Pozzolanic activity and durability of nano silica, micro silica and silica gel contained concrete

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Pozzolanic Activity and Durability of Nano Silica, Micro Silica and Silica Gel Contained Concrete

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Abstract. This paper aims to investigate the influence of replacement of cement with nano silica, micro silica and silica gel admixtures on pozzolanic activity, the replacement ratio was10% for all admixture, silica gel used in two forms (beads and crushed powder). Also, the water absorption test was investigated for obtaining the durability properties of concrete, in specimens for this test admixtures were added in four different dosages 1%, 2%, 3% and 4% by weight of the cementitious material into the concrete mixture. Experimental investigations of modified concrete were conducted after 28 days of water curing. Results showed that mixes of nano silica and crushed silica gel showed a higher pozzolanic activity index. For the water absorption test, all mixes incorporating nano silica, micro silica and silica gel showed lower absorption than control mixes best result were noticed with crushed silica gel and nano silica mixes. DTA analysis confirms the results for both poisonous activity and water absorption.

INTRODUCTION

Concrete is the most widely used material in the world. Its primary ingredient, cement, is also the costliest and environmentally unfriendly component in the concrete mix. The cement industry is one of two primary industrial producers of carbon dioxide (CO_2), creating up to 5 % of worldwide man-made emissions of this gas. Therefore, additives and admixtures are widely used in order to reduce the quantity of cement used and to obtain concrete of the same quality. [1]

Recent studies have explored the use of nanotechnology and nano-materials for enhancing the performance of cement paste. Nanoparticles have a high surface area to volume ratio, providing the potential for outstanding chemical reactivity. Nanoparticles can act as nuclei for cement phases (further promoting cement hydration due to their high reactivity), as the nano-reinforcement, and as a filler. Nano-SiO_2 has been found to improve concrete workability and strength, to increase resistance to water penetration [2]. Small amount of nano silica usually 0-6% replacement of cement is enough to enhance the properties of high strength concrete, addition of nano silica accelerated the hydration process and also reacts with Calcium Hydroxide to produce more amount of Calcium Silicate Hydroxide thereby improving the properties of concrete. [3]

YE Qing et.al have been studied the pozzolanic activity of nano silica and micro silica with content 3% of the weight of cement. Results indicate that the bond strength at the interface between aggregate and hardened cement paste, the compressive strength and the bonding strength of concrete incorporated with 3% nano silica increased more than those with silica fume. The pozzolanic activity of nano silica was much greater than that of micro silica. [4] 2006. *Alireza Naji Givi et.al.* Studied the size effect of nano silica on mechanical properties of concrete. 15nm and 80nm nano silica particles have been used with different contents 0, 0.5, 1, 1.5 and 2% by weight of cement. It was found that the concrete specimens with 15 NM, size harder and stronger than that of 80nm at an early age but this reversed at later ages. Results also showed that concrete with nano silica particles have higher compressive, flexural and split tensile strength than that without nanoparticles. [5] 2010. *Hasan Biricik et.al, 2014* investigated structural characteristics of cement mortars, impregnated with nano silica, silica fume and fly ash they found that the compressive strengths and flexural strengths developed in the mortar specimens containing nano silica particles

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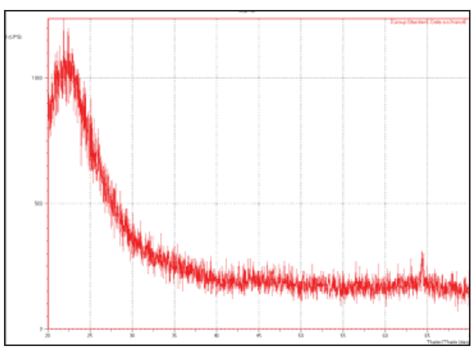
were found considerably higher than those of the corresponding specimen of silica fume and fly ash over and above the control at both ages. FTIR, TG-DTG and SEM analysis results were consistent with the remarkable increase in the mechanical strength of the mortars with NS. [6]. *Mohammed S.N. Et.al* studied the influence of nano silica and micro silica on the pozzolanic activity. Three proportions of NS 0.5, 5 and 10% of the weight of cement and three proportions of MS 5, 10 and 15 %, also three mixes of nano and micro silica were used results showed maximum enhancement was for 3% NS and for micro silica the improvement in 15%MS [7] 2016. *Peng Zhang et.al* used nano silica to reinforce high performance concrete Good development in both fracture toughness and water impermeability found by using nano silica. [8]2017.

Materials

Ordinary Portland Cement Type I, named Karasta, which are indicated that the cement is conformed to Iraqi specifications (I.Q.S.) No. 5/1984. The chemical and physical properties of the cement are shown in Table 1. Locally available natural sand with particles smaller than 0.5mm for mortar specimens and particles smaller than 2.5 mm for concrete mixes used as fine aggregate. Crushed gravel maximum size of 14mm and specific gravity of 2.8g/cm³ was used as coarse aggregate.

Micro silica is used as pozzolanic admixture. Physical and chemical specifications of micro silica are shown in Table 2. Nano silica is used as concrete admixture in this research, Table 3 which includes as received properties of nano silica. Silica gel purchased from Fluka Company is used as admixture, physical and chemical properties of silica gel (waste material used as desiccator for saving goods) are shown in Table 4. Superplasticizer Glenium 54 (G54) high range water reducing admixture, purchased form BASF Company, is used as workability adjusting material for concrete mixtures. Water Tap water is used for mixing and curing of all concrete mixes and specimens. Figure (1) showed the XRD spectra of admixtures and AFM analysis of nano silica



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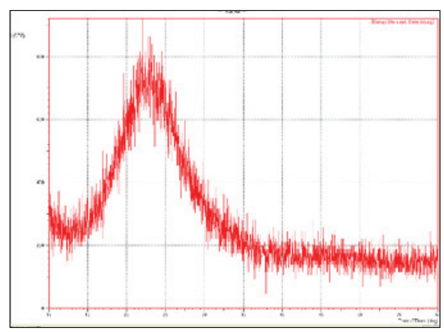

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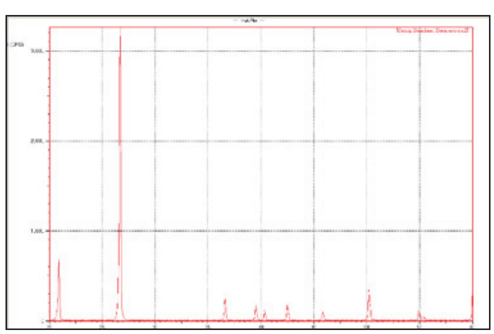


(c)

FIGURE 1. XRD spectrum of admixtures.

(a)XRD spectra of nano silica., (b XRD spectra of silica gel. (c) XRD spectra of micro silica.

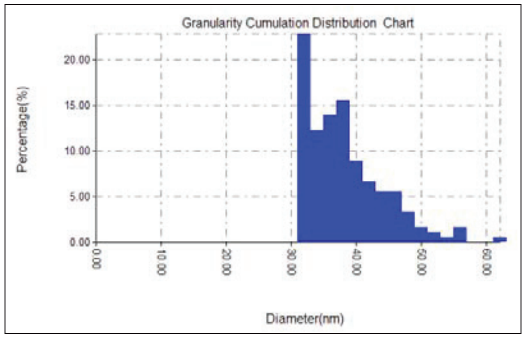


FIGURE 2. AFM analysis of nano silica.

TABLE 1. Chemical analysis of cement.

Oxide	**%**	**I.O.S. 5: 1984 [ ]Limits**
CaO	66.11	_
SiO_2	21.93	_
Al_2O_3	4.98	_
Fe_2O_3	3.10	_
MgO	2.0	< 5.0
K_2O	0.75	
Na_2O	0.35	
SO_3	2.25	< 2.8
Compound	**%**	**I.O.S. 5: 1984 [ ]Limits**
C_3S	50	_
C_2S	20.48	_
C_3A	4.0	_
C_4AF	13.17	_

TABLE 2. Properties of micro silica.

Property	**Value or description**
Material structure	Densified micro silica
Color	Gray
Density	1300 kg/m^3
M. Weight	60.08g/mol
Particle size**	≈ 40μm
Source	Fluka company –Switzerland

TABLE 3. Properties of nano silica.

Property	**Value or description**
Material structure	Hydrophilic Water-soluble SiO2
Color	White
Density	1300 kg/m^3
Purity	99.8%
Particle size**	30-60 nm
Source	Hwnanomaterial china

TABLE 4. Properties of silica gel.

Property	**Value or description**
Material structure	Transparent beads
Color	Violet
M.Wieght	60.08g/mol
Particle size	$\approx 2.5 - 4\,mm$
Abs. capacity	30% of its weight
Source	Fluka company- Switzerland

Mix Proportion

Mortar mixes details for nano silica, micro silica, silica gel and silica gel crushed pozzolanic activity investigation, are shown in Table 5. The proportion of admixture is10 %, as replacement of cement weight was depended for this test as in ASTM C 1240 **[9]** for testing pozzolanic activity index of silica fume**.** In absorption specimen's mixes, preparation the target design strength of 50, 70 MPa were designed according to British mix design method BS5328. Part 2:1991[10], seventeen types of concrete mixes is implemented in this study. The fixed parameters for all mixes are: water/cementitious, coarse and fine aggregate fractions, and superplasticizer contents. Mixes details and symbols can be seen in Table 6.

TABLE 5. Mortar mixes proportion for pozzolanic activity index test

Mix symbol	**Cement, g**	**Sand, g**	**NS g**	**MS G**	**SG G**	**SGC g**	**w/b%**	**G54/ cement %**	**Flow, mm**
Control	500	1375				---	0.485	0.5	160
NS	450	1375	50	-	-	-	0.485	0.75	153
MS	450	1375	-	50	-	-	0.485	0.5	155
SG	450	1375	-	-	50	-	0.485	0.6	160
SGC	450	1375	-	-	-	50	0.485	0.7	157

Specimens

The type and dimension of specimens that used in this research cubic with 50x50x50 mm for pozzolanic activity according to ASTM C109/109 [11] and cylindrical 100x50 mm for absorption test according to ASTM C 642[12] as shown in fig.2.

Tests

At the age (7) days with an accelerated curing at 65C^{o} the mortar specimens of the compressive strength were done in this research according to the B.S. 1881: part 116[13], also DTA analysis was done for each mortar mixes. For absorption test 28 days' age was considered with 2 specimens for each concrete mix this test is performed, according to ASTM C642 **[**9**]**.

TABLE 6. Concrete mixes proportion for water absorption test

Mix symbol	Cement, kg/m^3	Sand, kg/m^3	Gravel, kg/m^3	w/b%	G54 kg/m^3	MS kg/m^3 (rep.%)	SG kg/m^3 (rep.%)	SGC kg/m^3 (rep.%)	NS kg/m^3 (rep.%)
Control	515	721	1030	0.32	6.43	---	---	---	---
1MS	509.85	721	1030	0.32	6.43	5.15(1%)	---	---	---
2MS	504.7	721	1030	0.32	6.43	10.3(2%)			
3MS	499.55	721	1030	0.32	6.43	15.45 (3%)			
4MS	494.4	721	1030	0.32	6.43	20.6 (4%)			
1SG	509.85	721	1030	0.32	6.43		5.15 (1%)		
2SG	504.7	721	1030	0.32	6.43		10.3 (2%)		
3SG	499.55	721	1030	0.32	6.43		15.45(3%)		
4SG	494.4	721	1030	0.32	6.43		20.6(4%)		
1SGC	509.85	721	1030	0.32	6.43			5.15(1%)	
2SGC	504.7	721	1030	0.32	6.43			10.3(2%)	
3SGC	499.55	721	1030	0.32	6.43			15.45(3 %)	
4SGC	494.4	721	1030	0.32	6.43			20.6(4%)	
1NS	509.85	721	1030	0.32	6.43				5.15(1%)
2NS	504.7	721	1030	0.32	6.43				10.3(2%)
3NS	499.55	721	1030	0.32	6.43				15.45(3%)
4NS	494.4	721	1030	0.32	6.43				20.6(4%)

Where:
MS: Micro silica,
SG: Silica gel,
SGC: Crushed silica gel and
NS: Nano silica

(a) (b)

FIGURE 3. Specimens.

(a) Specimens of pozzolanic activity index test., (b) Specimens of absorption test

Experimental results and discussion

1. Pozzolanic Activity Index Test Results

For testing pozzolanic activity index mortar cubes of 50 mm side length are used for this test. One proportions of nano silica, micro silica, silica gel and crushed silica gel 10 %, as replacement of cement weight, are depended for this test. The ratio is as used in ASTM C 1240 **[9]** for testing pozzolanic activity index of silica fume. A Pozzolanic activity index can be determined using the following equation **[14]**:

$$\text{Strength activity index} = A_p / B_p \times 100$$

Where:

A_p: average compressive strength of mortar cubes of mixes incorporated admixture; and

B_p: average compressive strength of mortar cubes of without-adding (control) mixes.

The results are reported as averages of 3 replicates. The control mortar strength was 26 MPa, when replaced 10% of cement by nanosilica, crushed silica gel, micro silica and silica gel the strength became 47, 42, 40.8 and 36.4 MPa, respectively, so the pozzolnic activity indexes obtained from these results were 181, 161, 157 and 130 respectively as indicated in fig (3). Nano silica showed higher pozzolanic activity because nano silica reacts with the CH produced during cement hydration and results in more strength carrying C-S-H into the paste. As a more pozzolanic reaction occurs in the mix, more strength-carrying C-S-H is produced, which ultimately leads to a higher overall strength [15] these results were agree with Madhuwanthi Rupasinghe et.al 2017[15], and Wengui Li et.al. 2015[16], also YE Qing 2006 [4] found that the pozzolanic activity of nano silica was much greater than that of micro silica. The reaction rate of Ca $(OH)_2$ with nano silica and the velocity of C-S-H gel formation from $Ca(OH)_2$ with nano-SiO_2 were much quicker than that of $Ca(OH)_2$ with micro silica, in this work micro silica showed lower pozzolanic activity by about 13% with respect to that of nano silica. Crushed silica gel showed good pozzolanic activity, it was experimentally confirmed by Daunte Vaičiukynienėa et.al 2012 that the thermally activated silica gel could be used as an additive in hardened cement paste. This amorphous SiO_2 reacted with $Ca(OH)_2$ and form (C-S-H) type calcium silicate hydrates that additionally strengthened the hardened cement paste [17], so this quantity of silica gel additive enables to decrease the quantity of used cement and improved the mechanical properties of concrete. Silica gel also improved the pozzolan reactivity compared to control mix, but not as good as crushed silica gel's improvement.

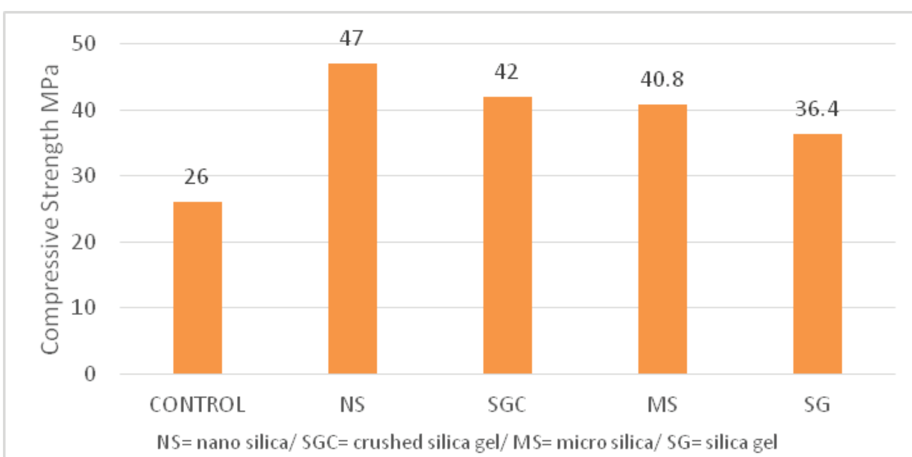


FIGURE 4. Compressive strength for cement mortar with different admixtures.

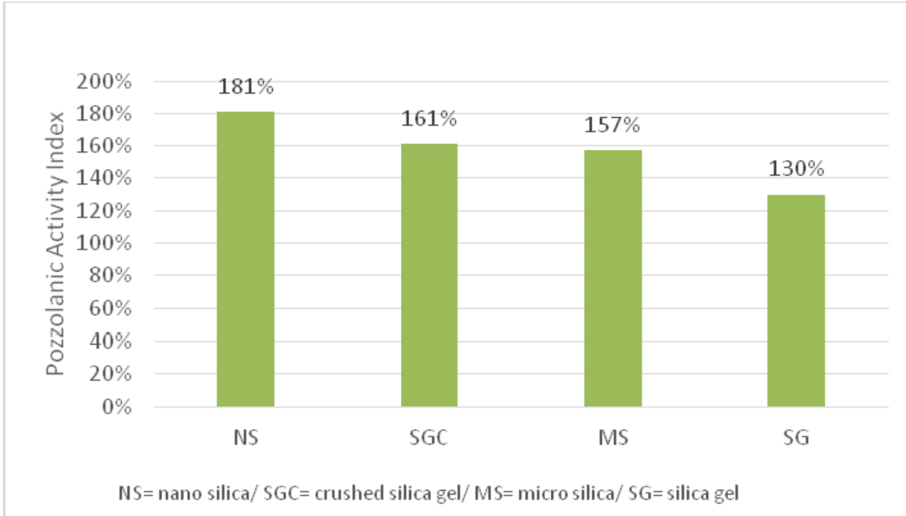


FIGURE 5. Pozzolanic activity index for cement mortar with different admixtures.

2. Absorption Test Results

The water absorption was conducted according to ASTM C642 [12], the water absorption test is carried out using (50x100 mm) cylinder specimens, testing procedure included removing the specimens from tap water at 28 days' age and placing it in an oven with a temperature set of 105 C^o for about 24 hours. After that, the oven is switched off and the specimens stilled in it for a specific period to be cooled then lifted from the oven and weighed. Thereafter, it returned to the oven and heated again as mentioned above. The process is repeated until the decrease in mass between two successive values becomes equal or less than 0.5%. The final dried mass is recorded. Then the oven-dried specimens placed in tap water for about 24 hours then removed from water and weighed. After that, they immersed in tap water for additional 24 hours, then removed and weighed. and the average water absorption of two samples was recorded and considered.

Absorption after immersion,% = 100*(B-A)/A

Where:
A = mass of oven-/dried sample in air, gm.
B = mass of saturated surface-dry sample in air after immersion, gm.

All mixes improved the durability of concrete through the reduction of water absorption. Nano silica implemented in this work generally exhibits a reduction in the water absorption potential of concrete this reduction, increased with the content of nano silica until reaching to 4% as replacement of cement as presented in fig (4-a), however, it's still lower than control mix this result agrees with the work achieved by S SANJU et.al in 2016 [18] where the addition of (0.5-1.5) % of nano silica reduced the water absorption by 54.2% in comparison to control specimen. The low water absorption values in nano silica mixes were attributed due to the higher pozzolanic effect of nano silica which made the concrete more compact and dense. Also the pore filling improved the pore structure of concrete, whoever S.Chirthra et.al[3], Ali Riza et.al[19], Peng Zhang et.al[20] and many researcher's work supported these results.

For micro silica mixes higher reduction recognized at 1%, while at 2, 3 and 4% showed higher water absorption, but it remains lower than control mixes, Mohammed S.N. [21] worker reported an improvement when using micro silica, while Leo Gu Ll et.al. [22] indicate that the combined effect of micro and nano silica enhance the durability of concrete by decreasing of the water absorption. Silica gel mixes results also showed good improvement in water absorption resistance we found that this resistance increased with the content of silica gel higher reduction observed at the percent of 4% while crushed silica gel give the same reduction at 1% and 2% percent of replacement which approximately same of nano silica result , so that means the crushed silica gel which considered as by product gave the same effect of nano silica in the improvement of water absorption resistance which reflected positively on the durability of concrete.

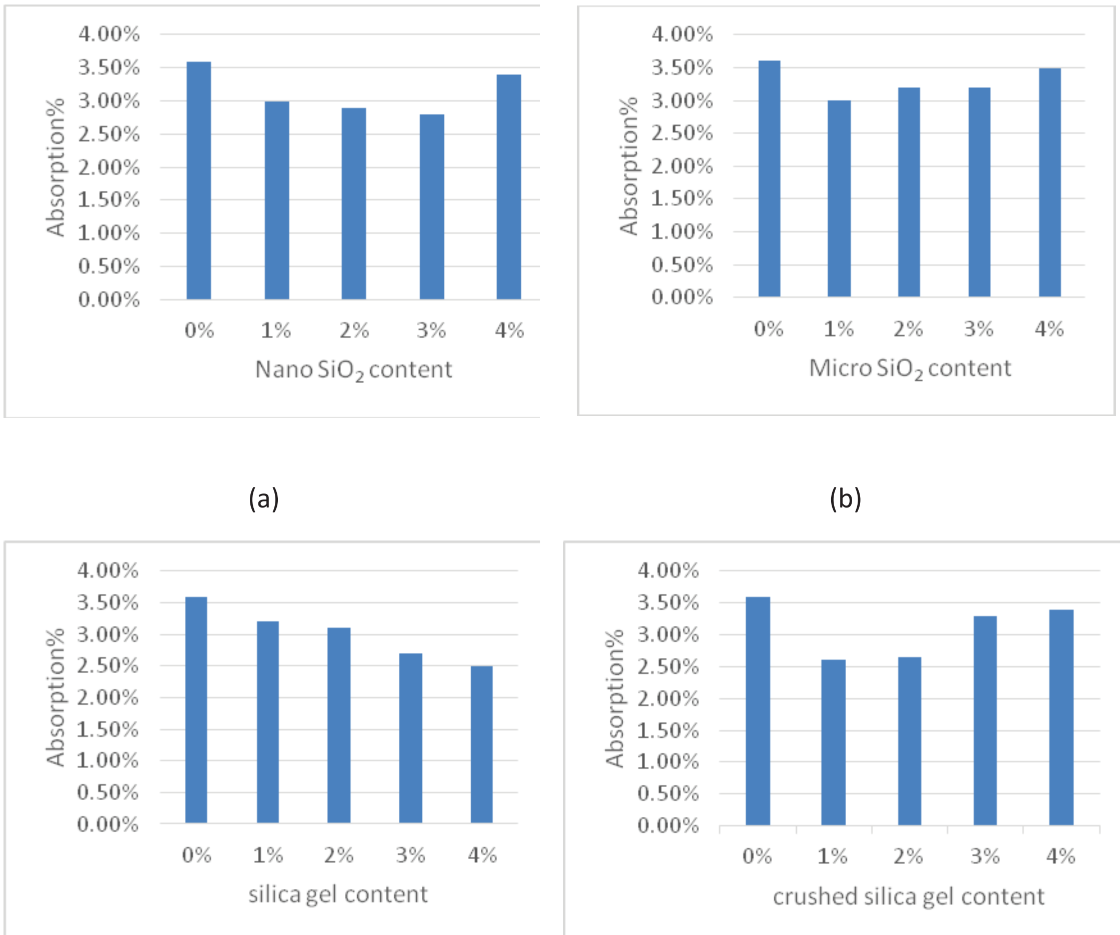
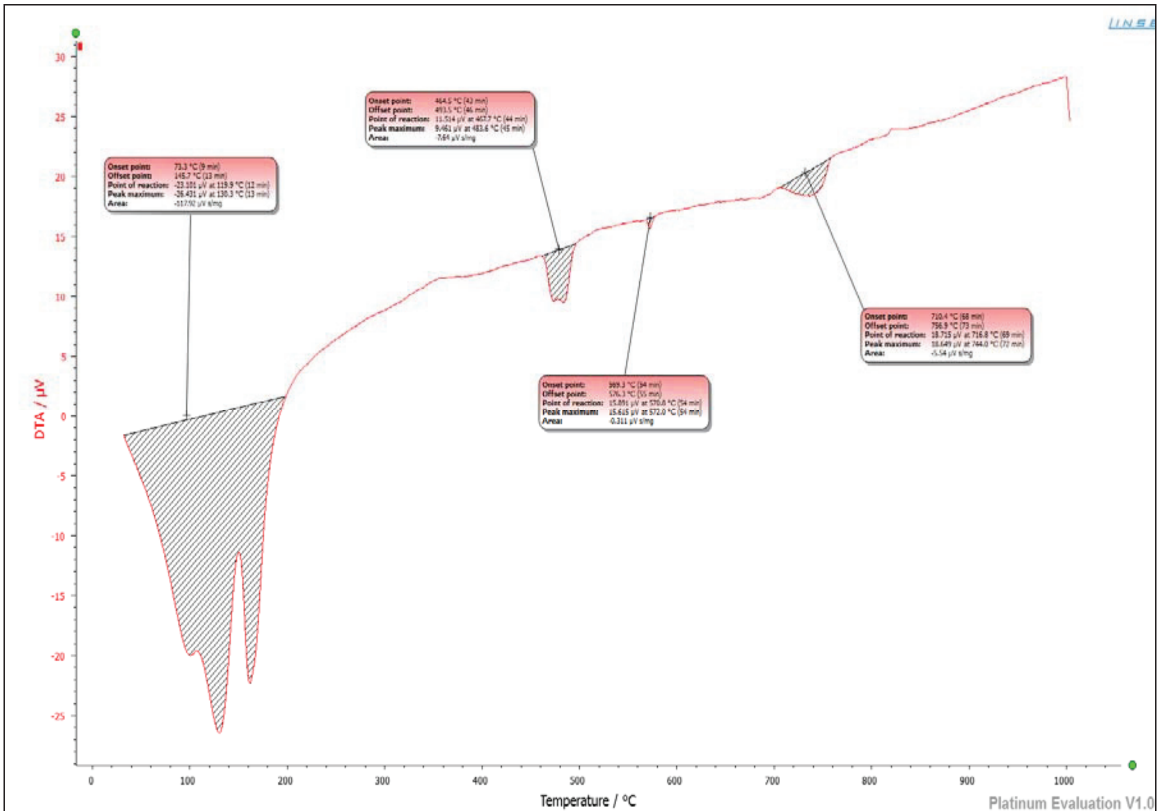


FIGURE 6. Results of absorption test.

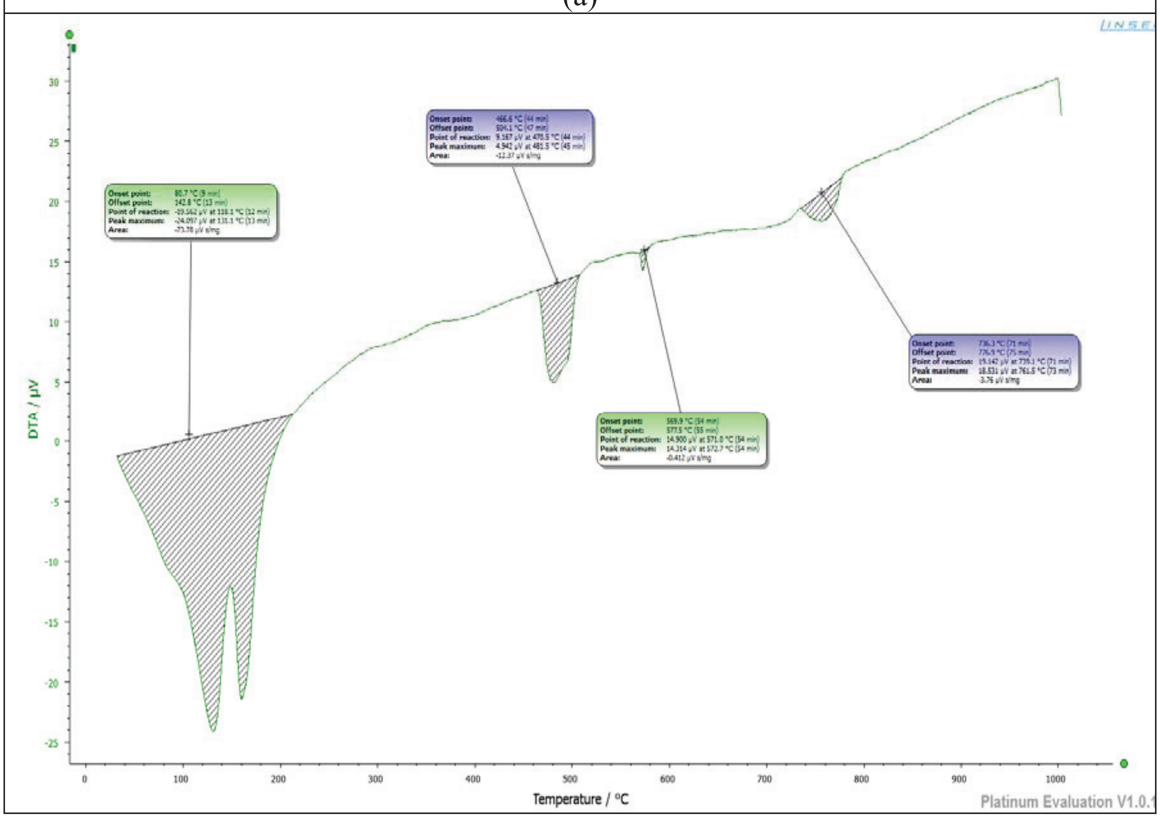
(a) Absorption results of nano silica addition. (b) Absorption results of micro silica addition.
(c) Absorption results of silica gel beads addition. (d) Absorption results of crushed silica gel addition.

3- DTA Analysis Results

DTA analysis was used to the hydration products using a LINSEIS L 70/2171 PC equipment. The tested powder samples, weighing up to 10 mg, were heated from 0 to 1000°C in a nitrogen atmosphere at a heating rate of 10°C/min. Figure (5) showed DTA curves of mortar specimens contained 10% wt. of nano silica, micro silica, silica gel silica gel crushed and control specimens after 28 days of curing. It can be seen that each curve consists of three zones which represent the typical reactions occurring in cement mortar. First zone represents the hydration of water molecules in C-S-H and ettringite took place within range from room temperature to 200C°, second, zone of thermal degradation for Ca (OH)2 occurred between 300-550C°, while the third zone explained the decomposition of $CaCO_3$ and escape of CO_2 from cement matrixes [23,24,25]. It can be seen from DTA curves that nano silica showed higher reactivity followed by crushing silica gel, micro silica and silica gel where comprised to the control mix this agree with Zemei Wa et.al [24] and Faiz U. A. Shaikh et.al [26] work, these results compatible with pozzolanic activity and absorption results in considering the nano silica and crushed silica gel more reactivity and made the best improvement in properties of concrete and cement paste than micro silica and silica gel.


DTA / µV
Temperature / °C
Platinum Evaluation V1.0

(a)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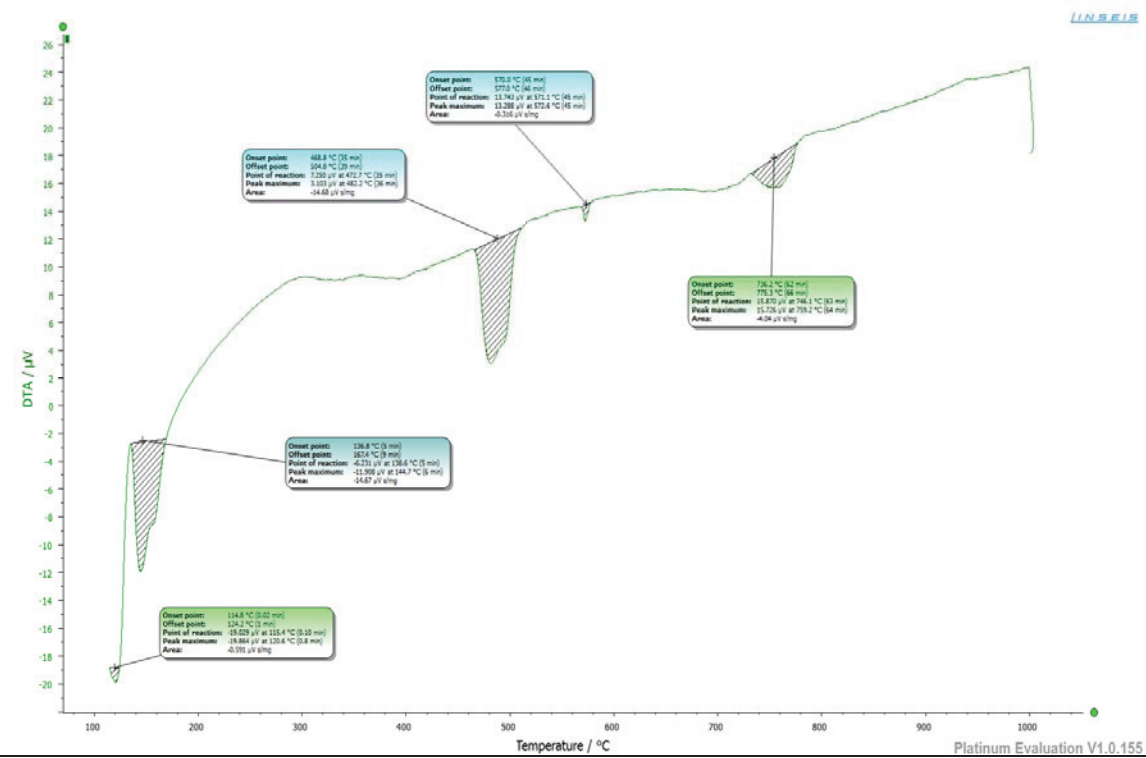
Platinum Evaluation V1.0.1

(b)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DTA / µV
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Temperature / °C
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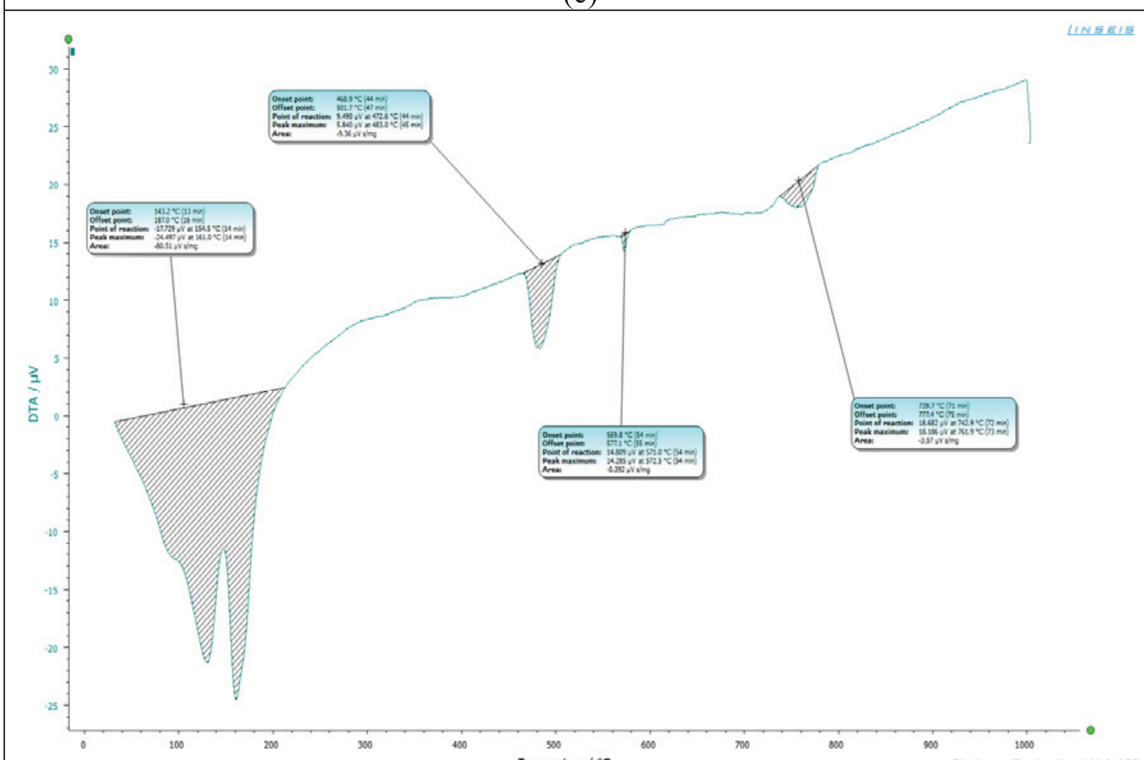
(c)


LINSEIS
DTA / µV
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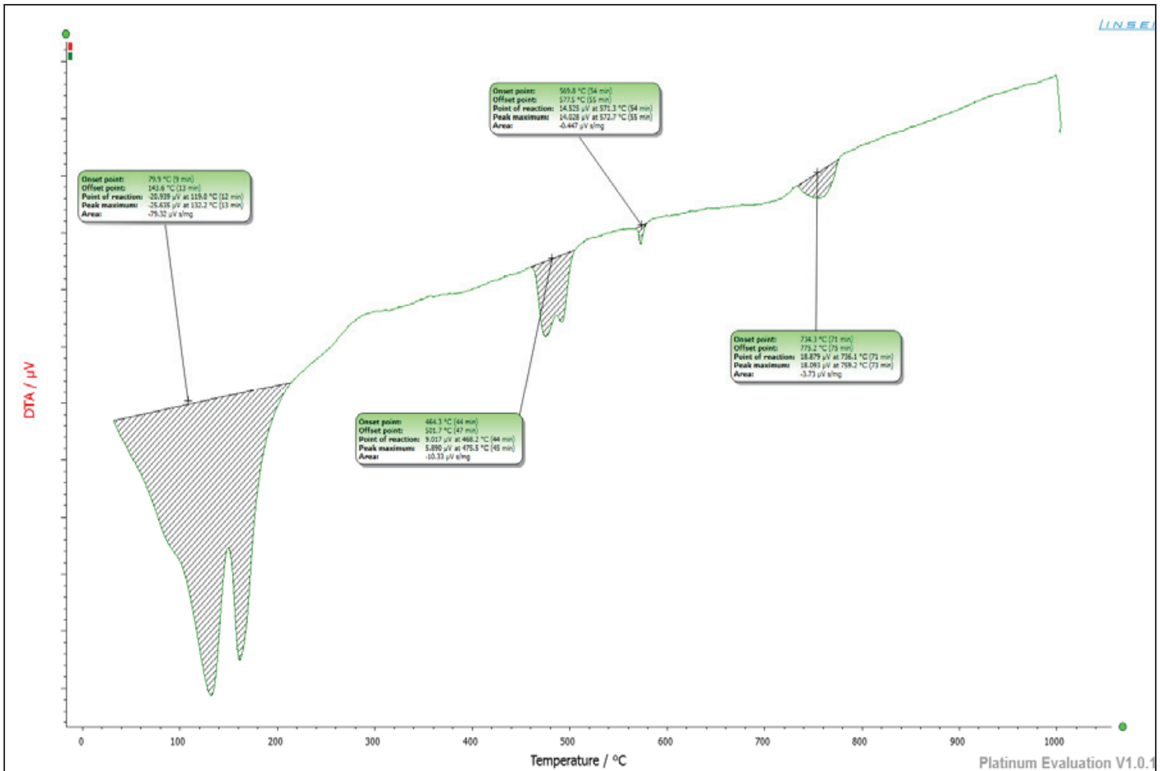
Temperature / °C
Platinum Evaluation V1.0.155

(d)



(e)

FIGURE 7. DTA analysis

(a) DTA curve of nano silica addition. (b) DTA curve of micro silica addition.
(c DTA curve of silica gel beads addition. (d) DTA curve of crushed silica gel addition (e) DTA curve of control mix.

Conclusion

Based on the results presented in this research, the following conclusions can be drawn.

- The compressive strength development of cement paste made by the $Ca(OH)_2$ and nano-SiO_2 was incomparably superior to that from Ca $(OH)_2$ and micro silica, so the pozzolanic activity of nano silica was much greater than that of micro silica. The reaction rate of $Ca(OH)_2$ with nano silica was greater than other additives.
- It was experimentally confirmed that the crushed silica gel could be used as an admixture in hardened cement paste. This amorphous SiO_2 reacted with $Ca(OH)_2$ and form (C-S-H) type calcium silicate hydrates that additionally strengthened the hardened cement paste, this admixture enhances both strength and durability of concrete and cement paste. From this study, we find that waste silica gel can give the same improvement made by nano silica on the properties of concrete with lower cost.
- All admixtures used in this work showed good resistance to water absorption in addition to strength enhancement.

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