship between water absorption and RCPT

The relationship between water absorption and rapid chloride permeability test of all the mixes at the age of 28, 90 and 365 days is depicted in Fig. 4.47. The value of R^2 comes to be 0.0591 at 28 days, 0.2063 at 90 days and 0.14 at 365 days. As there was increase in the age of specimens, the value of R^2 gets increases but at 365 days value again decreases. The overall value of R^2 comes to be 0.331.

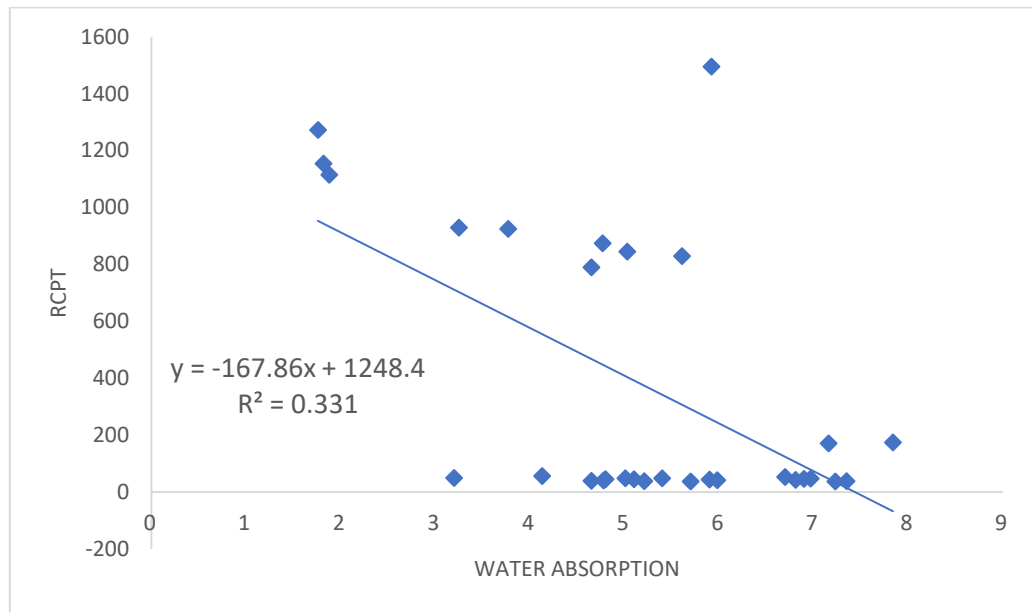


Fig. 4.47: Relationship between water absorption and RCPT of all SCC mixes at all ages

Table 4.30: Relationship between water absorption and RCPT of all SCC mixes at respective ages

Age	R^2 value	Y value
28 days	0.0591	$-34.929x + 1157.6$
90 days	0.2063	$14.435x - 27.361$
365 days	0.14	$12.325x - 13.963$

4.5.2.9 Relationship between sulphate resistance and RCPT

The relationship between sulphate resistance (in terms of strength loss) and rapid chloride permeability test of all the mixes at the age of 28, 90 and 365 days is depicted in Fig. 4.48. The value of R^2 comes to be 0.4707 at 28 days, 0.636 at 90 days and 0.7758 at 365 days. As there was increase in the age of specimens, the value of R^2 gets increases and is reaching close to value of 1. The overall value of R^2 comes to be 0.4638.

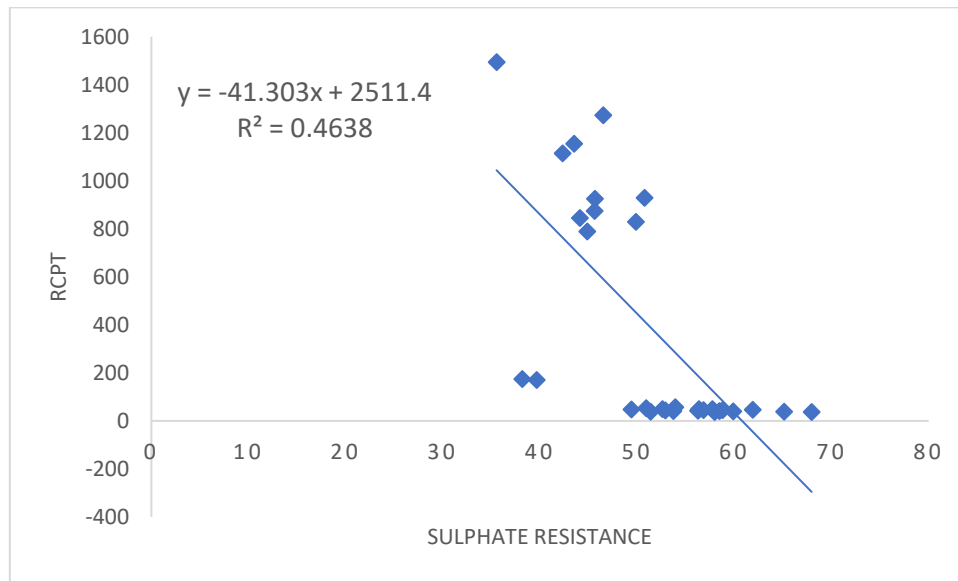


Fig. 4.48: Relationship between sulphate resistance and RCPT of all SCC mixes at all ages

Table 4.31: Relationship between sulphate resistance and RCPT of all SCC mixes at respective ages

Age	R^2 value	Y value
28 days	0.4707	$-37.333x + 2702.8$
90 days	0.636	$-5.2146x + 332.84$
365 days	0.7758	$-4.8389x + 334.39$

Chapter 5

Conclusions

This chapter presents the conclusions of all the tests done in experimental programme for the present study. It can be concluded from the test results that metakaolin and rice husk ash can be successfully used as partial replacement of cement and fine aggregates respectively in SCC. Based on test results following conclusions are drawn:

5.1 Fresh Properties

5.1.1 Slump flow

Slump flow values for all the SCC mixes were in the span of 650 – 755 mm and were within the prescribed limits (650 – 800 mm) as per EFNARC. When MK was used, slump flow first increases and then it starts decreasing as compared to the slump of control mix. It was in the span of 695 – 665 mm. Similar results were obtained for the addition of RHA also. It was in the span of 690 – 650 mm. While when MK and RHA were used in combination, slump flow decreased with the increasing percentages of MK and RHA. It was in span of 755 – 670 mm. The dosage of superplasticizer was increased to 2% from 1.5% for the mixes made with combined use of MK and RHA.

5.1.2 L – box

L – box values were in the range of 0.6 to 0.9. All the mixes satisfied the EFNARC criteria for L – box test, except mix M7 (30RHA). But, mix M7 satisfied EFNARC criteria for slump flow, V-funnel and U-box tests. It was observed that the blocking ratio decreased with the use of MK and RHA. Mix M2 (5MK) provided the best L – box ratio of 0.9. Thus, all the mixes have good filling ability.

5.1.3 U - box

U-box difference in height of concrete in two compartments was in the range of 8 – 30 mm, which was also within the limits prescribed by EFNARC (0 – 30 mm). The use of MK and RHA increased the U – box values of the mix. Control mix has U – box ratio of 12, while mix M2 (5MK) has U – box ratio of 19 which further increased with increasing percentages of MK. When both MK and RHA are used, test values were in span of 8 – 22.

5.1.4 V – funnel

The V- funnel time for all the mixes ranges from 8 to 12 seconds, which was within the span specified by EFNARC. So, all mixes satisfied the criteria laid by EFNARC thus indicating

good flow ability. V – funnel time for SCC mixes decreased with the use of MK and RHA when compared to mix made without MK and RHA. But at the same time V – funnel times also increased with the increase in the percentages of MK and RHA. When both MK and RHA were used V-funnel time decreased to 8 seconds from 11 seconds for mix without MK and RHA.

5.2 Strength Properties

5.2.1 Compressive strength

It was observed from the results that the strength increases with the increased percentages of MK, while in case of RHA, strength increases up to 10% replacement level and after that strength starts decreasing. The compressive strength results for mixes prepared with MK bespeaks of 24% increase in strength at 28 days, while mixes made with RHA showed 4.8% increase when correlated to control mix. When both MK and RHA are used in combination, highest strength was achieved by the mix M11, made with 10MK and 10RHA. It showed 26%, 27%, 42% and 48% increase in strength than the control mix at 7, 28, 90 and 365 days respectively. Also, all other mixes exhibited more strength than the control mix. So, using MK and RHA combination in SCC helps in strength upgradation.

5.2.2 Splitting tensile strength

Mixes prepared from the combination of MK and RHA displayed more strength at all ages when correlated to mix M1. The highest strength was achieved by the mix M11 (10MK10RHA), which showed the maximum strength of 2.74, 4.43, 5.12 and 5.96 MPa at 7, 28, 90 and 365 days respectively. It was observed that strength increased up to 10% RHA and after that it starts decreasing, while in case of MK it increases up to 10% and slightly decreases at 15%.

5.3 Durability Properties

5.3.1 Water absorption and porosity

The water absorption and porosity for mixes containing MK+RHA was lowered as against the control mix (M1). Also, there was decrease in porosity as the curing period was increased. The water absorption (after immersion) of the control mix was 5.81%, 7.25% and 6.80% at 28, 90 and 365 days respectively, while the water absorption of mix M11 (10MK 10RHA) was 3.93%, 4.90% and 4.10% at 28, 90 and 365 days respectively. For most of the mixes there was decrease in the values of water absorption and porosity between 90 and 365 days. But it was also detected

that the level of water absorption and porosity rises with the increased percentage of MK and RHA. Mix M16 (15MK30RHA) had the highest values of water absorption among all the mixes made with the use of MK and RHA.

5.3.2 Sulphate resistance

It was detected that the strength loss was maximum in control mix, while mix M11 had minimum of strength loss. All the mixes made with the use MK and RHA had better resistance to sulphate attack as against the control mix (M1). There was loss of 14, 16 and 17.9 % strength for the control mix at 28, 90 and 365 days respectively, while mix M11 had loss of 3.1, 4.5 and 4.8% at 28, 90 and 365 days respectively. The addition of MK and RHA improved the resistance to sulphate attack of SCC mixes.

Talking about the percentages of MK and RHA, it was observed that sulphate resistance improved up to 10% replacement level of MK, while at 15 % there was more loss of compressive strength as compared to 5 and 10% replacement levels, but it was still less than the control mix. While in the case of RHA, 10% replacement level was found to be optimum level.

5.3.3 Rapid chloride permeability

It was detected, that there was drop in chloride penetration with the use of MK and RHA. For mix M1, made without use of MK and RHA the permeability values were 1495, 175 and 171 coulombs at 28, 90 and 365 days respectively. When MK was used in percentages of 5, 10 and 15% with 10% constant percentage of RHA, permeability values decrease at all the ages. With 15%MK and 10%RHA there was 44%, 74% and 77% decrease in chloride permeability at 28, 90 and 365 days respectively. It can also be observed that permeability values keep on decreasing till mix M12, but there was a little rise for permeability values for mix M13, which was made with 10%MK and 30%RHA. Again, it can be observed that permeability values take a little rise for mix M16, made with 15%MK and 30%RHA.

5.4 Micro-Structural Properties

5.4.1 SEM

The SEM images clearly describe that the sample for mix without MK and RHA (M1) consists of voids and micro pores. Also, there is uneven spread of C-S-H gel in some areas along with the formation of ettringites. However, in the case of the samples for mixes containing RHA and MK a more homogeneous structure can be seen. The SEM images of mix M9 and M10

shows formation of calcium silicate hydrate (C-S-H) gel, and the gel is widely spread. This formation of C-S-H gel minimized the voids and results in the improved strength and durability properties.

As percentage of MK was increased, the structure becomes more uniform and denser. The pores were reduced to great extent. Also, in the cases when percentage of RHA is increased the mixes started disintegrating. The SEM images show inconsistent formation of C-S-H gel.

5.4.2 XRD

It can be observed from the XRD images, that for control mix, the amount of unused silica is maximum, reaching up to 3500. This can be directly linked to the performance of the control mix. For the mix M11 containing 10% MK and 10% RHA, the intensity of free silica is minimum. It was found that with the increased metakaolin percentages in concrete mixes, utilization of silica in C-S-H gel increased. This was observed up to 10% MK level. At 15 % MK level, this trend differs. The presence of calcium hydroxide is very less in all concrete mix, which confirms the maximum consumption in the hydration reaction. This leads to the dense microstructure and additional development of C-S-H gel, leading to the improvement of microstructure of the concrete, which has resulted in the improvement of the strength and durability properties.

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