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Research Article

Effect of Elevated Temperature on the Properties of Self-Compacting Mortar Containing Nanomaterials and Zircon Sand

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The present research work tries to assess the performance of a self-compacting mortar containing zircon sand as a substitute for river aggregate in combination with nanoalumina and nanosilica as cement replacements. The fresh state results, as observed through the mini slump cone and mini V funnel, showed positive effects of zircon sand on workability attainment. The EFNARC limits of workability were even satisfied at high substitution levels of the nanoparticle due to the contribution of zircon sand. The mechanical properties, durability, and microstructure of the mortar were evaluated by conducting experiments at room temperature and then at 200°C, 400°C, 600°C, and 800°C. Results show that there was a significant improvement in the thermal stability of the RPC mixes due to the synergistic effect of nanomaterials and zircon sand addition. The addition of nanomaterials and zircon sand accelerated the microstructural buildup and durability at elevated temperatures. The findings thus suggest a novel and effective approach to using zircon sand as a potential alternative to quartz sand in RPC in combination with nanomaterials to produce temperature-resistant concrete structures.

1. Introduction

Fire is one of the most serious threats that any concrete structure can face in its lifetime [1]. The damage caused by fire is vast, not only to the environment but also to life and property [2, 3]. Though concrete can sustain greater temperatures, its failure pattern is unpredictable, leading to a catastrophe [4, 5]. The durability studies of concrete also consider fire study as an integral part of their study [6, 7]. Perpetual exposure to heat weakens the integrity of concrete and thus makes it fail all of a sudden [8]. In addition, there are some concrete structures that are always subjected to temperatures higher than those intended for, namely refractory and nuclear purpose concretes. They can undergo various physical and chemical changes during their lifetime, and hence, their temperature stability has to be assessed before they can be used in field applications [9]. Well-developed hydration phases can make the concrete thermally stable [10]. The use of thermally stable aggregates can also contribute to the volume stability of concrete even at higher temperatures. The use of alternative sand should meet the design and ecological requirements as well as possess thermal stability [11]. One such material is zircon sand, which is available in plenty in nature, occurring along the ancient coastlines [12]. They are highly stable at elevated temperatures and are also chemically inert. The chemical and thermal stability of zircon sand makes it a widely used material for use in thermal applications and foundries [13].

The use of zircon sand as fine aggregates should reduce the demand for natural river aggregates while also meeting aggregate thermal requirements.

Though self-compacting concrete is a universally accepted material, it also possesses several disadvantages, such as the presence of voids and pores, which form channels for the ingress of harmful ingredients into concrete due to the lack of any external vibration techniques [7]. Because self-compacting concrete is commonly used as a filling agent in reinforced concrete structures, any formation of microcracks allows CO_2, chlorides, and moisture to enter the structure [14]. Concrete reinforced with steel is not very resistant to such harmful substances. Therefore, to minimize the risk caused to the reinforcements, the usage of ultrafine and nanomaterials for the design of self-compacting concrete becomes an inevitable choice [15–17]. The properties of the self-compacting concrete mainly depend on the mortar phase, and hence, it is important to study if mortar properties explain the concrete's behaviour [18]. Nanomortar is a general term used for mortar containing nanomaterial as an additive [19]. The bulk properties of concrete are significantly improved by the inclusion of nanosized particles in concrete through an improvement in packing capacity [20]. The micropores of concrete are reduced to the nanoscale and nanopores are completely nullified due to nanomaterial addition [21, 22]. The drawbacks found in concrete, such as permeability, porosity, and alkali-silica reaction, are also found to be reduced due to the nanomaterial addition [23, 24]. Hence, more durable and enhanced concrete performance can be attained through the filling capacity of nanoparticles and also through their involvement in the chemical hydration reaction [25]. Since the emergence of nanotechnology, several nanosized particles have been produced by limiting their size to nanolevel without affecting their original chemical composition and physical properties [26]. Nanomaterials have already been used in self-compacting mortars for the past decades to yield sufficient strength and stability. Highly innovative materials have been used in concrete that is of microscale and nanoscale and has yielded good thermal stability and mechanical strength of the mortar. Comparatively, the use of nanosized materials has yielded much more positive results than microsized particles [27]. The use of nanomaterials as a replacement or additive to cement also involves several technological implications. The proper dispersion of nanomaterials in cement should be taken with the utmost care; otherwise, it could lead to negative consequences [28]. The proportion of nanomaterials required to produce a high-quality concrete system also needs to be assessed before they can be incorporated into the concrete system [29].

Realizing the need to eliminate the use of natural aggregates for concrete production and utilising the benefit of the thermal stability of zircon sand for use in concrete structures exposed to extreme temperatures [30], the present research is directed accordingly. The significance of this research lies in examining the characteristics of SCC containing nanomaterials and zircon sand at elevated temperatures, which is the first study of its kind. The utilisation of zircon sand in mortar remains unexplored, and the results obtained from this study will provide enriched information on the contribution of nanomaterials and zircon sand towards the thermal stability of self-compacting mortar. Despite several studies on self-compacting mortar [28], no study has been attempted to characterise the combined effects of adding nanosilica and nanoalumina on the properties of self-compacting mortars with zircon sand aggregates. The study is an initiative to create confidence in the use of zircon sand as an ingredient for the production of self-compacting mortars. The research tries to harness the thermal stability of zircon sand to improve the fire-resistant behaviour of self-compacting mortars. The study also creates a deep understanding of the synergy between the nanomaterials and zircon sand in the strength improvement and workability of SCM. The study also creates confidence in the usage of zircon sand as fine aggregate in SCM and averts the environmental problems associated with the usage of natural sand in concrete production, leading to sustainability in concrete construction.

2. Materials

OPC 53 grade cement is used for the production of self-compacting mortar and conforms to IS 12269-1987. The cement properties were tested as per the requirement of Indian standard 4031-1988 and Indian standard 4032-1985. Analytical grade nanosilica and nanoalumina with 99% purity were used as cement additive. Natural river sand and zircon sand conforming to zone II of BIS 383-1970 are used as fine aggregates in the present study. Chemical oxide composition and physical properties of the raw materials are presented in Table 1.

Zircon sand is finer in nature compared to the natural river sand as shown in the particle size distribution of the aggregate and material images as presented in Figures 1 and 2. The EDS spectrum of river sand and zircon sand is shown in Figure 3. The low coefficient of expansion values of the zircon sand makes them thermally stable and maintains the dimensional stability of the specimens even at higher temperatures. The particle size of the fine aggregates plays a major role in the strength of the cementitious systems. The greater the fineness of the particle, the more improvement in the strength can be obtained up to a certain limit. The zircon sand exhibited a smaller size ($D_{50} = 0.435\,\mu$m) when compared to river sand ($D_{50} = 15.132\,\mu$m), and hence, fewer surface defects on the mortar, coupled with increased mechanical strength. The increase in fineness of zircon sand aggregates showed greater surface area, thereby increasing the area of contact between the cementitious components. Therefore, zircon sand in a smaller size fraction can aid in micropores that can be easily filled by the hydration products, causing a dense microstructure (Sadrmomtazi et al.). The coefficient of expansion of aggregates also plays a major role in the thermal stability of concrete. The silica content present in the aggregates defines the coefficient of thermal expansion values, and it can be seen that the coefficient of thermal expansion value of zircon sand is lower than that of river sand due to its relatively lower silica content. The silica phase of aggregates undergoes expansive phase change at elevated temperatures, and hence, zircon

Table 1: Chemical composition and physical properties.

Oxide	Cement	Zircon sand	River sand
SiO_2	21.56	21.20	96.18
Al_2O_3	6.67	6.911	2.76
Fe_2O_3	6.17	3.155	0.06
CaO	49.88	—	—
MgO	4.51	—	—
SO_3	2.75	—	—
K_2O	0.76	—	—
Na_2O	0.43	—	—
V_2O_5	—	0.384	—
Cr_2O_3	—	0.465	—
ZrO_2	—	67.685	—
Cl	—	<0.005	—
LOI	2.79	<0.01	0.67
Water absorption (%)	—	0.69	0.75
Water content (%)	—	0.07	0.09
Density (kg/m^3)	3150	2790	1570
Coefficient of thermal expansion (10^{-6}/°C)	—	7.20	11.75

(a) (b)

Figure 1: Materials images: (a) river sand and (b) zircon sand.

sand aggregates offer better thermal resistance with low quartz content and a coefficient of thermal expansion. The bulk density of the zircon sand is higher than river sand and hence makes them ideal for the production of low-porosity structures. The high abrasion resistance and high density of zircon sand in comparison to natural sand confer the improved properties for the concreting applications. The broken edges of the zircon sand during mixing with the natural sand aggregates can increase the number of sharp edges thereby enhancing the micromechanical interlocking of the aggregates with the cement paste.

3. Mix Proportion

The self-compacting mortar mixes were produced as per the concrete equivalent mortar (CEM) procedure stated by Schwartzeutruber and Catherine 2000. The mix proportion of the SCM mixes is presented along with their designation in Table 2. The binder was replaced by nanosilica and nanoalumina at different proportions (0.5, 1, 1.5, and 2% by weight), and the zircon sand was replaced instead of river sand up to 100% (increments of 25%). The binder content in the mixes was kept constant at 700 kg/m^3 to provide a high fraction of fine materials and to provide adequate powder content for the self-compacting mix. The w/b ratio was kept at 0.4, and the ratio of cementitious materials to fine aggregates was kept at 1 : 1.72. The dosage of polycarboxylate-based super plasticizer with density 1.08 kg/l was chosen as 4.5 kg/m^3 to attain the prescribed slump flow diameter values of 240 to 260 mm as per the EFNARC requirements. The mortar mixes were designated as shown in Table 2 depending on the proportion of zircon sand used as fine aggregate substitution (0%, 25%, 50%, 75%, and 100%).

3.1. Casting and Curing. The ingredients of self-compacting mortar mixes were initially weighed and mixed in a laboratory-type mortar mixer. The nanoalumina and nanosilica

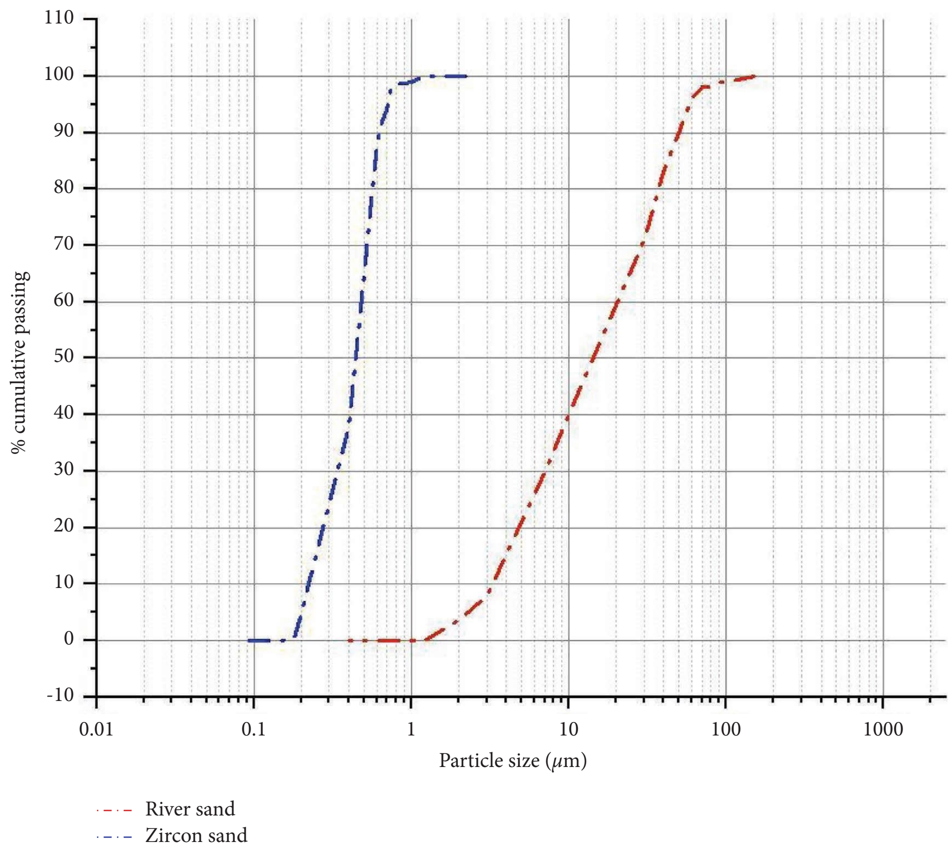


FIGURE 2: Particle size distribution curve of river sand and zircon sand.

were initially weighed and mixed with cement in its dry state before adding it to the mixer. Then, the calculated quantities of water are added to the mortar mix. The ingredients in their dry state were allowed to mix in the mixer for about 3 minutes, after which the water was added within the next 2 minutes. After thorough mixing of the ingredients, the mortar is placed in the different-sized moulds. The cast specimens with the moulds were placed at room temperature for about 24 hours, after which they were demoulded. The specimens were then kept under water for 28 days to undergo curing before they could be tested.

3.2. Heating of the Specimens. The self-compacting mortar specimens after 28 days of curing were taken and then heated in an electric furnace to the desired temperature. The refractory furnace has a temperature control panel that can automatically increase the temperature for the specified duration. The automatic control panel can also reduce the damage caused to the furnace when the temperature goes beyond the specified temperature. The muffle furnace is also equipped with a thermostat that can control the temperature and heating rate. The mortar specimens were subjected to heating and cooling cycles with the temperature set at a maximum of 200°C, 400°C, 600°C, and 800°C using the programmable muffle furnace. To prevent explosive spalling of the specimens due to the emission of excessive moisture after exposure to elevated temperatures, preheating of the specimens was done at 105°C for a period of 24 hours in an electric oven. This preheating procedure removes the excessive free moisture in the specimens, thereby reducing the explosive spalling of the mortar specimens due to the high vapour pressure buildup of the evaporated water from the specimens [31]. The heating of the SCM specimens was carried out in three stages. Initially, the specimens were heated to the target temperature at the rate of 5 C/min, and the specimens were maintained at that temperature for 2 hours and then cooled to attain room temperature. In Figure 4, the theoretical evolution of the heating and cooling rates of the specimens as a function of temperature is presented.

4. Experiments

The details of the experiments performed on the self-compacting mortar and the corresponding dimensions of the specimens used with their coded specifications for each test are shown in Table 3. The properties of the self-compacting mortar mixes were measured using the mini slump flow test and the mini V funnel test. The test was performed according to the EFNARC standards. The resistivity of the self-compacting mortar is determined using a 4-point Wenner probe tester, and the access of current flow can be indicated by the resisting power of the mortar against electric current flow under the influence of chloride ions (NT BUILD 492 1999). The electrical resistivity is a measure

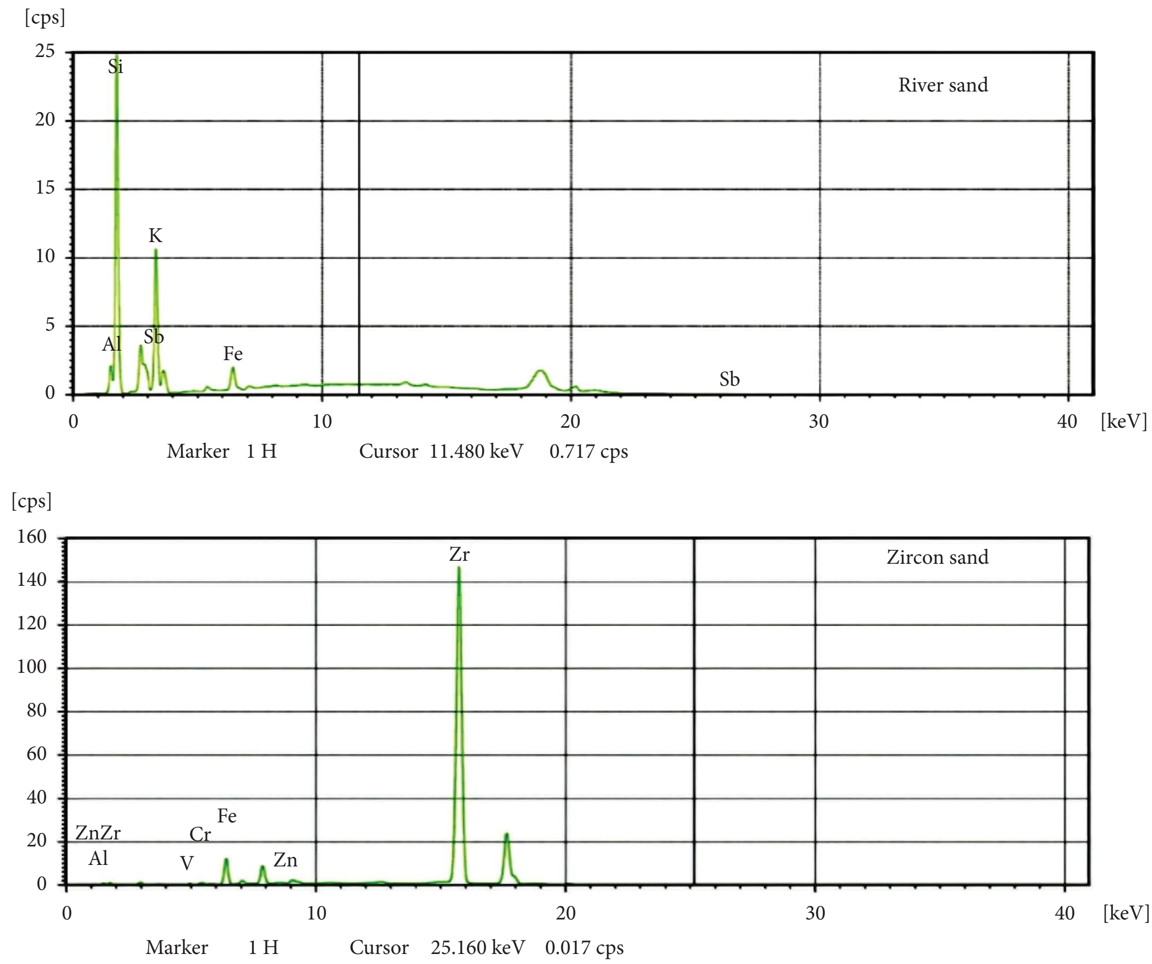


Figure 3: EDS spectrum of river sand and zircon sand.

Table 2: Mix details of developed SCM mixes.

Mix id	Binder			Fine aggregate	
	Cement (kg/m^3)	Nanosilica (kg/m^3)	Nanoalumina (kg/m^3)	Sand (kg/m^3)	Zircon sand (kg/m^3)
SCM	700	0	0	1210	0
SCM-Z25	693	3.5	3.5	907.5	302.5
SCM-Z50	686	7	7	605	605
SCM-Z75	679	10.5	10.5	302.5	907.5
SCM-Z100	672	14	14	0	1210

of the porous microstructure of the mortar and can give an idea of the permeability and transport properties of the mortar.

5. Results and Discussions

5.1. Fresh State Results. The workability results of the self-compacting mortar containing zircon sand and nanomaterials in Figure 5 clearly showed decreased flow values due to the incorporation of the nanoparticles in the self-compacting mortar mixes. Generally, the self-compacting nature of the mixes is expected to be increased with an increase in the zircon sand content. But the nanoparticles demand more water to wet their surfaces, which thereby reduces the free water availability, leading to decreased flowability. The zircon sands are nonwater-absorbing in nature and hence increase the free water availability for increasing the fluidity of mortar. Therefore, it can be affirmed that zircon sand is the sole contributor to the workability improvement in the self-compacting mortar mixes when w/b ratio and superplasticizer dosage are maintained constant. Moreover, the decreased flowability caused by the addition of nanomaterials was partially nullified by the increasing zircon sand content that possesses weak intermolecular cohesive force between them due to their glassy and smooth surface texture.

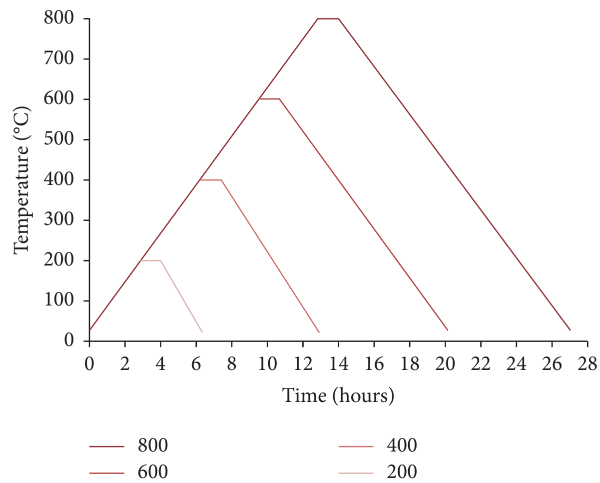


FIGURE 4: Heating temperature-time curve.

TABLE 3: Mortar specimen details for each test.

Experiments	Codal provision/standard	Details of the specimen
Strength tests		
Compressive strength	IS 516: 1959	150 mm cube
Flexural strength	IS 516: 1959	500 × 100 × 100 mm prism
Ultrasonic pulse velocity	IS 13311-I: 1992	150 mm cube
Durability tests		
Water absorption	ASTM C-642	50 mm thick × 100 mm dia
Porosity	ASTM C-642	50 mm thick × 100 mm dia
Electrical resistivity	—	50 mm thick × 100 mm dia
RCPT	ASTM C1202-1995	50 mm thick × 100 mm dia
RCMT	NT-build 492 1999	50 mm thick × 100 mm dia
Bulk diffusion	ASTM C 1556	50 mm thick × 100 mm dia
Microstructure		
SEM	—	Broken powdered specimens
XRD	—	Broken powdered specimens
FTIR	—	Broken powdered specimens

5.2. Compressive Strength. The compressive strength results in Figure 6 clearly showed that zircon sand replaced samples exhibited comparatively lower strength than the reference mortar, but these strength variations are, however, negotiable. The results proved the filler effect of nanosilica and nanoalumina that filled the voids created between the zircon aggregates, thereby minimizing strength reduction. The nonwater-absorbing nature of the zircon sand increases the amount of free water in the self-compacting mortar that can be utilised for wetting the nanoparticles. The higher surface area, fineness, and high reactivity of the nanoparticles also reduced the loss in compressive strength of the mortar even at higher substitution of zircon sand. The cohesion improvement between the cement pastes and zircon sand aggregates contributed by the nanoparticles is an important factor for this minimal decrease in the compressive strength. The results of the residual compressive strength of the self-compacting mortar after exposure to elevated temperatures also clearly showed that the compressive strength was increased at 200°C for all the mortar mixes. It can be said that after exposure to 200°C, the mortar gains strength due to the thermal activation of the nanoparticles. The mortar strength variation is due to the zircon sand that is highly thermally stable and hence is being used widely for the refractory purposes.

5.3. Flexural Strength. The flexural strength results of the self-compacting mortar mixes in Figure 7 clearly explained the favourable effects of nanoparticles in neutralizing the negative effect of the brittle zircon sand substitution on the flexural strength decrement of the mortar. The addition of nanomaterials improved the cohesion of zircon sand and the cement matrix thus partially contributing towards the flexural strength of the mortar at normal temperature. A slight decrement in the flexural strength was observed in the mortar at normal temperature and at 200°C when zircon sand was added beyond 25% with nanoparticle substitution. Intriguingly, this reduction in the flexural strength may be due to the formation of brittle regions in the mortar, creating a weaker interfacial zone between the aggregate and the mortar phase. Though zircon sand is highly thermally stable, the substitution of zircon sand beyond 25% causes a reduction in the flexural performance of the SCM at normal

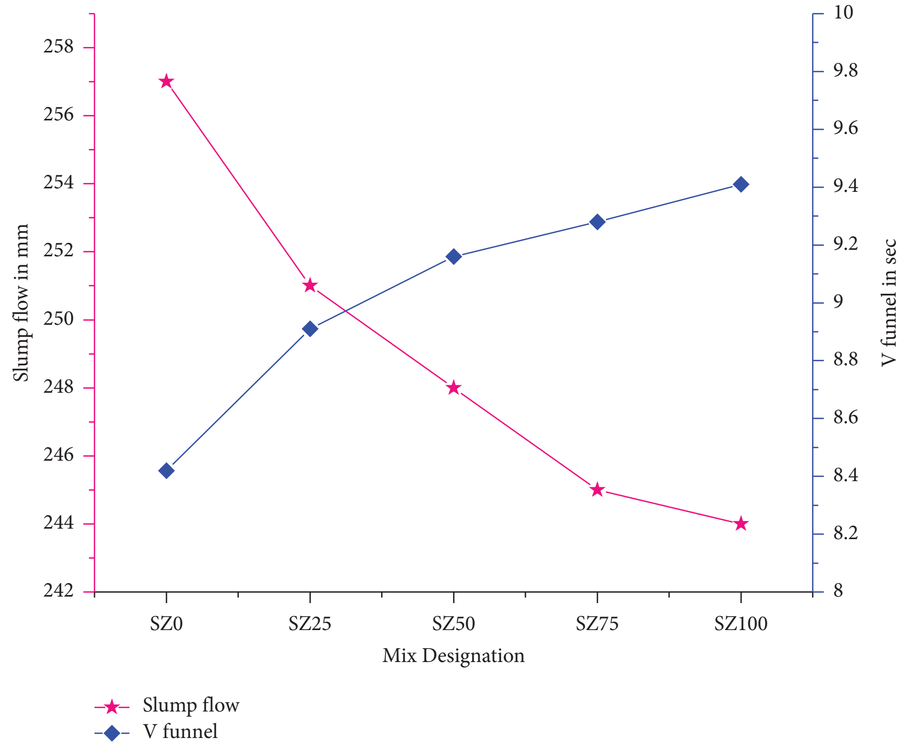


Figure 5: Fresh state results of self-compacting mortar containing zircon sand and nanoparticles.

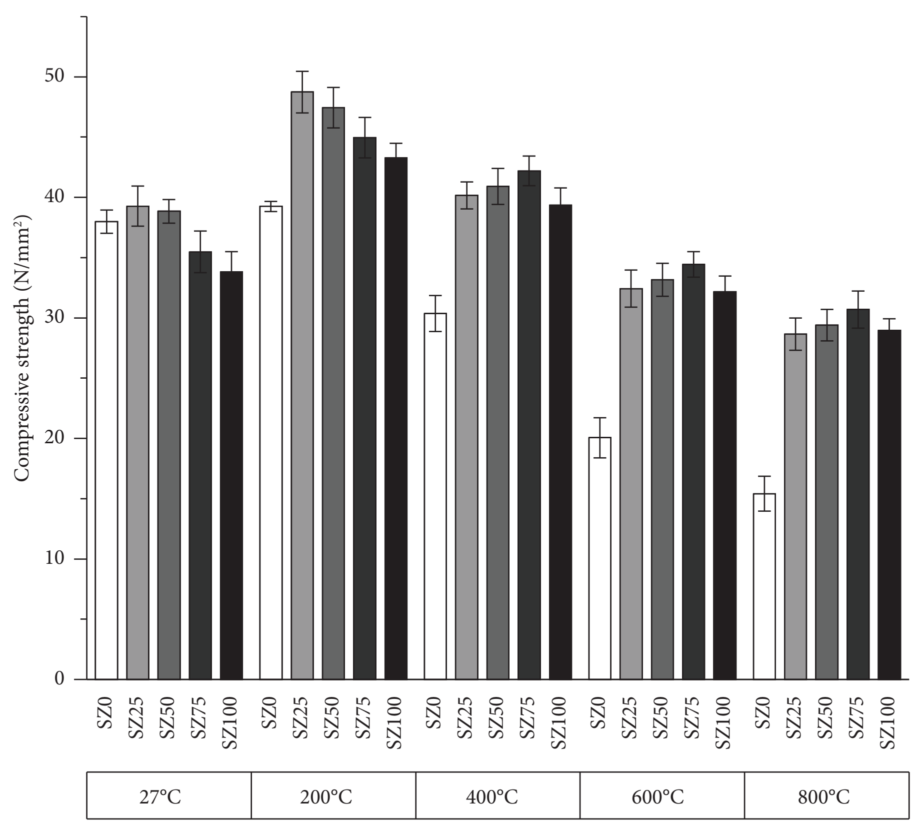


Figure 6: Variation of compressive strength of self-compacting mortars containing zircon sand and nanoparticles at normal and elevated temperatures.

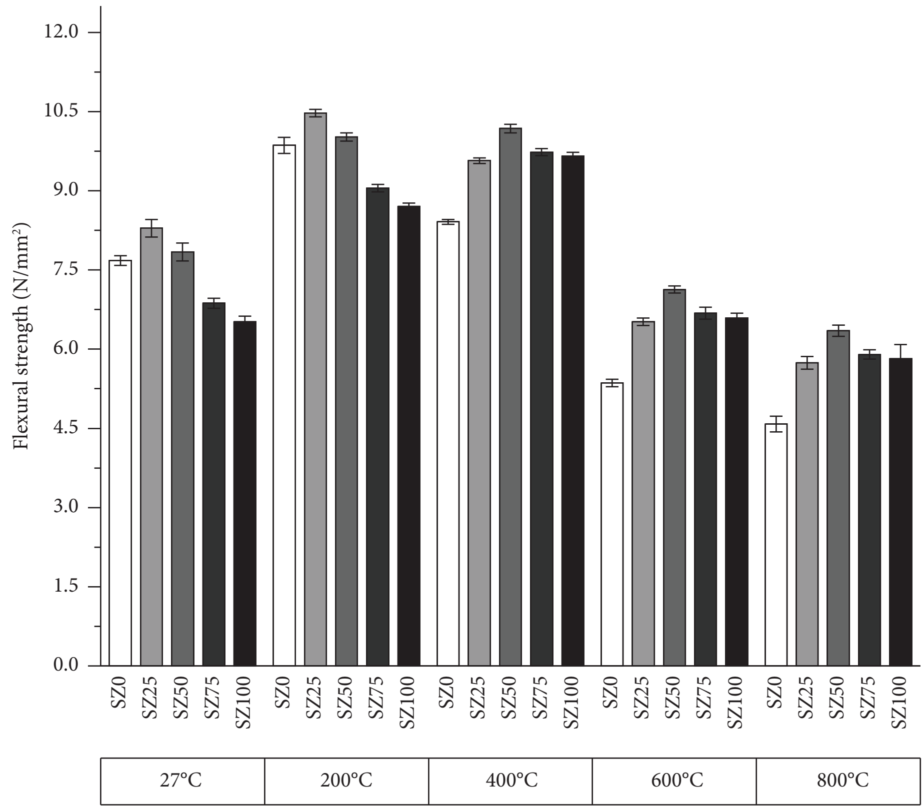


Figure 7: Variation of flexural strength of self-compacting mortars containing zircon sand and nanoparticles at normal and elevated temperatures.

temperature, whereas, after exposure to elevated temperature, only a marginal reduction in the flexural strength was observed due to the reduction in the difference in thermal gradients between the aggregate phase and the binder phase. Because zircon sand has a low coefficient of expansion, thermal cracks are less likely to form because the thermal strains caused by the shrinking cement paste and the expanding aggregate phase are less likely to be incompatible. The decreasing trends of flexural strength may also be reasoned by the increase in the brittleness of the mortar with increasing zircon sand substitution. Moreover, the zircon sand substitution at higher percentage levels creates a brittle zone that paves the way for the formation of microcracks. However, the nanoparticles acted as crack-arresting agents and delayed the formation of cracks in mortar. The flexural strength values of control mortar decreased significantly with increase in temperature for the control mortar whereas the strength reduction of the mortars containing zircon sand and nanoalumina was relatively lower at higher temperatures. The nanoparticles also acted as a nanoreinforcement material by providing strength to the mortar by resisting the propagation of microcracks. The nanoalumina is also highly resistant towards elevated temperature and improved the flexural strength of mortar at higher temperatures. The flexural strength increment with increasing zircon sand substitution and nanoparticles addition at a temperature range of about 200°C and 400°C may be explained by the reduction in pores caused due to the nanoparticles and zircon sand aggregates.

5.4. UPV. The UPV value of zircon sand substituted mortar mixes is found to be higher than normal self-compacting mortar for all the mixes as shown in Figure 8. The higher rate of hydration and the higher specific surface of the nanoparticles reduced the porosity of the mortar mixes, which subsequently influenced the ultrasonic pulse velocity. The denser internal structure of the mortar caused a subsequent increase in the pulse velocity, which in turn signifies the filling capacity of the nanomaterials in combination with zircon sand. The ultrasonic pulse velocities of the self-compacting mortar mixes at elevated levels show a significant improvement. Generally, a rapid reduction in ultrasonic pulse velocity is observed due to the increase in the pore structure and microcracking of the self-compacting mortar after exposure to higher temperatures. But only a gradual decrease in the ultrasound velocities of the self-compacting mortar was observed up to 600°C without a steep decrease, indicating the higher thermal stability of the zircon sand that can withstand the thermal effect even at elevated temperatures. When the quality of the produced mortar mixes was considered, all the zircon sand substituted mortar mixes exhibited improved ultrasound velocity than the control mortar at all temperatures and belonged to "good" quality grading up to 400°C as per the quality gradation given in IS 13311-1 (1992). This decrease in microcracks formed in mortar after exposure to elevated temperatures due to the zircon sand substitution may also be the cause of the improved UPV values.

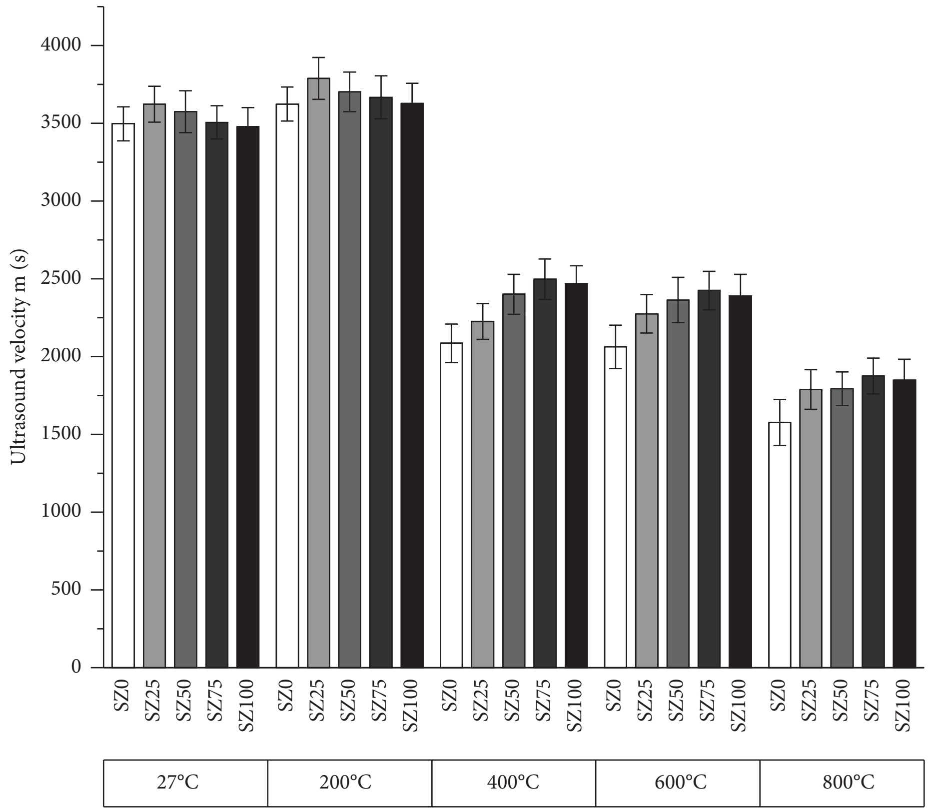


Figure 8: Variation of ultrasound velocity of self-compacting mortars containing zircon sand and nanoparticles at normal and elevated temperatures.

5.5. Water Absorption. The water absorption values of the mortar at normal temperatures and after being subjected to elevated temperatures are shown in Figure 9. From the results obtained, it can be clearly inferred that the water absorption values were reduced in the mortar that contained zircon sand when compared to the other mortar mixes. The decreased water absorption values of the mortar are a function of zircon sand and nanomaterials that minimize the free water available in the mortar, thus refining the pore structure of the mortar. The saturated water absorption values depend on the quantity of pores in the mortar. The minimization of pore sizes due to filling by nanoparticles also contributes to the decreased water absorption values. The attainment of a dense microstructure increases the compactness of mortar, leaving no spaces through which water can enter. The water absorption values of the mortar after exposure to higher temperatures showed a steady increase in their value at increasing temperatures. The significant contribution of zircon sand towards the water absorption reduction was observed in the mortar mixes when exposed to temperatures beyond 400°C. The zircon sand is thermally stable and prevents the creation of pores due to disruption of the aggregate phase of mortar. On the other hand, the use of nanomaterials prevents the disruption of the cement matrix by holding the ingredients together even at elevated temperatures and contributes to a reduction of channels available for the intrusion of moisture by blocking the water channels.

5.6. Porosity. The water porosity studies were done only to evaluate the pores in between the cement and zircon sand aggregates and hence are only a measure of open porosity. The porosity values also indicate the reduced pore spaces between the aggregates and the cement paste caused due to the addition of nanoparticles that caused a perfect binding of the cement and zircon sand as shown in Figure 10. The porosity values also clearly showed decreased values with increasing zircon sand substitution at all temperatures. The reduction in the microcavities of the mortar due to the high specific surface area of the nanomaterials is the main reason for the reduction in the porosity. The zircon sand, due to its surface characteristics, also effectively bonded with the cement paste, reducing the available pores in the self-compacting mortar. At increasing temperatures, the pozzolanic action of nanosilica also forms stabilised CSH gels that occupy the pores in the self-compacting mortar, thereby causing a reduction in the pore volume. The effect of nanoalumina also contributed to the porosity reduction, especially at elevated temperatures, by filling the voids created due to the loss of moisture from the self-compacting mortar at increasing temperatures, which creates pores after exposure to higher temperatures. The formation of pores due to the evaporation of free water available in the mortar at 200°C was reduced by the use of nanomaterials that occupied the interstitial spaces of the mortar in which free water can reside. The thermal activation of nanoparticles also creates additional CSH gels through the consumption of water,

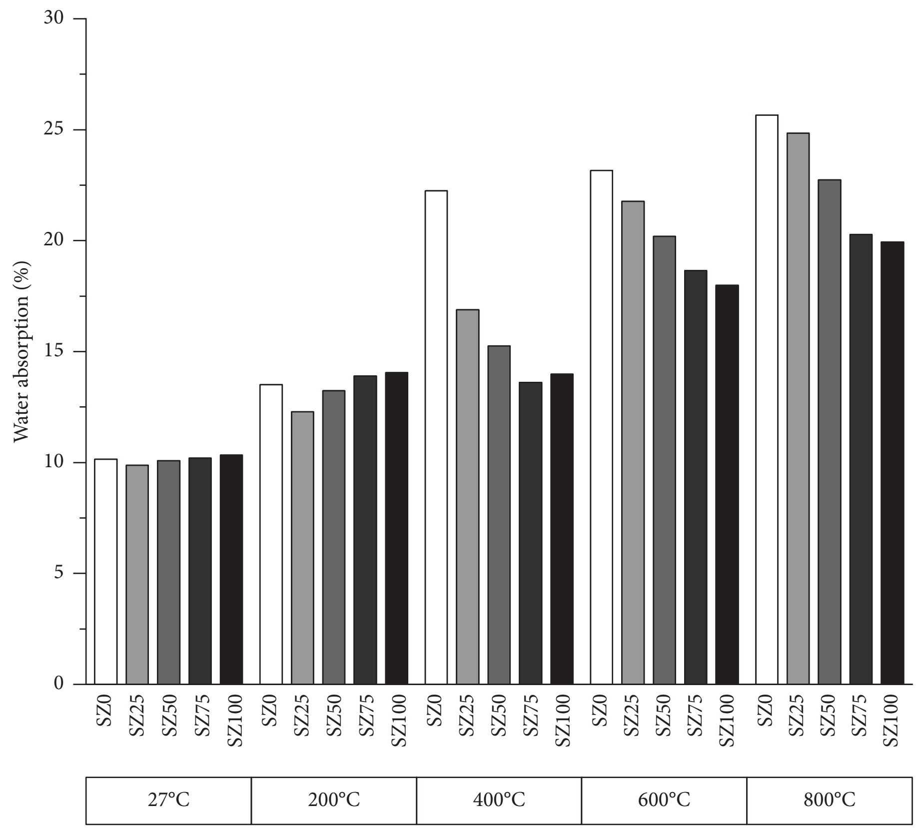


Figure 9: Water absorption of self-compacting mortars containing zircon sand and nanoparticles at normal and elevated temperatures.

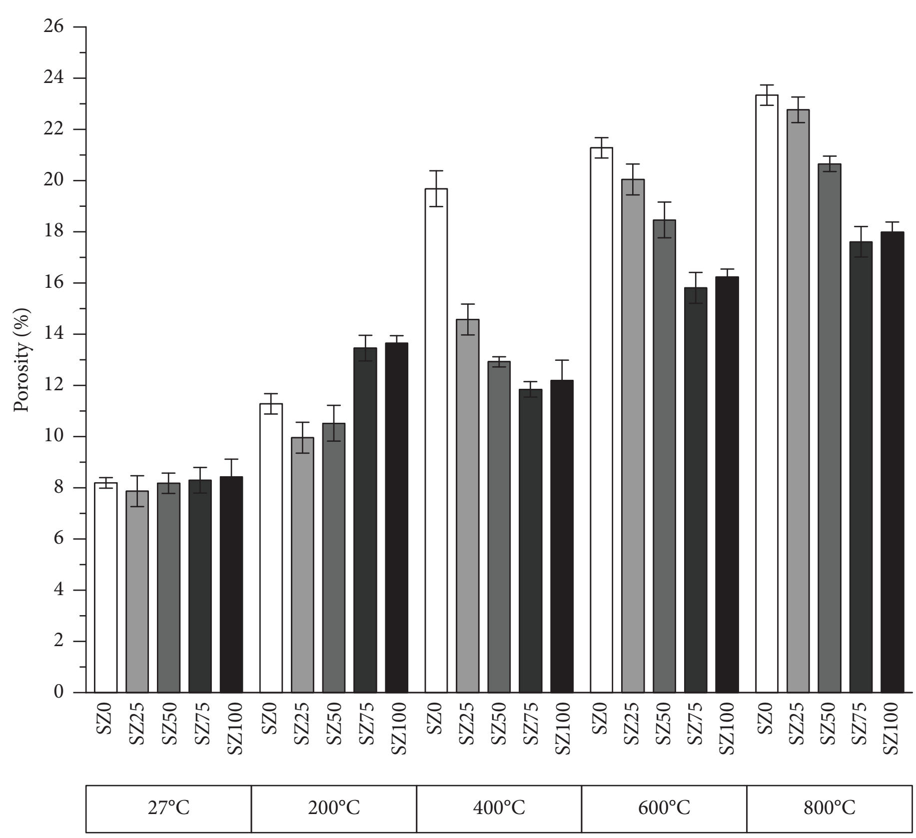


Figure 10: Porosity of self-compacting mortars containing zircon sand and nanoparticles at normal and elevated temperatures.

leading to the filling of the pores and reduced porosity. Therefore, the porosity values of the nanoparticles added to the mortar were much lower than the control mortar at 200°C. Beyond 400°C, the decomposition of the CSH gel takes place, which was minimized due to nanomaterial addition. The porosity reduction is essentially a function of two parameters, namely the extent of formed hydration products and the filling of pores. Both the parameters were achieved due to the addition of nanosilica and nanoalumina. Moreover, the stable nature of zircon sand towards elevated temperatures also held the ingredients of mortar together in place without leading to weathering of aggregates at elevated temperatures.

5.7. RCPT. The rapid chloride penetration test (RCPT) values of the self-compacting mortar at normal temperatures and after exposure to elevated temperatures are shown in Figure 11. The chloride penetration values measured as the quantity of charge passed through the mortar clearly showed increased values with increasing temperature in all the mortar mixes. The increased amounts of charge passed through the mortar at normal temperatures may be due to the increased porosity in the mortar as the amount of zircon sand and nanomaterials increased in the mortar. The hydration of ions through mortar is electrolytic through the cement paste, and the aggregates generally do not contribute to the passage of ions due to their high electrical resistivity. The conductivity of the ions through the pore solution in the mortar plays a significant role in the ion migration process. The OH-ions generally deplete due to the pozzolanic action, thereby improving the resistivity against the ionic migration, which directly reduces the passage of charge through the mortar. The addition of nanosilica in the mortar causes a decrease in the chloride penetration of the self-compacting mortars through their pozzolanic action. The inclusion of nanomaterials in the mortar caused a decrease in the amount of excessive free water content in the self-compacting mortar mixes, thereby reducing the separation of the materials present in the mortar. The nanoalumina inclusion effectively held the pore waters together, allowing them to be used in the hydration reaction. The compact nature of the mortar can also be visualised as the reason for the reduction in the charge passed through them. The angular surface texture of the zircon sand also strengthened the ITZ of the mortar, which reduced the travel of ions in the mortar, thereby avoiding the formation of microvoids.

5.8. RCMT. The rapid chloride migration test is also an essential durability test that measures the gradient of the passage of chloride ions in the mortar. The chloride migration coefficient values of the mortar are shown in Figure 12. The addition of nanoparticles improves the cohesiveness of the mortar by holding the ingredients altogether physically as well as chemically. The zircon sand also formed interlocks with the adhesive cement paste due to their physical nature. The high specific surface area of the nanoparticles in comparison with the cement paste increased the fineness content of the cement paste, thereby forming an impermeable layer around the aggregates. The mortar containing zircon sand was also highly resistant to elevated temperatures without allowing the microcracking of mortar at the aggregate surface and the cement paste. The increased dense CSH gel formations also screen the ingress of chloride ions, thereby reducing their migration coefficient. The addition of fine aggregates of varying sizes and types can alter the pore properties of mortar by reducing the migration coefficient. The coefficient of migration of chloride ions is also reduced due to the lower volume of voids and loss of ionic solution in the pores. The inert zircon sand aggregates do not form any charge on the surfaces, leading to reduced migration coefficient values, thereby modifying the porous mortar into a nonporous compact structure. The increasing temperatures cause the loss of free water in the mortar, increasing the void ratio of the mortar. However, because of the additions of nanoalumina and zircon sand, the amounts of water added were used to wet their surfaces as well as completely used for the hydration reaction. The reduced migration coefficient of mortar can also be attributed to the reduced separation of the ingredients of the mortar at elevated temperatures due to the thermal stability of the produced mortar.

5.9. Electrical Resistivity. The rapid chloride migration test is also an essential durability test that measures the gradient of passage of chloride ions in the mortar. The chloride migration coefficient values of the mortar are shown in Figure 13. The addition of nanoparticles improves the cohesiveness of the mortar by holding the ingredients altogether physically as well as chemically. The zircon sand also formed interlocks with the adhesive cement paste due to their physical nature. The high specific surface area of the nanoparticles in comparison with the cement paste increased the fineness content of the cement paste, thereby forming an impermeable layer around the aggregates. The mortar containing zircon sand was also highly resistant to elevated temperatures without allowing the microcracking of mortar at the aggregate surface and the cement paste. The increased dense CSH gel formations also screen the ingress of chloride ions, thereby reducing their migration coefficient. The addition of fine aggregates of varying sizes and types can alter the pore properties of mortar by reducing the migration coefficient. The coefficient of migration of chloride ions is also reduced due to the lower volume of voids and loss of ionic solution in the pores. The inert zircon sand aggregates do not form any charge on the surfaces, leading to reduced migration coefficient values, thereby modifying the porous mortar into a nonporous compact structure. The increasing temperatures cause the loss of free water in the mortar, increasing the void ratio of the mortar. However, because of the additions of nanoalumina and zircon sand, the amounts of water added were used to wet their surfaces as well as completely used for the hydration reaction. The reduced migration coefficient of mortar can also be attributed to the reduced separation of the ingredients of the mortar at elevated temperatures due to the thermal stability of the produced mortar.

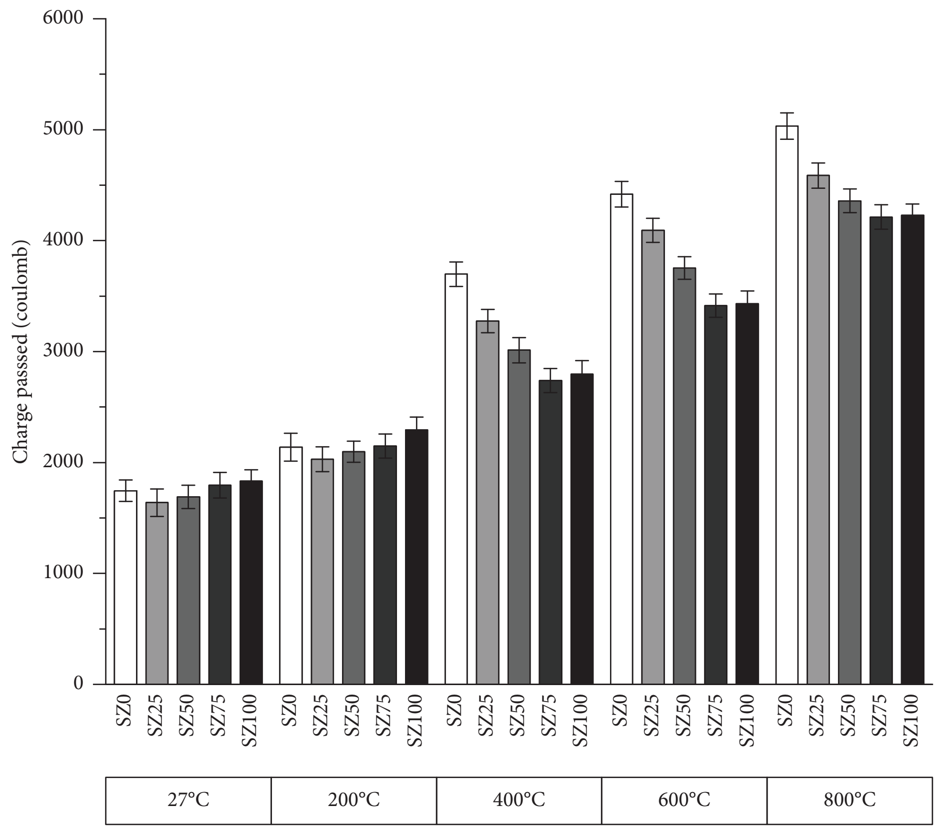


Figure 11: RCPT results of self-compacting mortars containing zircon sand and nanoparticles at normal and elevated temperatures.

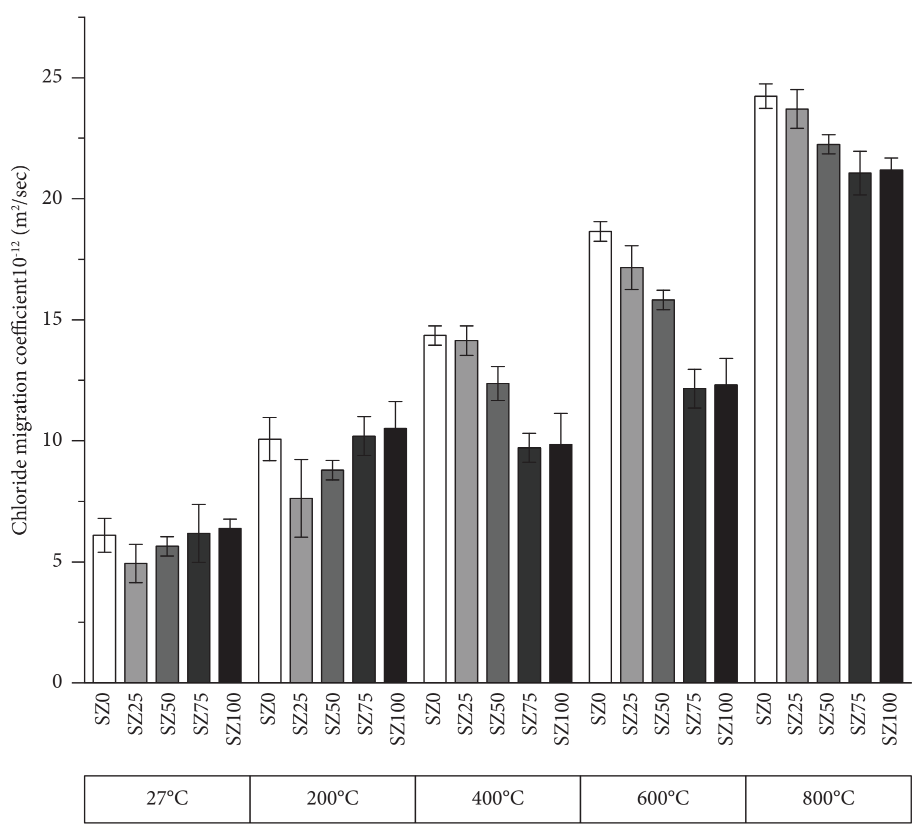


Figure 12: RCMT results of self-compacting mortars containing zircon sand and nanoparticles at normal and elevated temperatures.

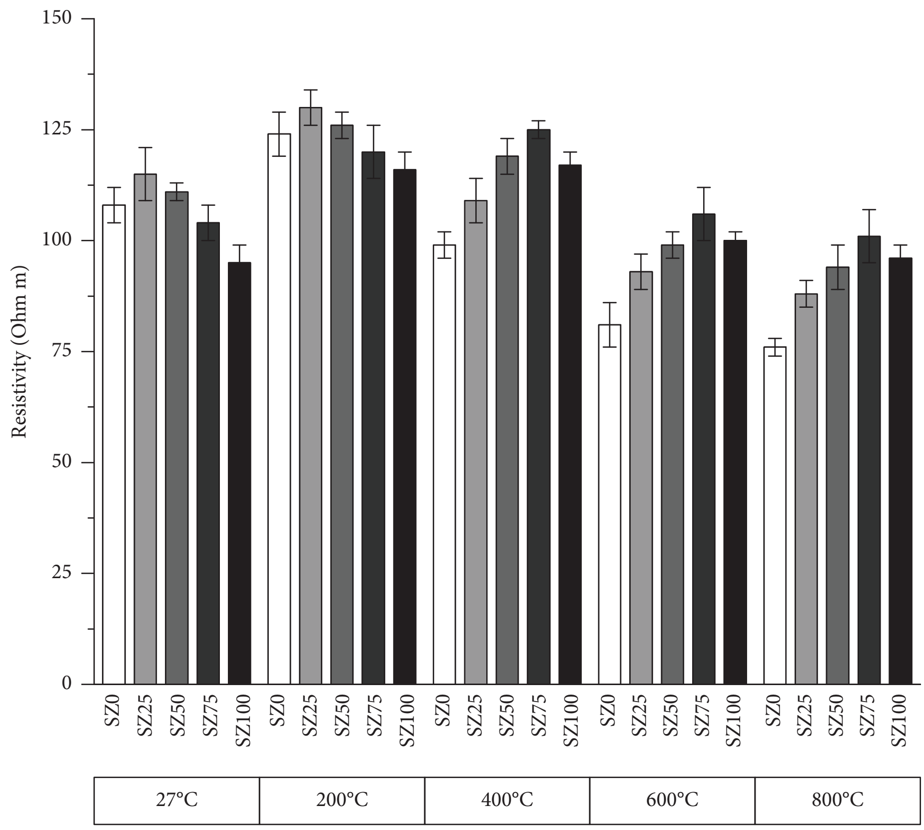


FIGURE 13: Electrical resistivity of self-compacting mortars containing zircon sand and nanoparticles at normal and elevated temperatures.

5.10. XRD. The XRD analysis done on the 28-day water-cured self-compacting mortar mixes is shown in Figure 14. The various mineralogical phases present in the mortar mixes are shown in Figure 10 by comparing them with the JCPDS XRD data file. It can be observed that portlandite, quartz, calcium silicate, and calcite are the major phases present in the mortar. The XRD pattern of the mortar mixes also shows the presence of quartz peaks, which arise from the inert aggregates. The presence of portlandite is deducted in all the patterns. However, the reduction in the portlandite phases indicates the consumption of CH leading to the formation of secondary hydration products. The patterns also clearly showed improved portlandite phases at a temperature of 200°C due to the thermal activation of nanosilica particles in the mortar. The other phases were almost similar in all the mixes, showing that the mineralogical phases were almost unaffected by temperature. The XRD patterns of the mortar mixes after exposure to 400°C clearly showed the disintegration of portlandite leading to the release of bound water from the CH and CSH phases. The XRD patterns after exposure to 600°C and 800°C, respectively, clearly showed only the quartz peak with minor calcite peaks in the control mortar (SCM). The XRD results thus show that the reduction in the strength of the mortar is due to the loss in the crystal structure of all the hydrated phases, CH and CSH crystals. The thermal activation of the hydration reaction due to the addition of nanomaterials has caused improved crystalline hydration products. Generally, CSH gels are amorphous in nature, and their identification in XRD is a little tough. At 400°C, the decomposition of portlandite peaks was clearly visible in the control mortar, whereas the portlandite peaks were visibly of high intensity in the mortar containing zircon sand. This shows the thermal stability of the produced mortar and the stable formation of portlandite. However, at temperatures above 600°C, all of the portlandite phases were completely reduced, indicating the decomposition of $Ca(OH)_2$. The portlandite phases decomposed severely at 800°C, and only the minor peaks of calcium silicate and quartz were found.

5.11. FTIR. The FTIR spectra of the mortar mixes after exposure to temperatures are shown in Figure 15. The decreasing broad vibrations of the water molecules around $3400\,cm^{-1}$ were found with increasing temperatures. The spectra diminished with increasing temperatures and were almost absent when the temperature increased beyond 400°C indicating the decomposition of portlandite. Less intense band around $1600\,cm^{-1}$ also indicates the decomposition of $Ca(OH)_2$. Only crystalline phases of silica were found in the FTIR spectra around $980\,cm^{-1}$ and $780\,cm^{-1}$ in all the mortar mixes after exposure to 600°C. The spectra thus clearly show the presence of highly stable CSH products in the mortar. The presence of silica also indicates the presence of thermally stable fine aggregates in the mortar. The peak around $980\,cm^{-1}$ which also indicates the CSH gel formations was found to be stable upto 600°C which at 800°C showed little disintegration. The modification of the

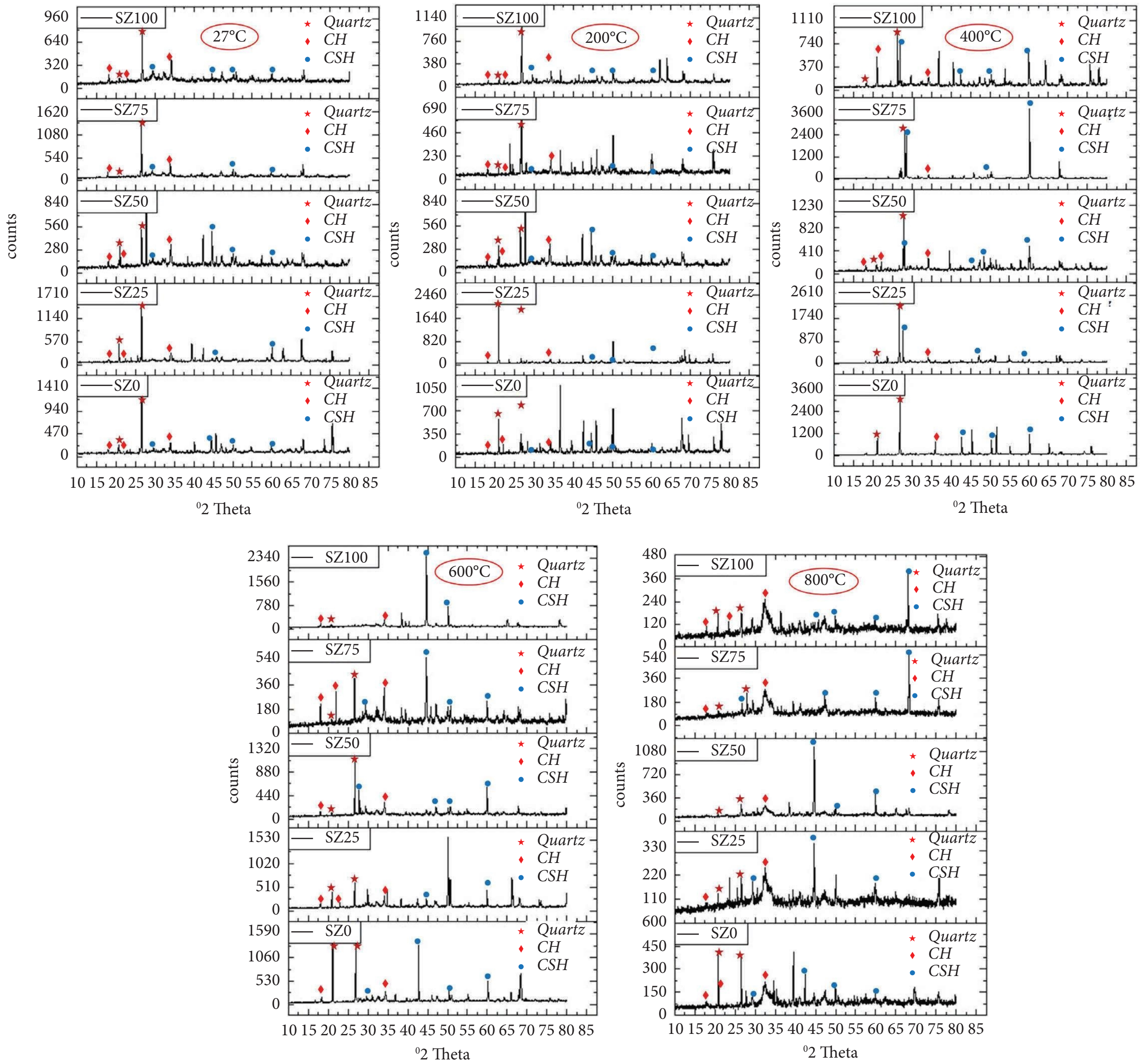


Figure 14: XRD spectra of self-compacting mortars containing zircon sand and nanoparticles at normal and elevated temperatures.

hydration products with increasing temperature causes increase or decrease in the strength of mortar. The decrement of the calcium silicates in the mortar was much lower in the mortars containing zircon sand in comparison to the control mortar as evident through FTIR spectral analysis which supports the results obtained from the mechanical and durability experiments.

5.12. SEM. The SEM images of the zircon sand substituted mortar mixes showed uniform distribution throughout the cement matrix, which is clearly evident from Figures 16(a) and 16(b). The denseness of the self-compacting mortar was also improved, as seen through the SEM images, due to the contribution of nanosilica. The images also show that the pore reduction may be caused by the filling effect of the nanoparticles. Moreover, the evidence of the CSH gel is shown by the flaky layers present in the images of mortar. The gel-like structure is clearly visible in the interface between the zircon sand and the aggregates, thereby increasing the bonding characteristics of the cement paste. The strengthening of the interfacial transition zone by the improved structure of CSH gel caused a significant reduction in the capillary pores in the mortar. Generally, the hydrated phases occur in grey, and the SEM images clearly show the grey phases, which are found to decrease with an increase in temperature. The decreasing thickness of the CSH gel was found with increasing temperatures, which is responsible for the loss in strength of the mortar with increasing temperatures. However, this effect was lowered in the zircon sand substituted mortar mixes due to the effect of the addition of nanomaterials. The homogeneous distribution of nanosilica and nanoalumina functioned as fillers in the mortar bonding agent due to their dilution effect. It can be seen that the

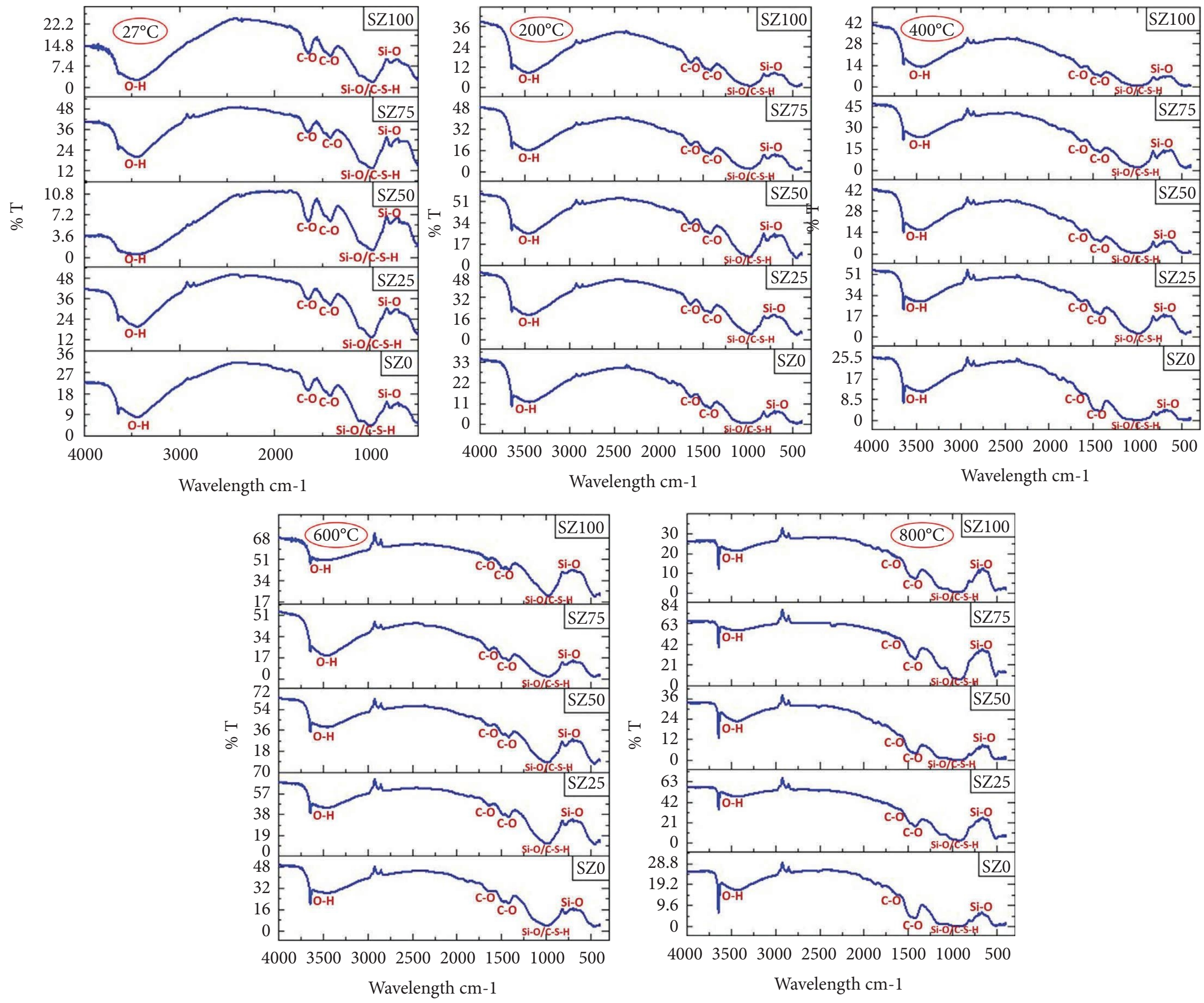
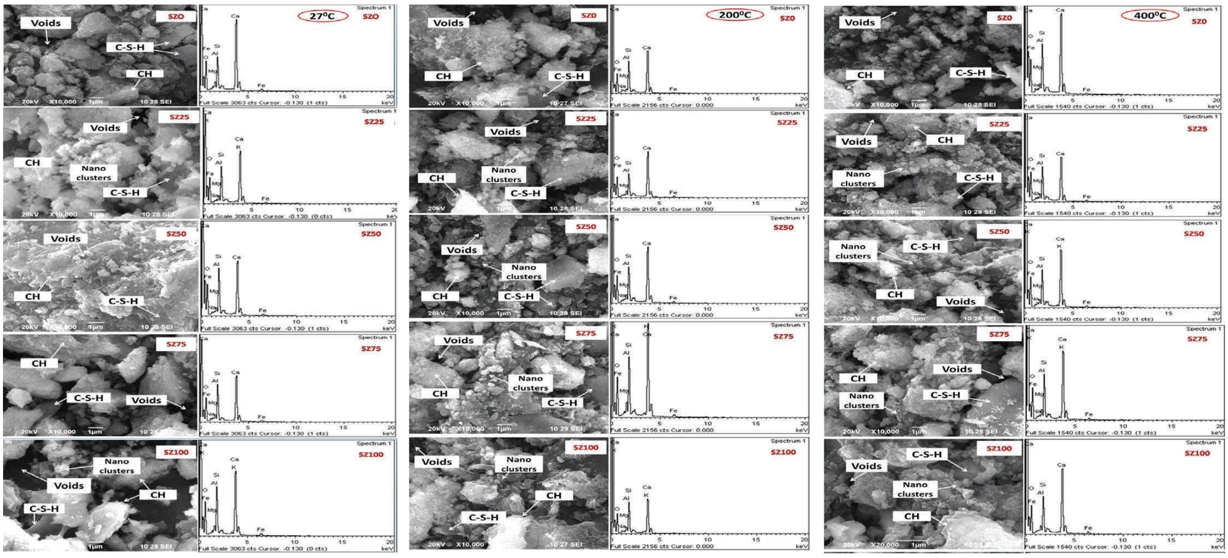


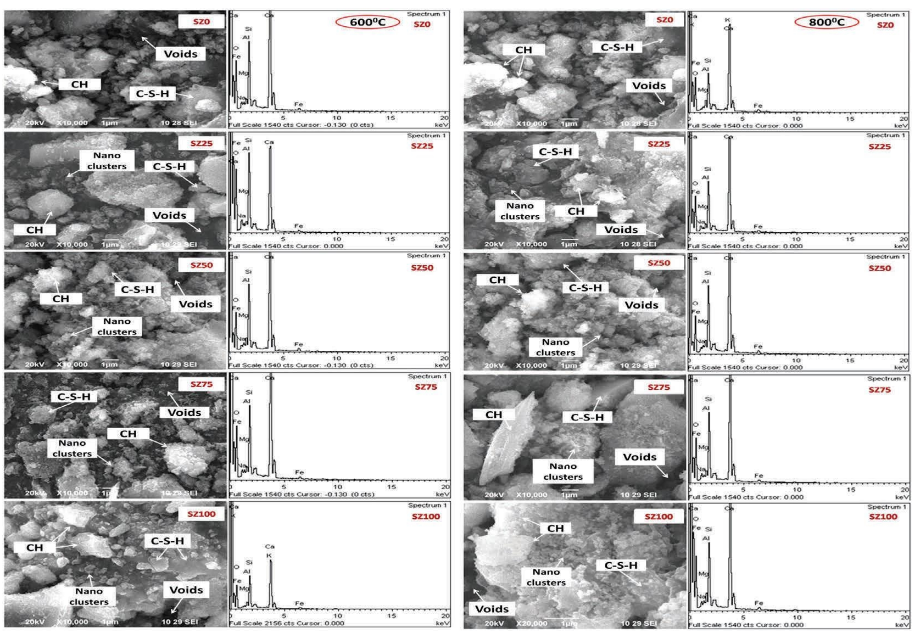
Figure 15: FTIR curves of self-compacting mortars containing zircon sand and nanoparticles at normal and elevated temperatures.

incorporation of nanoparticles transformed the CH crystals into CSH gels that surrounded the fine aggregates, thus forming a denser and more compact microstructure. The higher surface energy of the nanosilica also improved the contact between the cement paste and the aggregates, thereby reducing the distance between the aggregates and the cement particles. The SEM images also show the interlocking effect exhibited by the nanoparticles and the zircon sand aggregates even at elevated temperatures, which stands as evidence of the above stated mechanical strength results.

5.13. Cost Analysis and Ecological Performance. The main damage to the ecosystem caused by the concrete production is the extraction of natural resources and cement production. Also, the transportation of the raw materials also contributes to the economical and ecological concerns. A shift towards cement and fine aggregate replacement can decrease the extraction of natural resources and release the environmental behaviour. The economy index and ecological performance measurements have been done to recommend the optimum proportion of SCC. The economy index was obtained by estimating the cost required for the production of the mix including material transportation and procurement costs. The economy index is measured as a ratio of 28 days of compressive strength of the self-compacting mortar to the cost of production of 1 m^3 of self-compacting mortar. The economic index values presented in Table 4 clearly show that the SCM-Z25 showed a relatively higher index value and hence can be chosen as the optimal proportion from the economic point of view. Though the production cost of the mortar suggested in the present study increased due to the incorporation of nanomaterials, the real cost of conventional mortar production is hidden behind the ecological destruction caused by the usage of river sand. The self-compacting mortar mixes are produced using cement and river sand replacement, thereby minimizing their usage. Cement usage has caused significant issues in three damage categories, namely human health, ecosystem quality, and resource consumption. The material fine aggregate usage has also caused serious ecological disturbance due to overconsumption of natural resources. The self-compacting mortar mixes function well in terms of human health by minimizing the amount of CO_2 being released into the atmosphere which is produced during cement production. Apart from



(a)



(b)

Figure 16: (a) SEM images of self-compacting mortars containing zircon sand and nanoparticles at 27°C, 200°C, and 400°C. (b) SEM images of self-compacting mortars containing zircon sand and nanoparticles at 600°C and 800°C.

Table 4: Cost analysis and economic index of SCM specimens.

Materials used (INR/kg)	Mix id	Cost (INR)	Economical index				
			27°C	200°C	400°C	600°C	800°C
Cement (410/50 kg bag)	SCM	7880	0.0048	0.0049	0.0038	0.0025	0.0020
River sand (90/cft)	SCM-Z25	11575	0.0034	0.0042	0.0035	0.0028	0.0025
Nanosilica (525/kg)	SCM-Z50	15268	0.0025	0.0031	0.0027	0.0022	0.0019
Nanoalumina (900/kg)	SCM-Z75	18962	0.0019	0.0024	0.0022	0.0018	0.0016
Zircon sand (110/cft)	SCM-Z100	22656	0.0015	0.0019	0.0017	0.0014	0.0013

this, the emissions of hazardous gases such as sulphur dioxide and nitrogen oxide also affect human health and minimize the life years of humans. The particular matters emitted from cement clinker production and packing also play a dominant role in affecting human health. Even minute replacement of cement can cause maximum contributions to human health. Similarly, the replacement of natural fine aggregates minimizes the natural resource extraction, which contributes to ecosystem quality by minimizing the erosion of the river banks. Zircon sand used in SCM production is obtained from the nearby mineral sand mining industry and minimizes the environmental burdens of the conventional SCM mixes that require huge amounts of fine aggregates. Thus, the economic performance of the zircon sand containing SCM mixes seems to be an effective alternative to the conventional SCM for use in special purposes, especially elevated temperature applications.

6. Conclusions

Systematic research was conducted to explore the benefits of zircon sand and nanomaterial utilisation on the properties of SCM at normal and elevated temperatures. The synergistic filling effect of nanomaterials and the thermal stability of zircon sand were well exploited to increase the overall performance of SCM. The experimental results showed that the combined addition of nanomaterials effectively improved the strength and durability of the SCM. The SEM images also verified the improvement of the microstructure of SCM due to nanomaterial and zircon sand additions. The significant conclusions can be stated as follows.

The use of nanomaterials and zircon sand is more effective in increasing the strength of SCM at elevated temperatures than at normal temperatures. The relative effectiveness of zircon sand primarily results from its higher thermal stability and finer nature, which was further increased due to the higher degree of reactivity and chemical nature of the nanomaterials. Results also show that the nanomaterials in combination with zircon sand contribute to the lower permeability and increased resistance to water and chloride penetration even at higher temperatures up to 800°C. The data also show improved CSH formations with lower CH content, indicating a dense microstructure with low void volume. The comparison of the specimens made with and without zircon sand and nanomaterials after exposure to elevated temperatures showed a better economic index. The morphological characterization of the specimens after exposure to elevated temperatures also compensated for the mechanical results, confirming the synergistic effect of nanomaterials and zircon sand in strength preservation at elevated temperatures. Thus, significant effort has been made to improve the thermal resistance of SCM while demonstrating the advantages and limitations associated with the stipulated design proportions of SCM both economically and ecologically.

The final conclusion can thus be drawn that the use of zircon sand as a partial substitute for fine aggregate and nanoparticles (nano SiO_2 and nano Al_2O_3) in cement additives positively influenced the mechanical strength and durability properties of mortar at normal and elevated temperatures.

6.1. Potential Further Studies. The current study could lead to a variety of new studies on the use of zircon sand in the production of highly flexible self-consolidating concrete through the incorporation of fibers. The authors are also involved in the permeability and pore structure characterization of the SCM containing zircon sand and nanomaterials after being subjected to elevated temperatures. Performance assessment of the zircon sand containing SCM with combined usage of industrial by-products and agricultural wastes is highly limited, which will be explored in our future study.

Data Availability

All data generated or analysed during this study are included in this article.

Additional Points

Highlights. (i) Zircon sand supplies high thermal stability to sustain higher temperatures. (ii) Nanomaterials enhance the filling effect and promote the hydration reaction of cement. (iii) Both nanomaterials and zircon sand improve the dimensional stability of RPC after exposure to elevated temperatures. (iv) Utilisation of nanomaterials and zircon sand enhances durability, mitigating the ingress of deleterious ions.

Consent

Additional informed consent was obtained from all individual participants for whom identifying information is included in this chapter.

Conflicts of Interest

The authors declare no conflicts of interest.

Authors' Contributions

Sahaya Ruben conceived and designed the conceptual ideas. M. Sophia performed the analysis and prepared the manuscript. M.A.Raja collected and contributed the data for analysis. Chandran Masi drafted the original manuscript and supervised it completely.

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