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Sustainable Alternate Materials for Concrete Production from Renewable Source and Waste

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Abstract: Natural resources are being continuously extracted for the production of concrete which leads to degradation of the ecosystem. This is also a challenge for sustainability to save Nature. This study seeks to identify a suitable replacement material for river sand and stone aggregate for the sustainable utilization of renewable sources. Manufactured sand (M-sand) from industrial by-products and coconut shell (CS), an agricultural waste, are the resources selected as replacement materials for sustainability. This study uses M-sand as fine aggregate and CS coarse aggregate in place of river sand (R-sand) and crushed stone aggregate (CSA) for concrete production, respectively. To prove that M-sand and CS are sustainable alternate materials, this study focused on the microstructural characteristics on concrete constituents and CS aggregate and also conducted on concrete produced using R-sand, M-sand and CS. Also, this study focused on the microstructural characteristics and properties of conventional concrete (CC) and coconut shell concrete (CSC) produced using both R-sand and M-sand. Since this study aims to find sustainable alternative materials for R-sand and CSA by M-sand and CS, its properties are studied and compared since microstructural characterization is very significant for concrete compatibility. Microstructural studies revealed that the use of M-sand does not affect the microstructural properties of concrete compared to R-sand concrete and rather it improves the strength of concrete. A similar same trend was observed when CS was used with M-sand compared to CS used with R-sand. Hence, this study strongly suggests that the use of M-sand in its place of R-sand and CS in its place of CSA are sustainable alternatives for the production of concrete so that natural resources can be saved and hence sustainability could be sustained.

Keywords: sustainability; sustainable material; R-sand and M-sand; coconut shell; concrete; properties

1. Introduction

Sustainability is one of the major concerns for the entire world at present. The construction world is not exempt from this also. In the construction world, concrete is the supreme commonly used material which consumes a lot of natural aggregate resources such as river sand (R-sand) and crushed stone aggregate (CSA). Aggregates occupy nearly 70–80% of the concrete volume. Therefore, natural aggregate resources are being continuously extracted whenever the infrastructural development takes place for any industries for concrete production. Due to this sustainability issues arise for sustainable development. Therefore, it is the duty of the concrete researchers to find suitable replacement materials to save the natural resources. If the replacement materials also come from renewable sources then it could be an advantage for both the construction industries and sustainability. In light of this, many researchers have tried to incorporate additional or replacement materials from industrial, domestic and agricultural wastes in concrete [1–17]. Also, the use of recycled aggregates is also considered another source for alternate material to be used in concrete. Many researchers have studied the use of recycled aggregates in different concrete production processes and tested their properties and characteristics. Some people have done their research at an advanced level by developing hybrid tube columns containing structural recycled aggregate concrete filled—GFRP-steel composite tested under axial...
compression [18]. Surface deformation and strain in recycled aggregate concrete-filled steel tubular columns via four-ocular vision in real time has also been detected [19].

One of the alternate materials found for CSA is coconut shell (CS). The use of CS in place of CSA will definitely lead to improved sustainability. To justify this there are some applications of CS in practically implemented structural and non-structural element production. CS is used in concrete as a coarse aggregate for the production of non-pressure pipe [20], paver blocks [21], manhole cover slabs [22], prefabricated unipaver concrete blocks embedded with CS particles [23], prefabricated fencing posts embedded with CS particles [24] and hollow blocks produced using CS as coarse aggregate [25]. Apart from these applications, precast reinforced concrete slabs in which CS was used as coarse aggregate followed by layman methodology were described without any technical suggestions in 2007 [26] and these slabs were supported with CS-containing hollow blocks and allowed normal routine usage like people sitting on these slabs and sometime used them as weights. After 14 years, they continue in service without any experienced damages. This practical example gives more confidence that CS can be used as coarse aggregate in its place of CSA without any hesitation. This is also a proven example that the CS is an alternate sustainable material and can justify the use of CS in the field of sustainability.

Many studies are carried out using CS as coarse aggregate to produce coconut shell concrete (CSC) [27–32] using R-sand, but now the situation has changed for two major reasons: one is the overexploitation of natural sources and hence scarcity of R-sand; the other one is to save the sources for future generations and also environmental degradation. Therefore, there is a need to find an alternate or replacement material for R-sand [33–39]. Though many possible materials have been suggested to replace R-sand, quarry dust (QD) and manufacturing sand (M-sand) are the two major resources from one origin point of view.

Since the combination of using QD and CS in the production of CSC was already done by others [40–43], also the study of using M-sand and CS in the production of CSC is limited and hence this study has been taken. Concrete is a heterogeneous substance and though it is a mixture of cement and aggregates on a macroscopic level, cement paste itself may consists of unhydrated components, some amorphous hydrated products such as needles of ettringite, calcium hydroxide (CH) phase gel/crystals, calcium silicate hydrates (C-S-H) fibrous crystals and pores which are factors affecting the strength and durability of concrete. Therefore, use of any other material other than the CC constituents should not affect the pozzolanic and physical action of cement and concrete. The enhancement of the microstructure of concrete happens not only because of pozzolanic reactions but also due to filler effects. Since the use of M-sand and CS combinations is new to CSC, their microstructural analysis study is very important because pozzolanic reactions and filler effects are two factors which decide the microstructure of concrete density, homogeneity and uniformity, leading to improved concrete strength and durability. There is a research gap since not many studies have been done on CSC using M-sand. Hence, information on the effect of M-sand as fine aggregate and CS as coarse aggregate on concrete properties is limited. It was found that microstructural characteristics and mechanical properties data are also limited. Therefore this study proposed to develop M-sand concretes using CS as coarse aggregate and analysed their properties.

The paper is structured as follows: in Section 2, the concrete constituents used for this study and their physical and chemical properties and also the process of using CS as aggregate are described with tables and photographs. Section 3 describes the concrete mixes and proportions of both CC and CSC with R-sand and M-sand. Section 4 illustrates the microstructural studies carried out using different methods, the sample preparation methods, equipment types and conditions for running the microstructural studies. Section 5 describes the complete experimental programme followed in this study such as microstructural methods of study and mechanical properties of both CC and CSC with R-sand and M-sand. Section 6 discusses the test results of the microstructural studies on concrete constituents, concrete mixes, workability and fresh concrete density, hardened.
concrete density and strength and mechanical properties. Major conclusions drawn from the results and discussion are finally presented in Section 7.

2. Concrete Constituents

Conforming to IS 12269: 2013 [44], 53 grade ordinary Portland cement (OPC) with specific gravity 3.11 was utilized. Since for the production CSC, the literature recommends the use of 12.5 mm maximum size CS [20–29,40–43], in this study the same size was also used and similarly for CC using CSA for comparison. Local sources of both R-sand and M-sand were used to produce CC and CSC, both of them conforming to IS 383: 2016 [45] zone II grading. Discarded CS was collected from a coconut industry and crushed using a CS crusher available on our university premises. Crushed CS was processed and then used as suggested in earlier studies [20–29,40–43]. Table 1 gives the specific gravity, bulk density and fineness modulus of the aggregates used. Table 2 provides different chemical compositions of OPC, R-sand and M-sand in terms of percentages. Figure 1a shows M-sand collecting site and Figure 1b shows the raw CS collected. Crushed CS samples shown in Figure 2a,b indicate the sizes of CS compared with a scale. Figure 3a,b show the sieve analysis graph of both R-sand and M-sand with the indication of lower and upper limits of passing % of zone II classification as recommended in IS 383: 2016 [45]. It can be seen that the sieve analysis graphs of both R-sand and M-sand lie in between the lower and upper limits of zone II and also it can be seen that the graphs of both R-sand and M-sand are very close to each other.

Table 1. Properties of aggregates used.

| Parameters          | CSA   | CS    | R-Sand | M-Sand |
|---------------------|-------|-------|--------|--------|
| Specific gravity    | 2.83  | 1.14  | 2.63   | 2.66   |
| Bulk density (kg/m³) | 1660  | 645   | 1685   | 1710   |
| Fineness modulus    | 6.66  | 6.05  | 2.84   | 2.89   |

Table 2. Chemical composition of OPC, R-sand and M-sand.

| Chemical Composition (%) | OPC   | R-Sand | M-Sand |
|--------------------------|-------|--------|--------|
| Ca                       | 37.18 | 0.65   | -      |
| O                        | 29.70 | 49.27  | 28.79  |
| Si                       | 8.03  | 26.69  | 22.83  |
| C                        | 7.10  | 6.44   | 15.25  |
| Fe                       | 3.78  | 6.29   | 6.38   |
| Al                       | 2.68  | 6.63   | -      |
| S                        | 1.56  | 1.25   | -      |
| Mg                       | 0.16  | 0.26   | -      |
| K                        | -     | 2.45   | 3.05   |
| Na                       | -     | -      | 1.17   |
| Others                   | 2.65  | -      | -      |
| Loss on ignition         | 2.44  | -      | -      |
Figure 1. (a) Quarry source of M-sand (b) raw coconut shell collected.

Figure 2. (a) Crushed coconut shells (b) scale sized coconut shells.
3. Concrete Mixes

As stated in the previous Section 2, CSC and CC produced with R-sand were considered as control mixed concretes to produce 25 N/mm$^2$ as minimum strength. Since most of the earlier studies [20–29,40–43] adopted the mix proportions for CC as 1:2.22:3.66:0.55 and for CSC as 1:1.47:6.5:0.42 in which 320 kg/m$^3$ and 510 kg/m$^3$ cement content for the respective mixes, in this study also the same mixes were taken although the materials’ properties have some slight variations. However, keeping the same cement content, an equal volume
of M-sand was replaced by R-sand in both CC and CSC and this equal volume of M-sand weights were included in the concrete mixes as 1:2.40:3.65:0.60 for CC with M-sand and designated as CCM. Similarly, the same concept was adopted for CSC with M-sand, in this case the mix proportion is 1:1.60:0.65:0.42 and the product is designated as CSCM.

4. Microstructural Studies

The microstructural characteristics are studied through scanning electron microscopy (SEM) and energy dispersive spectroscopy (EDS) for OPC, R-sand and M-sand chemical element identification. For the purpose of quantitative analysis, peak positions and intensities associated with the patterns were analysed. The chemical compositions and their quantities of OPC given in Table 2 are with respect to the peak positions and intensities associated with the pattern of the EDS. The Fourier transform infrared spectroscopy (FTIR) technique finds the infrared (IR) absorptions or emissions of a solid or liquid substance in the wavelength ranging between 400 cm\(^{-1}\) and 4000 cm\(^{-1}\) that could be used to find the bonding nature of materials. The materials having wavelengths 400–1500 cm\(^{-1}\), 1500–2500 cm\(^{-1}\) and above 2500 cm\(^{-1}\) are said to be single bonded, double bonded and triple bonded in nature [46,47], respectively. SEM, EDS and X-ray diffraction analysis (XRD) were conducted on four mixes CC, CCM, CSC & CSCM.

For the benefit of the readers, the sample preparation methods, the equipment and conditions of running SEM/EDS and XRD are presented here. Surface topography of the sample can be observed through SEM. In general, SEM imaging gives only a visual representation of how the sample surface looks and it is difficult to draw discernable conclusions from the images. Energy dispersive spectroscopy (EDS) detects emitted backscattered electrons and gives the elemental composition of the analyzed area. Calcium-silicate-hydrate (C-S-H) gel is a primary nano-crystalline phase present in hydrated ordinary Portland cement (OPC) that is responsible for strength and durability. SEM and EDS were used to find out the C-S-H phases in a composite sample as the hydrated sample has different other phases present. Therefore, the data obtained from these experiments would be used to identify C-S-H phases in the samples.

At the end of the appropriate curing period, concrete prisms of approximately 10 mm square were cut from samples using a concrete cutting machine. Core samples were taken from the concrete cubes and also some chunks were taken from concrete samples and tested for its microstructural characteristics in SEM and EDS analysis at every period of time. XRD is a technique for the identification of the mineral content of powdered samples or the determination of the chemical structure of crystalline materials. The process involves directing X-ray beams on the powdered sample, and diffracted x-rays are recorded as characteristic of crystalline phases of the specimen. The results are interpreted by comparing the peaks with standards available to identify the phases present in the sample. For this analysis, concrete samples were ground to a fine powder of less than 63 µm. X-mineralogical analysis was carried out using X’Pert PRO equipment (PANalytical, city, country) at the SRM Institute of Science and Technology Nanotechnology Laboratory and SRM SCIF services.

The SEM image of the sample was obtained first. Once a clear SEM image is obtained, a target area on the specimen was selected to obtain the spectra. EDS was done on the same image to obtain the spectra for different locations. A backscatter electron detector was used to obtain the spectra. Combining EDS with SEM imaging provides spatially resolved elemental analysis. EDS detects the emitted backscattered electrons and gives the elemental composition of the analyzed area when the electron beam hits the sample surface and hence EDS detects the presence of elements in a scanned area. Data evaluation gives a spectrum which is evaluated to determine the elements present in the sample. The y-axis represents counts, which is the number of X-rays received and processed by the detector and the x-axis represents the different energy levels of the counts. Each peak in the spectrum denotes the presence of the element in the total scan area of the image. A smart map of different elements present in the sample was obtained using the smart mapping
option in the analysis software. From the smart maps required elements are identified. For C-S-H gel to be identified means three elements: oxygen (O), silica (Si) and calcium (Ca) can be found with the help of smart maps. From the smart maps, locations where Si, Ca and O are in abundance represent the C-S-H phase. Similarly, the presence of Ca and O corresponds to areas with calcium hydroxide (CH). Likewise, with the help of the smart map option in the analysis software any required compounds such as C-A-S-H, CH, and the atomic ratios between the components can be identified.

X-ray powder diffraction (XRD) is a rapid analytical technique primarily used for phase identification. The principle that is used in this XRD is the production of spectra consisting of several components that have different wavelengths and the specific wavelengths are characteristic of the target material (generally, copper Cu). Then the X-rays are collimated and directed onto the sample. As the sample and detector are rotated, the intensity of the reflected X-rays is recorded and the peaks in intensity occur. A detector records and processes this X-ray signal and converts the signal to a count rate which is then output to a device such as a printer or computer monitor. The geometry of an X-ray diffractometer is such that the sample rotates in the path of the collimated X-ray beam at an angle $\theta$ while the X-ray detector is mounted on an arm to collect the diffracted X-rays and rotates at an angle of $2\theta$. The intensity of diffracted X-rays is continuously recorded as the sample and detector rotate through their respective angles while testing and a peak in intensity occurs was captured by the recorder was taken for analysis of each sample.

The main aim of this research was to study the microstructural characteristics and also the hydration processes mainly responsible for the internal microstructure of cement and concrete. The hydration process starts once water is added to the cement, hence we wanted to track the hydration and hence microstructure. Hence we chose and considered 0, 1, 3, 7, and 28 as the observation day age so that the formation of primary long and slender ettringite, aggregation of fibrous crystals and clusters and a reticular network of C-S-H are verified from 0–28 days on both CC and CSC mixes.

5. Experimental Programme

In addition to the SEM, EDS, and XRD microstructural analyses conducted on concrete mixes, to measure the workability, slump and compaction factor tests were conducted too. Tests of properties of concrete in its hardened state such as compressive and flexural strength, split and impact strength resistance were conducted at 3, 7 and 28 days. The guideline IS 1199: 2018 recommended procedure was followed for conducting slump and compaction factor tests [48]. As per clause 5 of IS 516:1959 (reaffirmed in 2004) compressive strength was tested on 100 mm concrete cube specimens [49]. Using a simple beam with a middle third-point loading flexural strength test conducted as per the procedure recommended in ASTM C78-18 [50] and for this a beam of 100 $\times$ 100 $\times$ 500 mm size was used. A typical tested flexural specimen and its failure at the middle third point is displayed in Figure 4a,b showing a failed cross section of a specimen as a typical example.

Using cylindrical specimens splitting tensile strength tests were conducted as per the procedure recommended in ASTM C496-11 [51] and for this the size of cylinder is 100 mm in Ø and 200 mm in length. Figure 5a was taken during testing of specimens at the time of diametrical crack formed. Figure 5b was taken after the test when the cylinder specimen split into two parts and Figure 5c shows the tested specimen of one of the CSCs with M-sand under split tensile conditions.

Using a cylindrical disc specimen the impact resistance of the concrete mixes used in this study were tested as per the procedure recommended in ACI committee 544.1R-96 [52]. For this test, the size of cylinder disc specimen used is 152.4 mm in Ø and 63.5 mm in thickness. Figure 6a illustrates the equipment devised as per ACI committee 544.1R-96 and used for impact resistance test. Figure 6b shows a failed concrete cylinder disc specimen. An impact energy of one blow is calculated as 19.89 Joules with respect to the mass of the hammer (4.45 kg) and height of the fall (457 mm).
Figure 4. (a) Simple beam tested under third-point load (b) sectional failed specimen.

Figure 5. (a) Split tensile test (b) Splitting of specimen (c) failed specimen.

Figure 6. (a) Equipment devised for impact test (b) concrete cylinder discs and tested specimens.
6. Results and Discussions

Results on the different tests such as microstructural characteristics, concrete consistency, strength of concretes under compression, flexure, split tensile and impact resistance are discussed in this section.

6.1. Microstructural Studies on Concrete Constituents

Figure 7 shows SEM images of: (a) OPC and (b) R-sand. Similarly, Figure 8 shows SEM images of (a) M-sand and (b) of CS. It can be seen that there are some pores present in the CS aggregate from Figure 8b compared to CSA, R-sand and M-sand and is the reason for the higher water absorption capacity of CS compared to conventional aggregate.

Figure 7. Internal microstructural image of (a) OPC (b) R-sand.

Figure 8. Internal microstructural image of (a) M-sand (b) CS.

Figures 9–11 illustrate the EDS analysis of OPC, R-sand and M-sand, respectively. In general, R-sand and CSA are inert materials and hence their chemical components are mostly inactive except in some exceptional cases, hence this study is not focused on the impact of the chemical components present in R-sand and M-sand on the concrete
production. Rather, some significant properties of M-sand compared to R-sand and their influences are discussed here. R-sand has a smooth texture and better shape (Figure 7b) and hence requires less water, whereas M-sand has a rough texture and an angular shape (Figure 8a) and hence requires more water. Moisture can be trapped in between the particles of R-sand but moisture is generally not available unless M-sand is washed in water. In the case of R-sand there are some chances of getting silt content and the maximum allowable silt is only 3%. A silt presence in R-sand beyond 3% is dangerous for durability. Sometimes, 5–20% presence of silt in R-sand is possible but in case of M-sand the presence of silt content is zero. This zero silt in M-sand is an advantage for the long term performance of concrete structures. Since M-sand is artificially manufactured it leads to less probability of adulteration and hence oversizing can be avoided which cannot be avoided in the case of R-sand where a minimum presence of 1-6% of oversized pebble stones is possible. Beyond all, although M-sand uses regular aggregate resources, it causes less harm to the environment compared to R-sand, because, extraction of R-sand is dangerous to the environment, causing eco-inequalities, decrease groundwater levels and hence dried rivers [53].

Figures 12 and 13 show the FTIR analyses of R-sand and M-sand, respectively. In general, the FTIR signal is presented as a spectrum, ranging from 4000 cm\(^{-1}\) to 400 cm\(^{-1}\), in lieu of a molecular fingerprint of the samples tested. FTIR analysis is a great tool for chemical identification because each molecule/chemical structure will yield a distinctive spectral fingerprint. From Figures 12 and 13, it can be seen that the FTIR patterns wavelength of R-sand and M-sand are very similar to each other. In fact, some of the peak wavelengths of both R-sand and M-sand like the band at 788.89 cm\(^{-1}\) are found to be the same, which shows that the chemical structures of both R-sand and M-sand are almost similar. It can also be seen that the main bands of both R-sand and M-sand lie between 400 and 1500 cm\(^{-1}\), hence it can be stated that these materials have only single bonds [46,47].
Figure 9. EDS analysis result of OPC.

Figure 10. EDS analysis result of R-sand.

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Figure 11. EDS analysis of result of M-sand.

Figure 12. FTIR analysis of R-sand.

Figure 11. EDS analysis of result of M-sand.

Figure 10. EDS analysis result of R-sand.

Figure 11. EDS analysis of result of M-sand.
The results of microstructural studies carried out on the concrete samples made from different mixes using SEM images, EDS and XRD analysis are presented in this section. At the end of different curing periods, core samples were taken and studied for the purpose of identification of chemical elements through SEM, EDS and XRD analyses. It is difficult to draw discernable conclusions from SEM images in general because they give only a visual representation of how the sample looks. EDS on the other hand detects the back scattered electrons emitted from the analysed area and gives the elemental composition. A primary nano-crystalline phase present in hydrated OPC i.e., calcium-silicate-hydrate (C-S-H) gel is responsible for its strength and durability. SEM, EDS and XRD are used to determine the C-S-H phases in a sample since a hydrated sample also has other different phases. Therefore, data obtained using these experiments would be considered to detect C-S-H phases.

6.2. Microstructural Studies on Concrete Mixes

To obtain evolution images for the samples on the four mixes CC, CCM, CSC and CSCM at an age 0, 1, 3, 7 and 28 days SEM was done and illustrated in Figures 14–18,
respectively. It can be noted that there are some factors that affect the cement hydration system starting from the sample preparation up to the images taken by the operators since very fine particles are used for taking images after the magnification and sizes may be adjusted by the operator without being aware and not knowing the importance of the images. We found that a slight movement and adjustment gives different images, where one image shows cement hydrated properly and the other image from the same sample shows it is not properly hydrated, hence it can be stated that the SEM images taken from the sample and their corresponding inferences should be considered with respect to that particular image and not extrapolated to other regions.

In general, C-S-H phase formation morphology varies from poorly crystalline fibers to clusters and reticular networks. These network clusters of C-S-H amount to 50–60% of the volume of solids when Portland cement paste is completely hydrated and is its most significant phase that determines the paste properties. In this study C-S-H phase is also formed is almost the complete area of the images shown in Figure 18 since it shows only the clusters and reticular networks and no ettringite is found. All these images (Figures 14–18) show that a systematic hydration process takes place in both R-sand and M-sand containing mixes. Hence the replacement of M-sand for R-sand does not affect conventional cement hydration processes in both CC and CSC mixes.

Figure 14. SEM Images taken at zero day on all the mixes.
Figure 15. SEM Images taken at one day on all the mixes.

(a) CC mix
(b) CCM mix
(c) CSC mix
(d) CSCM mix

Figure 16. SEM Images taken at three days on all the mixes.

(a) CC mix
(b) CCM mix
(c) CSC mix
(d) CSCM mix
6.2.2. EDS Analysis

For EDS analysis, SEM images should be obtained on the sample first. A target location needs to be fixed on this image to obtain the spectra. EDS can be performed on the same image to obtain the spectra for different locations using a backscatter electron detector. Combining SEM and EDS will provide spatially resolved elemental analyses. When an electron beam hits the sample surface EDS detects the elemental composition of the analyzed area [55]. To determine the elements present in the sample spectrum a data evaluation must be done. The number of X-ray counts received and processed by the detector should be represented on the $y$-axis and the different energy levels of the counts are represented on the $x$-axis. In the total scan area of the image of each peak in the spectrum represents the presence of an element. For C-S-H gel to be identified it means three elements namely: oxygen (O), silica (Si) and calcium (Ca) should be found. Locations wherever Si, Ca and O are in abundance represent the C-S-H phase. Similarly, the occurrence of Ca and O is attributed to the areas with calcium hydroxide (CH). EDS was done to obtain evolution images for the samples on the four mixes (CC, CCM, CSC and CSCM) at age zero, one day, three days, seven days and 28 days and they are illustrated in Figures 19–23, respectively.

Figure 17. SEM Images taken at seven days on all the mixes.

Figure 18. SEM Images taken at twenty eight days on all the mixes.
Needle-shaped crystals of calcium trisulfoaluminate hydrate, called ettringite, first appear within a few minutes/hours of cement hydration as a result of interaction between calcium, sulfate, aluminate, and hydroxyl ions. Ettringite formation can be seen from Figures 14 and 15 in all the four mixes during the early phase of cement hydration that normally consumes most of the sulfate in the cement. The mechanism which controls stiffening of concrete is the formation of ettringite. For concrete specimens, the peak intensities of Al, S, Si and Ca are usually associated with the formation of ettringite. Formation of primary ettringite during early stages is a necessary and beneficial component of Portland cement systems [54]. Similarly, it can be seen in Figures 16 and 17 that the long and slender ettringite formed earlier is converted into aggregations of fibrous crystals in which there are some long prismatic crystals that indicate that the formation of calcium hydroxide (CH) and very small fibrous crystals indicating the formation the C-S-H phase.

6.2.2. EDS Analysis

For EDS analysis, SEM images should be obtained on the sample first. A target location needs to be fixed on this image to obtain the spectra. EDS can be performed on the same image to obtain the spectra for different locations using a backscatter electron detector. Combining SEM and EDS will provide spatially resolved elemental analyses. When an electron beam hits the sample surface EDS detects the elemental composition of the analyzed area [55]. To determine the elements present in the sample spectrum a data evaluation must be done. The number of X-ray counts received and processed by the detector should be represented on the y-axis and the different energy levels of the counts are represented on the x-axis. In the total scan area of the image of each peak in the spectrum represents the presence of an element. For C-S-H gel to be identified it means three elements namely: oxygen (O), silica (Si) and calcium (Ca) should be found. Locations wherever Si, Ca and O are in abundance represent the C-S-H phase. Similarly, the occurrence of Ca and O is attributed to the areas with calcium hydroxide (CH). EDS was done to obtain evolution images for the samples on the four mixes (CC, CCM, CSC and CSCM) at age zero, one day, three days, seven days and 28 days and they are illustrated in Figures 19–23, respectively.

A table of atomic counts (in %) is usually generated automatically at the time of analysis from which the ratio of Ca:Si could be calculated. The EDS analysis confirms that the quantities of C-S-H in all the mixes depends on the pozzolanic reactions at different ages. A lesser value of atomic Ca:Si shows that a major part of the cements reacted with portlandite (CH) [56,57]. In fact C-S-H is hyphenated and is a not a well-defined compound hence it is suggested that the Ca:Si ratio varies between 1.5 to 2.0 for the well hydrated C-S-H phase according to the literature [54]. The Ca:Si ratios of different mixes of this study at different ages are listed in Table 3.

Table 3. Ratio (Ca: Si) for different mixes at different days.

| Mixes | Ratio (Ca:Si) | Zero Day | One Day | 3 Days | 7 Days | 28 Days |
|-------|---------------|----------|---------|--------|--------|---------|
| CC    | 3.58          | 3.07     | 2.57    | 1.94   | 1.86   |
| CCM   | 3.43          | 2.95     | 2.49    | 1.93   | 1.71   |
| CSC   | 3.28          | 2.86     | 2.38    | 1.91   | 1.69   |
| CSCM  | 3.23          | 2.64     | 2.11    | 1.89   | 1.54   |

Table 3 shows that the ratio Ca:Si is higher during the early days and decreases as the age increases. At an age of 28 days, the ratio Ca:Si for all the four mixes lies in between 1.5 to 2.0, especially in case of CSC and CSCM compared to CC and CCM mixes which indicates and stresses that the systematic hydration process takes place in both R-sand and M-sand-containing mixes.
Figure 19. EDS analysis at zero day on all the mixes.
Figure 20. EDS analysis at one day on all the mixes.
Figure 21. EDS analysis at three days on all the mixes.
Figure 22. EDS analysis at seven days on all the mixes.
Figure 23. EDS analysis at twenty eight days on all the mixes.

6.2.3. XRD Analysis

X-ray diffraction analyses were conducted on samples of the four mixes CC, CCM, CSC and CSCM at 0, 1, 3, 7 and 28 days of age and they are shown in Figures 24–28, respectively.
to study phase nature of the materials and identified the main reacting compounds like ettringite, C-S-H and (Ca(OH)$_2$). The main compounds detected by XRD are C-S-H, Ca(OH)$_2$ and CH, there is an increase in the intensity of C-S-H peaks (Table 4) and a decrease of the other peaks. Table 4 shows the intensity count ranges for C-S-H, Ca(OH)$_2$ and ettringite compounds of all the four mixes at different ages. The XRD results supported the SEM and EDS results of mixed CC, CCM, CSC and CSCM. Compared to the formation of the compounds Ca(OH)$_2$, C-S-H compound intensity counts are high, which shows that there is no interruption of the pozzolanic reactions and this has a significant positive influence on the reduction of Ca(OH)$_2$–(CH) in all the mixes, which is beneficial for the quality of concrete strength.

A lesser value of atomic Ca:Si shows that a major part of the cements reacted with portlandite (CH) [56,57]. In fact C-S-H is hyph enated and is not a well-defined compound hence it is suggested that the Ca:Si ratio varies between 1.5 to 2.0 for the well hydrated C-S-H phase according to the literature [54]. The Ca:Si ratios of different mixes of this study at different ages are listed in Table 3.

Table 3. Ratio (Ca: Si) for different mixes at different days.

| Mixes | Ratio (Ca:Si) | Zero Day | One Day | 3 Days | 7 Days | 28 Days |
|-------|---------------|----------|---------|--------|--------|---------|
| CC    | 3.58          | 3.07     | 2.57    | 1.94   | 1.86   |
| CCM   | 3.43          | 2.95     | 2.49    | 1.93   | 1.71   |
| CSC   | 3.28          | 2.86     | 2.38    | 1.91   | 1.69   |
| CSCM  | 3.23          | 2.64     | 2.11    | 1.89   | 1.54   |

Table 3 shows that the ratio Ca:Si is higher during the early days and decreases as the age increases. At an age of 28 days, the ratio Ca:Si for all the four mixes lies in between 1.5 to 2.0, especially in case of CSC and CSCM compared to CC and CCM mixes which indicates and stresses that the systematic hydration process takes place in both R-sand and M-sand-containing mixes.

6.2.3. XRD Analysis

X–ray diffraction analyses were conducted on samples of the four mixes CC, CCM, CSC and CSCM at 0, 1, 3, 7 and 28 days of age and they are shown in Figures 24–28, respectively to study phase nature of the materials and identified the main reacting compounds like ettringite, C-S-H and (Ca(OH)$_2$). The main compounds detected by XRD are C-S-H, Ca(OH)$_2$ and CH, there is an increase in the intensity of C-S-H peaks (Table 4) and a decrease of the other peaks. Table 4 shows the intensity count ranges for C-S-H, Ca(OH)$_2$ and ettringite compounds of all the four mixes at different ages. The XRD results supported the SEM and EDS results of mixed CC, CCM, CSC and CSCM. Compared to the formation of the compounds Ca(OH)$_2$, C-S-H compound intensity counts are high, which shows that there is no interruption of the pozzolanic reactions and this has a significant positive influence on the reduction of Ca(OH)$_2$–(CH) in all the mixes, which is beneficial for the quality of concrete strength.

![Figure 24. XRD analysis at zero day on all the mixes.](image-url)
Figure 24. XRD analysis at zero day on all the mixes.

Figure 25. XRD analysis at one day on all the mixes.

Figure 26. XRD analysis at three days on all the mixes.
Figure 26. XRD analysis at three days on all the mixes.

(a) CC mix (b) CCM mix

(c) CSC mix (d) CSCM mix

Figure 27. XRD analysis at seven days on all the mixes.

(a) CC mix (b) CCM mix

(c) CSC mix (d) CSCM mix

Figure 28. XRD analysis at twenty eight days on all the mixes.

(a) CC mix (b) CCM mix

(c) CSC mix (d) CSCM mix
Table 4. Maximum intensity of compounds from XRD analysis.

| Compounds (Peak Intensity) | Zero Day | One Day | 3 Days | 7 Days | 28 Days |
|---------------------------|----------|---------|--------|--------|--------|
| CC Mix                    |          |         |        |        |        |
| C-S-H                     | 350–400  | 750–800 | >2500  | 650–675| 950–1000 |
| Ca(OH)\textsubscript{2}   | 300–325  | 500–550 | <500   | 350–375| 750–800 |
| Ettringite                | 150–175  | 100–150 | <250   | 100–125| Nil    |
| CCM Mix                   |          |         |        |        |        |
| C-S-H                     | 300–325  | 550–600 | 375–400| 1400–1450| 900–950 |
| Ca(OH)\textsubscript{2}   | 275–300  | 350–400 | 225–250| 1300–1350| 600–650 |
| Ettringite                | 075–100  | <100    | 100–125| 100–150| 100–150 |
| CSC Mix                   |          |         |        |        |        |
| C-S-H                     | 400–425  | 450–500 | 475–500| 400–425| 400–425 |
| Ca(OH)\textsubscript{2}   | 200–225  | 400–450 | 350–375| 425–450| 250–275 |
| Ettringite                | 175–200  | <100    | 125–150| Nil    | 150–175 |
| CSCM Mix                  |          |         |        |        |        |
| C-S-H                     | 525–550  | 400–450 | 525–550| 400–425| 650–700 |
| Ca(OH)\textsubscript{2}   | 275–300  | 250–300 | 300–325| 350–375| 200–250 |
| Ettringite                | 125–150  | 050–100 | 125–150| 100–125| 100–150 |

6.3. Concrete Mixes’ Workability and Density

True slump patterns were found for all mixes used in this study during the conducted slump tests. That is the concrete mass evenly dropped all around without any disintegration. Therefore, it can be specified that use of M-sand in place of R-sand produces cohesive mixes and does not show any segregation characteristics, even in case of CSC mix. For the CC, CCM, CSC and CSCM mixes used, slump values are found to be 8, 0, 5 and 5 mm, respectively. Likewise, the compaction factor values are found to be 0.91, 0.88, 0.89 and 0.86, respectively, for the CC, CCM, CSC and CSCM mixes. From these two different tests, according to the slump test, the degree of workability of concrete mixes are low [54] since these slump values are less than 25 mm, but, when the compaction factor value is considered, the degree of workability of concrete mixes are medium [57]. However, a vibrating technique was used for compaction purposes and hence none of the mixes experience any difficulties for compaction and finishing of concrete specimens.

The workability of concrete mixes produced with M-sand is reduced compared to R-sand-containing concrete mixes and it this was observed for both CC and CSC mixes. The reason is that the R-sand has a smooth texture and better shape and hence renders more workability, while and shows less workability. Another reason is that in general it is considered about the water absorption of coarse aggregate and moisture content of fine aggregate should be used for the production of concrete. Normally, water absorption of fine aggregate is negligible compared to coarse aggregate, but in this study M-sand is used as a fine aggregate in which its parent material is crushed rocks and therefore the water absorbing nature is influenced to reduce the concrete workability compared to R-sand. The concrete density in the fresh state of the mixes CC, CCM, CSC and CSCM was 2480, 2585, 2035 and 2165 kg/m\textsuperscript{3}, respectively. Though an equal volume of M-sand is replaced for R-sand as a fine aggregate, because of the higher density of M-sand compared to R-sand, the density of the concrete mixes produced with M-sand is higher. Similar trends are found in the literature [58–64]. If CCM fresh concrete mix density is compared with CC fresh mix concrete density, then the CCM mix density is 4.23% higher than the CC mix density. Similarly, if CSCM concrete mix density is compared with CSC mix concrete density, then CSCM mix density is 6.39% higher than CSC mix density.
6.4. Concrete Density and Strength

Hardened concrete density and compressive strength of concrete CC, CCM, CSC and CSCM are presented in Table 5. If at 28 days, CCM hardened concrete mix density is compared with CC hardened mix concrete density, then CCM mix density is 3.55% higher than CC mix density. Similarly, if CSCM concrete mix density is compared with CSC mix concrete density, then CSCM mix density is 8.27% higher than CSC mix density. This increase of hardened concrete density as the age increases is due to the enhancement of microstructural characteristics of pozzolanic reaction of concrete cementitious like the conversion of long and slender ettringite during the early hours into large prismatic crystal (CH) and then into short fibrous crystals (C-S-H) and then into close clusters and reticula network due to C-S-H formation in later stages which reduces the pores present and also removes the pore water present earlier and hence the density of hardened concrete increases. This is also proved from the SEM analysis discussed in the Section 6.2.1. Generally, when CS is used it may lead to a lightweight concrete (LWC). Various standards classify LWC differently. As per ACI 318-14, concrete having dry density lesser than 1840 kg/m$^3$ can be classified as LWC [65], the British Standard EN 13,055—Part 1 categorize LWC as that whose dry density is less than 2000 kg/m$^3$ [66] or not greater than 2200 kg/m$^3$ as given in clause 11.1.1 of BS EN 1992-1-1 [67]. Therefore, any concrete produced with a density less than 2200 kg/m$^3$ concrete can be considered as LWC and hence both CSC and CSCM mixes can be grouped under LWC.

Table 5. Hardened concrete properties of mixes used.

| Test Age | CC Mix | CCM Mix | CSC Mix | CSCM Mix |
|----------|--------|---------|---------|----------|
| Density (kg/m$^3$) | Strength (N/mm$^2$) | Density (kg/m$^3$) | Strength (N/mm$^2$) | Density (kg/m$^3$) | Strength (N/mm$^2$) |
| 3-days | 2440 | 16.95 | 2610 | 19.80 | 1965 | 16.80 | 2095 | 17.40 |
| 7-days | 2445 | 21.40 | 2620 | 25.65 | 1975 | 19.90 | 2110 | 22.30 |
| 28-days | 2535 | 28.80 | 2625 | 33.15 | 1995 | 26.95 | 2160 | 29.80 |

The use of M-sand in place of R-sand does not encourage a reduction of the strength of the concrete mixes because M-sand has sharp-edged, angular texture particles and this creates better particle to particle interlocking which also develops bonding with the cement mortar and thereby increases the strength. Also, strength development due to increase in age is traditional and in absolutely no doubt regarding the strength development of M-sand-containing mixes. Strength of CSCM at 28 days increased by 10.57% compared to CSC and CCM at 28 days increased by 15.10% compared to CC mix. Therefore, there is no need to hesitate to use M-sand as a 100% replacement alternative for R-sand in the production of concrete on an equal volume basis for the selected mix proportion from a strength point of view. The same trends of enhancement of strength of concrete mixes when using M-sand in place of R-sand were found in the reports published on the use of this kind of M-sand and quarry dust by other researchers [58–64].

6.5. Mechanical Properties

Apart from the compressive strength test results of other properties such as flexural strength, splitting tensile strength and impact resistance of concrete mixes results are presented in Table 6.
Table 6. Hardened concrete properties of mixes used.

| Test Age | CC Mix   | CCM Mix  | CSC Mix  | CSCM Mix |
|----------|----------|----------|----------|----------|
|          | Flexural strength (N/mm$^2$) |          |          |          |
| 3 days   | 2.55     | 2.95     | 2.46     | 3.20     |
| 7 days   | 3.42     | 4.10     | 3.06     | 3.70     |
| 28 days  | 4.78     | 5.38     | 4.60     | 5.20     |

|          | Splitting tensile strength (N/mm$^2$) |          |          |
| 3 days   | 2.25     | 2.36     | 1.80     | 1.88     |
| 7 days   | 2.64     | 2.82     | 2.30     | 2.36     |
| 28 days  | 3.42     | 3.70     | 2.70     | 2.92     |

| Impact resistance in (Joules) | Initial crack | Final crack | Initial crack | Final crack | Initial crack | Final crack | Initial crack | Final crack |
|-------------------------------|---------------|-------------|---------------|-------------|---------------|-------------|---------------|-------------|
| 3 days | 159         | 179         | 198           | 218         | 218           | 278         | 298           | 377         |
| 7 days | 238         | 318         | 318           | 377         | 377           | 477         | 457           | 556         |
| 28 days | 318        | 417         | 397           | 477         | 497           | 636         | 616           | 735         |

6.5.1. Flexural Strength

Flexural strength of CSCM mix at 28 days is 5.20 N/mm$^2$ (17.45% of its compressive strength) which is increased by 13.04% compared to CSC mix and flexural strength of CCM at 28 days is 5.38 N/mm$^2$ (16.23% of its compressive strength) which is increased by 12.55% compared to CC mix. In general it is traditional that the flexural strength of concrete may lie in between 10 to 15% of the respective compressive strength of CC mixes [40] and the same was approximately seen in this study also, but in the case of CSC and CSCM mixes it is slightly higher, which is due to the fibrous nature of CS which increases the flexural strength and hence the abovementioned statement about a 10 to 15% range is not applicable for concrete mixes in which CS is used. The Indian Standard IS 456: 2000 [68] provides a theoretical formula to calculate the approximate flexural strength with respect to compressive strength of concrete, i.e., flexural strength $f_b$ equal to $0.7 \sqrt{f_{ck}}$, where $f_{ck}$ is compressive strength. Compared to IS 456: 2000 recommendation, flexural strength of CC, CCM, CSC and CSCM mixes are increased by an amount of 27.13, 33.50, 26.72 and 36.12%, respectively. Therefore, to predict the flexural strength of CS and M-sand-containing concrete, this IS 456:2000 standard recommendation can be used by considering the compressive strength of concrete so that the actual flexural strength of those mixes will be definitely more than the predicted value. From this flexural study, it can also be stated that the general assumption reinforces the notions about the behavior of CSCM concrete mix in comparison with CC, CCM and CSC mixes.

6.5.2. Splitting Tensile Strength

Splitting tensile strength of CSCM mix at 28 days is 2.92 N/mm$^2$ (9.80% of its compressive strength) which represents an increase by 8.15% compared to CSC mix and the splitting tensile strength of CCM at 28 days is 3.70 N/mm$^2$ (11.16% of its compressive strength) which is an increase by 8.19% compared to CC mix. From this splitting tensile strength study it can also be stated that the general assumption reinforces the behavior of the concrete mix CSCM in comparison with CC, CCM and CSC mixes.

6.5.3. Impact Resistance

At 28 days, the average impact resistance of CC mix (16 blows) to form initial cracks is 318 Joules and 417 Joules (21 blows) to final failure of specimens. Likewise, at 28 days, the
The average impact resistance of CSC mix was 497 Joules (25 blows) to form initial cracks and 636 Joules (32 blows) to final failure of specimens. Similarly, at 28 days, the average impact resistance of CCM mix was 398 Joules (20 blows) to form initial cracks and 477 Joules (24 blows) to final failure of specimens. Likewise, at 28 days, the average impact resistance of CSCM mix was 616 Joules (31 blows) to form initial cracks and 736 Joules (37 blows) to final failure of specimens. The enhancement of impact resistance both in initial and final failure of both CSC and CSCM mixes are due to the presence of long continuous (Figure 29a) and discrete fibrous material (Figure 29b) in the internal microstructure of CS used which is not available in the case of CCA. However, overall use of M-sand both in CCM and CSCM mixes impacts resistance more compared to CC to CSC mixes. This is because of the strength enhancement of M-sand concrete. Therefore, it can be stated that both the strength and nature of the internal structure of aggregates used are also the deciding parameters for impact resistance of concrete mixes. Figures 30 and 31 show the graphical representation of impact resistance in terms of Joules to form initial cracks and final failure at different ages of concrete mixes used in this study, respectively.

![Figure 29. (a) Long continuous (b) discrete fibrous present in CS.](image)

![Figure 30. Impact resistance of concrete mixes at initial failure.](image)
Figure 31. Impact resistance of concrete mixes at final failure.

For the benefit of the readers, the characteristics of R-sand and M-sand are compared in table form and presented in Table 7 in order to make very clear about the present study findings.

Table 7. Comparison of R-sand and M-sand characteristics.

| Characteristics                  | R-Sand          | M-Sand          |
|----------------------------------|-----------------|-----------------|
| Surface structure                | Smooth texture  | Rough texture   |
| Moisture                         | May be available| Not available unless it is wet sieved |
| Silt content                     | Possible to present 5–20% | Not available (0%) |
| Adulteration                     | More possibility | Less possibility |
| Over sizes                       | Cannot be avoided | Can be avoided |
| FTIR pattern                     | Similar patterns |                 |
| Bonding nature                   | Single bonding  |                 |
| Cement hydration processes       | Not affected    |                 |
| Ratio (Ca: Si) at 28 days for CSA used concrete | 1.86 | 1.71 |
| Ratio (Ca: Si) at 28 days for CS used concrete | 1.69 | 1.54 |
| Segregation characteristics      | Does not happened |                 |
| Workability                      | Increases       | Decreases       |
| Concrete density                 | Less            | More            |
| Concrete strength                | Less            | More            |

Results of microstructural studies on this new type of composite material with M-sand and CS in concrete production is concurrent with the traditional concrete material behaviour and hence it has advantages because these two materials are innovative products
made from renewable resources and waste as sustainable alternate materials. Also, it can be noted that the mechanical properties such as compressive strength, split tensile strength, flexural strength and impact resistance of concrete made with M-sand both in CC and CSC are higher compared to the other studies in which R-sand and quarry dust (QD) were used with CS [40–43]. However, this study is limited to only mechanical properties and hence other significant properties such as bonding, durability, plastic and drying shrinkage, deflection characteristics, cracks and strain compatibility of structural elements require further study to draw firm conclusions about the advantages.

7. Conclusions

This study found that M-sand is a sustainable alternate fine aggregate from a renewable source that can be used in place of R-sand and CS is a sustainable alternate coarse aggregate in place of CSA for concrete production from agricultural waste. Although M-sand is produced from usual aggregate resources, it causes less harm to the environment compared to R-sand because extraction of R-sand is bad for the environment and ecobalance, decreasing groundwater levels and drying rivers. Similarly, the use of CS in the production of concrete has dual advantages: one is minimizing the solid waste going to landfills and simultaneously solving the environmental degradation issue. Finally, a strong recommendation is that M-sand can be used in its place of R-sand as an alternate sustainable material for the production of both CC and CSC and there should be no doubt that this study has found sustainable alternate materials for concrete production from renewable resources and waste.

From the research carried out the following conclusions are drawn from an engineering properties point of view:

- M-sand has zero silt, is not oversized and less probability of adulteration compared to river sand (R-sand). M-sand has a rough surface texture and an angular shape which are advantages for strength development compared to R-sand.
- Both R-sand and M-sand have similar chemical structures. The major IR bands of both R-sand and M-sand lay between 400 and 1500 cm$^{-1}$, hence it can be stated that these materials have single bonded structures.
- SEM images analysis show that a systematic hydration process takes place in both R-sand and M-sand mixes. Hence it can be specified that the use of M-sand in place of R-sand does not affect the conventional cement hydration process both in CC and CSC.
- EDS analysis proves that Ca:Si is higher during early days and decreases as the age increases. At an age of 28 days, the Ca:Si ratio for all four tested mixes lies in between 1.5 to 2.0, especially in the case of CSC and CSCM compared to CC and CCM which indicates and stresses that the systematic hydration process have taken place in mixes containing both R-sand and M-sand.
- The XRD results supported the SEM and EDS results of the CC, CCM, CSC and CSCM mixes. Compared to the formation of the compounds Ca(OH)$_2$, C-S-H compounds’ intensity counts are high, which shows that there is no interruption of pozzolanic reactions and that has a significant positive influence on the reduction of Ca(OH)$_2$–(CH) in all the mixes, which is beneficial for the quality of concrete strength.
- The use of M-sand reduces the workability and increases the density in both CC and CSC. Use of M-sand intensified the mechanical properties of both CC and CSC compared to R-sand.
- The flexural strengths of CSC and CSCM mixes are slightly higher than the traditional range of 10 to 15% of compressive strength of the respective mixes. This is because of the fibrous nature of CS and hence this is not applicable for concrete mixes where CS is used.
- Both flexural strength and splitting tensile strength studies show that the general assumptions reinforces the behavior of concrete mix CSCM in comparison with CC, CCM and CSC mixes.
• There is an enhancement of impact resistance both in initial and final failure of both CSC and CSCM mixes due to the presence of long continuous and short discrete fibrous material present in the CS internal microstructure.

• Both the strength and nature of the internal structure of aggregates are also the deciding parameters for the impact resistance of concrete mixes.

Overall, this study finds that the use of M-sand as fine aggregate and CS as coarse aggregate in the production of concrete is feasible and these two materials are definitely a sustainable alternative materials for concrete manufacture. Microstructural study results on M-sand and R-sand in combination with CS are the most important contribution of this study. This study was only limited to the mechanical properties and hence bonding properties, durability properties, structural elements like beams under flexure, shear, torsion, plastic shrinkage and drying shrinkage, deflection characteristics of slabs, compression members under both static and dynamic loads could be some of the future research lines in this area. Finally, it can be strongly recommended without any hesitation that in practice M-sand be used in place of R-sand and CS be used in place of CSA for both structural and non-structural concrete elements.

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