CHAPTER 3

MATERIALS AND EXPERIMENTAL PROGRAM

3.1 Introductions:

This chapter contains the properties of used materials, experimental program mixing procedure, fresh concrete tests and hardened concrete tests.

3.2 Fine aggregate:

Natural siliceous sand of 2.60 fineness modulus was used. Table (3-1) shows the properties of this sand.

Table (3.1) Properties of used sand

Properties	Test result
Unit weight kg/m^3	1.79
Specific gravity	2.60
Fineness modulus	2.60
Fine material passing sieve No. 200	2.4%

Table (3-2) and Figure (3-1) shows the sieve analysis of the used sand and the limits of ASTM C33-84.

Table (3-2) Sieve analysis of used sand

Sieve No.	4	8	16	30	50	100
Passing %	98	86	80	53	20	2
ASTM limits	90-100	80-100	50-85	25-60	10-30	2-10

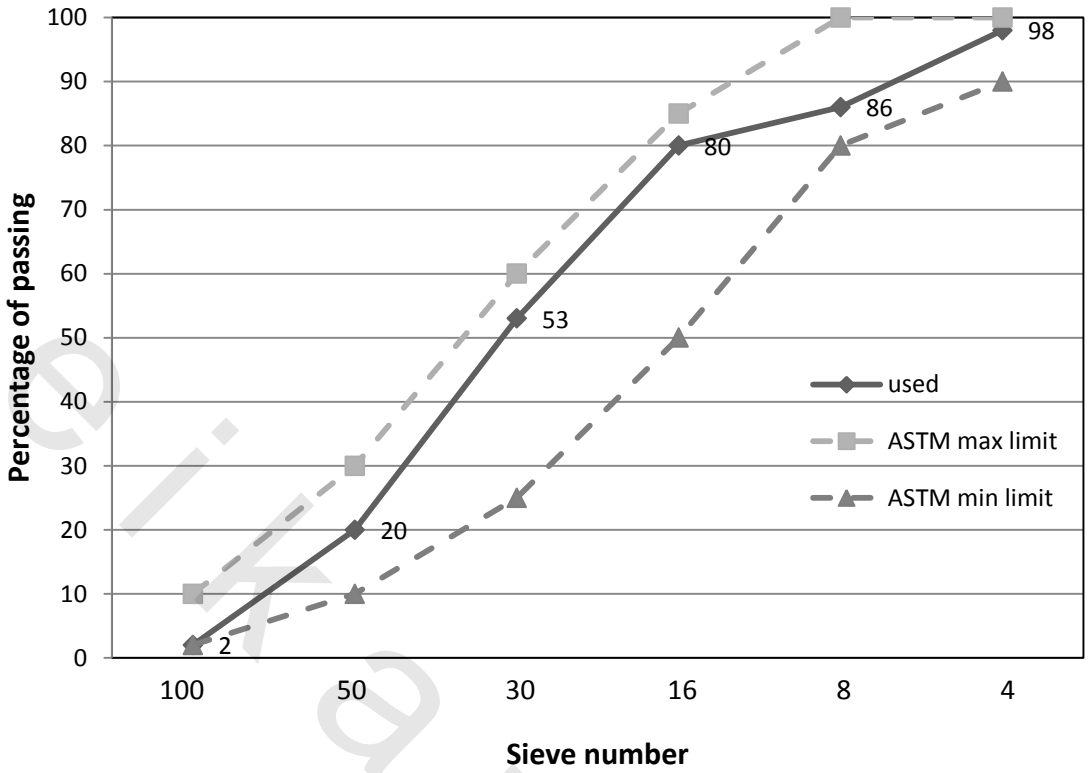

Figure (3-1) Sieve analysis curve of sand

3.3 Coarse aggregate:

A hard crushed pink lime stone aggregate was used. Table (3-3) contains the properties of used coarse aggregate.

Table (3-3) Properties of crushed pink coarse lime stone.

Properties	Coarse aggregate
Apparent specific gravity	2.583
Unit weight kg/m^3	1.44
Water absorption %	1.95
Loss Angelus %	27
Fine material passing from sieve No. 200	2.10

Table (3-4) and Figure (3-2) shows the sieve analysis of used coarse aggregate with 10 mm nominal maximum aggregate size.

Table (3-4) Sieve analysis of used coarse aggregate.

Sieve size No.	Passing %	ASTM C33 limits
$\frac{1}{2}$	100	100
$\frac{3}{8}$	95	85-100
$\frac{3}{16}$	28	10-30
8	9	0-10
16	3	0-5

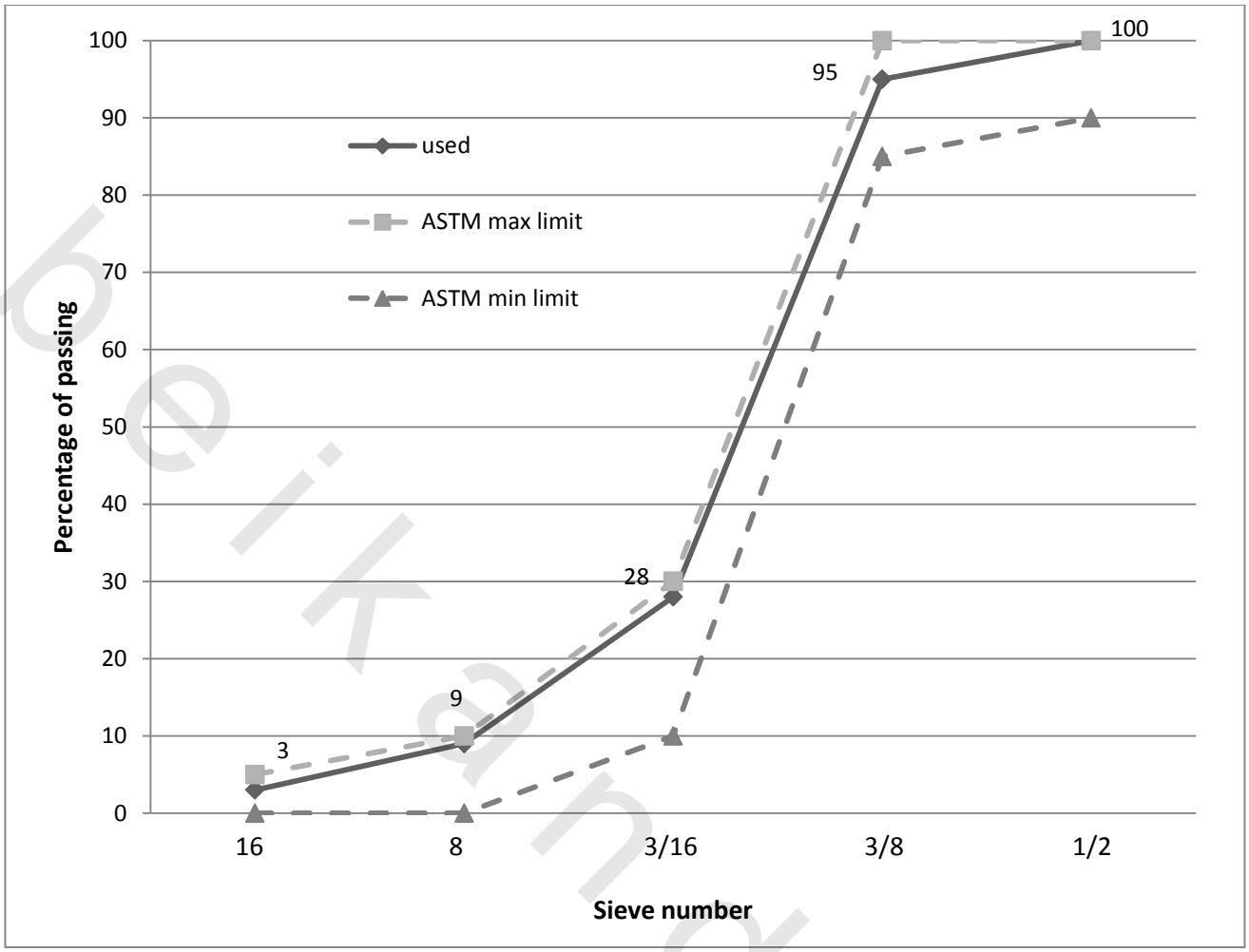

Figure (3-2) Sieve analysis curve of coarse aggregate

3.4 Cement:

Portland cement (OPC) and sulfate resisting cement (SRC) were used in this study. Three different sources of OPC and two different sources of SRC were considered in this study. The physical and mechanical properties of used cements meet the requirements of ESS4756 -2006

The properties and chemical analysis of these cements are given in tables (3-5) and (3-6).

Table (3-5) Physical and mechanical properties of used cement.

Properties	OPC Alex 1	OPC Asuite 2	OPC Amria 3*	SRC Alex (source 1)	SRC Amria (source 2)
Initial setting time (min)	160	152	185	135	190
Final setting time (min)	310	305	330	275	305
% retained sieve No.200	6	8	11	6.5	7.5
Compressive strength 3days 7days	 20.3 28.3	 19.5 27.5	 18 29.7	 24.7 32.3	 18 30.1

Table (3-6) Chemical composition of used cement.

Oxide	OPC Alex 1	OPC Asuite 2	OPC Amria 3*	SRC Alex (source 1)	SRC Amria (source 2)
Sio2	20.1	20	19.85	19.57	19.73
$Al_2 O_3$	5.50	4.41	4.44	4.0	3.48
Fe_2O_3	3.42	5.54	3.29	5.40	4.73
CaO	61.1	63.17	62.35	62.4	62.82
MgO	4.03	1.41	2.84	3.1	3.3
SO_3	2.57	2.73	2.75	2.20	2.17
K_2O	0.27	0.23	0.27	0.3	0.30
Na_2O	0.56	0.54	0.57	0.48	0.47
Cl	0.03	0.05	0.04	0.02	0.02
Free CaO	0.66	0.96	0.77	0.55	0.64
LOI	2.9	2.85	3.50	2.41	2.81
C_3S	46.85	61.26	60.597	64.434	69.465
C_2S	22.28	11.121	11.196	7.498	4.16
C_3A	8.79	4.01	6.2	1.463	1.22
C_4AF	10.41	13.815	10.0115	16.432	14.393
$SO_3/(C_3A+C_3S)$	0.04	0.044	0.043	0.035	0.032
SO_3/C_3A	0.312	0.681	0.444	1.504	1.778

- The grade of all used cement is CEM I 42.5 except OPC (Amria 3*), Where the grade of this cement is 32.5 N

The chemical compounds are calculated using Pogue equations.

- The first cement is CEM I 42.5 N (ESS 47 56), produced by Alex cement company (Titan).
- The second cement is CEM I 42.5 N (second source of OPC) produced by Assute company.
- The third source of OPC cement is CEM I 32.5 N (produced by El Amria company).
- The fourth cement is sulfate resisting cement of grade 42.5 (Alex cement company).
- The fifth cement is sulfate resisting cement of grade 42.5 (El Amria company).

3.5 Silica fume:

Silica fume satisfied the requirements of ASTM C1240. The average particle size is 0.1 micron and the surface area is 150000 cm^2/gm. The physical properties and chemical composition of used silica fume are presented in table (3-7).

Table (3-7) Physical properties and chemical composition of silica fume.

Property	Silica fume	ASTM limits
Specific gravity	2.2	-
Silicon dioxide	90.5	$\geq$ 85%
Aluminum oxide	0.88	-
Ferric oxide	0.39	-
Calcium oxide	17.5	-
Sulfur trioxide	0.8	
Loss in ignition	1.5	$\leq$ 6%

3.6 Chemical admixture:

Four types of high range water reducing admixture (HRWR) were used, the first is Type G based on modified lignosulfonates(G-ML), the second is Type F based on sulfonated naphthalene formaldehyde condensates(F-Na), the third is Type F based on sulfonated melamine formaldehyde condensates(F-Me), while the fourth is Type G based on polycarboxylates(G-Poly). Type C is also used, it is based on a mixture of organic and inorganic complex agent and meets the requirement for hardening accelerators according to EN 934-20. These chemical admixtures meet the requirement of ASTM C94.

3.7 Experimental program:

In order to study the effect of over dosage of Type G admixture in retempering of concrete, the following variables were considered:

- Cement types, five cement sources were used. They are two sources of CEM I 42.5 N, one source of CEM I 32.5 N, and two sources of sulfate resisting cement, type V, grade 42.5.
- Retempering time, where 30 and 60 minutes were used.
- Chemical admixture used in concrete mix, Type G according to ASTM C494, mixing of Type G and Type F (based on sulfonated melamine), mixing of Type G and Type F based on (sulfonated naphthalene), and Type F (based on sulfonated melamine) were considered.
- Chemical admixture for retempering, no retempering, and retempering with Type G, Type F (based on melamine), Type F(based on naphthalene), accelerating admixture (sika rapid), mixing of Type G and accelerating admixture, polycarboxylic Type G admixture, and mixture of Type G and Type G (polycarboxylic) were used.
- Cement content, four cement contents of 400, 350, 300 and 250 kg/m^{3}were used with CEM1 42.5 N (source 1)
- Presence of silica fume, four concrete mixes were tested using silica fume.

The previous variables can be followed using Figure (3.3), and tables (3.8) through (3.10). Table (3-8) shows mixes with different initial admixture and different retempering admixture. Table (3-9) shows mixes of CEM I 42.5 N using different cement contents and the presence of silica fume.

Table (3-10) contains the mixes of other source of CEM I 42.5 N and CEM I 32.5 N to study the effect of redose using Type G admixture after 60 minutes. Table (3-11) presents mixes with sulfate resisting cement, and mixes containing silica fume.

Table (3-8) Mixes of CEMI 42.5 N (source 1*) cement content of 400 kg/m^3

Mix	Initial admixture		Time of redose	Redose admixture %				
	type	dose %		G	F-Na	F-Me	C-Rapid	G-Poly
M1	0.0	0.0	0.0					
M2	G	1.5	0.0					
M3	G	1.5	30	0.75				
M4	G	1.5	60	1.5				
M5	G	1.5	30		0.75			
M6	G	1.5	60		1.5			
M7	G	1.5	30			0.75		
M8	G	1.5	60			1.5		
M9	G+F-Na	1+0.5	0.0					
M10	G+F-Na	1+0.5	30		0.75			
M11	G+F-Na	1+0.5	60		1.5			
M12	G+F-Me	1+0.5	0.0					
M13	G+F-Me	1+0.5	30			0.75		
M14	G+F-Me	1+0.5	60			1.5		
M15	G	1.5	30	0.375			0.375	
M16	G	1.5	60	0.75			0.75	
M17	G	1.5	60	0.375			1.125	
M18	G	1.5	60				1.5	
M19	G	1.5	60	0.75				0.2

*Source 1 OPC Alex

Table (3-9) Mixes of CEM I N 42.5 (source 1) for different cement contents

Mix	**C.C kg/m^3	Silica fume kg/m^3	Water kg/m^3	Sand kg/m^3	Coarse agg.	Intial admix G %	Redose G	
							time min.	dose%
M20	250	0.0	200	739	1108	1.5	0.0	0.0
M21	250	0.0	200	739	1108	1.5	60	1.5
M22	300	0.0	195	728	1098	1.5	0.0	0.0
M23	300	0.0	195	728	1098	1.5	60	1.5
M24	350	0.0	191	723	1081	1.5	0.0	0.0
M25	350	0.0	191	723	1081	1.5	60	1.5
M26	460	40	180	600	1120	*2	0.0	0.0
M27	460	40	180	600	1120	*2	60	2

* other source of Type G of maximum dose of 2% was used
**c.c = Cement content

Table (3-10) Mixes of different cement grade and sources of cement content of 400 kg/m^3

Mix	Cement type	Sources	Intial admix G %	Redose G	
				time min.	dose%
M28	CEM1 42.5 N	2*	1.5	0.0	0.0
M29	CEM1 42.5 N	2*	1.5	60	1.5
M30	CEM1 32.5 N	3**	1.5	0.0	0.0
M31	CEM1 32.5 N	3**	1.5	60	1.5

*2 = OPC Asuite Grade of 42.5N
**3 = OPC Amria Grade of 32.5N

Table (3-11) Mixes of sulfate resisting cement CEM I 42.5 of different sources

Mix	C.C kg/m^3	Silica fume kg/m^3	Intial admix G %	Redose G%	Redose admixture %		
				time min.	G	F-Na	F-Me
S.R.C Alex (Source 1)							
M32	400		1.5				
M33	400		1.5	30	0.75		
M34	400		1.5	60	1.5		
M35	400		1.5	60		1.5	
M36	400		1.5	60			1.5
M37	400		1.5 F-Me	60			1.5
S.R.C Amria (Source2)							
M38	400		1.5				
M39	400		1.5	30	0.75		
M40	400		1.5	60	1.5		
M41	340	60	1.5				
M42	340	60	1.5	60	1.5		

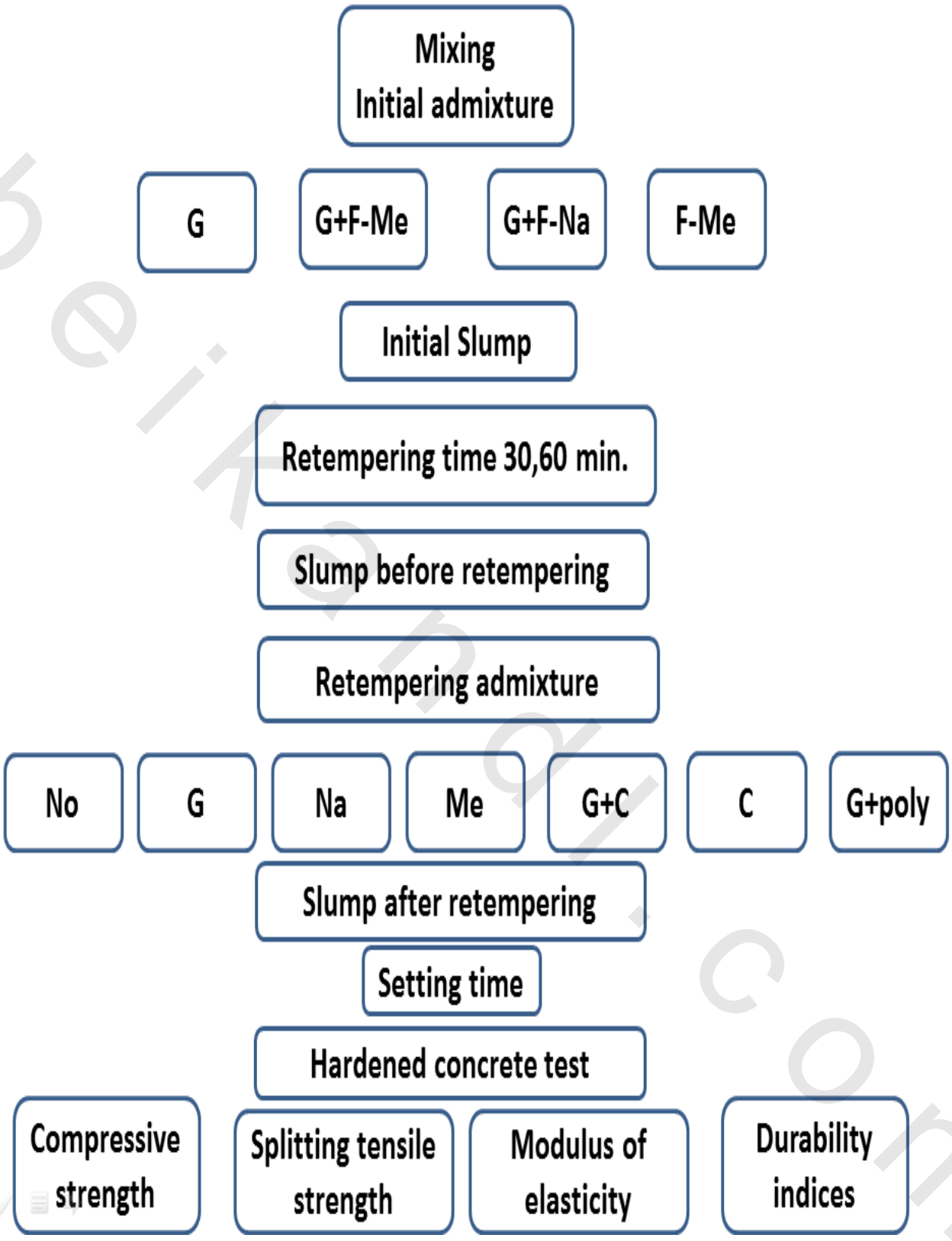

Figure (3-3) Flow chart of experimental program of CEM I 42.5N source 1

3.8 Symbols of used mixes:

A concrete mix shown in previous tables is represented using the symbols of M n (1, 2, 3) where:

- M refers to mix
- n refers to mix number
- 1 refers to initial admixture Type and dosage%
- 2 refers to retempering time after continuous mixing
- 3 refers to retempering admixture and dosage%

As examples:

- M1(0, 0, 0), it means that mix No. one hasn't any admixture, no retempering time and no retempering admixture.
- M8(1.5G, 60, 1.5F-Me), it means that mix no.8 having initial admixture of 1.5% Type G, retempering after 60 minutes, and retempering using 1.5% of Type F based on melamine.
- M10(1G+0.5F-Na, 30, 0.75F-Na) it means mix No.10 having initial admixture of 1%Type G and 0.5% Type F based on naphthalene, retempering after 30 minutes, and retempering using 0.75% of Type F based on naphthalene.
- M17(1.5G, 60, 0.375G+1.125 C) it means mix no.17 having initial admixture of 1.5% Type G, retempering after 60 minutes, and retempering using 0.375% of Type G and 1.125%C.

3.9 Mix proportions

Table (3-12) presents the proportions of most mixes and table 3.8 contains the proportions of the other mixes in gradients. All mixes have almost a slump ranged from 14 to 18 cm. All mixes were designed using the absolute volume equation and trial mixes.

Table (3-12) Proportions of concrete mixes

*Mix	C.C kg	Water kg	Sand kg	Coarse agg. kg	Silica fume kg
M1	400	235	720	982	
M2 to M19	400	189	720	1055	
M28 to M31	400	185	725	1060	
M32 to M40	400	189	720	1055	
M41 to M42	340	189	710	1055	60

*The other used mixes are given in Tables (3.8) through (3.11)

3.10 Mixing, preparing and curing

The concrete was mixed using a laboratory drum mixer for 4 minutes for concrete without retempering. The mixing continued for retempered concrete for 30 or 60 minutes. The concrete was cast in molds according to E.S.S 1947 and left till concrete hardening.

The molds were demolded and the concrete was cured for 7 days and left in laboratory conditions till testing date.

3.11 Fresh concrete tests:

Slump test was carried out after mixing (4 minutes), before retempering and after retempering according to ASTM C143.

3.12 Setting time

The initial and final setting time test was carried out according to ASTM C403-80.

3.13 Absorption, Voids ratio and Density tests

In order to study the effect of over dose of retarding admixture on durability of concrete, absorption, capillary pores, apparent specific gravity and bulk were determined. This procedure can be summarized as follows, two specimens at least was used for each test these specimens were prepared from splitting concrete strength test specimens after test. Any friable parts were removed before test.

The specimens were oven dried (100-110 ^{o}C) to reach a constant weight. After removing each specimen from oven, it was allowed to cool in air. The dry weight was designated (A).

The concrete specimen was immersed in water for 48 hours at least. The surface moisture was removed with a towel. The weight is measure and denoted (B).

The specimen was immersed in boiled water (according to ASTM C642 for 5 hours. The specimen is allowed to cool for not less than 14 hours the concrete is fully saturated. The surface water was removed with a towel. This weigh is designated (C).

The specimen is suspended in water using a rope. The suspended weight called (D). the test results are calculated using the following equations:

Absorption after immersion% (An = natural absorption) $= \frac{(B-A)}{A} \times 100$----------(2-1)

Absorption after immersion and boiling % (At = total absorption)

$= \frac{(C-A)}{A} \times 100$ ----------(2-2)

Capillary pores % (V = capillary voids) $= \frac{(C-A)}{(C-D)} \times 100$ ---------- (2-3)

Bulk specific gravity (γ) $= \frac{A}{(C-D)} \times 100$ ---------- (2-4)

Apparent specific gravity (Gs) $= \frac{A}{(A-D)} \times 100$ ---------- (2-5)

3.14 Compressive strength test

Cube compressive strength using standard cubes of $150 \times 150 \times 150$mm was evaluated at 1, 3, 7, 28 and 90 days age according to E.S.S (1947). If concrete did not hard after 1 day, test at 2 days will be carried out.

3.15 Splitting tensile strength test.

Splitting tensile strength cylinder of 150×300 mm was used to obtain splitting tensile strength at 90 days age, this test was carried out according to ASTM 1658, this strength (Fts) is calculated as follows:

$$Fts = \frac{2P}{\pi DL} \text{----------} (2\text{-}6)$$

3.16 Modulus of elasticity test.

Cylinder of 150×300 mm was used to obtain modulus of elasticity (E) at 90 days age according to E.S.S (1947) (E) at 90 days age. This is calculated as follows;

$$E = \frac{fa-0.5}{eu-eo} \text{----------Mpa}$$

$$f_a = \frac{\text{cube compressive strength}}{3} = \frac{fcu}{3} \text{ Mpa}$$

e_u = strain of concrete corresponding to $\frac{fcu}{3}$

e_o = strain of concrete corresponding to 0.5 Mpa