

Elevated Temperature Effects on the Properties of Self Compacting Mortar Containing Nano Materials and Zircon Sand

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Research Article

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Abstract

The present research work tries to assess the performance of self compacting mortar containing zircon sand as substitute for river aggregate in combination with nano alumina and nano silica as additive for cement. The fresh state results as observed through slump cone and mini V funnel showed positive effects of zircon sand balancing the negative effect of nano particles addition on workability. The mechanical properties, durability and microstructure of the mortar were assessed by conducting experiments at normal temperature and after subjecting to temperatures of 200⁰C, 400⁰C, 600⁰C and 800⁰C. The results indicate that addition of nano alumina and nano silica contributed towards the mechanical strength enhancement at elevated temperatures in combination with zircon sand which is a very good refractory material. The durability of the self compacting mortar at elevated temperatures enhanced due to the combined action of nano materials and zircon sand which is evident through microstructure analysis.

Highlights

- Performance of nano silica and nano alumina modified zircon sand self compacting mortar
- Strength and durability of mortar enhanced due to nano particles
- Promotion of hydration at higher temperatures by nano materials by consumption of Calcium hydroxide
- Microstructure modification due to nano materials and thermal stability enhancement due to zircon sand

Introduction

Fire is one of the serious threats that any concrete structure can face in its lifetime (Wasim and Hammad, 2015). The damage caused by fire is vast not only to the environment but also to life and property (Ulrich, 1988; Rashad, 2015). Though concrete can sustain high temperatures their failure pattern is unpredictable leading to a catastrophe (Nima et al, 2013; Chan et al, 2000). The durability studies of concrete also consider fire study as an integral part of their study (Ali et al, 2020). The prolonged exposure to heat weakens the integrity of concrete and thus makes them fail all of a sudden (Mohamed Salhi et al, 2020). In addition these are some concrete structures that are always subjected to temperatures higher than that intended for namely refractory and nuclear purpose concretes (Yanfeng et al, 2008). 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 (Hanaa et al, 2009). Well developed hydration phases can make the concretes thermally stable (Nuruzzaman et al, 2020). 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 must possess thermal stability. One such material is zircon sand that are available plenty in nature occurring along the ancient coastlines (Umarajyadav and Vahini, 2017). They are highly stable to high

temperatures and are also chemically inert (Richard et al, 2004). The chemical and thermal stability of the zircon sand makes them a widely used material for use in thermal applications and foundry (Rand et al, 2018). The use of zircon sand as fine aggregates shall provide a solution for reducing the demand on natural river aggregates as well as meeting the thermal requirements of aggregates (Renisha et al, 2019).

Though self compacting concrete is a universally accepted material they also possess several disadvantages such as presence of voids and pores which forms channels for the ingress of harmful ingredients into concrete due to the lack of any external vibration techniques (Hosseini and Farhad, 2020). Generally self compacting concrete is used as filling agents for reinforced concrete structures and hence any formation of micro cracks causes the penetration of CO₂, chlorides and moistures weakening the structure of the concrete (Brahim Safi et al, 2013). The concrete reinforced using steel is not highly resistant towards such deleterious substances. Therefore to minimize the risk caused to the reinforcements, the usage of ultra fine and nano materials for the design self compacting concrete becomes an inevitable choice (Zapata et al, 2013). The properties of the self compacting concrete mainly depend on the mortar phase and hence study if mortar properties explain the concrete behaviour (Tung-Chai Ling et al, 2012). Nano mortar is a general term used for the mortar containing nano material as an additive (Zhan et al, 2019). The bulk properties of concrete are significantly improved by the inclusion of nano sized particles in concrete through improvement in packing capacity (Zhenhua et al, 2006). The micro pores of concretes are reduced to nano scale and nano pores are completely nullified due to nano material addition (Shaikh et al, 2014; Nazari and Riahi, 2011). The drawbacks found in concrete such as permeability, porosity and alkali silica reaction are also found to reduce due to the nano material addition (Singh et al, 2013; Muhd Norhasri et al, 2017). Hence more durable and enhanced concrete performance can be attained through the filling capacity of nano particles and also through their involvement in the chemical hydration reaction (Sobolev and Ferrada Gutierrez, 2005). Since the emergence of nano technology, several nano sized particles have been produced by limiting their size to nano levels without affecting their original chemical composition and physical properties (Rahmat et al, 2015). Nano materials have already been used in self compacting mortars for the past decades to yield sufficient strength and stability (Ehsan et al, 2015). Highly innovative materials have been used in concrete that are of micro and nano scale have yielded good thermal stability and mechanical strength of the mortar (Nestor et al, 2014). Comparatively the use of nano size materials has yielded much more positive results than the micro particles (Berra et al, 2012). Use of nano materials as replacement or additive to cement also involves several technological implications (Elzbieta et al, 2017). The proper dispersion of nano materials in cement should be taken utmost care otherwise which could lead to negative consequences. The proportion of nano materials required to produce high quality concrete system also needs to be assessed before they can be incorporated in concrete system (Deyu Kong et al, 2012).

This research work mainly aims at utilization of zircon sand in self compacting mortar with the combined effect of nano-silica and nano alumina as additive for cement. Despite several studies on self compacting mortar, no study has been attempted to characterize the combined effects of adding nano silica and nano alumina on the properties of self compacting mortars with zircon sand aggregates have

also not been studied so far. The study is an initiative to create confidence on the use of zircon sand as an ingredient for the production of self compacting mortars. The research also tries to harness the thermal stability of zircon sand to improve the fire resistant behaviour of self compacting mortars.

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 nano silica and nano alumina with 99% purity were used as cement additive. Natural river sand and zircon sand conforming to zone II of BIS 383–1970 is used as fine aggregates in the present study. Chemical oxide composition of the raw materials is presented in Table 1.

Table 1
Chemical composition of cement
and zircon sand

Oxide	Cement	Zircon sand
SiO ₂	21.56	21.20
Al ₂ O ₃	6.67	6.911
Fe ₂ O ₃	6.17	3.155
CaO	49.88	-
MgO	4.51	-
SO ₃	2.75	-
K ₂ O	0.76	-
Na ₂ O	0.43	-
V ₂ O ₅	-	0.384
Cr ₂ O ₃	-	0.465
ZrO ₂	-	67.685
Cl	-	< 0.005
LOI	2.79	< 0.01

MIX PROPORTION

Five different self compacting mortar mixes were produced by varying the proportions of zircon sand as fine aggregates and nano particles (nano silica and nano alumina) as cement additives. A constant

binder content of 700 kg/m³ was maintained throughout the mixtures. The ratio of nano particles were adopted as 0.5%, 1%, 1.5% and 2% by weight of cement in binary combinations. The super plasticizer dosage was chosen as 4.5 kg/m³ to attain the prescribed slump flow diameter values of 240 to 260 mm (Ehsan et al, 2015). The water binder ratio was maintained as 0.4. 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%).

Table 2
Mix Design

MIX ID	BINDER			FINE AGGREGATE	
	CEMENT	NANO SIICA	NANO ALUMINA	SAND	ZIRCON SAND
	(kg/m ³)				
SCM	700	0	0	1210	0
SCM-Z25	700	3.5	3.5	907.5	302.5
SCM-Z50	700	7	7	605	605
SCM-Z75	700	10.5	10.5	302.5	302.5
SCM-Z100	700	14	14	0	1210

CASTING and CURING

The ingredients of self compacting mortar mixes were initially weighed and mixed in a laboratory type mortar mixer. The nano alumina and nano silica 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 its dry state were allowed to mix in the mixer for about 3 minutes after which the water is added within the next 2 minutes. After thorough mixing of the ingredients the mortar is placed in the different sized moulds. The casted specimens with the moulds were placed in room temperature for about 24 hours after which they are demoulded. The specimens were then kept under water for 28 days duration to undergo curing before they can be tested.

HEATING OF THE SPECIMENS

The self compacting mortar specimens after 28 days curing were taken and then heated in an electric furnace to the desired temperature. The refractory furnace has 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 thermostat that can control the temperature and heating rate. The mortar specimens were placed inside the furnace and the temperature is increased from room temperature in increments of 200⁰C to the temperature of 800⁰C. The uniform heating of the specimens were ensured through keeping the specimens at the target temperature inside the furnace for about 2

hours. The specimens were then removed from the furnace and maintained at laboratory to attain room temperature before the testing is carried out.

Experiments

The details of the experiments performed on the self compacting mortar and the corresponding dimensions of the specimens used with their codal specifications for each test are shown in Table 3. The fresh state properties of the self compacting mortar mixes were measured using mini slump flow test and mini V funnel test. The test was performed confirming to the EFNARC standards.

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 x 100 x 100 mm prism
Ultrasonic Pulse Velocity	IS 13311- I: 1992	150 mm cube
Durability tests		
Water absorption	ASTM C-642	50 mm thick x 100 mm dia
Porosity	ASTM C-642	50 mm thick x 100 mm dia
Electrical Resistivity		50 mm thick x 100 mm dia
RCPT	ASTM C1202-1995	50 mm thick x 100 mm dia
RCMT	NT-Build 492 1999	50 mm thick x 100 mm dia
Bulk diffusion	ASTM C 1556	50 mm thick x 100 mm dia
Microstructure		
SEM	-	Broken powdered specimens
XRD	-	Broken powdered specimens
FTIR	-	Broken powdered specimens

Results And Discussions

Fresh state results

The workability results of the self compacting mortar containing zircon sand and nano materials in Fig. 1 clearly showed decreased flow values due to the incorporation of the nano particles in the self compacting mortar mixes. Generally the self-compacting nature of the mixes is expected to be increased

with increase in the zircon sand content. But the nano particles demand more water to wet their surface thereby reduces the free water availability leading to decreased flowability. The zircon sands are non water absorbing in nature and hence increases the free water availability for increasing the fluidity of mortar. Therefore it can be affirmed that zircon sand is the sole contributor of the workability improvement in the zircon sand substituted self compacting mortar mixes containing nano materials when w/b ratio and superplasticizer dosage are maintained constant. Moreover the decreased flowability caused by the addition of nano materials was partially nullified by the increasing zircon sand content that possesses weak inter molecular cohesive force between them due to their glassy and smooth surface texture.

Compressive strength

The compressive strength results in Fig. 2 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 nano-silica and nano alumina that filled the voids created between the zircon aggregates thereby minimizing strength reduction. The non water absorbing nature of the zircon sand increases the amount of free water in the self compacting mortar that can be utilized for wetting the nano particles. The higher surface area, fineness and the high reactivity of the nano particles also reduced the loss in compressive strength of the mortar even at higher percentage substitution of zircon sand. The cohesion improvement between the cement pastes and zircon sand aggregates contributed by the nano particles 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 high temperatures also clearly showed that the compressive strength was increased at 200⁰C for all the mortar mixes. It can be said that after exposure to 200⁰C the mortar gains strength due to the thermal activation of the nano particles. The mortar strength variation is due to the zircon sand that is highly thermally stable. The zircon sand can withstand the thermal shock to a great extent and hence is being used widely for the refractory purposes.

Flexural Strength

The flexural strength results of the self compacting mortar mixes in Fig. 3 clearly explained the favourable effects of nano particles in neutralizing the negative effect of the brittle zircon sand substitution on the flexural strength decrement of the mortar. The addition of nano materials improved the cohesion of zircon sand and the cement matrix thus partially contributing towards the flexural strength of the mortar at normal temperature. The decrement in the flexural strength beyond 25% substitution of zircon sand and increasing nano particle addition may be due to the self desiccation of the CSH gels that are formed due to increased reaction sites presenting the unavailability of the spaces for further hydration to take place. The decreasing trends of flexural strength may also be reasoned from the increase in the brittleness of the mortar with increasing zircon sand substitution. Moreover the zircon sand substitution at higher percentage levels creates brittle zone that paves the way for the formation of micro cracks. However the nano particles 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 nano alumina was relatively lower at higher temperatures. The nano particles also acted as a nano reinforcement material by providing strength to the mortar by resisting the propagation of micro cracks. The nano alumina is also highly resistant towards high temperature and improved the flexural strength of mortar at higher temperatures. The flexural strength increment with increasing zircon sand substitution and nano particles addition at a temperature range of about 200°C and 400°C may be explained by the reduction in pores caused due to the nano particles and zircon sand aggregates.

UPV

The UPV value of zircon sand substituted mortar mixes are found to be higher than normal self compacting mortar for all the mixes as shown in Fig. 4. The higher rate of hydration and the higher specific surface of the nano particles reduced the porosity of the mortar mixes which subsequently influenced the ultrasonic pulse velocity. The denser internal structure of the mortar caused subsequent increase in the pulse velocity which in turn signifies the filling capacity of the nano materials in combination with zircon sand. The ultrasonic pulse velocities of the self compacting mortar mixes at elevated shows a significant improvement. Generally a rapid reduction in ultrasonic pulse velocity is observed due to the increase in the pore structure and micro cracking 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 also observed upto 600°C without a steep decrease indicating the higher thermal stability of the zircon sand that can withstand the thermal effect even at high temperatures.. When the quality of the produced mortar mixes were considered all the zircon sand substituted mortar mixes exhibited improved ultra sound velocity than the control mortar at all temperatures and belonged to 'good' quality grading upto 400°C as per the quality gradation given in IS 13311-1 (1992). This decrease of micro cracks formed in mortar after exposure to high temperatures due to the zircon sand substitution may also be the cause for the improved UPV values.

Water Absorption

The water absorption values of the mortar at normal temperatures and after subjected to high temperature is shown in Fig. 5. From the results obtained it can be clearly inferred that the water absorption values were reduced in the mortar which contained zircon sand when compared to the other mortar mixes. The decreased water absorption values of the mortar is a function of zircon sand and nano materials that minimized the free water available in the mortar thus refining the pore structure of mortar. The saturated water absorption values depend on the quantity of pores in the mortar. The minimization of pore sizes due to filling by nano particles also contributes to the decreased water absorption values. The attainment of 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 its 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 400⁰C. 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 nano materials prevent the disruption of cement matrix by holding the ingredients together even at high temperatures and contributed to reduction of channels available for the intrusion of moisture by blocking the water channels.

Porosity

The water porosity studies were done only to evaluate the pores in between the cement and zircon sand aggregates and hence is 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 nano particles that caused a perfect binding of the cement and zircon sand as shown in Fig. 6. The porosity values also clearly showed decreased value with increasing zircon sand substitution at all temperatures. The reduction in the micro cavities of the mortar due to the high specific surface area of the nano materials 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 nano silica also formed stabilized CSH gels that occupy the pores in the self compacting mortar thereby causing reduction in the pore volume. The effect of nano alumina also contributed to the porosity reduction especially at high 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 loss of water molecules at 200⁰C creates empty spaces in mortar thereby leading to increased porosity of mortar. The addition of nano materials had left no free water that can be evaporated. Moreover the formation of CSH gels occurs more actively around 200⁰C utilizing the water molecules due to thermal activation of nano particles. Therefore the porosity values of the nano particles added mortar were much lower than the control mortar at 200⁰C. Beyond 400⁰C the decomposition of the CSH gel takes place which was minimized due to nano materials addition. The porosity reduction is essentially a function of two parameters namely extent of formation hydration products and filling of pores. Both the parameters were achieved due to addition of nano silica of nano alumina. Moreover the stable nature of zircon sand towards high temperatures also held the ingredients of mortar together in place without leading to weathering of aggregates at high temperatures.

RCPT

The rapid chloride penetration test (RCPT) values of the self compacting mortar at normal temperatures and after exposure to high temperatures are shown in Fig. 7. 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 nano materials increased in the mortar. The hydration of ions through mortar is electrolytic through

the cement paste and the aggregates generally do not contribute to 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 nano silica in the mortar causes a decrease in the chloride penetration of the self compacting mortars through their pozzolanic action. The inclusion of nano materials 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 nano alumina inclusion also effectively held the pore waters together utilizing them for hydration reaction. The compact nature of the mortar can also be visualized 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 that reduced the travel of ions in the mortar thereby avoiding the formation of micro voids.

RCMT

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 Fig. 8. The addition of nano particles improve 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 nano particles in comparison with the cement paste increased the fineness content of the cement paste thereby forming impermeable layer around the aggregates. The mortar containing zircon sand was also highly resistant to high temperatures without allowing the micro cracking of mortar at the aggregate surface and the cement paste. The increased dense CSH gel formations also screens the ingress of chloride ions thereby reducing their migration coefficient. The additions of fine aggregates with 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 less 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 non porous compact structure. The increasing temperatures cause the loss of free water in the mortar increasing the void ratio of mortar. But due to the additions of nano alumina and zircon sand the amounts of water added were utilized to wet their surface and also completely utilized for hydration reaction. The minimized separation of the ingredients of the mortar at high temperatures due to the thermal stability of the produced mortar can also be reasoned as the fact for the reduction of migration coefficient of mortar.

Electrical Resistivity

The electrical resistivity of the mortar at various ages is shown in Fig. 9. Since self compacting mortars are essentially used for filling in the reinforcements the determination of electrical resistance becomes mandatory. The electrical resistivity of the mortar mixes increased as the replacement level of the fine aggregate using zircon sand and nano particles increased but only upto a certain extent. This variability

in the electrical resistivity was mainly due to the increase in the denseness and decrease in the porosity of the mortar mixes due to the synergistic effect of zircon sand and nano particles. The increment in the electrical resistivity of the self compacting mortar with increase in the temperatures may be attributed to the improved hydration products and extra CSH gel formation due to the pozzolanic action of the nano silica and thermal activation of nano alumina. This increment in the electrical resistivity with increasing temperatures may also be contributed by the decreased porosity, tortuosity and chemistry of the pore solution and pore network. The positive effect of nano alumina in increasing the amounts of denser and thicker CSH gels has formed barriers that prevented the ingress of electric charges. The passage of electric current mainly depends on the pore water present in mortar. The nano particles addition has led to decrease in the quantity of free water available for conducting the electric current. Moreover the increased reaction sites of nano silica also have led to the utilization of water for the complete hydration of CH and hence no free water can be available. The pore filling characteristics of nano alumina and zircon sand also effectively plugged the pores of mortar thereby removing the spaces through which water can reside. The zircon sand substituted self compacting mortar mixes showed good resistivity values indicating the quality of CSH gels produced. The addition of nano silica and nano alumina acts as thermal activator that produces additional CSH gels at 200⁰C. Beyond 400⁰C the zircon sand due to its thermal stability well functioned as inhibitor of passage of ions into the mortar by maintaining the structural stability.

XRD

The XRD analysis done on the 28 days water cured self compacting mortar mixes is shown in Fig. 10. The various mineralogical phases present in the mortar mixes is shown in Fig. 10 by comparing with 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 arises from the inert aggregates. The presence of portlandite is deduced in all the patterns however the reduction in the portlandite phases indicates the consumption of CH leading to formation of secondary hydration products. The patterns also clearly showed improved portlandite phases at the temperature 200⁰C due to the thermal activation of nano silica particles in the mortar. The other phases were almost similar in all the mixes showing that the mineralogical phases were almost unaffected due to temperature. The XRD patterns of the mortar mixes after exposure to 400⁰C clearly showed the disintegration of portlandite leading to the release of bound water from –CH and CSH phases. The XRD patterns after exposure to 600⁰C and 800⁰C 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 nano materials has caused the improved crystalline hydration products. Generally CSH gels are amorphous in nature and their identification in XRD is a little tough. At 400⁰C the decomposition of portlandite peaks was clearly visible in the control mortar whereas the portlandite peaks were 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 beyond 600⁰C

all the portlandite phases were completely reduced showing the decomposition of Ca(OH)_2 . The portlandite phases decomposed severely at 800°C and no peaks were found. At 800°C only the minor peaks of calcium silicate and quartz were found.

FTIR

The FTIR spectra of the mortar mixes after exposure to temperatures are shown in Fig. 11. The decreasing broad vibrations of the water molecules around 3400 cm^{-1} were found with increasing temperatures. The spectra diminished with increasing temperatures and was 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 hydration products with increasing temperature causes increase or decreases 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.

SEM

The SEM images of the zircon sand substituted mortar mixes showed uniform distribution throughout the cement matrix which is clearly evident from Fig. 12(a) and Fig. 12(b). The denseness of the self compacting mortar was also improved as seen through the SEM images due to the contribution of nano silica. The images also show that the pore reduction may be caused by the filling effect of the nano particles. 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 colour and the SEM images clearly shows the grey phases which are found to decrease with increase in the 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 addition of nano materials. The homogeneous distribution of nano silica and nano alumina functioned as fillers in the mortar bonding agent due to their dilution effect. It can be seen that the incorporation of nano particles transformed the CH crystals into CSH gels that surrounded the fine aggregates thus forming denser and more compact microstructure. The higher surface energy of the nano-silica 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 nano particles and the zircon sand aggregates even at high temperatures which stands as evidence to the above stated mechanical strength results.

Conclusions

From the experimental results obtained the following conclusions can be arrived:

1. The workability of the mortar increased due to zircon sand substitution nullifying the effect of additions of nano alumina and nano silica. The visual observation of the fresh mortars containing zircon sand and nano materials showed no segregation and bleeding.
2. The compressive strength of mortar increased at all temperatures due to the substitution of zircon sand as fine aggregate and nano particles as cement additive. The decrement in the strength value of the zircon sand substituted mortar mixes at higher temperatures was also minimal due to the thermal stability of zircon sand.
3. The flexural strength of the mortar mixes after exposure to high temperatures was also much increased in comparison to control mortar mix. The flexural strength increment is a result of the thermal stability of zircon sand in combination with nano alumina that functioned as significant filler in mortar.
4. The increment in the split tensile strength at higher temperatures may be attributed by the addition of nano alumina and zircon sand substitution that increase the strength of interfacial transition zone leading to reduced bleeding and shrinkage.
5. The water absorption values of the mortar containing zircon sand as fine aggregate and nano particles as cement additive showed lesser values when compared to the control mortar after exposure to high temperatures.
6. The higher amounts of hydration products present in the mortar due to nano particles substitution and thermal stability of zircon sand contributed to the reduced porosity at all temperatures.
7. The chloride ion penetration was also much lower for the mortar at all temperatures due to the increased pore structure refinement due to nano alumina and nano silica addition. The low transport properties of the mortar aided by the nano particles addition and zircon sand substitution have contributed to the reduced chloride ion penetration in the mortar even at high temperatures.
8. The UPV values of the mortar mixes containing zircon sand and nano particles at 200⁰C showed values well above 4.5 km/sec and thereby making the mortar categorize under excellent quality. The mortars even at high temperatures upto 400⁰C were of good quality and above 600⁰C showed average quality whereas the control mortar were poor category when exposed to high temperature.
9. The electrical resistivity of mortar mixes was also found to increase with increasing zircon sand substitution. The temperature increase decreased the electrical resistivity of mortar but the reduction was only marginal due to the pore filling characteristics of nano particles and thermal stability of zircon sand that made the pores discontinuous.

10. The XRD patterns of the mortar mixes showed well developed crystalline hydration peaks of CSH and CH. The phases of formed hydration products peaks were found in all the mortar mixes containing zircon sand and nano particles indicating no hindrance to hydration process. The presence of hydration products as crystalline peaks even at high temperatures indicate that the produced mortar mixes with zircon sand were highly thermally stable.
11. The FTIR spectral curves of the mortar containing zircon sand and nano alumina also confirmed the presence of well developed hydration products. The presence of the bands of silicate at high temperature indicates the high thermal stability of the hydration products formed in the mortar. The maintenance of the chemical structure of mortar is also visible at high temperatures due to the effect of zircon sand and nano alumina.
12. The SEM images of the mortar mixes with zircon sand as fine aggregate and nano alumina as cement additive showed dense microstructure with minimal void volume. The rosettes of CSH was also evident in the SEM images of the mortar containing zircon sand at high temperatures indicating the high temperature stability of the produced hydration products in the zircon sand mortar. The lesser amount of pores indicates the higher contribution of nano particles and zircon sand towards the minimal strength reduction at higher temperatures.

The final conclusion can thus be obtained that the use of zircon sand as partial substitute for fine aggregate and nano particles (nano SiO₂ and nano Al₂O₃) cement additive positively influenced the mechanical strength and durability properties of mortar at normal and elevated temperatures.

Declarations

Competing Interests

The authors declare there are no competing interests.

Funding Statement

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Data Availability

The authors confirm that the data supporting the findings of this study are available within the article.

Authors Contributions

Author 1: Conceived and designed the conceptual ideas.

Author 2: Collected and contributed the data for analysis.

Author 3: Performed the analysis and prepared the manuscript.

Compliance with Ethical Standards

Conflict of Interest: The authors declare that they have no conflict of interest.

Research involving human participants and/ or animals:

This chapter does not contain any studies with human participants or animals performed by any of the authors.”

Informed consent

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

Consent for Publication

All the three authors give their consent for the publication of identifiable details, which can include photograph(s) and/or videos and/or case history and/or details within the text to be published in the SILICON Journal and Article.

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Figures

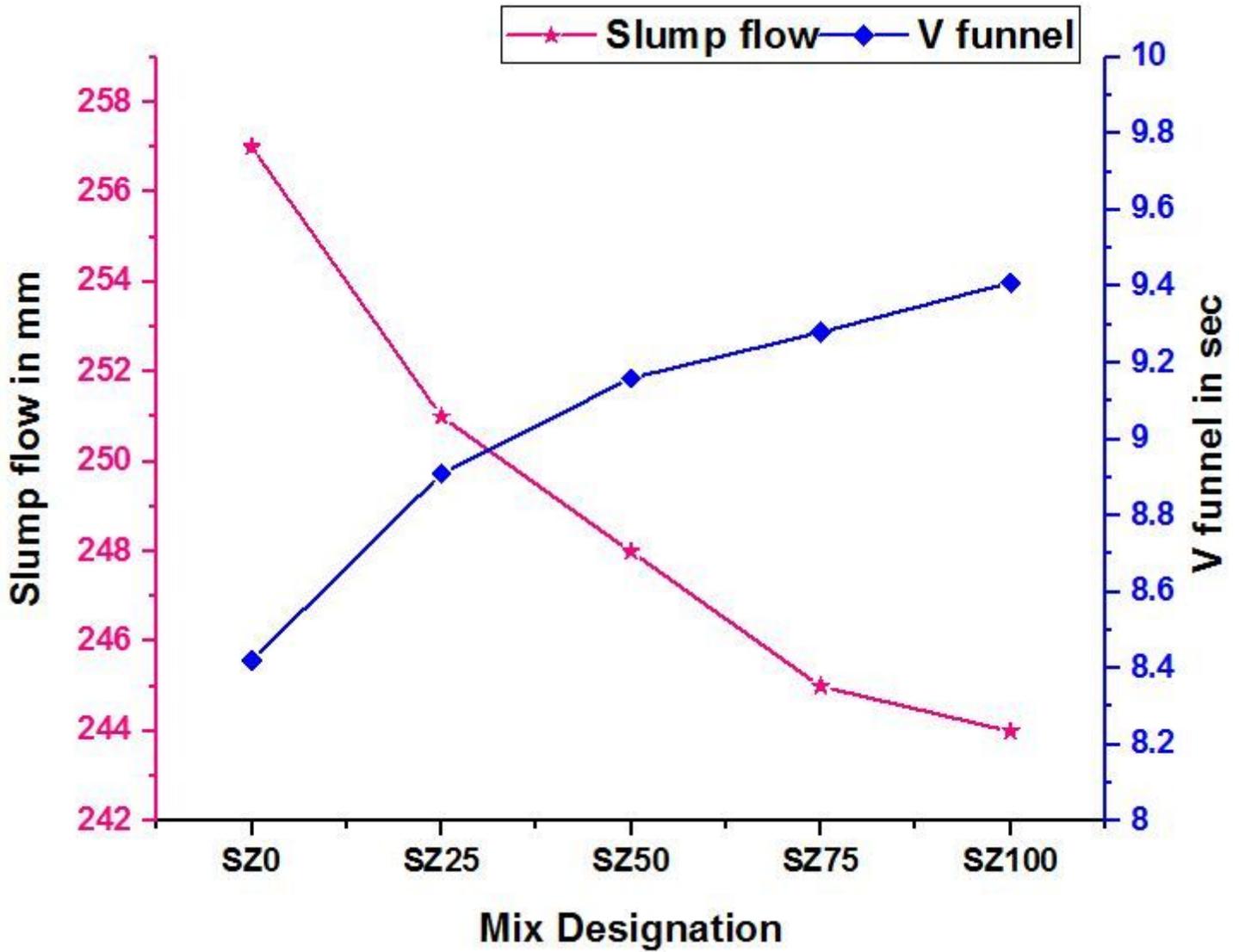


Figure 1

Fresh state results of self compacting mortar containing zircon sand and nano particles

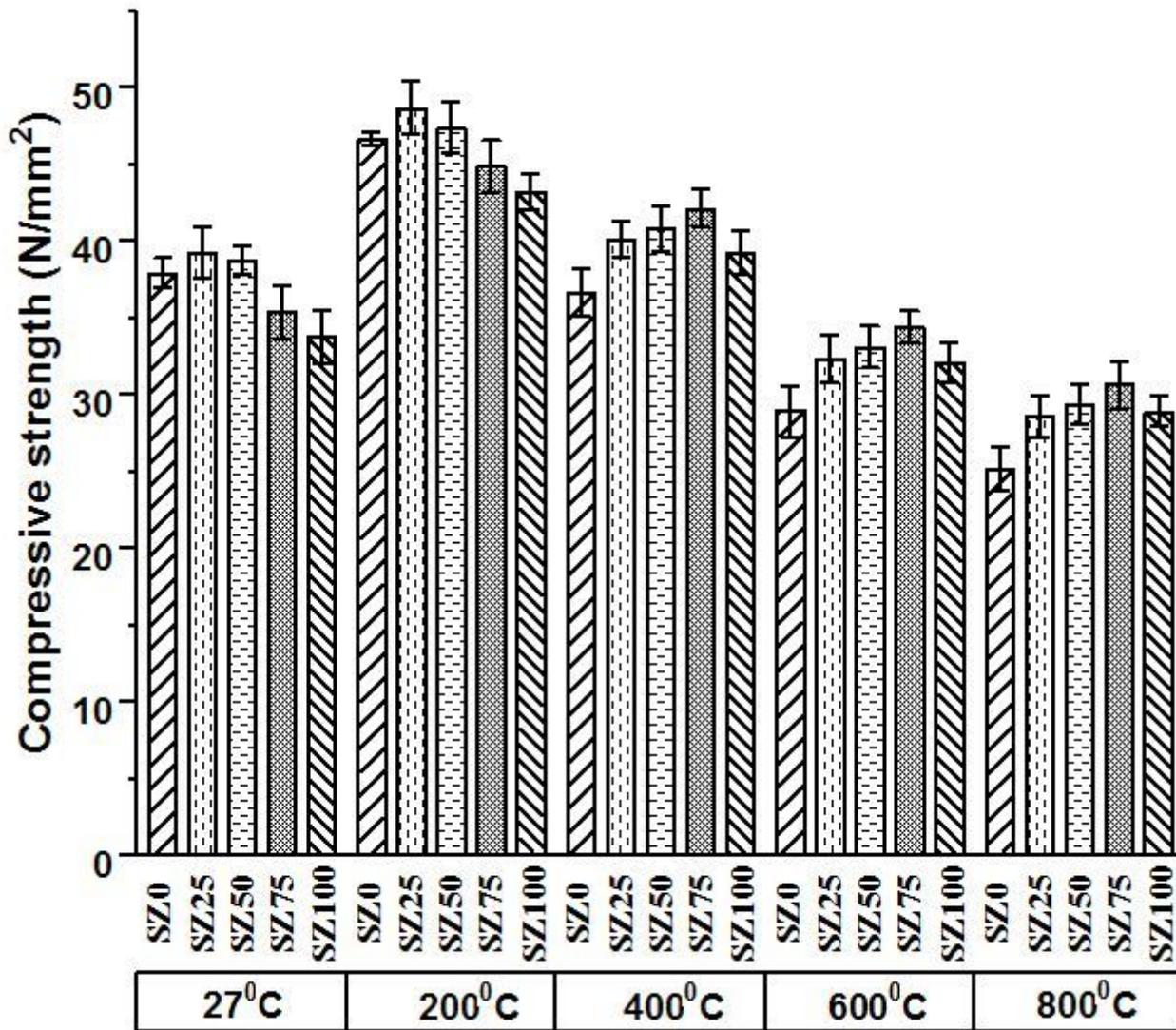


Figure 2

Variation of compressive strength of self-compacting mortars containing zircon sand and nano particles at normal and elevated temperatures

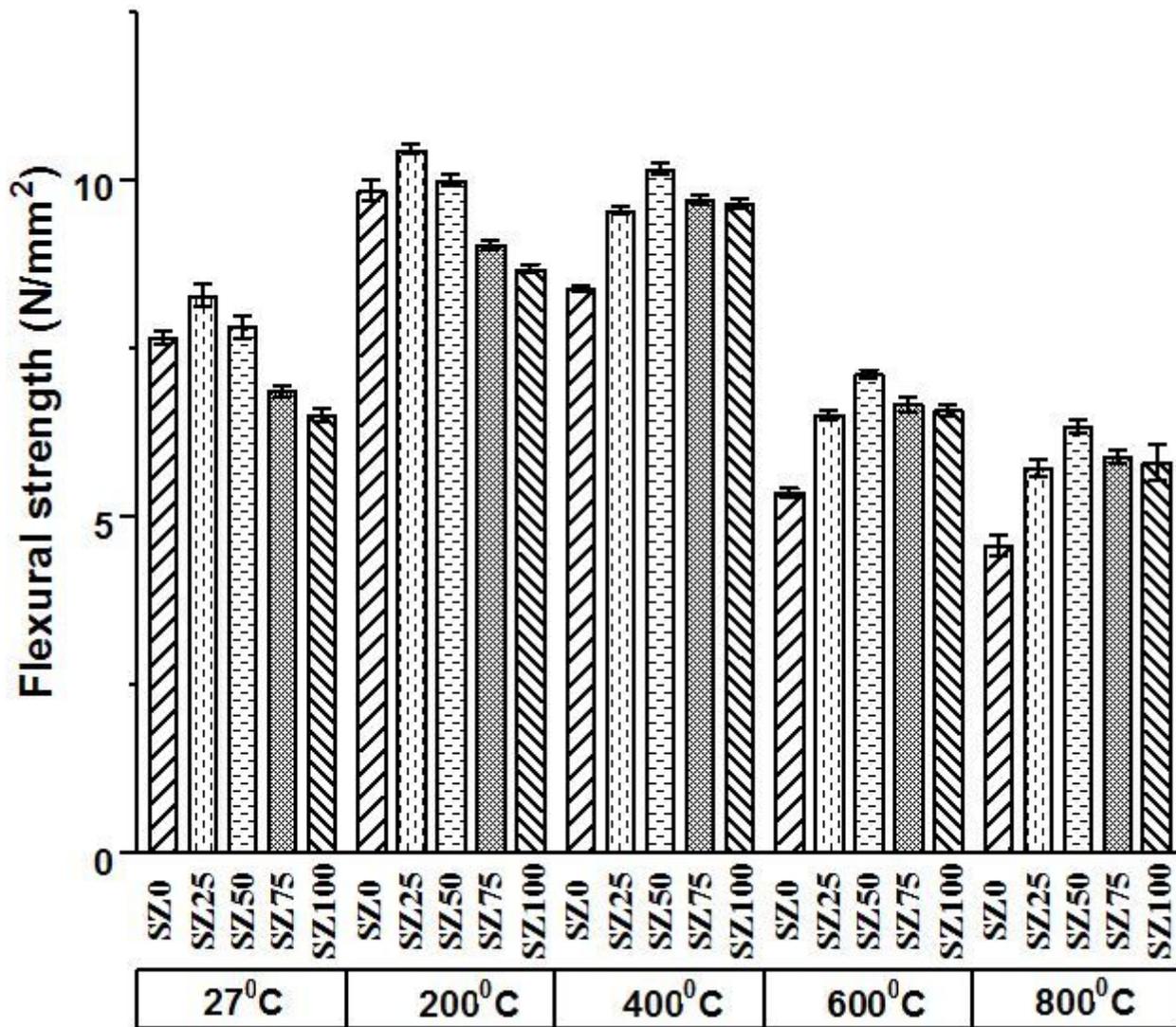


Figure 3

Variation of flexural strength of self-compacting mortars containing zircon sand and nano particles at normal and elevated temperatures

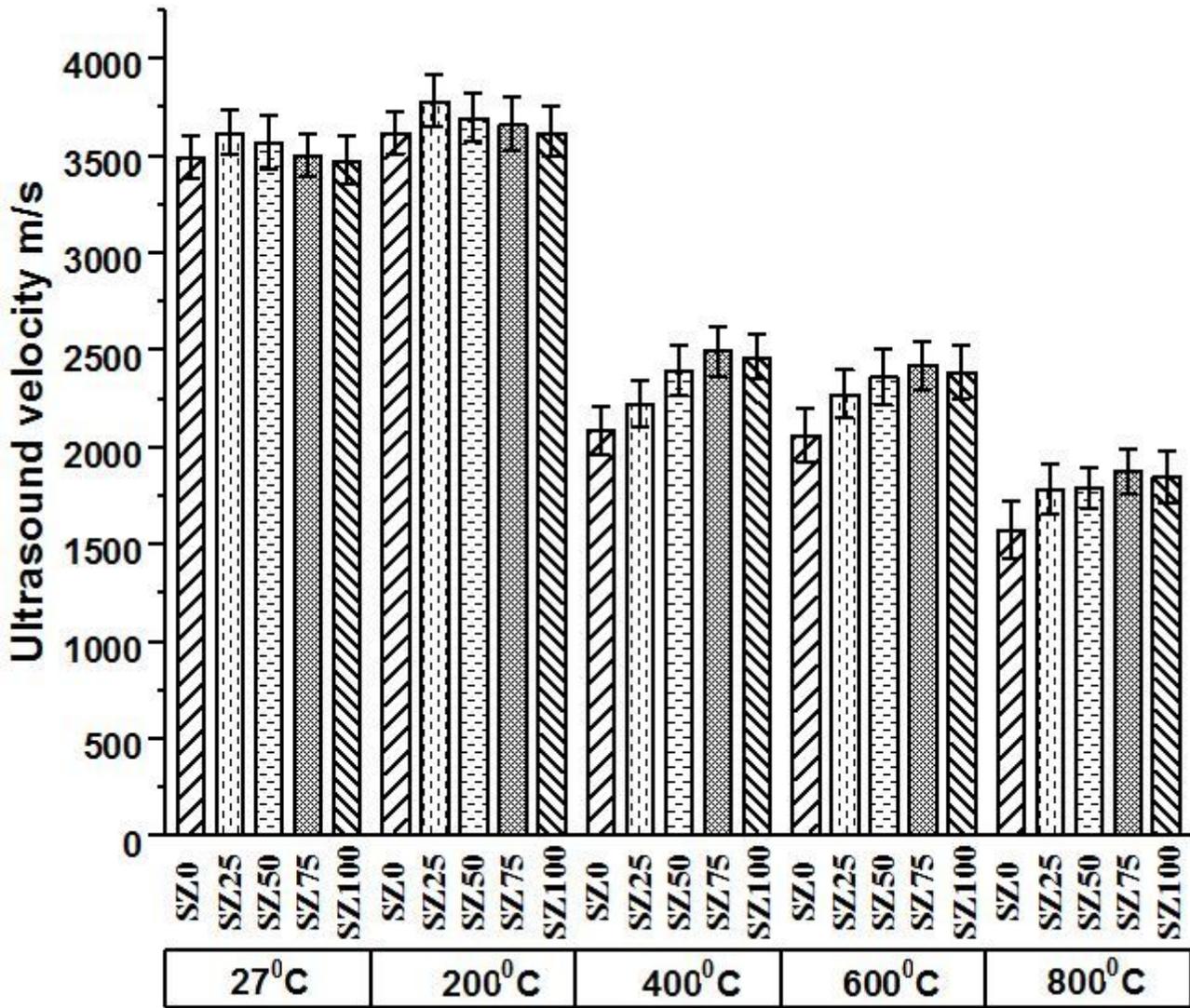


Figure 4

Variation of ultrasound velocity of self-compacting mortars containing zircon sand and nano particles at normal and elevated temperatures

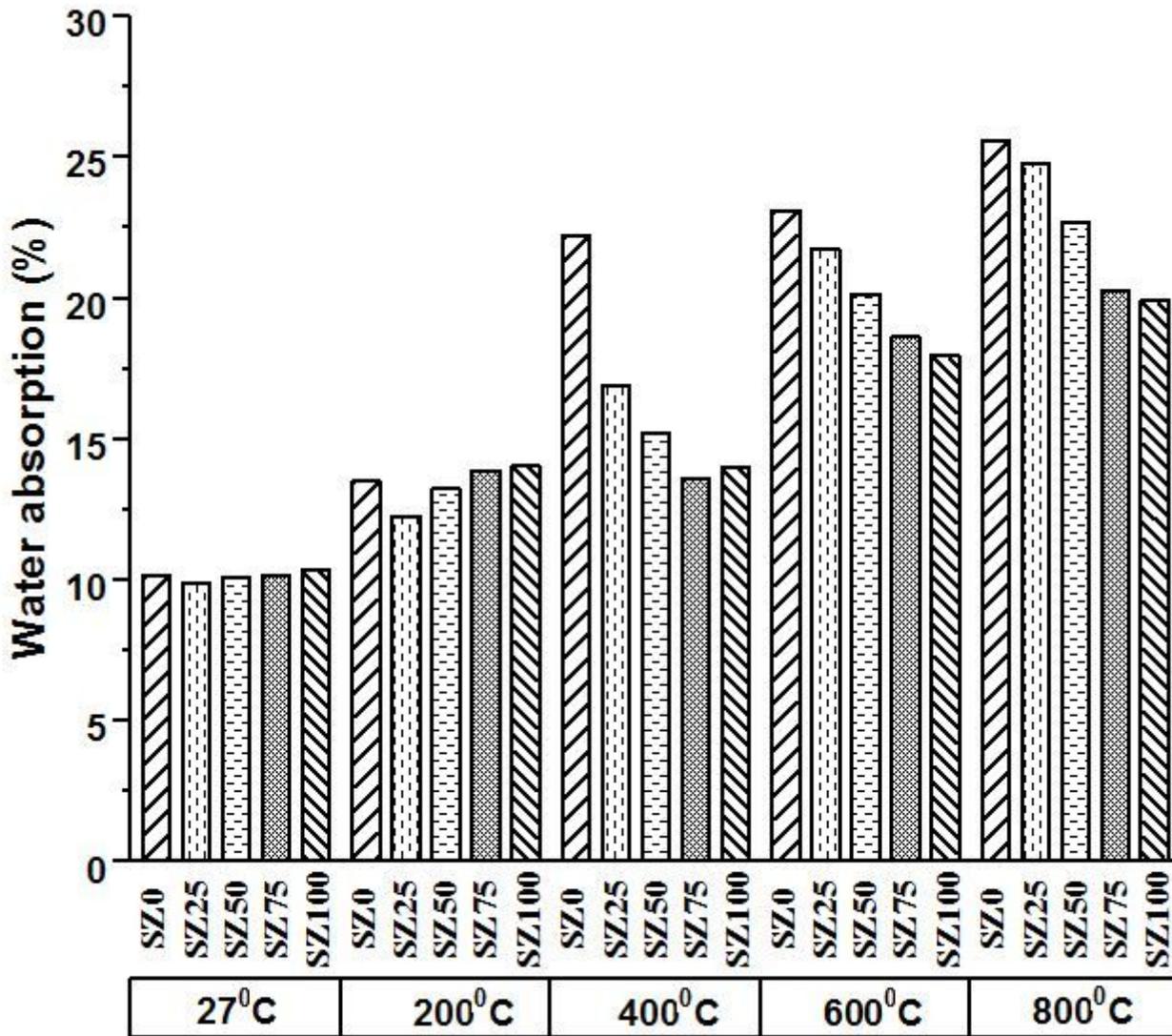


Figure 5

Water absorption of self-compacting mortars containing zircon sand and nano particles at normal and elevated temperatures

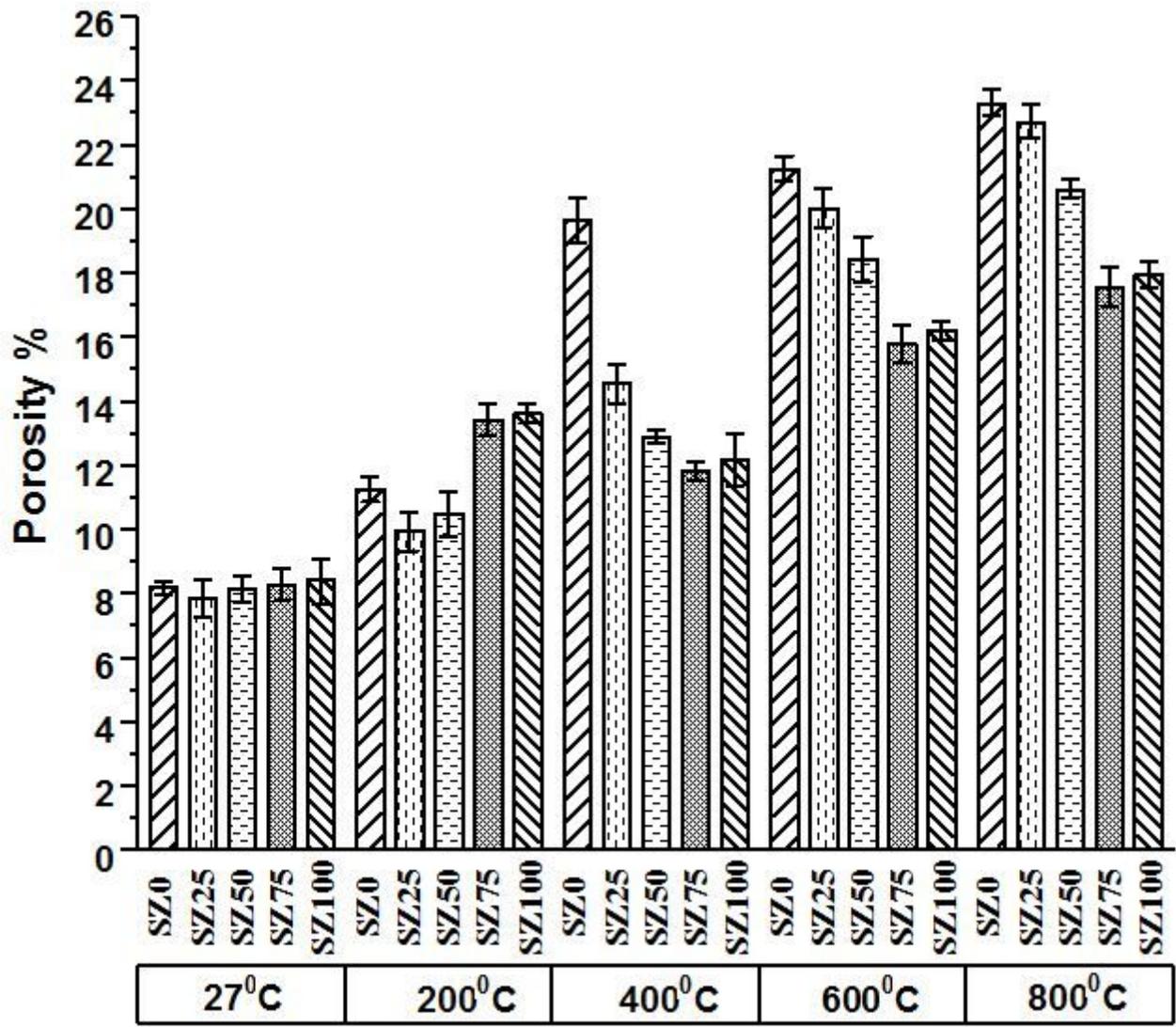


Figure 6

Porosity of self-compacting mortars containing zircon sand and nano particles at normal and elevated temperatures

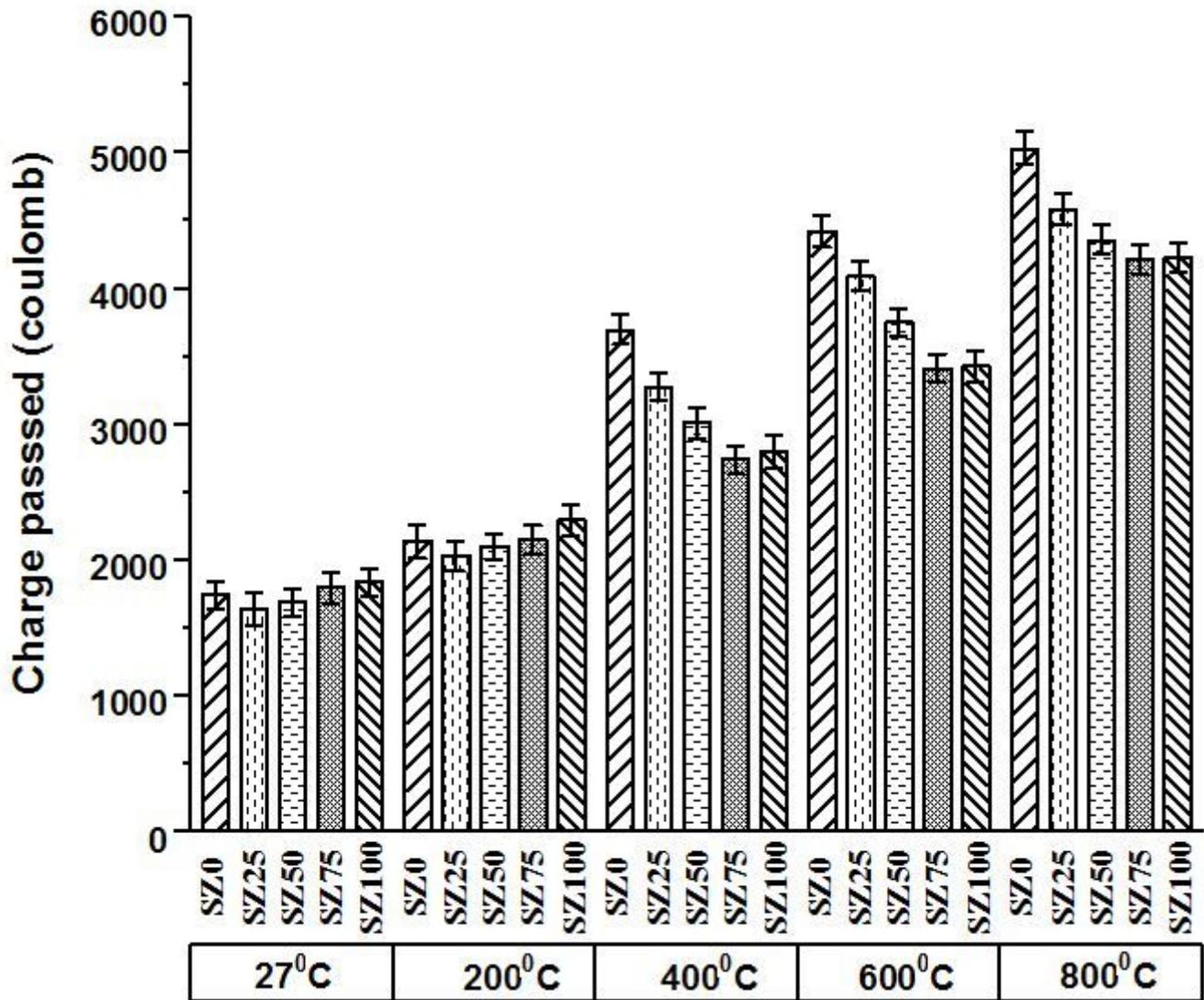


Figure 7

RCPT results of self-compacting mortars containing zircon sand and nano particles at normal and elevated temperatures

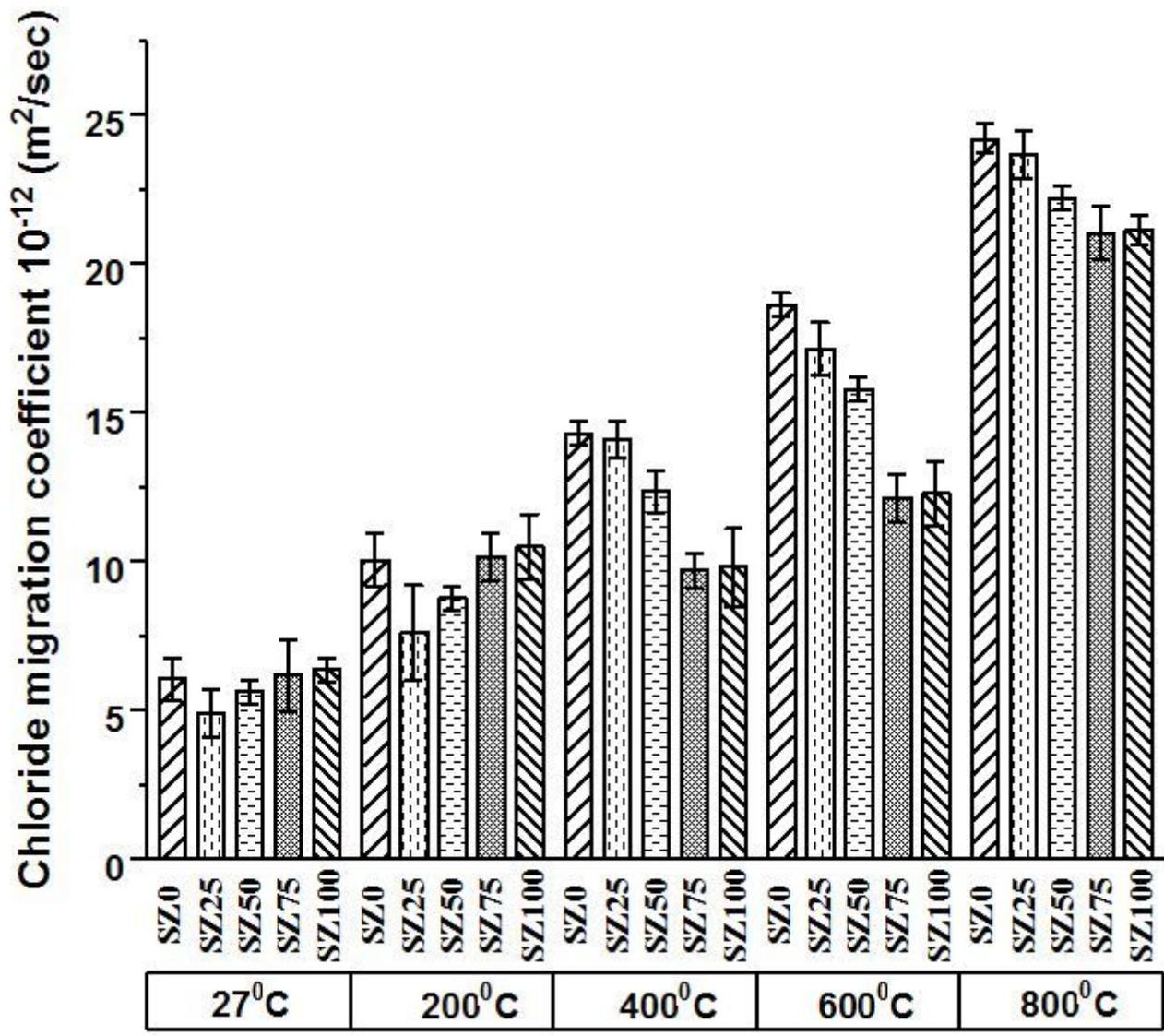


Figure 8

RCMT results of self-compacting mortars containing zircon sand and nano particles at normal and elevated temperatures

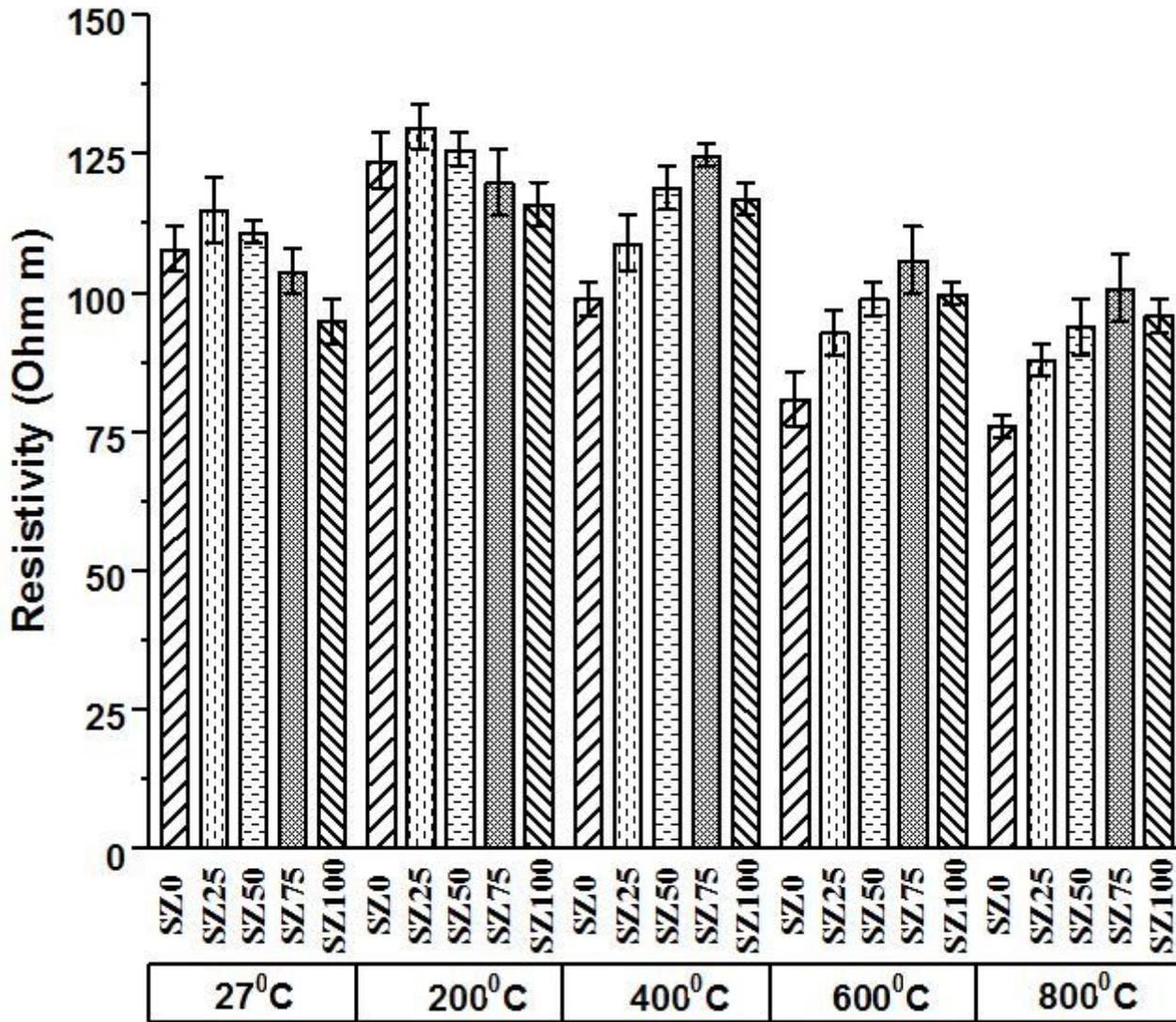


Figure 9

Electrical resistivity of self-compacting mortars containing zircon sand and nano particles at normal and elevated temperatures

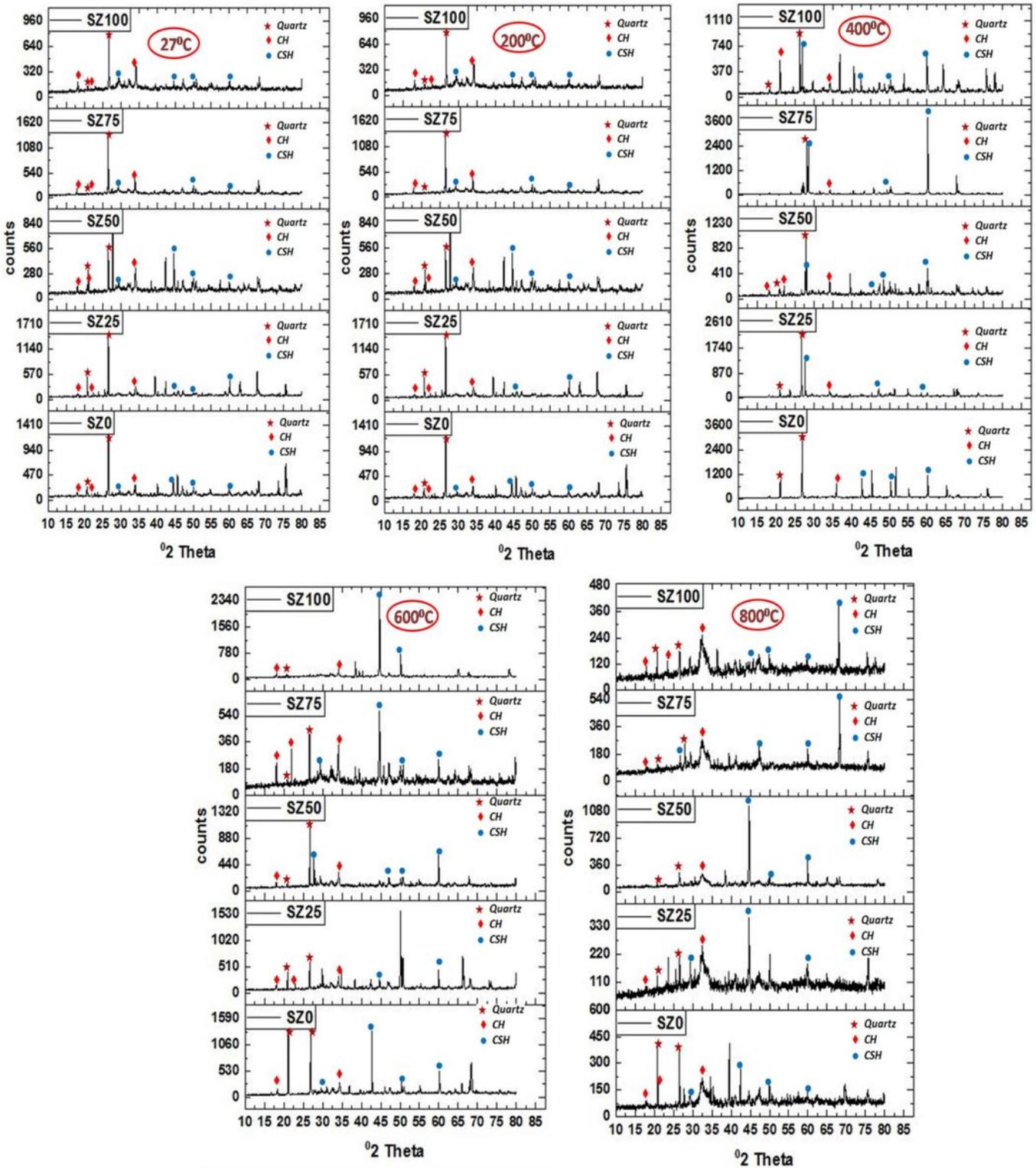


Figure 10

XRD spectra of self-compacting mortars containing zircon sand and nano particles at normal and elevated temperatures

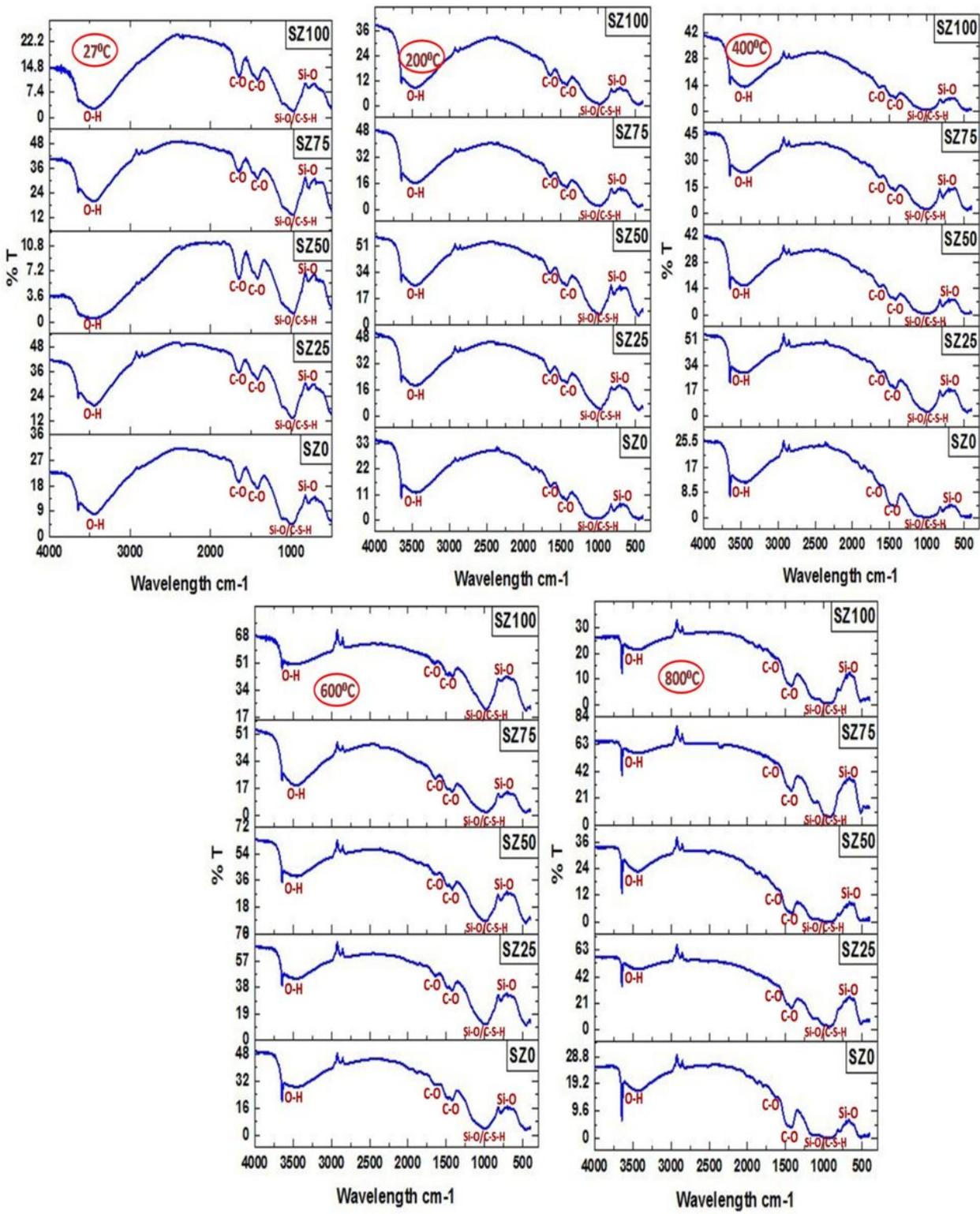


Figure 11

FTIR curves of self-compacting mortars containing zircon sand and nano particles at normal and elevated temperatures

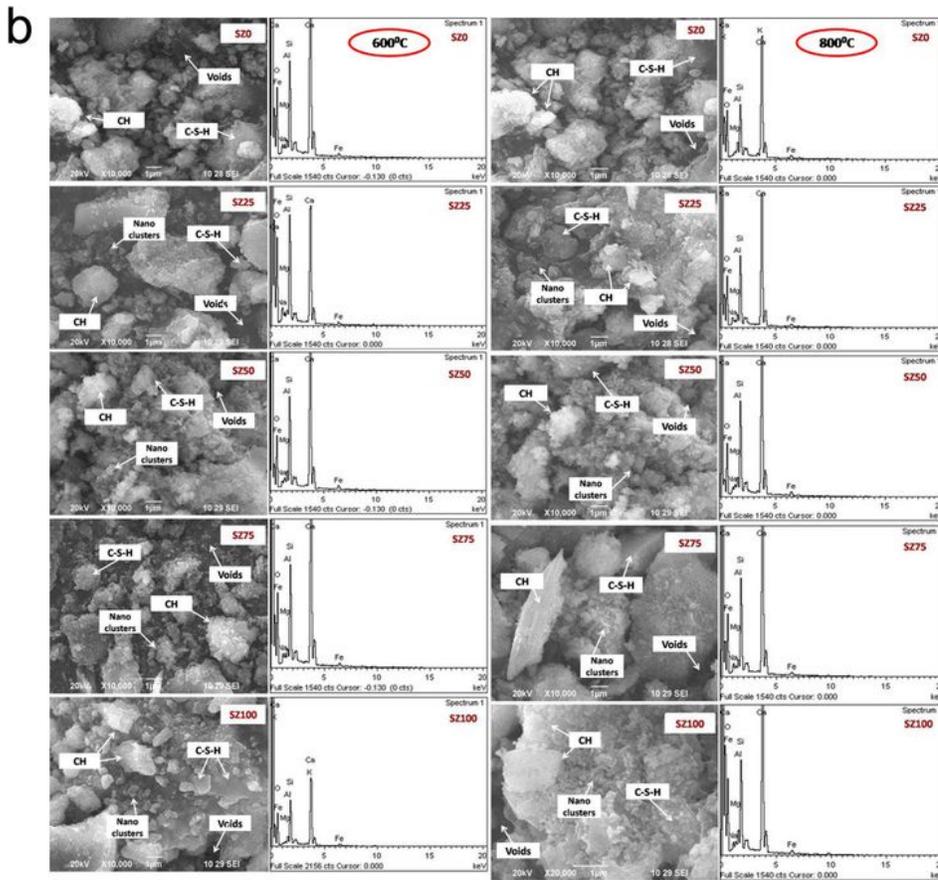


Figure 12

(a) SEM images of self-compacting mortars containing zircon sand and nano particles at 270C, 2000C and 4000C. (b) SEM images of self-compacting mortars containing zircon sand and nano particles at 6000C and 8000C.