Flexural Strength of Crushed Rock Dust Concrete at Elevated Temperatures

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Abstract. This experimental study presents the effect of partial replacement of Ordinary Portland Cement (OPC) by crushed rock dust (CRD) as filler material on the flexural strength of concrete when subjected to elevated temperatures of 200 °C, 400 °C, 600 °C and 800 °C for duration of 2 hours using an electrically controlled furnace. The OPC replacement percentages are: 0% (C40), 10% (C41), 20% (C42), 30% (C43) and 40% (C44) by weight. Ultrasonic pulse velocity (UPV), Mass loss, flexural strength, and scanning electron microscope (SEM) are evaluated at the targeted elevated temperatures. At ambient temperature, up to 40% CRD, a dense microstructure with less pores is observed using SEM micrographs. No visible cracks are observed on the beams specimens of C40 and CRD concrete beams, when exposed up to 400 °C. Both C40 and CRD concrete beams begin to crack when temperature reached to 600 °C and pronounced surface cracks are observed at 800 °C. UPV values obtained with C40 and CRD concrete beams at elevated temperatures are in good agreement with flexural strength and mass loss values. SEM micrographs signify the use of CRD in concrete at elevated temperatures. The results of C40 and CRD concrete beams at elevated temperatures are found to be acceptable when exposed up to 400 °C.

1. Introduction
Concrete when exposed to fire or elevated temperature creates a severe potential problem to the safety of the structure. Concrete is an incombustible construction material than wood and steel, but when exposed to elevated temperatures, its cement matrix and constituent properties are affected, resulting in the deterioration of its physical, mechanical and durability properties [1]. The main factors influencing the behaviour of concrete at elevated temperatures are moisture content, type of aggregates, relative proportions of cement and aggregates, thermal compatibility between cement paste and aggregate, peak temperature and exposed duration as well as size and shape of member [2]. The deterioration of concrete at higher temperatures includes crack formations, causing spalls, large pores and reduction of bonding between cement matrix and aggregates. This results in the development of high internal tensile stresses causing damage, cracking and a significant reduction in compressive strength. Hence, the residual compressive strength of concrete exposed to fire plays an important role to signify the suitability [3].

The thermal parameters such as specific heat, the coefficient of thermal expansion, thermal conductivity and diffusivity are similar to normal strength concrete and high strength concrete, but some studies showed poor performance and high risk of spalling of high strength concrete at elevated temperatures due to its high brittleness and low permeability. Concrete spalling at elevated temperatures is majorly influenced by moisture gradients and free water. However, concrete with 3
4% of moisture by weight has a high risk of spalling than concrete having moisture content less than 3% by weight. On the other hand, high performance concrete or dense microstructure concrete having zero moisture content may spall at elevated temperatures [4-5]. It is well known that concrete has great vulnerability towards spalling when prepared with low water to cement ratio than high water to cement ratio. The changes occur in cement paste when exposed to fire or elevated temperature is as follows: (1) The eviccation of evaporable water at 100°C temperature. (2) At a temperature of 180°C, the hydrates of calcium silicate start dehydration. (3) The disintegration of calcium hydroxide takes place at 500°C. (4) At around 700°C, the decomposition of calcium-silicate-hydrate begins. (5) At the elevated temperature of 400°C, calcium hydroxide in hardened cement paste of concrete starts dissociation, and the change persists up to the entire obliteration of the calcium-silicate-hydrate gel at around 900°C [5-7].

From the extensive review of literature, it may be noticed that the flexural strength of CRD concrete at elevated temperatures are not studied till date. Hence, in the present experimental study, the flexural strength of concrete at elevated temperatures with partial replacement of OPC with CRD is studied. The targeted elevated temperatures of 200 °C, 400 °C, 600 °C and 800 °C are maintained for 2 hours to attain a thermal constancy of specimens between the inner core and outer part of the concrete specimens.

2. Experimental Program

2.1. Materials

Ordinary Portland Cement (OPC) 53 grade conforming to IS 12269:1987 [8] was used. Its standard consistency is 32% and specific gravity is 3.1. Locally available Krishna river sand (maximum size 4.75mm) conforming to zone III of IS 383:1970 [9] was used. Crushed stone aggregate of maximum size 20 mm conforming to IS 383:1970 [9] was used. CRD collected from nearby stone processing units was used. CRD passing through 150µm sieve was used for partial replacement of cement in concrete. The physical properties of fine aggregate, coarse aggregate and CRD are shown in Table 1.

| S.No. | Physical Property | Result |
|------|-------------------|--------|
| 1.   | Fine aggregate    |        |
|      | (a) Specific gravity | 2.62   |
|      | (b) Fineness modulus | 2.65   |
|      | (c) Water absorption (%) | 1.0 % |
| 2.   | Coarse aggregate  |        |
|      | (a) Specific gravity | 2.88   |
|      | (b) Fineness modulus | 6.88   |
|      | (c) Water absorption (%) | 0.50 % |
| 3.   | Crushed Rock Dust |        |
|      | (a) Specific gravity | 2.52   |
|      | (b) Water absorption (%) | 1.52 % |

2.2. Concrete Mix design

Concrete target strength of 40 MPa designed as per latest IS code, IS 10262: 2009 [10] was considered as control concrete (C40) mix. The concrete mixture proportion obtained was 1:1.39:2.91 (OPC: natural sand: coarse aggregate) with 0.38 as water to binder ratio. The details of concrete mixes are given in Table 2.
Table 2. M40 Grade: Concrete mix proportions

| Mix designation | CRD (%) | CRD (kg/m³) | Cement (kg/m³) | Fine aggregate (kg/m³) | Coarse aggregate (kg/m³) |
|-----------------|---------|-------------|---------------|------------------------|-------------------------|
| C40             | 0       | 0           | 450           | 625.5                  | 1309.5                  |
| C41             | 10      | 45          | 405           | 625.5                  | 1309.5                  |
| C42             | 20      | 90          | 360           | 625.5                  | 1309.5                  |
| C43             | 30      | 135         | 315           | 625.5                  | 1309.5                  |
| C44             | 40      | 180         | 270           | 625.5                  | 1309.5                  |

2.3. Experimental Procedure

After mixing the concrete constituents uniformly, the fresh concrete is poured into 100 x 100 x 500 mm beam moulds and was vibrated for one minute on a table vibrator to remove air voids. The specimens were kept undisturbed in the laboratory for 24 hours from casting and then de-moulded, cured for 28 days using water immersion method and then both C40 and CRD concrete beams were kept air dried for 7 days and then heated in an electrically controlled furnace, which is capable of up to 1200 °C. The targeted temperatures considered were 200 °C, 400 °C, 600 °C, and 800 °C for 2 hours of exposure duration with a heating rate of 10 °C /min to achieve thermal stability between outer and inner layers of specimens [11]. The power supply of furnace was switched off at the end of 2 hours of exposure at the targeted temperatures, and then the specimens were left in furnace with door opened for slow cooling in air for 24 hours. The mass loss of specimens was estimated through measuring the mass of specimens before and after heat exposure. A fully computerized universal testing machine, having a maximum loading capacity of 1000 kN was used to measure the flexural strength of both heated and unheated beam specimens. A uni-axial load of 0.5 MPa/sec was applied. The UPV values of concrete beam specimens are measured before and after subjection to targeted temperatures in accordance with IS: 13311 (Part 1): 1992 [12]. The UPV testing equipment consists of one transmitter and one receiver head of 54 kHz. The X-ray diffraction analysis (XRD) was performed on CRD used for partial replacement of cement by using XRD meter Rigaku Miniflex 600. SEM analysis of C40 and CRD concrete was conducted before and after subjection to elevated temperatures by using SEM having Tungsten heated filament as electron source. Three test samples are collected from each specimen with less than 1 cm in dimension having blocky shape with smooth and regular surface.

3. Results and Discussion

3.1. Crushed rock dust

Fig. 1 shows the XRD pattern and SEM micrograph of CRD used for partial replacement of cement. From the XRD pattern of CRD, it may be noticed a peak intensity count between 20° and 30°, indicated the presence of quartz (SiO₂). The SEM morphology of CRD showed that it consists of angular and irregular particles. The chemical compositions of CRD used are presented in Table 3. The sum of Al₂O₃, SiO₂ and Fe₂O₃ that are present in CRD is 87.86% which is greater than 70%, satisfying the requirement of ASTM C 618 [13]. The sum of SiO₂ and Al₂O₃ that are presented in CRD is 84.46% of the total mass. The percentages of aluminate and silicate in the CRD indicate some pozzolanic reactivity.
3.2. Mass loss

The mass loss of M40 grade concrete specimens after exposure to elevated temperatures are shown in Table 4. The average mass loss of the five samples (C40, C41, C42, C43 and C44) at 200 °C, 400 °C, 600 °C and 800 °C was 2.89%, 4.70%, 6.66% and 8.83%, respectively. The reduction in mass of C40 and CRD concrete samples at elevated temperatures conform the deterioration of structural integrity of samples. Mass loss in the beam specimens at below 400 °C is less due to the evaporation of free water and capillary water. When concrete is exposed to above 400 °C, the decomposition of C-S-H gel takes place and hydrated water in C-S-H gel releases resulting in mass loss of concrete samples. At 800 °C, both C40 and CRD concrete beams experienced highest mass loss, which may be attributed to dissociation of the interaction transition zone between aggregates and cement paste. In general, the mass loss of heat exposed concrete specimens occurs due to the eviction of hydrated water from hardened cement matrix, which leads to the formation of air voids in the concrete.

Table 4. Mass loss of M40 grade concrete at elevated temperatures

| Mix designation | CRD Content (%) | Mass loss (%) @ 200 °C | Mass loss (%) @ 400 °C | Mass loss (%) @ 600 °C | Mass loss (%) @ 800 °C |
|-----------------|-----------------|------------------------|------------------------|------------------------|------------------------|
| C40             | 0               | 2.62                   | 4.32                   | 6.43                   | 8.62                   |
| C41             | 10              | 2.74                   | 4.44                   | 6.51                   | 8.67                   |
| C42             | 20              | 2.76                   | 4.63                   | 6.55                   | 8.74                   |
| C43             | 30              | 3.09                   | 4.96                   | 6.87                   | *E.S                   |
| C44             | 40              | 3.28                   | 5.18                   | 6.96                   | *E.S                   |

3.3. Flexural strength

Table 5 shows the flexural strength of M40 grade concrete beam specimens at ambient temperature and after exposure to elevated temperatures. At ambient temperature, the 28-day flexural strength of C41, C42, C43 and C44 concrete beams are 105.23%, 106.92%, 103.36% and 97.57% that of C40 (5.35 MPa), respectively. The 28-day flexural strength of C40, C41, C42, C43 and C44 concrete beams at 200°C are retained by 86.92%, 89.70%, 88.11%, 88.07% and 85.06%, respectively when compared with the flexural strength at ambient temperature. The 28-day flexural strength of C40, C41, C42, C43 and C44 concrete beams at 400°C are retained by 65.42%, 58.97%, 61.71%, 56.42% and 56.32%, respectively when compared with the flexural strength at ambient temperature. The 28-day flexural strength of C40, C41, C42, C43 and C44 concrete beams at 600°C are retained by 41.50%, 38.72%, 42.48%, 35.62% and 35.06%, respectively when compared with the flexural strength.
at ambient temperature. The 28-day flexural strength of C40, C41 and C42 concrete beams at 800°C are retained by 21.31%, 18.65%, and 16.43%, respectively when compared with the flexural strength at ambient temperature. At 800°C, the flexural strength values of C43 and C44 specimens are not obtained since these specimens are broken due to spalling.

Table 5. Flexural strength of concrete

| Mix designation | CRD (%) | Average flexural strength @ 28 days (MPa) | Average flexural strength (MPa) after exposed to elevated temperatures |
|-----------------|---------|------------------------------------------|---------------------------------------------------------------------|
| C40             | 0       | 5.35                                     | 4.65, 3.50, 2.22, 1.14                                               |
| C41             | 10      | 5.63                                     | 5.05, 3.32, 2.18, 1.05                                               |
| C42             | 20      | 5.72                                     | 5.04, 3.53, 2.43, 0.94                                               |
| C43             | 30      | 5.53                                     | 4.87, 3.12, 1.97, *E.S                                               |
| C44             | 40      | 5.22                                     | 4.44, 2.94, 1.83, *E.S                                               |

*E.S = Explosive spalling

The decrease in the flexural strength values of concrete between 200°C and 400°C, could be due to the steady dehydration that occurs within the cementitious material, thus changing the saturated surface of concrete to dry state. The loss in flexural strength between 500°C and 600°C is attributed to the cement matrix volumetric expansion caused due to the desiccation of C-S-H gel and the change in chemical composition of Ca(OH)₂ to CaO. Furthermore, the shrinkage of the cement paste occurs due to the release of hydrated water and aggregates expansion, resulting in the impairing of the cohesion between cement paste and aggregate. Fig. 2 shows a view of C40 and C42 beam specimens exposed to a target temperature of 600°C for 2 hours of duration. Fig. 3 shows a view of beam specimens exposed to 800°C.

**Figure 2.** A view of beam specimens exposed to 600°C for 2 hours of duration.
Between 400 °C and 800 °C, concrete microstructure disintegrates due to the disintegration of calcium hydroxides and also the desiccation of CSH gel beyond 450 °C thus declining the aggregates and cement matrix strength. Concrete, when exposed to 800 °C, undergoes high physic-chemical transformation which leads to the formation of new re-crystallized compounds and also tends to increase shrinkage or expansion between concrete constituents. The results revealed that the flexural strength values of concrete decreased gradually with the increase in temperature. In brief, the thermal incompatibility between cementitious material and the aggregates at elevated temperatures results in the physical decomposition of cohesion between them which tends to further strength loss of concrete.

3.4. Ultrasonic pulse velocity
Table 6 shows the UPV values and quality grading of M40 grade concrete specimens, in accordance to IS 13311 (Part 1): 1992 [12]. At 800 °C, UPV values of C43 and C44 specimens are not obtained since these specimens are broken due to spalling. The UPV values of M40 grade concrete beam specimens at 28-day curing age lies within the range of excellent quality. A good relationship is observed between the flexural strength and UPV values with the related factor ($R^2$) of 0.983 as shown in Fig. 4. As flexural strength values, UPV values also decreased with the increase in elevated temperature. This is due to the development of porosity in concrete when exposed to high temperature. A minimal decrease in UPV values is noticed at 200 °C temperature which may be due to the dehydration of cementitious material and loss of free water. A sharp decrease in UPV values is noticed between 400 °C and 600 °C which could be due to dehydration of CSH gel and also due to the aggregates phase transformation. In brief, the concrete UPV values decreases due to the degradation of concrete microstructure at elevated temperatures.

| Mix designation | CRD (%) | Average UPV (km/s) @ 28 days | Average UPV values (km/s) after exposed to elevated temperatures |
|------------------|---------|-----------------------------|---------------------------------------------------------------|
|                  |         | @ 200°C @ 400°C @ 600°C @ 800°C |                                                               |
| C40              | 0       | 4.74 (E) 4.42 (G) 3.72 (G) 2.53 (D) 1.76 (D) |
| C41              | 10      | 4.82 (E) 4.41 (G) 3.64 (G) 2.55 (D) 1.64 (D) |
| C42              | 20      | 4.93 (E) 4.41 (G) 3.61 (G) 2.74 (D) 1.66 (D) |
| C43              | 30      | 4.85 (E) 4.32 (G) 3.48 (M) 2.23 (D) E.S |
| C44              | 40      | 4.73 (E) 4.12 (G) 3.26 (M) 2.06 (D) E.S |

E = Excellent; G = Good; M = Medium; D = Doubtful; E.S = Explosive spalling
Figure 4. Correlation between flexural strength and UPV values of M40 grade concrete exposed to elevated temperatures

3.5. Scanning Electron Microscope Analysis

Fig. 5 shows the SEM micrographs of M40 grade concrete, at ambient temperature. From the SEM micrographs obtained at ambient temperature, it can be observed that the internal microstructure is dense and intact with less pores resembles the concrete in good condition. The formation of hydration products, secondary CSH gel and the micro filling capacity of CRD resulted in the dense microstructure of concrete. At ambient temperature, up to 40% CRD replacement, a dense microstructure with less pores is observed using SEM micrographs. This shows that the use of CRD as filler material in concrete is favorable for increasing the internal microstructure which results in increase of properties of concrete.

From the SEM micrographs of concrete exposed to 200 °C, it is observed that the internal microstructure is still dense with less micro pores developed due to the steady dehydration that occurs in CSH gel. The change in the internal microstructure of concrete after exposure to 200 °C could be due to the steady dehydration that occurs within the cementitious material, thus changing saturated surface of concrete to dry state leading to the development of micro pores. From the SEM micrographs of concrete exposed to 400 °C, it is observed that there is a little deterioration in the microstructure of concrete with micro-spaces and micro-pores. This could be due to steady dehydration and loss of chemically bonded water and free water in the cement matrix. Furthermore, the decomposition of CSH gel occurs at 400 °C that leads to the development of internal cracks and pores.

From the SEM micrographs of concrete exposed to 600 °C, it is observed that there is a significant deterioration in microstructure bonding with macro-spaces and macro-cracks. This could be attributed to the cement matrix volumetric expansion that was caused due to the desiccation of CSH gel and the change in chemical composition of Ca(OH)$_2$ to CaO. Furthermore, the shrinkage of the cement paste occurs due to the release of hydrated water and aggregates expansion, resulting in the impairing of the cohesion between cement paste and aggregates.

Fig. 6 shows the SEM micrographs of M40 grade concrete at 800 °C target temperature. From the SEM micrographs it can be observed that the concrete exposed to 800 °C target temperature that there is enlarged cracks with more macro-pores and with less intact. This could be due to the disintegration of the microstructure of concrete due to the disintegration of calcium hydroxides and also the desiccation of CSH gel arises beyond 450 °C that declines the strength of cement matrix and coarse aggregates. Furthermore, at 800 °C exposure temperature, the concrete undergoes high physico-chemical transformation which leads to the formation of new re-crystallized compounds also tend to increase shrinkage or expansion between concrete constituents.
Figure 5. SEM micrographs of M40 grade concrete at ambient temperature with
(a) C40 (b) C41 (c) C42 (d) C43 (e) C44
4. Conclusions

- At ambient temperature, up to 30% CRD replacement ratio, both flexural strength and UPV values are found higher than those of C40.
- At ambient temperature, up to 40% CRD replacement ratio, a dense microstructure with less pores is observed using SEM micrographs. This shows that the use of CRD as filler material in concrete is favorable for increasing the internal microstructure which results in increase of properties of concrete.
- No visible cracks are observed on the beams of C40 and CRD concrete surface exposed up to 400 °C. Both C40 and CRD concrete beams begin to crack when temperature reached to 600 °C and pronounced surface cracks are observed at 800 °C.
- Mass loss in concrete beams below 400 °C is less due to the evaporation of capillary water and free water. A higher mass loss is observed between 600 °C and 800 °C with C40 and CRD concrete beams.
• A minimal decrease in UPV values is noticed at 200 °C temperature. Between 400 °C to 600 °C a sharp decrease in UPV values is noticed. UPV values obtained with C40 and CRD concrete beams at elevated temperatures are in good agreement with flexural strength and mass loss values.

• In particular, the sharp decrease in flexural strength of C40 and CRD concrete at 600 °C and 800 °C than at 400 °C, may be the result of development of enlarged cracks with less intact of microstructure of concrete at elevated temperatures and the results are supported by SEM analysis.

• SEM micrographs signify the use of CRD in concrete at elevated temperatures. The results of C40 and CRD concrete beams at elevated temperatures are found to be acceptable when exposed up to 400 °C.

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