

4.1 GENERAL

The properties of cement paste, mortar and concrete are highly dependent on the individual features of various materials that constitute the hardened cement paste, mortar and concrete. The effects of hydrated cement paste on the physical properties of mortar and concrete are much more significant in comparison with the impact of the type of aggregates used in mortar and concrete. Therefore, to understand the effects of RSA and MS on properties of paste, mortar and concrete, it was essential to study the behaviour of RSA and MS particles as well. In this chapter, the results highlighting various properties of RSA and MS particles and the properties of other constituents of concrete have been studied and discussed in detail.

4.2 AGGREGATE

Basalt based crushed coarse aggregates of a maximum nominal size of 20 mm and 10 mm as per IS 383 and IRC 44 [226], [251] were obtained from Dalla quarry in Sonbhadra district of the state of Uttar Pradesh, India. Riverbed sand was used as fine aggregate conforming to Zone II as per IS 383 [251] and was obtained from Chopan quarry in Sonbhadra district of the state of Uttar Pradesh, India.

The results of sieve analysis of coarse and fine aggregates are shown in Table 4.1 and their mechanical properties are mentioned in Table 4.2. All the mechanical properties like fineness modulus, specific gravity, water absorption, crushing and impact value, flakiness and elongation index, abrasion value were within their specified limits.

Table 4.1 Percentage passing of coarse and fine aggregates for PQC

Sieve Size (mm)	Percentage Passing								
	Coarse Aggregate						Fine Aggregate		
	20 mm			10 mm			Sand (Zone II)		
	Min. Limit	Max. Limit	Obtained	Min. Limit	Max. Limit	Obtained	Min. Limit	Max. Limit	Obtained
20	85	100	96.84	-	-	-	-	-	-
12.5	-	-	-	-	100	100	-	-	-
10	0	20	14.29	85	100	88.86	-	-	-
4.75	0	5	0	0	20	2.86	90	100	97
2.36	-	-	-	0	5	0	75	100	94.4
1.18	-	-	-	-	-	-	55	90	82.4
0.6	-	-	-	-	-	-	35	59	54.6
0.3	-	-	-	-	-	-	8	30	24
0.15	-	-	-	-	-	-	0	10	7

Table 4.2 Mechanical properties of coarse and fine aggregates

Properties	Specifications	Coarse Aggregate			Fine Aggregate	
		Limit	Obtained		Limit	Obtained
			20 mm	10 mm		
Fineness Modulus	IS 383 [251]	6 - 6.9	6.89	6.08	2.1 - 3.37	2.41
Specific Gravity	IS 2386 (III) [228]	2.5 - 3	2.78	2.72	2.5 - 3	2.65
Water Absorption (%)	IS 2386 (III) [228]	< 2	0.6	0.75	0.3 - 2.5	1.1
Unit Weight (kg/m ³)	-	-	1610	1525	-	1480
Crushing Value (%)	IS 2386 (IV) [227]	< 30	10.6		-	
Impact Value (%)	IS 2386 (IV) [227]	< 30	9.5			
Flakiness Index (%)	IS 2386 (IV) [227]	< 30	25.6			
Elongation Index (%)	IS 2386 (IV) [227]	< 35	31.1			
Abrasion Value (%)	IS 2386 (IV) [227]	< 35	29.6			

Fineness modulus depicts average size of aggregates. Coarse aggregates used in this research were highly resistant to crushing and impact. The pavement with aggregates of lower crushing value generally has a higher service life as compared to

aggregates of higher crushing value. The aggregates used in this study had relatively higher specific gravity and lower water absorption which portray its superior quality. The presence of too elongated and too flaky aggregate particles reduces the strength of the rigid pavements. They are liable to break under heavy vehicular load. The aggregates which constitute the top layer of the pavements are subjected to wear due to abrasive action of tyres. Therefore, only those aggregates were considered in the present investigations, which were highly resistant to abrasion.

4.3 CEMENT, RICE STRAW ASH AND MICROSILICA

The cement utilized was OPC of 43 grade confirming to IS 8112 [252]. 920D-grade microsilica was used which was obtained from Elkem South Asia Pvt. Ltd. Rice straw was obtained from the agricultural farm of the Banaras Hindu University. It was burnt in the open air without any enhanced burning technique. The burning temperature of rice straw was around 290 ± 5 °C as was measured by an infrared thermometer. It was comparable to the temperature at which the combustion of most types of wood take place (300 °C) [253]. The open air burning of rice straw leads to emission of greenhouse gases like CO₂, CO, CH₄ and N₂O [254]–[257]. The emission of CO, CH₄ and N₂O plays a significant role in increasing global warming while emission of CO₂ due to burning of rice straw is not taken into consideration for calculating net amount of greenhouse gases which affects the atmosphere, based on the hypothesis that the CO₂ will be absorbed again by the rice plants during the next cultivation [255]. As per previous studies [258], [259], the open-air burning of rice straw emits around 57-81% CO₂, 5-9% CO, 0.43-0.9% CH₄ and 1.16-1.5% N₂O. In open-air burning of 100 kg of rice straw, 19.4-32.2 kg of CO₂ is released into the atmosphere while 90 kg of CO₂ is emitted in the production of 100 kg of cement [12], [258], [259]. It can be concluded

that open-air burning of rice straw does affect the atmosphere however, to a much lower extent as compared to the production of cement.

Therefore, studies concerning part replacement of cement hold economic as well as environmental importance. In the current study, RSA was collected carefully to avoid any unwanted particles like dust. It was then sieved through 90 μm sieve and stored in air tight bags for use as pozzolanic material. No further processing like pulverisation etc. was done thereby saving energy. The 90 μm size sieve was chosen in conformity with similar procedure adopted by other studies for preparation of supplementary cementitious material [64], [260], [261]. As per the literature, the cementitious materials of higher fineness were found to be more reactive as compared to the cementitious materials of lower fineness [262]–[264]. However, according to Mehta 1979 [265], higher fineness of cementitious materials ought to be avoided as their reactivity will depend solely on the surface area of the particles and not their composition.

The density, specific gravity, specific surface area and mean particle size of the OPC, RSA and MS are shown in Table 4.3. It was found that the specific gravity of RSA and MS was lower than the OPC. The lower specific gravity of RSA and MS as compared to OPC signifies that the volume of RSA and MS, which replaces the same mass of OPC, will be higher. The mean particle size of the microsilica was much smaller than that of the OPC and RSA particles. In other terms, MS and RSA particles were 28 times and 5 times respectively, finer than the OPC particles, which was as per reported by other researchers as well [22]. The particle size distribution of OPC, RSA and MS is shown in Figure 4.1. The specific surface area of MS was more than that of the RSA particles and the OPC particles. On mixing RSA and MS with OPC, there is an

increase in the surface area of the cementitious materials which helps in achieving early strength of concrete [266].

Table 4.3 Properties of OPC, RSA and MS

Material	Density (kg/m ³)	Specific Gravity	Fineness (%)	Specific Surface Area (m ² /g)	Mean Particle Size (µm)
OPC	3190.4	3.2	7.2	0.3	17
RSA	2243.25	2.25	-	1.846	3.3
MS	2223.31	2.23	-	16.14	0.6

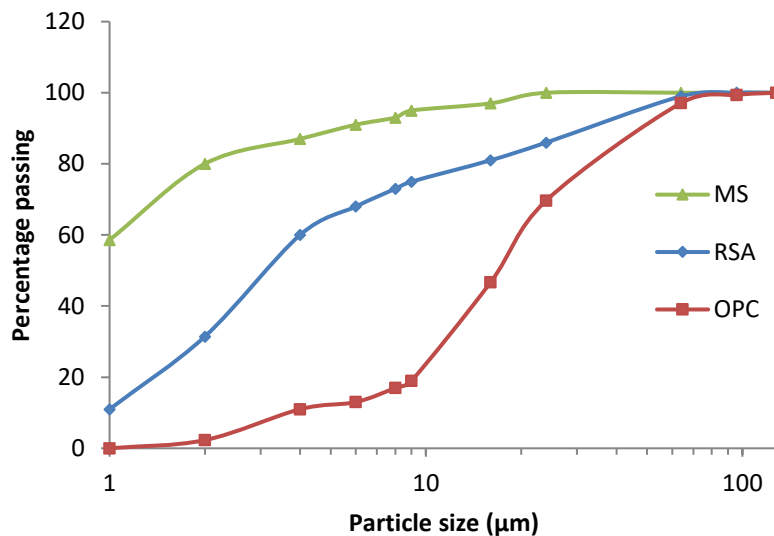


Figure 4.1 Particle size distribution of OPC, RSA and MS

The chemical composition of OPC, RSA and MS is shown in Table 4.4. RSA can be listed as class F pozzolan as it satisfies the criteria listed in ASTM C618-78 [43]. Microsilica and rice straw ash had a negligible amount of CaO and high SiO₂ content of 91.3% and 79.82%, respectively. It shows the significance of their role as pozzolans as they had very little or no cementitious value. Since RSA contains high amount of SiO₂ (79.82%), it could be used as a source of silica in glass production [267]. β-wollastonite could also be produced from RSA by the process of autoclaving and sintering due to the presence of a high amount of silica [35]. It was found in the literature that Si (Silicon), Mg (Magnesium), K (Potassium) and Ca (Calcium) are the main elements of sunflower husk ash due to which it is used as a raw material in the production of ceramic products

[268]. Since the amount of Si, Mg, K and Ca in rice straw ash were similar to the sunflower husk ash; rice straw ash could also be used as a precursor in ceramic industries. Rice straw ash also contains elements of K and Ca; therefore, it could also be used as a crude fertilizer to improve the properties of agricultural soil [36]. According to the literature, if the amount of magnesium oxide (MgO) in the cementitious material is more than 5%, the chances of cracking in concrete are higher [142]. The lime saturation factor (LSF), alumina ratio (AR) and silica ratio (SR) of OPC were 0.91%, 1.66% and 2.07% respectively, well within the range specified in IS 8112.

Table 4.4 Chemical composition of OPC, RSA and MS

Compound (% by wt.)	Material		
	OPC	RSA	MS
SiO ₂	20.34	79.82	91.3
CaO	62.16	0.37	0.171
MgO	0.79	7.54	1.29
Al ₂ O ₃	6.13	1.13	0.616
Fe ₂ O ₃	3.7	0.245	1.47
P ₂ O ₅	2.03	3.75	-
SO ₃	1.73	-	3.13
Na ₂ O	1.33	0.501	0.082
SrO	0.45	-	-
Cl	0.305	4.06	-
TiO ₂	0.189	-	-
S	-	1.16	-
K ₂ O	-	1.07	0.653
P	-	-	0.38
ZnO	-	-	0.244

4.3.1 Mineralogical, Microstructural and Thermo Gravimetric analysis of OPC, Rice Straw Ash and Microsilica

XRD analysis is a non-destructive mineralogical study to identify the different phases present in a substance. The XRD patterns of OPC, RSA and MS are shown in Figure 4.2. According to XRD analysis, the primary phase in OPC is alite (C₃S) while minor phases are belite (C₂S), ferrite (Fe₂O₃) and silica (SiO₂) in the form of celite.

Similarly, the primary phase in RSA and MS is silica (SiO_2) in the form of quartz. A small amount of silica in the form of cristobalite was also observed in RSA. On comparing the XRD patterns of RSA and MS, it was observed that MS has a comparatively compact structure. XRD pattern of MS demonstrates a narrow peak hump which implies that it contains highly reactive silica in the amorphous form.

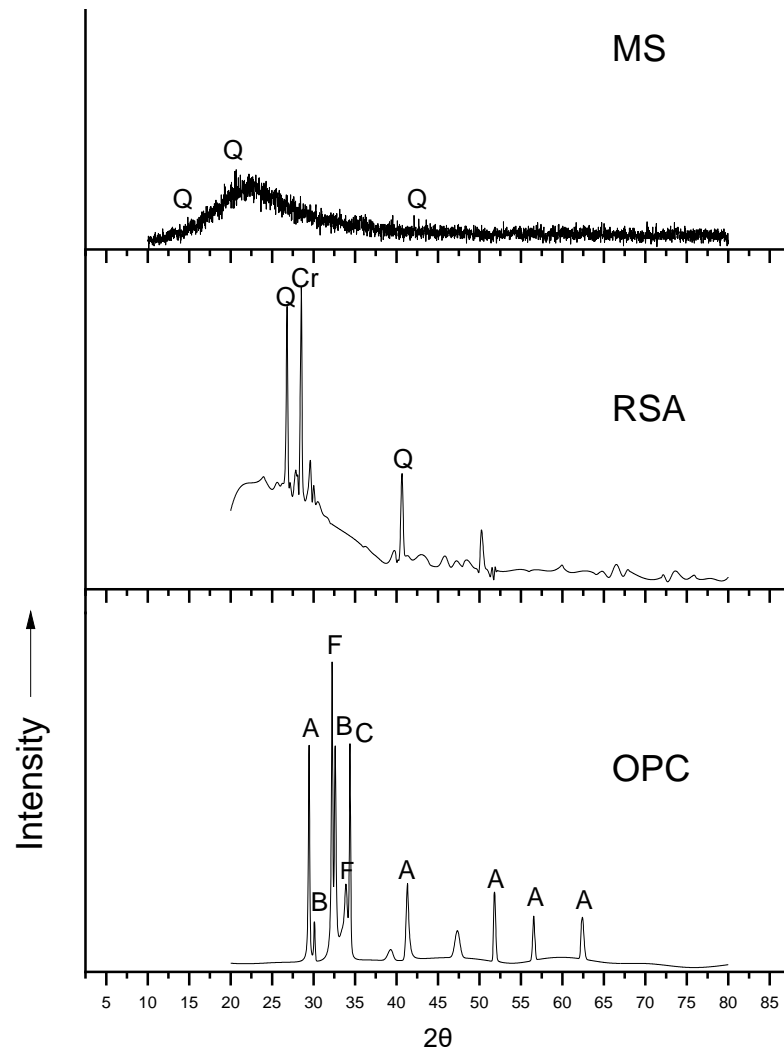
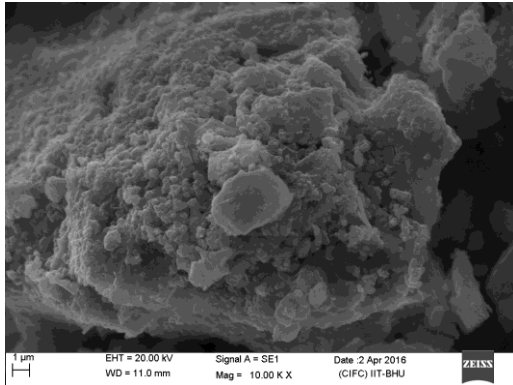


Figure 4.2 XRD pattern of OPC, RSA and MS

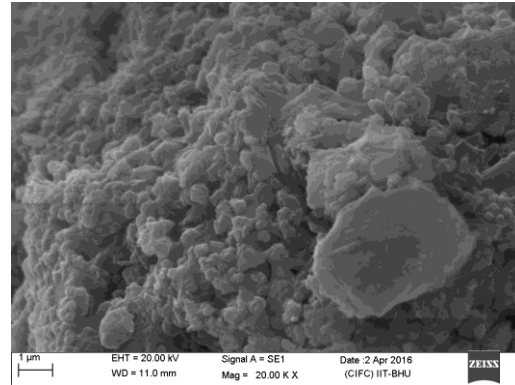
(Q - Quartz, Cr - Cristobalite, A - Alite, B - Belite, F - Ferrite, C - Celite)

Figure 4.3 shows the SEM micrographs of OPC, RSA and MS. It can be seen that the shape of the OPC particles (Figure 4.3 (a) and (b)) is irregular somewhat similar to the form of aggregates. The RSA particles are acicular with aspect ratio of about

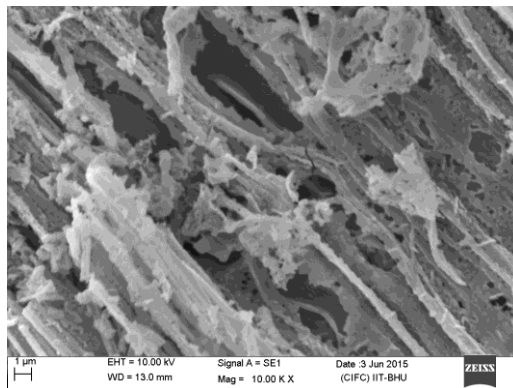
8.5:1 (Figure 4.3 (c) and (d)) [37]. The MS particles are spherical in shape as can be seen in Figure 4.3 (e) and (f). The MS particles are combination of numerous ultrafine particles. RSA particles were more porous as compared to OPC and MS particles.



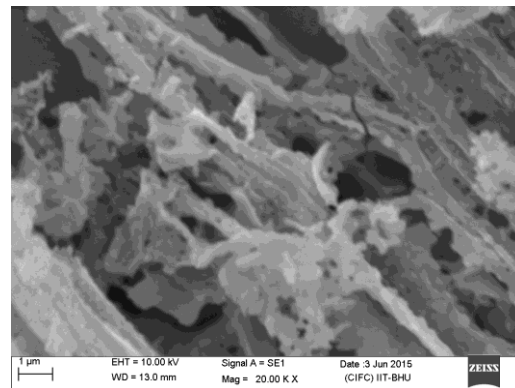
(a) OPC at 10,000 times magnification



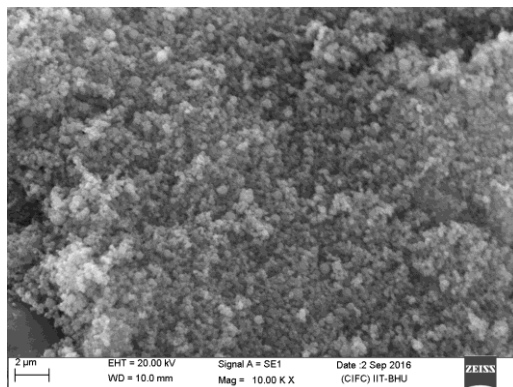
(b) OPC at 20,000 times magnification



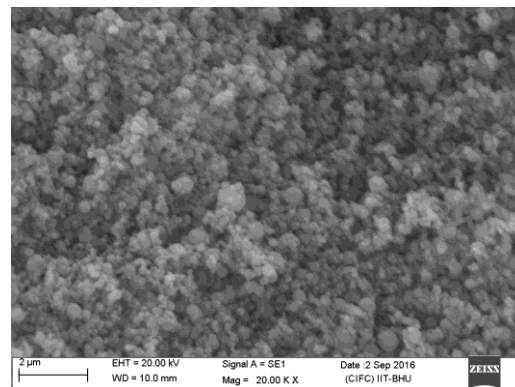
(c) RSA at 10,000 times magnification



(d) RSA at 20,000 times magnification



(e) MS at 10,000 times magnification



(f) MS at 20,000 times magnification

Figure 4.3 SEM micrographs of OPC, RSA and MS at different magnification levels

TGA is a process of analysing the change in physical and chemical properties of material when subjected to a constant rate of increase in temperature. Only TGA analysis of RSA was studied. It was observed that loss of mass of RSA due to exposure

to rising temperature occurred in four stages as shown in Figure 4.4 and Table 4.5. The least rate of weight loss (0.015 % per min.) was observed in stage I with range of temperature varying between 25 and 60 °C. It was because RSA was produced by burning rice straw in an open air and therefore temperature as low as 60 °C did not make RSA to loose weight. However in stage II, the rate of weight loss (0.467 % per min.) in the temperature range of 60 to 220 °C was highest amongst all the stages. This loss of weight was due to removal of moisture from RSA. As RSA particles are porous in nature, they tend to absorb moisture from the atmosphere. It signifies the importance of pre-heating of RSA in an oven before its usage as a cementitious material. The rate of weight loss in stage III (0.161 % per min.) in the temperature range of 220 to 860 °C was due to dismissal of volatile matter. Similar nature of RSA was also observed by Chakraverty and Kaleemullah (1991) [32]. Weight loss in stage IV (0.311 % per min.) in the temperature range of 860 to 1200 °C was due to slow oxidation of unburned carbon. As RSA was produced without using any enhanced techniques of burning, high amount of unburned carbon was expected in the present study. A similar result was observed by Jaya et al. (2013) [269] for rice husk which when burnt at 600 °C, underwent change in colour from greyish white to white when the burning temperature was increased to 800 °C due to slow oxidation of unburned carbon at higher temperatures.

Table 4.5 Thermal analysis of RSA

Condition	Stage			
	I	II	III	IV
Temperature Range (°C)	25 - 60	60 - 220	220 - 860	860 - 1200
Time Range (min.)	0 - 4	4 - 10	10 - 42	42 - 59
Weight Loss Range (%)	0 - 0.06	0.06 - 2.86	2.86 - 8.03	8.03 - 13.32
Rate of Weight Loss (% per min.)	0.015	0.467	0.161	0.311

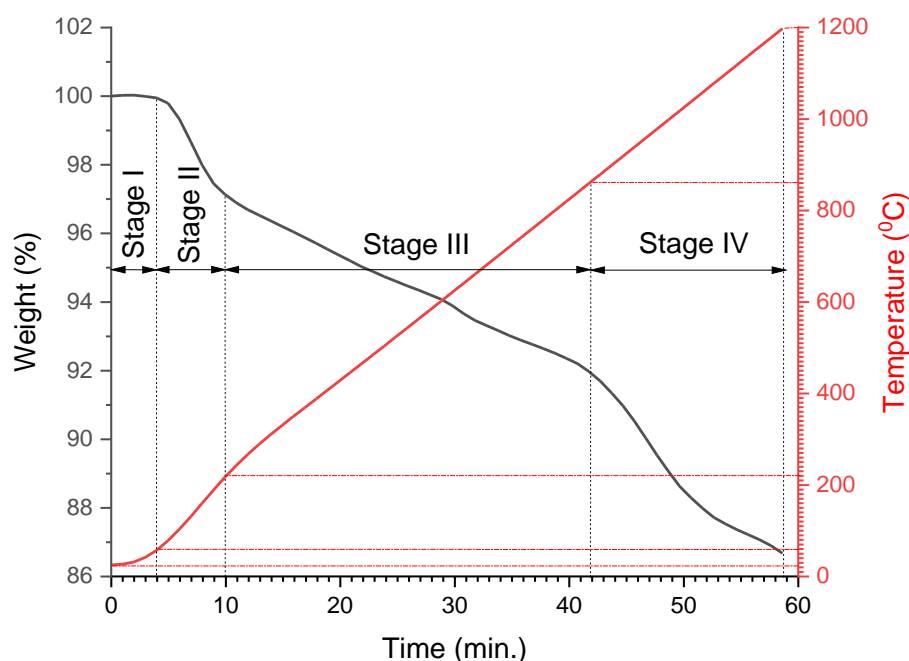


Figure 4.4 TGA analysis of RSA

4.4 CHEMICAL ADMIXTURE AND WATER

The decrease in workability due to the incorporation of RSA and MS was compensated by the usage of sulphonated naphthalene polymer-based high range water reducer (HRWR). Conplast SP430 HRWR was procured from Fosroc Chemicals (India) Pvt Ltd. The datasheet about the characteristics of Conplast SP430 superplasticizer as obtained from the supplier is given in Table 4.6. Potable water was used for mixing and curing as per IS 456 [270]. Its temperature was maintained at 27 ± 2 °C.

Table 4.6 Characteristics of superplasticizer

Standard Compliance	Complies with IS 9103 [271] and BS EN 934 (II) [272]
Specific Gravity	1.220 - 1.225 at 30 °C
Chloride Content	Nil
Air Entrainment	Roughly 1% extra air is entrained
Compatibility	Compatible with all types of cement except high alumina cement

4.5 CHAPTER SUMMARY

This chapter discussed the various properties of the materials used in the experiments carried out in the current investigation. The sources of aggregates, OPC, RSA, MS and HRWR, along with their physical and chemical properties, were discussed in detail. Subsequently the results of microstructural and mineralogical analysis of OPC, RSA and MS were analysed. Also, the thermal analysis of RSA was discussed in detail. The detailed analysis of the reactivity of RSA and MS was done in the current chapter, which justified their usage as a cementitious material.