CHAPTER 3

MATERIALS AND EXPERIMENTAL PROGRAMME

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3.1 INTRODUCTION.

This chapter describes the materials, mixing procedures, specimen's fabrications, curing, test parameters, equipments and test methods adopted in the research work.

3.2 SPECIMENS DETAILS.

3.2.1 MATERIALS.

3.2.1.1 Cement

Two types of cement were considered in this thesis. Portland cement (CEM I) meets the **ESS 2335-1/2008** requirements, and limestone portlandcement according to **ASTM C595**The chemical analysis was carried out for CEM Iand the results are given in**Table 3.1**.

3.2.1.2 Aggregate types

A. Fine aggregate (Sand).

Natural siliceous sand has been used as fine aggregate in concrete with specific gravity 2.5 and fineness modulus (F.M.) of 2.54. To find out the physical properties of fine aggregate the following tests were done: -

1- Specific gravity and absorption according to **ASTM C 128** requirements for fine aggregate.

2- Unit weight according to **ASTM C 29.**

3-Clay and materials finer than sieve No.200 (75μm) by washing according to **ASTM C 142/78** and **B.S. 882:1992.**

4-Sieve analysis of fine aggregate according to **ASTM C 33/84.**

-The results of these experiments are given in **Table 3.2.**The percentages passing by weight is shown in **Table 3.3**andthe grading curve of sand meets the **ASTM C 33/84** requirements as shown in **Fig 3.1**.

B. Coarse aggregate (P.L.S.).

The coarse aggregate was pink limestone with specific gravity and nominal maximum size (N.M.S.) of 2.56 and 1" respectively.

To find out the physical properties of coarse aggregate the following tests were performed:

1-Specific gravity and absorption meets **ASTM C 127** requirements for coarse aggregate**.**

2-Unit weight according to **ASTM C 29.**

3-Clay and materials finer than sieve No.200 (75µm) by washing according to**ASTM C 142/78** and **B.S.882:1992.**

4-Hardness of coarse aggregate (crushing value) according to **B.S. 812Part110-1990.**

5-Sieve analysis of coarse aggregate according to **ASTM C33/84.**

The results of these experiments are given in **Table 3.2**.The percentage passing by weight is shown in **Table 3.4** and the grading curve meets the**ASTM C 33/84** requirements as shown in **Fig 3.2**.

3.2.1.3 Water.

Tap water was used for mixing and curing the concrete. The chemical analysis of water is given in **Table 3.5**.

3.2.1.4 Silicafume.

Silicafume was used as a pozzolanic material with bulk density 650 kg/m³. It was used as cement replacement. Silicafume meets the **ASTM C 1240/2000** requirements. The chemical composition and physical properties of silicafume are presented in **Table 3.6**.

3.2.1.5 Bentonite.

Locally available brown bentonite is also used as partial cement replacement material for concrete and mortar.**Table 3.6** represents the chemical component and physical properties of bentonite.

3.2.1.6 Limestone powder.

Lime stone powder was used as an additive and cement replacement by mass for concrete. The physical properties and XRD analysis of used limestone powder are presented in **Table 3.7** and **Fig. 3.3** respectively.

3.2.1.7 Ceramic powder.

Also ceramic powder was used in the third part of the experimental program as a cement replacement of the plaster mortar. EDAX analysis of used ceramic powder was presented in **Table 3.8**and **Fig. 3.4** respectively.

3.2.1.8Melamine powder.

Melamine is a white crystalline powder that contains carbon, hydrogen, and nitrogen.Melamine is widely used to produce melamine resins, for applications in plastics, adhesives,laminated countertops, molded dishware, whiteboards, coatings, paper, textiles, dyes, flame-retardants and as superplastisizer for concrete. It was used as a partial replacement of cement content in mortar that may be used as external protection. EDAX analysis of used melamine powder was presented in **Table 3.9**and **Fig. 3.5** respectively.

3.2.1.9Brick clay powder.

The waste bricks blocks that used in this study were obtained from recycled bricks. Cracked pieces of bricks were crushed by a jaw crusher. Andat laboratory scale the bricks wastes were ground with an air jet mill to obtain bricks powder.The resulting powders were sieved through a 45-µm (325 mesh) sieve. The resulting brick clay powderwas used as a partial replacement of cement content in the plaster mortar. Also the EDAX analysis of brick clay was tabulated in **Table 3.10** and **Fig. 3.6** respectively.

3.2.1.10 Polypropylene fibers.

Polypropylene fiber from Sika Company was used as an additive to concrete mix with 0.10 Volume fraction. The properties of used PP fibers were concluded in **Table 3.11.**

3.2.1.11Steel fibers.

Steel fibers with 0.40 mm thickness and12 mm length with volume fraction 1.2 were used in this study to enhancement the fire endurance of conventional concrete.

3.2.1.12 Admixtures.

One type of Superplasticizer Type F admixture was used to obtain a constant slump of 10 ± 2.5 cm. The admixture complies with the ASTM C 494/82 andB.S. 5075 Part 3 requirements.

Table 3.1: Chemical analysis of CEM I.

	CEM I
Silicon dioxide(SiO_2) %	17.8
Aluminum oxide (Al_2O_3) %	4.82
Ferric oxide (Fe_2O_3) %	1
Magnesium oxide (MgO)%	3
Sulfur trioxide(So_3)%	2.7
Calcium oxide (CaO)%	60.9
Loss in ignition %	4.65
Insoluble residue%	4.34
Tricalciumsilicate(C_3S)%	59.73
Tricalciumaluminate(C_3A)%	11
Tetracalciumaluminoferrite (C_4AF) %	3.043

Table 3.2: Physical properties of fine and coarse aggregate according to ASTM specifications.

Property	Sand	Pink Limestone	Allowable limit
Bulk Specific gravity	2.46	2.56	-
Absorption, %	2.4	1.8	3%
Unit weight	1.71	1.40	-
Material Finer than 75µm, %	0.8	0.4	Less than 3%
Hardness of Coarse Aggregate Crushing value, %	-	21	Less than 30%

Table 3.3: ASTM C 33/84 requirements and percentage passing by weight of Fine aggregate (F.M. = 2.54).

Sieve Size or No.	**3/8"**	**No.4**	**No.8**	**No.16**	**No.30**	**No.50**	**No.100**
	10mm	**5mm**	**2.36mm**	**1.18mm**	**600µm**	**300µm**	**150µm**
ASTM REQUIREMENTS % C 33/84	100	95-100	80-100	50-85	25-60	10-30	2-10
PERCENTAGE PASSING BY WEIGHT (%)	100	97.2	92.6	82.2	57.9	14.4	2.1

Table 3.4: ASTM C 33/84 requirements and percentage passing by weight of coarse aggregate (N.M.S. = 1").

Sieve Size or No,	**3/2"**	**1"**	**3/4"**	**1/2"**	**3/8"**	**No.4**	**No.8**
	37.5mm	**25.4mm**	**20mm**	**14mm**	**10mm**	**5mm**	**2.36mm**
ASTM REQUIREMENTS % C 33/84	100	95-100	60-80	25-60	12.5-35	0-10	0-5
PERCENTAGE PASSING BY WEIGHT (%)	100	100	77.3	59.9	23.83	1.56	0.39

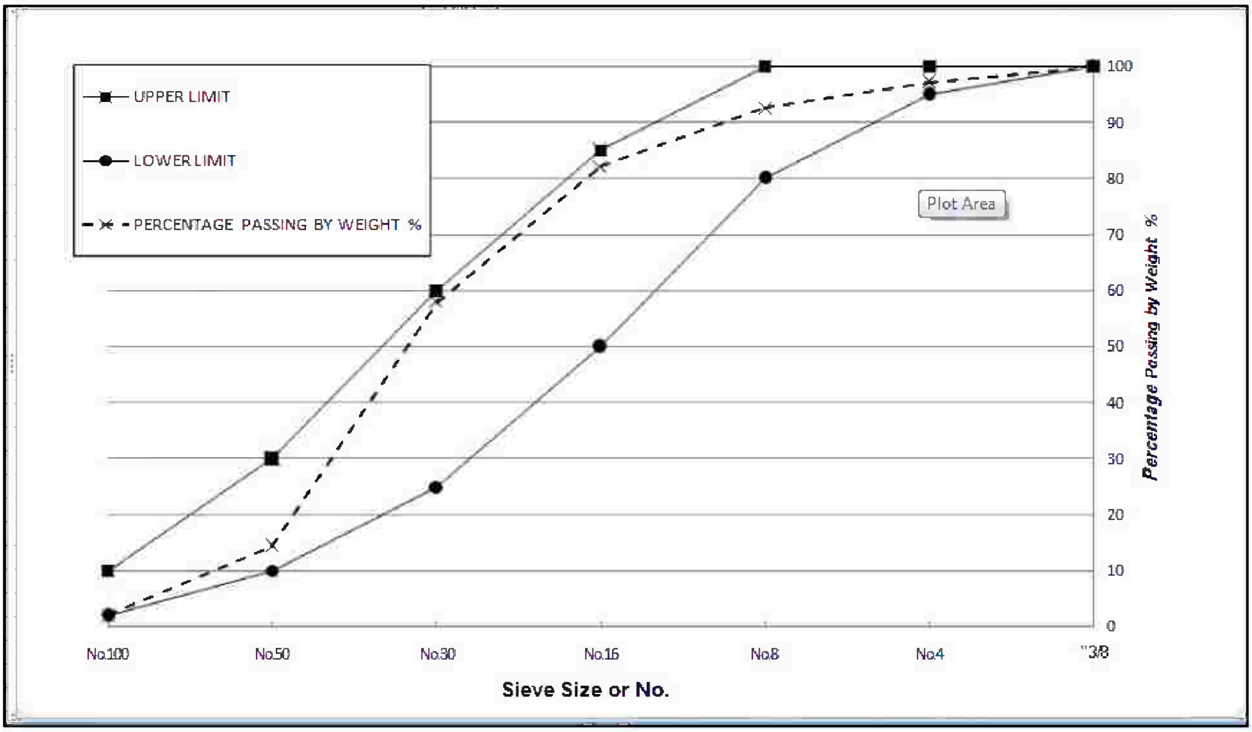

Fig. 3.1-Grading of sand according to ASTM specifications.

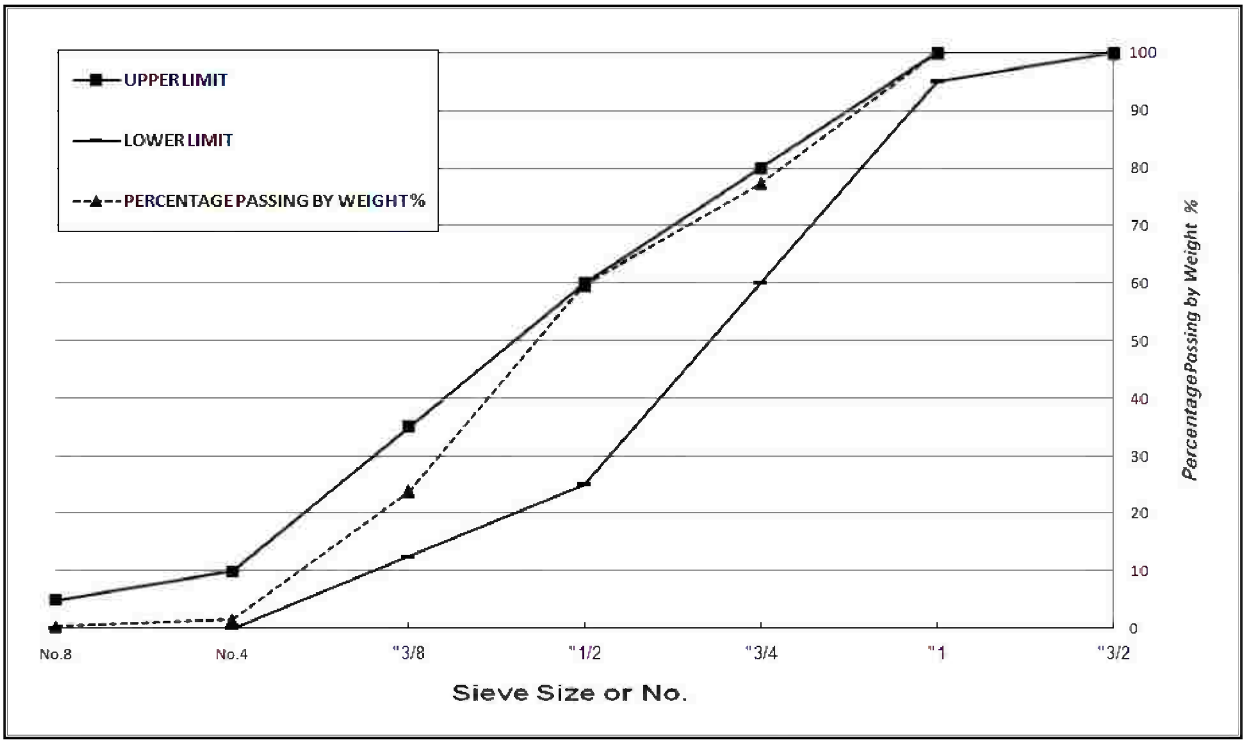

Fig. 3.2-Grading of pink lime stone according to ASTM specifications.

Table 3.5: Maximum allowable chemical impurities in water used for mixing and curing of concrete(**Adel A. El-kurdi, et al. 2007**).

Type	Allowable Limit (p.p.m)	Water used in mixing and curing (p.p.m)
Soluble as solid materials	Less than 2000	280
Alkaline carbonates and soluble bicarbonates	Less than 1000	183
Sodium and Potassium Alkaline (Na2o+0.658K2O)	Less than 600	Trace
Chlorides **-Prestressed concrete and bridge slabs** **-Reinforced concrete**	Less than 500 Less than 1000	42
Sulfates **-as SO4** **- as SO3**	Less than 3000	53 44
Sodium chloride	Less than 20000	69
Calcium bicarbonate	Less than 400	183
Magnesium chloride	Less than 40000	59
Magnesium Sulfate	Less than 25000	75
Sodium phosphate	Less than 500	Nil
Sodium borate	Less than 500	Nil
Sodium sulfate	Less than 100	78
Sodium hydroxide	Less than 5000 or 5% wt of cement	Trace
Potassium hydroxide	Less than 1500 or 5% wt of cement	Trace
Sugar	Less than 500	Trace
Silt and Clay	Less than 2000	Trace
PH Value	More than 4	7.1

Table 3.6: Chemical compositions and physical properties of silicafume and bentonite.

Chemical composition (%)	Silicafume	Bentonite
(SiO_2) %	96.1	48.11
(Al_2O_3) %	0.5	14.56
(Fe_2O_3) %	0.7	7.48
CaO%	0.21	1.93
MgO%	–	2.73
Na_2O%	0.31	2.98
K_2O%	0.49	0.92
SO_3%	0.1	-
C_3A%	-	-
Loss on ignition	1.14	19.63
Specific gravity	2.32	2.65
Specific surface (cm^2/gm)	225000	52300
Bulk density (kg/m^3)	650	660

Table 3.7: Physical properties of limestone powder (LP)(**Aymanet al.2014).**

Properties	Value
Blaine	3400 cm2/gm
Specific gravity	2.55
Bulk density (kg/m^3)	٥٥٠

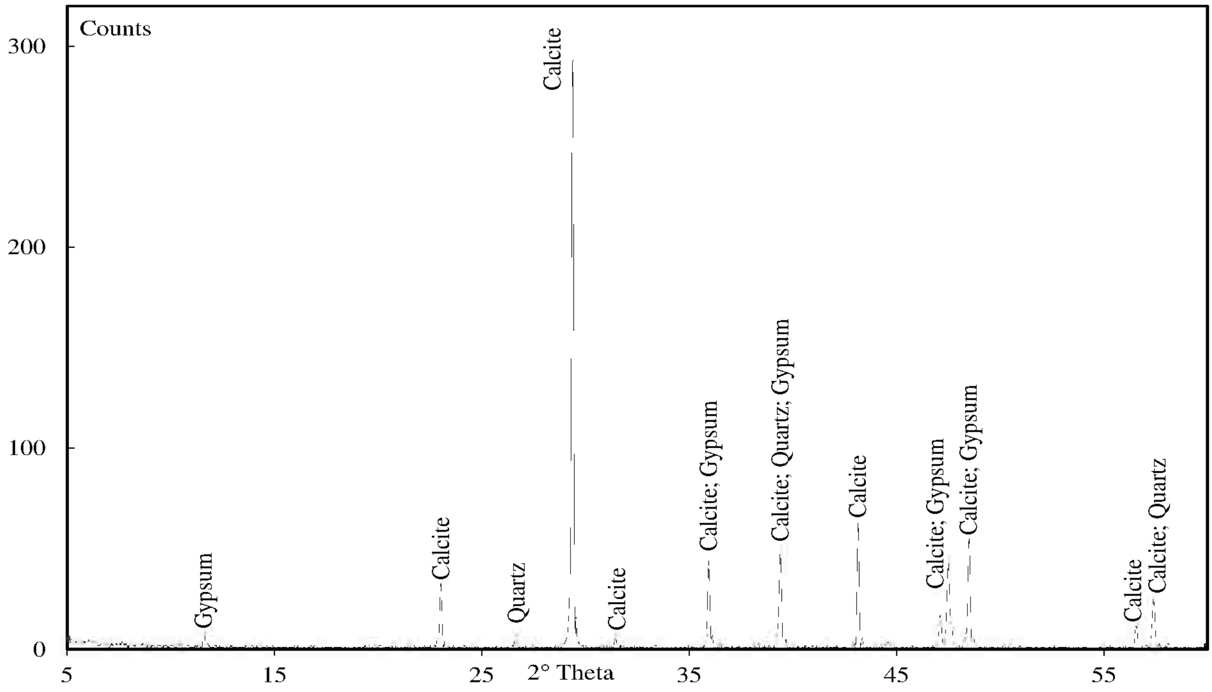

Fig. 3.3: XRD analysis of used limestone powder (LP).

Table 3.8: EDAX analysis of ceramic powder.

Label	Range (keV)	% Total
AlKa	1.388 to 1.587	2.5
SKa	2.188 to 2.428	0.2
CaKa	3.568 to 3.828	84.9
CaKb	3.888 to 4.148	10.5
CuKa	7.887 to 8.208	1.2
ZnKa	8.467 to 8.807	0.6

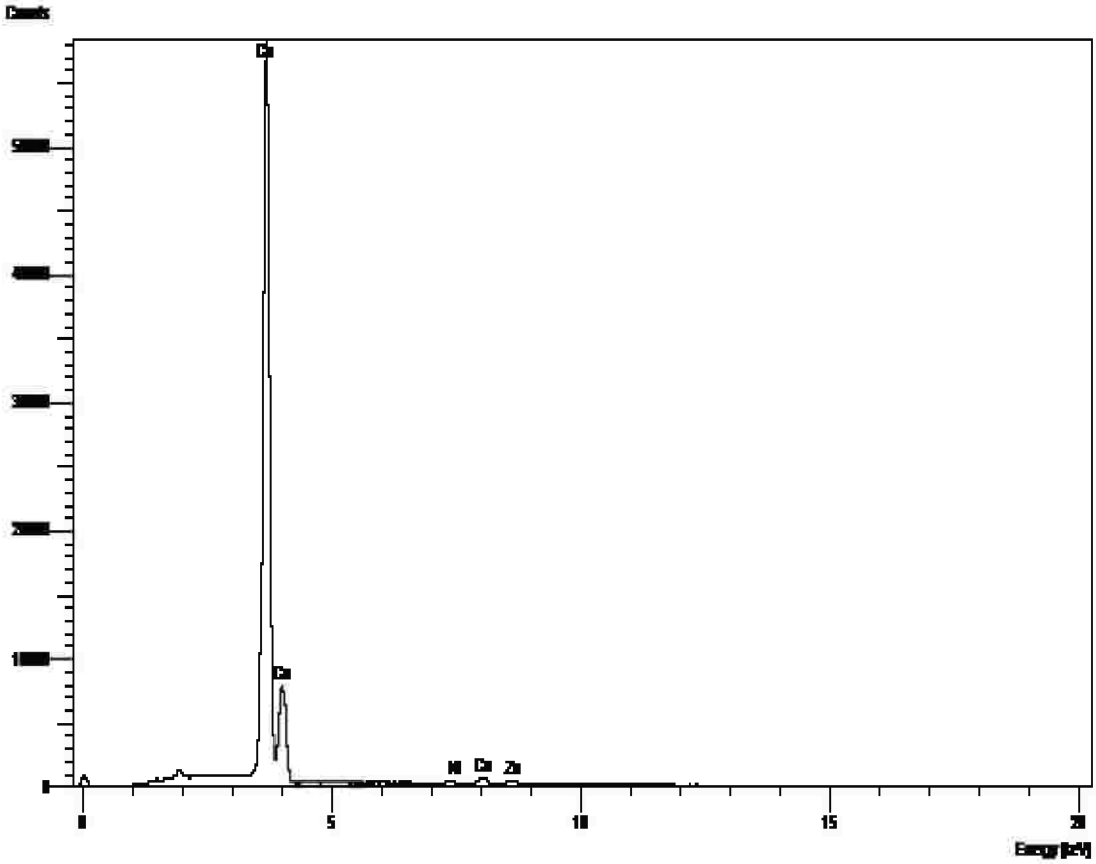

Fig. 3.4: EDAX analysis of used ceramic powder (C.P).

Table 3.9:EDAX analysis of melamine powder.

Label	**Range (keV)**	**% Total**
AlKa	1.388 to 1.587	0.6
SiKa	1.628 to 1.847	1.7
SKa	2.188 to 2.428	0.1
CaKa	3.568 to 3.828	93.4
FeKa	6.247 to 6.548	0.3
CuKa	7.887 to 8.208	2.3
ZnKa	8.467 to 8.807	1.6

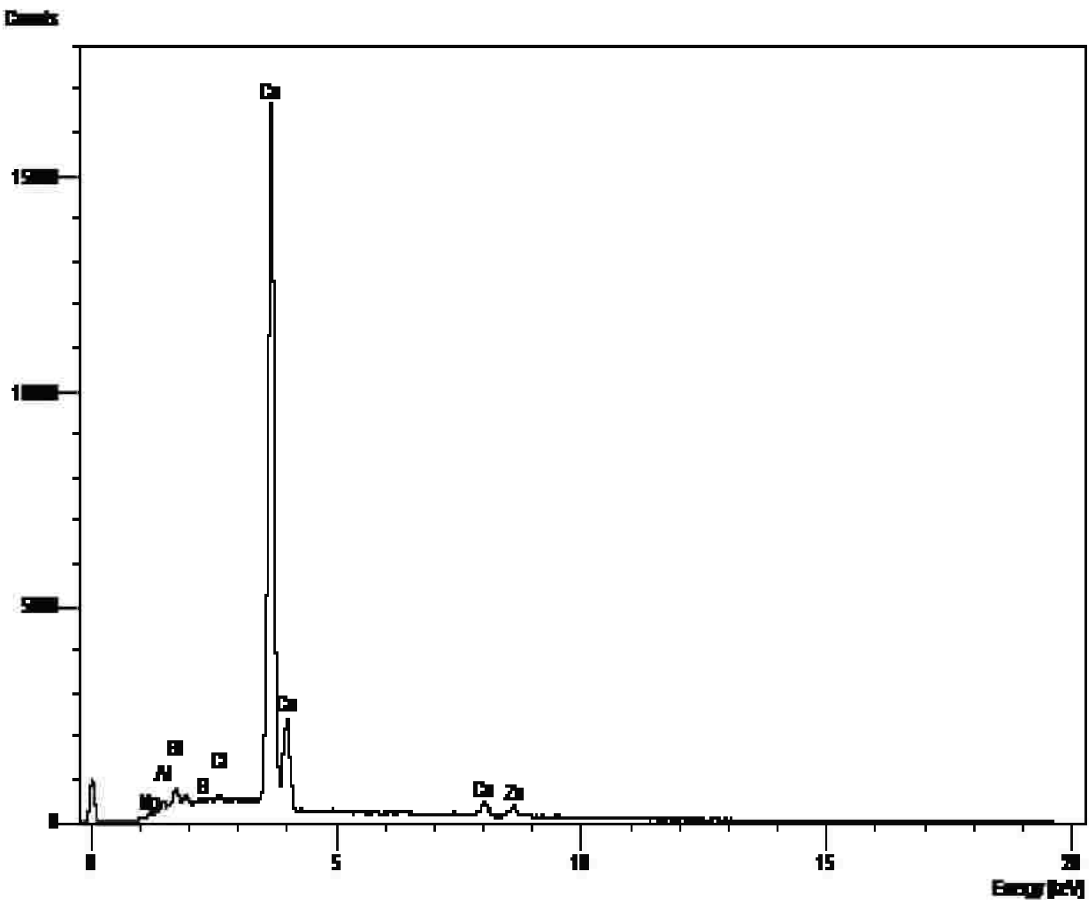

Fig. 3.5: EDAX analysis of used melamine powder (M.P).

Table 3.10: EDAX analysis of brick clay powder.

Label	Range (keV)	% Total
AlKa	1.388 to 1.587	10.6
SiKa	1.628 to 1.847	18.8
SKa	2.188 to 2.428	4.9
CaKa	3.568 to 3.828	38.4
FeKa	6.247 to 6.548	10.9
CuKa	7.887 to 8.208	9.6
ZnKa	8.467 to 8.807	6.8

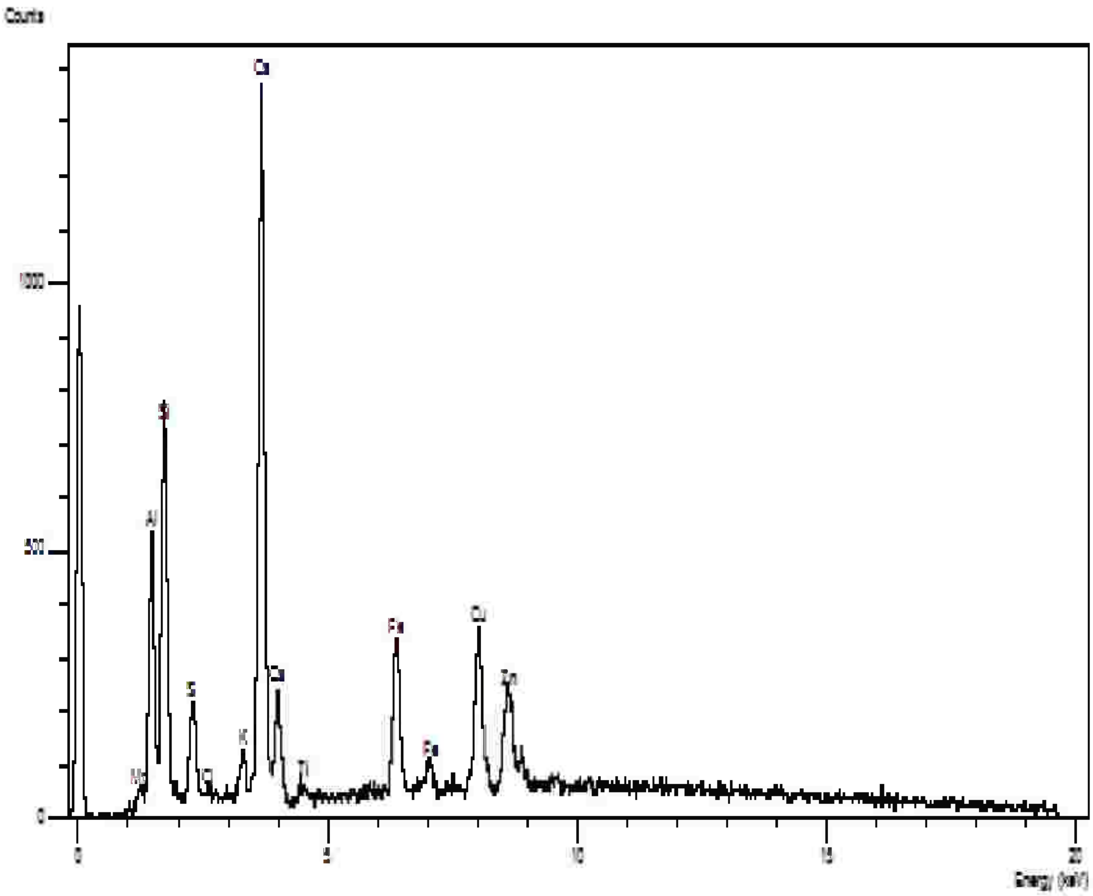

Fig. 3.5: EDAX analysis of used brick clay (B.C).

Table 3.11: Physical properties of polypropylene fibers.

Property	Description
Length	6 mm
Diameter nominally	18 μm
Elongation at rupture	15 %
Density:	0.91 kg/dm^3
E-module:	3500-3900 N/mm^2
Tensile strength	300 N/mm^2
Melting point:	160 - 165° C
Moisture absorption	0%
Color	Transparent white

3.2.2 PREPARATION OF SPECIMENS.

3.2.2.1 Test specimens for concrete.

1-Cubes 100 x100 mm.

Cubes 100 mm side lengths were used to determine cube compressive strength,weight loss, TGA/DTG and SEM for all concrete mixtures.Twelvecubes were carried out for every mix.

2-Cylinders 100 x200 mm.

Cylinders 100 mm diameter and 200 mm length were used to determine both of splitting tensile strength and pull out test by put steel bar 10 mm diameter in the center of the concrete cylinder.

3-Beams 100 x100 x500 mm.

120 beams with square section of 100 mm side length and 500 mm long were used to discover the flexural strength for all concrete mixtures as presented in **Fig. 3.7**. Twelve beam for every mix where divided into three beams for each degree of temperature.

Fig. 3.7- Beams before flexural test.

4-Reinforced concrete slabs 250x300 x50 mm.

Reinforced concrete slabs with 250 mm width, 300 length with 50 mm thickness were used,without materials scale down, to study the effect of the studied materials on the concrete fire protection, see **Fig. 3.8**. Thermocouples with 0.8mm solid wire, type K(nickel-chrome, nickel), ceramic insulation with maximum temperature about 1200º C are located with connection to steel bars to develop their temperature during the fire exposure test.

Fig. 3.8- Small scale slabs.

5-Conventional concrete slabs 250x300 x100 mm.

Also six conventional reinforced concrete slabs with 250 mm width, 300 length and 100 mm thickness were casted with 25, 50 and 75 mm concrete cover to investigate the effect of the cover thickness on the reinforcement´s temperatureas seen in **Fig. 3.9**.

Fig. 3.9- Reinforced concrete slab with embedded thermocouples to investigate the effect of the cover thickness on the reinforcement´s temperature.

<u>3.2.2.2 Test specimens for mortar</u>

<u>1-Cubes 25 x25mm.</u>

Twelve cubes with 25 mm side length were casted for each mortar mixture to evaluate the 28-day cube compressive strength, weight loss, TGA/DTG and SEM.

<u>2-Conventional concrete slabs 250x300 x50 mm.</u>

Conventional reinforced concrete slabs with 250 mm width, 300 length with 50 mm thickness were casted and a 25 mm plastering from the examine mortars was done to study the effect of the different studied materials on the concrete fire protection as an external protection as shown in **Fig. 3.10[a,b].**

(a) (b)

Fig. 3.10- a) Slabs during pouring the plaster b) Slab after plastering.

3-Cubes 100 x100mm.

Conventional concrete cubes with 100 mm side length were poured, these cubes werefrom all sides covered by 25 mm from studied mortars. The purpose is to study that how these materials can affect the conventional concrete cubes in term of compressive strength after exposed to 600ºC for 2 hrs.

3.2.2.3 Mixing procedures.

The mixer for the concrete was a pan-type roller, 0.15 cubic meter capacity. The dry pink limestone and sand was allowed to mix in for 2 minutes, cementitious materials were added until the blend was uniform. The High Range Water-Reducing Admixture was added to the water and gradually added the water to the dry mix and the mixing continued for a further five minutes. The mixing procedures were according to the **ASTM C 192.** Then the concrete is charged out from the mixer. The concrete was placed in molds in approximately three equal layers, and compacted on a vibrated table. All specimens after casting were left covered with polythene sheet for 24 hours, the molds were then stripped and the specimens were placed in a temperature controlled curing tank at 20º C for a further 27 days, after that specimens were taken from the curing tank, wiped clean to remove any loose grit or extraneous material.The cubes were dried at 105º C for 24 hours then were weighed before exposed to the fire test. In unstressed residual strength test method, the temperature was kept at fixed temperatures of 200, 400, and 600º C each maintained for 2 hours exposure time. The specimens were then taken out from the oven. Cooled down to room temperature by keeping it in room temperature, then the specimens were weighted and coded to be ready for the test. Each three cubes were tested and the average value reported for compressive strength, weight loss and surface cracks. To determine the temperature of the exposed and unexposed surface of slabs, **Fluke 568 IR thermometer laser apparatus** was used. Two Thermocouples with 0.8mm solid wire, type K(nickel-chrome, nickel), ceramic insulation were used to measure the temperature of Rft inside the concrete slabs.

3.3 TEST PARAMETERS

In the mixture proportion of all concrete mixtures, water to cementitious materials ratios of 0.40 was used. The cementations materials were constant at 400kg/m³ except the mix containing 10 and 15% L.P as additive to cement content. **Table 3.11**, shows at the beginning the abridged terms of the concrete mixtures which may be used as follow to describe the mixtures. The detailed concrete mixture constituents used in this study were presented in**Table 3.12**

Table 3.11: Abridged terms of the concrete mixtures.

Abridged term	Description
M-C	Control mix
M-(Silica)	Concrete mix with 10% silicafume, as a cement replacement by mass.
M-(PPF)	Concrete mix with 0.90 kg/m³ polypropylene fibers.
M-(Sil.PPF)	Concrete mix with 10% silicafume, as a cement replacement + 0.90 kg/m³ polypropylene fibers.
M-(Steel.F)	Concrete mix with 20 kg/m³ steel fibers.
M-(L.P.10R)	Concrete mix with 10% limestone powder, as a cement replacement by mass.
M-(L.P.15R)	Concrete mix with 15% limestone powder, as a cement replacement by mass.
M-(L.P.10A)	Concrete mix with 10% limestone powder, as a cement additive by mass.
M-(L.P.15A)	Concrete mix with 15% limestone powder, as a cement additive by mass.
M-(Ben.10R)	Concrete mix with 10% bentonite, as a cement replacement by mass.

Table 3.12: Details of concrete mixes.

Materials	Cement, kg/m³	Coarse. agg(kg)	Fine agg.kg/m³	Water, kg/m³	Superpla., kg/m³	Silicafume kg/m³	PP-fibers, kg/m³	S. fibers, kg/m³	L.P kg/m³	Ben, kg/m³	w/c (ratio)
M-C Control	400	1060	708	160	1.1	-	-	-	-	-	0.4
M-Silica	360	1058	705	160	1.1	40	-	-	-	-	0.4
M-PPF	400	1060	708	160	2.6	-	0.9	-	-	-	0.4
M-Silica+ PPF	360	989	659	160	2.6	40	0.9	-	-	-	0.4
M-Steel.F	400	1060	708	160	1.1	-	-	20	-	-	0.4
M-L.P.10R	360	1059	707	160	1.1	-	-	-	40	-	0.4
M-L.P.15R	340	1057	705	160	1.1	-	-	-	60	-	0.4
M-L.P.10A	400	1016	676	176	1.1	-	-	-	40	-	0.4
M-L.P.15A	400	990	660	184	1.0	-	-	-	60	-	0.4
M-Ben.10R	360	1060	707	160	1.1	-	-	-	-	40	0.4

3.4 TEST METHODS.

3.4.1 Fresh state of concrete.

1) Method of mixing and sampling fresh concrete in the laboratory, according to **ASTM C 192.**
2) Method of determination of slump, according to **B.S 1881: PART 102.**

3.4.2 Hardened state of concrete and mortar.

The most common way to study the influence of elevated temperatures on the properties of concrete is to expose the material to high temperature, cool it down to room temperature and then carry out tests. This method yields the (post exposure to the high temperature) or (residual) properties of concrete.In unstressed residual strength test method in this thesis, the temperature was kept at fixed temperatures of 200, 400, and 600º C, each maintained for 2 hours exposure time. The specimens were then taken out from the oven, cooled down to room temperature by keeping it in room temperature, then the cube specimens only were weighted and coded to be ready for the test.MTI –KSL -1700X electric oven was used for cubes and cylinders with the heating rate 8°C/min,Gas oven was also used for beams that used to determine flexural strength and for cylinders that used to evaluate pull out strength as presented in **Figs. 3.11 and 3.12** respectively.

Fig. 3.11- Electric oven

Fig. 3.12-Gas oven

3.4.2.1 Compressive strength.

The method for determination of compressive strength of concrete and mortars cubes is according to **B.S. 1881:Part 116.** A universal testing machinecapacity 2500 KN as shown in **Fig 3.13**was available for the compression. The machine meets the **B.S. 1881: Part 115.**

Fig. 3.13- Compression testing machine.

3.4.2.2 Splitting tensile strength.

Splitting tensile strength was done to evaluate the impact of elevated temperature on conventional concrete. The indirect tensile tests were performed on 100 × 200 mm cylinders using universal testing machine as per ASTM C496 as seen in **Fig. 3.14**. Three samples per batch tested and the average strength is reported. The indirect tensile strength is determined as follows:

$\sigma t = 2Pmax/\pi DL$

Where σt is the indirect tensile strength, Pmax is the maximum applied load, D is the diameter and L is the length of cylinder.

Fig. 3.14- Concrete cylinder under compression machine to evaluate splitting tensile strength.

3.4.2.3 Flexural strength.

Flexural testing machine was used to obtain the flexural strength of concrete beams, shown in **Fig. 3.15.** The flexural strength of all concrete mixtures was done (using simple beam with four points loading) according to **ASTM C 78.**

Fig. 3.15- Flexural testing machine.

3.4.2.4 The impact of fire on reinforced concrete slabs.

Small scale reinforced concrete slabs of different mixtures were exposed tofire; the degrees of fire temperatures were 600, 800 and 1200ºC. Thermocouples with 0.8mm solid wire, type K(nickel-chrome, nickel), ceramic insulation with maximum temperature about 1200º C are located with connection to steel bars to develop their temperature with time.To determine the temperature of the exposed and unexposed surface of slabs, Fluke 568 IR thermometer laser apparatus was used as shown in **Fig. 3.16**. Also visual inspection was carried out on slabs to investigate the spalling and the surface cracks.

Fig. 3.16-Fluke 568 IR thermometer laser apparatus.

3.4.2.5 Effect of concrete cover thickness on reinforcement protection.

100 mm thickness small scale conventional reinforced concrete slabs with different concrete cover thickness to protect the reinforcement inside the slabs were exposed to 800ºC for 2 hrs. The temperatures of the reinforcement were recorded with time up to 2 hr from exposure and until 2hr of air cooling, which it means that the readings were taken for4 hrs to incidence more accuracy.

3.4.2.6 The impact of water coolingregime on residual compressive strength of conventional concrete.

After heating, cubes were cooled down in quick cooling. For quick cooling, specimens were cooled down by placing them in water for one day. The cubes were removed and dried at 105ºC for 24 hours then were coded to be ready for the compression test.

3.4.2.7Deceitful pull out test.

Pull out test was done to study the effect of elevated temperature on the correlation between steel bars and the concrete. **Fig. 3.17** shows the specimens under the testing machine.

Fig. 3.17 – Pull out specimen under testing machine.

3.4.2.8Thermogravimetric Analysis and differential thermal analysis (TGA/DTG).

The TGA/DTG was carried out on both studied **concrete and mortar**. Thermogravimetric analysis is a technique in which the mass of a substance ismonitored as a function of temperature or time as the sample specimen is subjected to acontrolled temperature program in a controlled atmosphere.The datafrom the test is displayed as TGA (weight loss as a function oftemperature) and as DTG (derivative thermal gravimetry,weight loss rate as a function of temperature).A TGA instrument consists of a sample pan that is supported by a precision balance. That panresides in a furnace and is heated during the experiment. The mass ofthe sample is monitored during the experiment. A sample purge gas controls thesample environment. This gas may be inert or a reactive gas that flows over thesample and exits through an exhaust. In this thesis, samples were taken from inside the cubes just about 1 mm from the surface and were analyzed by thermogravimetric analysis (TGA/DTG). TGA and DTG were done simultaneously. The maximum heating temperature was 1000°C with the heating rate20°C/min using air as a medium under static condition. The thermogrevimetric analyzer was (Thass-TGA I 1000) as shown in **Fig. 3.18.** The mass of sample was about 40 mg.

Fig. 3.18-The thermogrevimetric analyzer(Thass-TGA I 1000).

<u>3.4.2.9 Scanning electron microscope (SEM).</u>

In this work a SEM instrument, JEOL-JSM-5300, as shown in **Fig.3.19**is used toinvestigate the surface morphology of the **concrete and mortars**. In combination with other analyticaltechniques this provides an understanding of the hydration process of cement and the fire impact on the microstructure of the concrete and mortar.

The samples were coated, before the testcarried out,with a conducting nano-layer of conducting gold to lower charge build-up as shown in **Fig. 3.20**.

Fig. 3.19-SEM instrument, JEOL-JSM-5300.

Fig. 3.20 Coating samples with nano- layer of gold before test.

3.4.2.10Visual inspection.

Visual inspection was carried out on all concrete cube specimens after air cooling to investigate the propagation of surface cracks with elevated temperature. Two amplifies lens were used to aid in cracks exploration as appeared in **Fig. 3.21.**

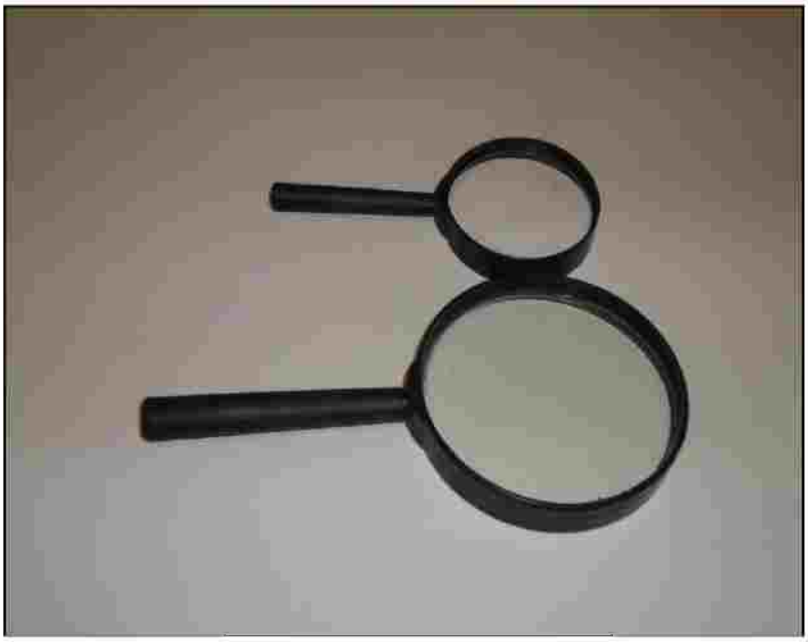

Fig. 3.21-Two amplifies lens.

3.4.2.11 Weight loss.

The spalling of the concrete and mortar was investigated as a function of weight loss. The concrete and mortar cubes were weighted before and after exposure to elevated temperature to determine the weight loss.