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Experimental Study of fire effects on compressive strength of normal-strength concrete supported with nanomaterials additives

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Abstract: *Fire in buildings is nearly always man- made, i.e. resulting from negligence or error, which can cause immense damage in terms of lives and property. Concrete is widely used as a primary structural material in construction due to numerous advantages, such as strength, durability, ease of fabrication, but one of the most important advantages of concrete over other buildings materials (steel, wood,) is its fire resistive properties. Fire resistance can be defined as the ability of concrete to enable the structural elements to withstand fire or to give protection from it [1]. This includes the competence to resist a fire or to continue to perform a given structural function, or both. Concrete regard as a fireproof because of its incombustibility and its ability to withstand high temperature without collapse. However, its properties can change dramatically when exposed to high temperature and many problems were experienced with concrete in fire such as deterioration in mechanical properties. A number of complex physicochemical reactions take place when concrete is heated, causing mechanical properties as compressive strength and stiffness to deteriorate. Using nanomaterials as additives into concrete production to improve its mechanical properties has emerged as a promising research field nowadays. A better understanding of complex structure of concrete based materials incorporating supplementary cementing materials at Nano-level may result in a new generation of concrete, stronger and more durable, with desired stress-strain behavior and possibly, with the whole range of newly introduced "smart" properties. Nanomaterials are very reactive because of the particle 's small size and large surface area and have great potential in improving concrete and cement properties such as compressive strength, permeability cement mortar, flexural resistance [2]. This study investigates the effect of nanoparticles on the compressive strength of normal-strength concrete after fire exposure. The experimental program focused on the effect of using Nano silica, Nano clay, and hybrid from both of them on some durability properties of concrete produced with the three materials as a partial replacement of cement at different ratios for each one.*

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I. Introduction

Concrete containing mineral additives is used extensively throughout the world for their good performance and for ecological and economic benefits. Researchers are very keen to explore the concrete behavior. Recently, many types of materials are progressed to use as supplementary materials cementitues with concrete to improve the durability properties of concrete. One of these new supplementary materials cementitues is using the traditional pozzolanic materials such as kaolin and silica at Nano scale particles. Recently, nanotechnology has attracted considerable scientific interest due to the new potential uses of particles in nanometer scale. Thus industries may be able to re-engineer many existing products that function at unprecedented levels. Due to these developments in Nano science and technology, various forms of Nano-sized amorphous materials have become available. Many literature studies confirmed that, these nanomaterials have enhancing effect on cement mortar blended with nanomaterials. A few of these studies tried to study the nanomaterial effect on some durability properties of blended concrete, but it still insufficient. Approximately, few studies inside Egypt focusing entirely on the impact of some Nanomaterials (Nano silica, Nano clay and hybrid of them) on the most important properties of blended concrete durability such as, fire resistance. The aim of this study is to investigate the influence of using Nano materials as well as other pozzolanic materials as partial replacement of cement on mechanical as well as durability properties of normal concrete, specially the fire resistance property.

Fire resistance can be defined as the ability of concrete to enable the structural elements to withstand fire or to give protection from it. This includes the competence to resist a fire or to continue to perform a given structural function, or both. As known, the rise in temperature causes a decrease in the strength for the concrete. However, the rate at which the strength decrease depends on many factors such that the rate of increase in the temperature of the fire, the insulating properties of concrete, heating rate, dehydration of C–S–H gel, and thermal incompatibility between aggregates and cement paste [3]. Fire test methods are used to determine the fire resistive properties of concrete. The most widely used and nationally accepted test procedure is that developed by ASTM-E119. The effect of fire on concrete supported with natural pozzolans has not been investigated in detail. Researchers and previous studies differ in their opinion about the changes in the characteristics of concretes, particularly in the range of 100–250 ˚C. Whereas for temperature above 300 ˚C, there is uniformity in opinion that there will be a decrease in mechanical properties [4–5]. This study hopes to give researchers a better vision about concrete behavior under different conditions of heat exposure, with and without being supported with Nano additives.

II. Experimental work

Specimens of different concrete mixes with different ratios of Nano particles additives were prepared, and after different times (7-28-56-90 days) the strengths were obtained and measured at the room temperature (25^{o}C), specimens of (28days) were also measured at elevated temperatures. The research investigates the effect of elevated temperature degrees at (200 ^{o}C,400 ^{o}C, 500 ^{o}C,600 ^{o}C,700 ^{o}C, 800 ^{o}C) for an exposure periods equal (60-120 minutes) with different ratios of Nano particles additives on the concrete compressive strength. After exposure to elevated temperature, the specimens were cooled naturally in a closed furnace chamber. The reason of using the slow cooling rate was to minimize differential temperature gradient effects on specimens [6]. The compressive strengths were measured for the specimens after exposure to the fire and compared with the strength of reference specimens. The compressive strength tests were performed in accordance with ASTM C109 [7], using cubic specimens of 100×100×100 mm. The mixes were molded in the cubes for one day, then they were demolded to put in curing for different ages (7 – 28 – 56 – 90) days.

One of the most important points is how to put the Nano materials in concrete mixes keeping their original composition and physical characteristics from any change, its known that Nano particles are very active and they going to be gathered producing a bigger particles size after put in water, therefor a dispersion technique must be used to keep the particles in Nano size. In this study wet mix technique was applied using vane motor where Nano particles were mixed with half amount of the water and superplasticizer for two minutes until the mixture was homogeneous, then the other amount of water and superplasticizer were mixed and added gradually to the mix.

This experimental study focused on ordinary strength concrete as it is the most used type of concrete in our buildings. Moreover, many researchers concerned with high strength concrete in their studies about elevated temperature effect on concrete, while few studies focused on ordinary strength concrete. All used materials, mix proportions, and tests results are described in details.

i. Materials Properties.

This part includes a detailed description of the materials used through this study, these materials are; cement, aggregates, Nano silica (NS), Nano clay (NC), water, and admixtures (superplasticizer).

Cement: In this study, CEM I 42.5 N. Testing of cement was carried out as the Egyptian Standard Specifications ESS 4756-1/2009 [8], with the specific gravity of 3.15 was used. The properties shown in Table (1).

Table 1: Chemical Composition of cement

Oxide Composition	Mass %
Silicon dioxide (SiO2)	20.22
Aluminum oxide (Al2O3)	6.05
Ferric oxide (Fe2O3)	3.3
Calcium oxide (CaO)	62.75
Magnesium oxide (MgO)	2.00
Sulphur trioxide (SO3)	2.25
Potassium oxide (K2O)	0.85
Sodium oxide (Na2O)	0.79
Loss on Ignition	1.77

Coarse Aggregates

There are three types of coarse aggregates which commonly used in concrete mixes(natural gravel – basalt – dolomite), and to choose one of them which must have better thermal properties to resist fire than others, three mixes were designed and tested at temperature of 600 ^{o}C for 2hrs so as to determine the best type to use at the reference mix of this research.

The following tables shows the Sieve analysis test results for each type of coarse aggregate, different mix design of each type before burning, and the final results of compressive strength of concrete before and after burning for each type.

Table (2): Sieve Analysis of Coarse Aggregates

Sieve Size (mm)	37.5	19	9.5	4.75
% Passing basalt	100	100	95.31	46.38
% Passing dolomite	100	97.48	22.31	6.07
% Passing gravel	97.19	88.36	13.44	2.7

Table (3): Mixes designs

Mixes	Cement content (Kg/m3)	w/c ratio	Water, kg/m3	Aggregate Content kg/m3	
				Sand, kg/m3	Coarse aggregate
Basalt	400	0.45	180	650	1250
Dolomite	400	0.45	180	656	1170
Crushed Gravel	400	0.45	180	677	1245

Table (4): Results of specimens before & after fire at 600°C at 28 days

Types of aggregates	Compressive strength kg/cm3			
	reference		At 600 degree	
	Specimens	Average	Specimens	Average
Crushed gravel	328	335	266	268
	355		286	
	322		252	
dolomite	448	482	352	346
	476		336	
	482		350	
basalt	440	447	341	331
	446		338	
	455		314	

Results shows that mixes prepared using dolomite (crushed limestone) have the best resistance of fire effect on concrete, they also have the highest compressive strength at ordinary conditions. Consequently, the coarse aggregate used at this experimental study was local crushed limestone (dolomite) with a specific gravity of 2.66, bulk unit weight of 1618 Kg/m3, void percentage 39.2%, fineness modulus of 6.35, according to the requirement of ESS 1109/2002 [9].

Fine Aggregates

Fine aggregate used in this experimental work was natural siliceous sand, clean and rounded fine aggregate with a specific gravity of 2.65, a bulk unit weight of 1675 Kg/m3, and a void percentage 36.8%, and fineness modulus of 2.92, according to the requirement of ESS 1109/2002 [9].

Nano Silica

Amorphous Nano silica (SiO_2) with particle size ranged from 13-75 nm was used in preparation the Nano silica blended concrete specimens in this study. The Nano-silica used is powder type, Colorless (White), and with average density 2.2 - 2.6 g/mL at 25 °C. The following table shows the chemical properties of used Nano silica.

Table (5) chemical composition of Nano silica

Oxide Composition	Mass %
Silicon dioxide (SiO_2)	99.85
Aluminum oxide (Al_2O_3)	0.02
Ferric oxide (Fe_2O_3)	0.01
Calcium oxide (CaO)	0.03
Magnesium oxide (MgO)	0.01
Potassium oxide (K_2O)	0.01
Sodium oxide (Na_2O)	0.01
Loss on Ignition	0.04

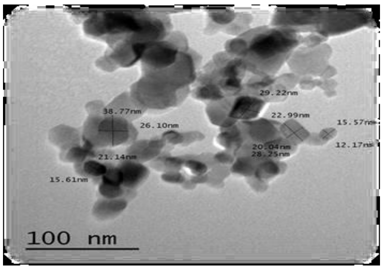

Figure (1) TEM micrograph for the Nano silica

Nano Clay

The Nano-clay used in this work is metakaolin which produced from heat treatment of kaolin. This heat treatment at about 900 ^{o}C breaks down the structure of kaolin such that the alumina and silica layers become puckered and lose their long – term order, producing metakaolin MK.

The Nano-clay used is powder with (off white) color, and with average density 0.6 – 0.8 gm/cm3 at 25 °C. The following table shows the chemical properties of used Nano clay.

Table (6) chemical composition of Nano clay

Oxide Composition	Mass %
Silicon dioxide (SiO2)	51.52
Aluminum oxide (Al2O3)	40.18
Ferric oxide (Fe2O3)	1.23
Calcium oxide (CaO)	2.00
Magnesium oxide (MgO)	0.12
Potassium oxide (K2O)	0.53
Titanium dioxide (TiO2)	2.27
Sodium oxide (Na2O)	0.08
Loss on Ignition	2.01

Superplasticizer

A superplasticizer based on a modified polycarboxylic ether was employed to obtain a satisfactory workability for the mixes and accelerates the cement hydration. A commercial superplasticizer (GLENIUM ACE 30) produced by chemicals company (BASF) in Egypt was used at all mixes with fixed proportion.

ii. Mix proportion

To achieve the objectives of this work three groups with a total numbers of 12 concrete mixes were prepared and investigated. Table (9) illustrates the mixes design of all mixes. The mixes were divided into three groups representing the key variables in the experimental study. The first group was designed using different ratios of Nano silica as a cement replacement with percentages 0, 1, 2, 3, and 4%, respectively. The second group with different ratios of Nano clay as a cement replacement with percentages 1, 3, 5, 7, 9 %. The third group withhybrid mix between Nano silica and Nano clay using different ratio, and to answer about the question why that hybrid mix was investigated, the answer is related aboutcharacteristics of each material. Previous studies showed that Nano silica is more amorphous than the Nano clay, which means that Nano silica is very effective and contribute in increasing of the C-S-H gel during the hydration process of cement [10]. However, Nano clay can be classified as filler material that help in making concrete dense and durable by decreasing voids ratio [11]. Thence, design combination between Nano silica and Nano clay can make a moderate area between the two materials.

Table (7) The mixes design

Mix	Symbol	Cement (kg)	Aggregate (kg)		Water (lit)	S.P (lit)	Nano Silica(kg)	Nano Clay(kg)
			coarse	fine				
Control mix	M0	400	1170	656	180	5	----	----
1%NC	M1	396	1170	656	180	5	----	4
3%NC	M2	388	1170	656	180	5	----	12
5%NC	M3	380	1170	656	180	5	----	20
7%NC	M4	372	1170	656	180	5	----	28
9%NC	M5	360	1170	656	180	5	----	36
1%NS	M6	396	1170	656	180	5	4	----
2%NS	M7	392	1170	656	180	5	8	----
3%NS	M8	388	1170	656	180	5	12	----
4%NS	M9	384	1170	656	180	5	16	----
0.5% NS+ 4.5% NC	M10	380	1170	656	180	5	2	18
1% NS + 4% NC	M11	380	1170	656	180	5	4	16
1.5% NS +3.5% NC	M12	380	1170	656	180	5	6	14

III. ResultandDiscussion

Compressive strength results before elevated temperature exposure:

The following table shows the test results of the compressive strength at age 7 ,28 ,56, 90 days for all mixes mentioned in table 7.

Table (8) compressive strength test results at different ages.

Property	Age	Control mix	Nano silica %				Nano clay%					Hybrid %(NS+NC)		
			1	2	3	4	1	3	5	7	9	0.5+4.5	1+4	1.5+3.5
Compressive strength (kg/cm^2)	7	295	333	348	338	327	322	356	386	294	289	315	318	338
	28	380	426	415	397	388	394	419	445	386	370	423	437	431
	56	388	435	446	424	412	411	440	468	435	397	441	456	450
	90	398	446	459	435	422	423	451	484	446	409	452	469	462

Result shows that all types of Nano materials used in this study makes remarkable improvement in concrete compressive strength, while this improvement have a different ratios according to concrete age and Nano materials proportion.

Obviously, Nano clay gives the optimum improvement in compressive strength at 5% replacement of cement ratio, followed by the hybrid mix at (1%NS + 4% NC), and Nano silica at 2% respectively. Also its remarkable that Nano clay gave a better improvement in concrete compressive strength at early ages, while the rate of improvement decreases later with age increasing. The opposite case is with Nano silica where the improvement in compressive strength rate increases with time, while the hybrid mix located in a moderate area between Nano clay and Nano silica.

The following table and figures showing the Percentage of improvement in compressive strength at different ages for the three types of additives.

Table (9) percentage of compressive strength improvement according to age and Nano materials additive

Type of Nano additive	Optimum percentage of replacement	Percentage of improvement in compressive strength(%)			
		7 days	28 days	56 days	90 days
Nano silica	2%	17.9	11	14.9	15.3
Nano clay	5%	30.8	17.1	20.6	21.6
Hybrid mix	(1% + 4%)	7.8	15	17.5	17.8

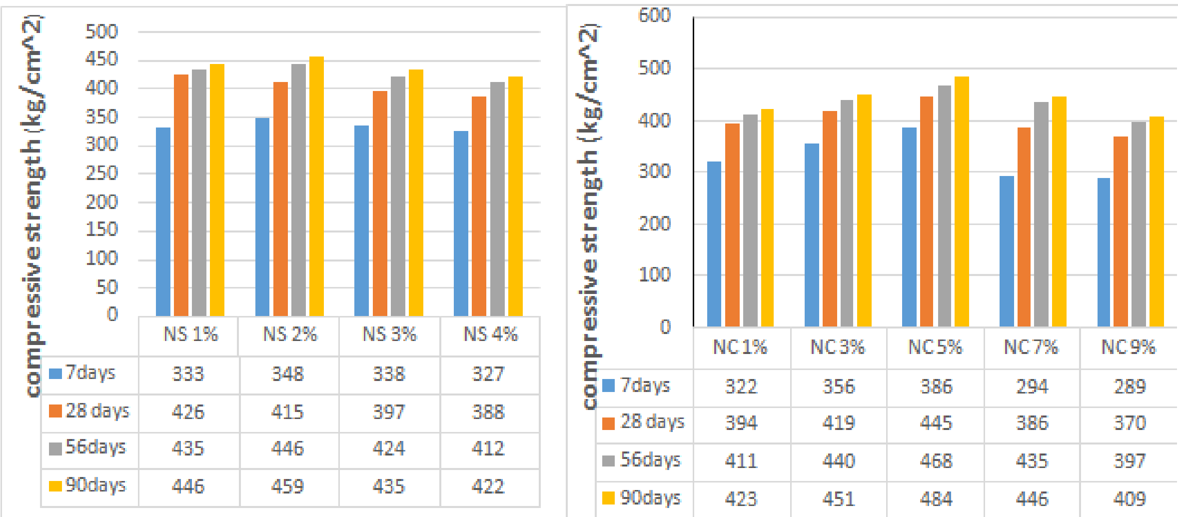

Figure (2) compressive strength according to different Nano additives with different ratios and ages.

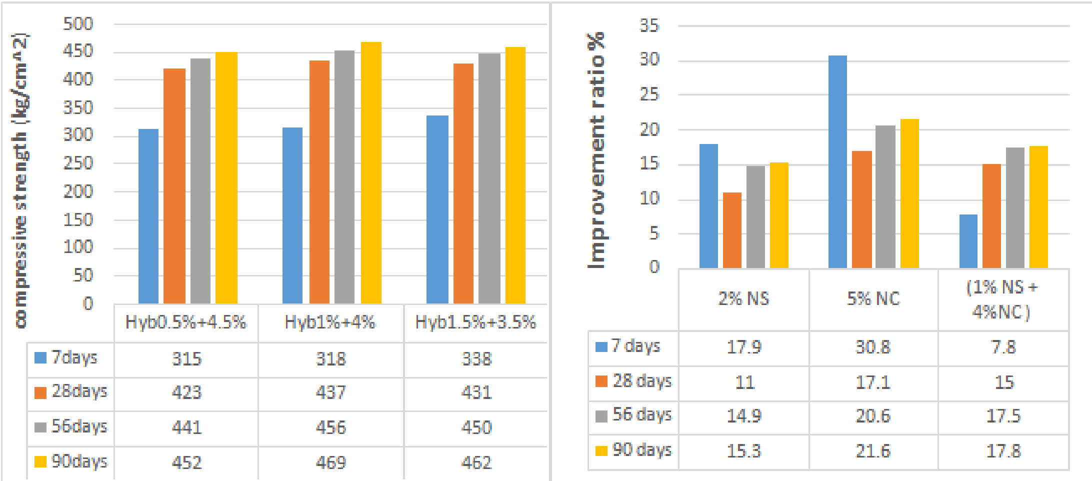

Figure (3) compressive strength according to different hybrid mixes, and compressive strength improvement according to optimum replacement ratios

compressive strength results after elevated temperature exposure:
The following tables shows the test results of the compressive strength at elevated temperature with 1, 2 hours burning time for all mixes mentioned in table (7) at 28 days.

Table (10)compressive strength test results at different temperature degree and Nano materials proportion after1 hour burning.

28 days/1hr		Control mix	Nano silica (NS)%				Nano clay (NC)%					Hybrid (NS + NC)%		
property			1	2	3	4	1	3	5	7	9	0.5+4.5	1+4	1.5+3.5
Compressive strength (kg/cm^2)	25 °C	380	426	415	397	388	394	419	445	386	370	423	437	431
	200 °C	371	441	455	460	456	407	438	470	456	438	440	454	448
	400 °C	356	427	448	454	449	408	435	465	454	432	421	436	426
	500 °C	348	400	436	445	438	370	410	451	425	402	396	413	401
	600 °C	326	361	398	409	404	337	382	411	381	357	353	373	359
	700 °C	252	280	304	311	307	262	288	316	298	278	280	287	281
	800 °C	212	229	243	251	245	221	238	255	245	227	231	241	233

Table (11) compressive strength test results at different temperature degree and Nano materials proportion at 2 hours burning.

28 days/2hrs		Control mix	Nano silica (NS)%				Nano clay (NC)%					Hybrid (NS + NC)%		
property			1	2	3	4	1	3	5	7	9	0.5+4.5	1+4	1.5+3.5
Compressive strength (kg/cm^2)	25 °C	380	426	415	397	388	394	419	445	386	370	423	437	431
	200 °C	364	432	445	451	447	399	429	462	447	429	431	445	439
	400 °C	346	415	435	441	436	396	422	452	441	419	409	425	414
	500 °C	324	377	411	417	413	349	386	420	401	379	373	385	378
	600 °C	286	325	359	371	364	304	341	374	345	322	318	328	323
	700 °C	206	233	253	259	256	218	240	261	248	232	233	238	234
	800 °C	156	172	183	187	184	166	179	192	184	171	174	179	175

Comparisons between results can demonstrate the elevated temperatures effect on specimens with or without Nano materials, also it's obvious that Nano additives have apositive effect on compressive strength, while the improvement proportion varies according to Nano additive type, replacement ratio, exposure time, and temperature degree. The optimum additives ratios were 3% for Nano silica, 5% for Nano clay, and (1% + 4%) for the hybrid mix.

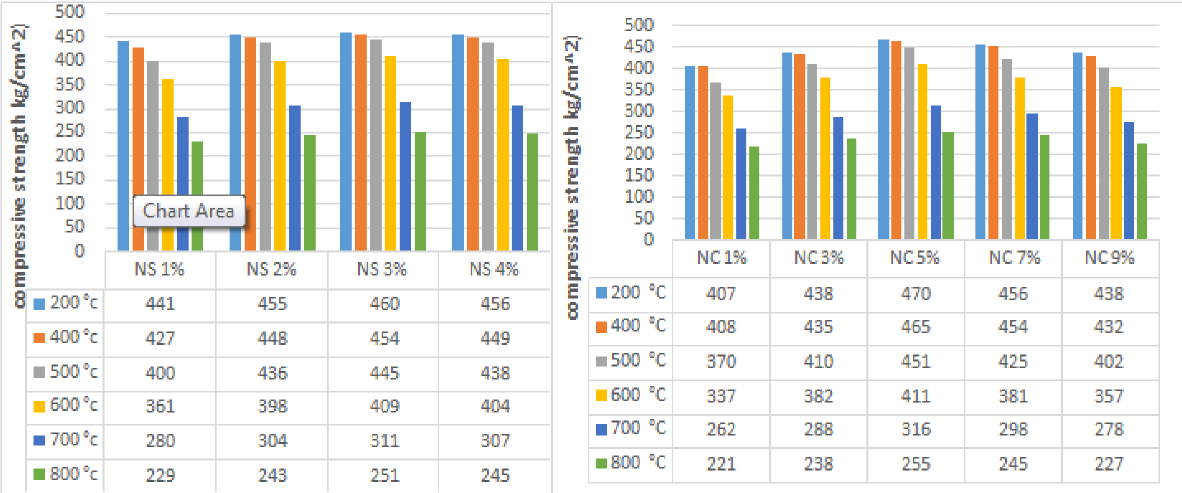

Figure (4) compressive strength after 1-hour exposure with different Nano additives ratios.

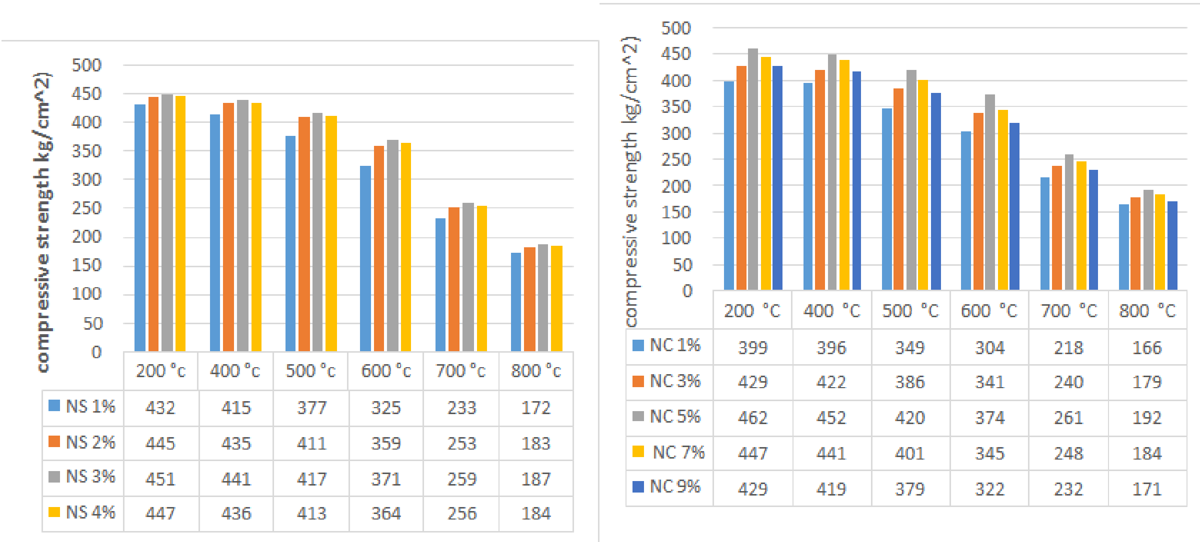

Figure (5) compressive strength after 2 hours expousre with different nano additives ratios

Table (12) percentage of compressive strength improvement according to temperature degree and Nano materials additive after 1 hour

Type of Nano additive	Optimum percentage of replacement	Compressive strength improvement ratio %					
		200 °C	400 °C	500 °C	600 °C	700 °C	800 °C
Nano silica	3%	24.3	27.5	27.8	25.5	23.4	18.4
Nano clay	5%	26.7	30.6	29.6	26	25.4	20.2
Hybrid mix	(1% + 4%)	22.4	22.5	18.7	14.4	13.9	13.7

Table (13) percentage of compressive strength improvement according to temperature degree and Nano materials additive after 2 hours

Type of Nano additive	Optimum percentage of replacement	Compressive strength improvement ratio %					
		200 °C	400 °C	500 °C	600 °C	700 °C	800 °C
Nano silica	3%	23.9	27.4	28.7	28.9	25.7	19.8
Nano clay	5%	26.1	30.6	30	29.7	26.7	23
Hybrid mix	(1% + 4%)	22.3	22.8	19.2	16	15.5	14.7

From the perspective of compressive strength of concrete, the heating conditions can be divided into two stages as (25 - 200°C), and (200 - 800°C). For the control mix there is little loss in strength at the stage from (25 - 200°C) followed by sharp decrease in the higher heating degrees. On the other hand, specimens which have Nano additives showed a different behavior, they had an increase in compressive strength in (25 -

200°C) stage than control specimens, this increase may be because of the hydrothermal interaction of Nano materials particles as a result of temperature rise with the liberated free lime during hydration reaction. Moreover, Nano particles activates pozzolanic reactions which led to the create additional amount of hydration particles, that reactions help concrete to gain more strength.

At high temperatures, the thermal effect might cause water migration whereas dehydration of moisture supply from outside is insufficient. Internal stress and thus micro and macro cracks are generated due to the heterogeneous volume dilatations of ingredients and the buildup of vapor in the pores. Therefore, at higher temperature, especially above 200°C, the observed decrease in compressive strength of blended concrete containing Nano materials may be because of internal thermal stress generated around pores which generate micro cracks.

Mass loss:

Mass loss can affect the fire resistance and the overall strength of the structural element. Mass loss due to thermal effect of high temperature exposure may cause spalling of concrete element, which cause a destructive effect on the element rigidity, especially if it exposes the element core and reinforcing steel. The following tables showing mass loss for the control mix and the optimum replacement ratios of Nano additives mixes at different temperature degrees.

Table (14)mass(gm)of the control mix and the optimum replacement ratios of Nano additives mixes at different temperature degrees

Type of Nano additive	Mass(gm) before burning	Mass(gm) after 400 °C	Mass(gm) after 600 °C	Mass(gm) after 800 °C
Control mix	2569	2443	2316	2068
Nano silica 3%	2635	2565	2494	2339
Nano clay 5%	2673	2588	2486	2311
Hybrid mix (1+4) %	2649	2572	2491	2323

Table (15) mass loss as a percent ratio for the control mix and the optimum replacement ratios of Nano additives mixes at different temperature degrees

Type of Nano additive	Mass loss after 400 °C	Mass loss after 600 °C	Mass loss after 800 °C
Control mix	4.9%	9.85%	19.5%
Nano silica 3%	2.65%	5.35%	11.2%
Nano clay 5%	3.18%	6.99%	13.5%
Hybrid mix (1+4) %	2.9%	5.95%	12.3%

Results showing a remarkable enhancement for mixes which have Nano additives in its composition with different ratios.

Electrical conductivity:

Three samples of Nano silica, Nano clay, and calcium hydroxide solution $Ca(OH)_2$ as a control sample were tested to measure the electrical conductivity. calcium hydroxide is known with its high electrical conductivity (6. 26mm.S/cm), it also does not have a high pozzolanic activity, so we can say that the relation between pozzolanic activity and electrical conductivity is a reverse one. Results of electrical conductivity test were as the following:

Table (16) Electrical conductivity test results

Material	EC (mS/cm)
Ca(OH)2	6.26
Nano silica	5.91
Nano clay	6.28

The value of 6mm.S/cm can be represented as the upper limit for **EC** to ensure efficiency of nanomaterial [12], so it's clear that Nano silica is more active as apozzolanic material than Nano clay, while Nano clay can be considered as a filler material which help to make the concrete denser.

On the other hand, previous studies which investigated the relationship between electrical conductivity and steel bar corrosion rate confirmed that there is proportional relation between them, but that relation is conditional with the existence of chlorides,sulphates, carbonates, ornitrates solution. All previous substances are not existing at chemical composition of Nano silica or Nano clay as described at tables 5,6, so we can conclude that there is no direct relation between adding Nano silica or Nano clay to concrete and corrosion rate of steel bars.

Compressive strength predicting equation:

Regression analysis is a set of statistical processes which aims to estimate the relationships between one dependent and one or more independent variables.Regression analysis were made to create an empirical equation which help to predict the residual compressive strength. The empirical formula was as the following:

Fcu (after burning) = X0 – X1(temperature degree) – X2(exposure time) + X3 (Nano clay%) +X4(Nano silica%).

The equation has a value of regression (R Square) equals 0.79, that value represent a measure of correlation between outputs and targets. when R Square value close to one that means close relationship, while the value zero or closer values means random relationship. According to the high number of variables and conditions the value of 0.79 of R Square represent a very good one. The values of equation constants are as the following

X0	X1	X2	X3	X4
567	0.387	31.1	5.5	10.1

IV. Conclusion

Based on the results of this experimental study the following conclusion can be made:

1- Nano technology and its applications in engineering is a promising field to study.
2- Nano materials which have pozzolanic activity like Nano silica, or Nano clay which considered as a filler material can be very useful in civil engineering field, we can use it as a replacement materials of cement which consider as one of the main sources of carbon dioxide emission, moreover its proved that they can improve the mechanical and durable properties of concrete.
3- The 5 % Nano clay, hybrid (1% Nano silica + 4% Nano clay), and 2% Nano silica as a partial replacement of cement increase the concrete compressive strength at 28 days more than concrete without Nanomaterials (control mix) with 17.1 % 15 % and 11 % respectively.
4- The Nano materials (NS and NC) enhance the mechanical properties of concrete at room temperature up to 200 °C, then Increasing the temperature decrease the residual compressive strength.
5- Exposure for 2 hours to 800 ^{o}C temperature degree make concrete lose about 60% of its strength, that ratio is very dangerous and may cause structure failure for the buildings, this must be strong motive for researchers to continue their researches in this field.
6- The 5 % Nano clay, 3% Nano silica, and hybrid (1% Nano silica + 4% Nano clay) as a partial replacement of cement found to be the optimum ratios to improve concrete resistance to fire, at 28 days and exposure for 2 hours to 800 ^{o}C temperature degree the improvement ratios were 23 % 19.8 % and 14.7 % respectively.
7- Nano clay is more effective than Nano silica in increasing concrete compressive strength and concrete resistance for fire, 5% is the optimum ratio of cement replacement at most cases.
8- At 9% replacement of cement with Nano clay, the compressive strength increased with 2.7% than control mix, that may have great environmental effect if companies could produce Nano clay at commercial level which means we can decrease cement production with about 10% and reduce carbon dioxide emission with the same ratio.
9- Using Nano additives as a partial replacement with cement enhance concrete resistance of spalling and mass loss at all conditions.
10- a predicting equation of compressive strength can be developed for each mix conditions using data analysis and regression technique, or other techniques like neural artificial networks or fuzzy models.

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