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Using Citric Acid As An Admixture and It's Influence on Some Properties of Concrete

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Abstract

The objective of this investigation is to find the effectiveness of the citric acid as retarding admixture. The experimental results indicate that the optimum dosage of citric acid is 0.02% by weight of cement. This dosage causes a delay in initial and final setting time of 1:42 and 3:18 hour: minute, respectively; and a reduction in water-cement ratio of 13% relative to reference concrete mix. So, citric acid can be classified according to ASTM-C494 as water-reducing and retarding chemical admixture type D.

The investigation also extends to evaluate the effect of using citric acid on properties of concrete such as, compressive strength, splitting tensile strength, modulus of rupture and dynamic modulus of elasticity at normal conditions and after exposure to salt solutions. Generally the results indicate that using citric acid in concrete enhance it's properties at normal conditions, the percentage increase in compressive strength, splitting tensile strength, modulus of rupture and dynamic modulus of elasticity at age 180 days was about 56%, 13%, 24% and 8% respectively relative to reference concrete. Also it was observed that concrete containing citric acid has good performance after 180 days exposure to salt solution in comparison with reference concrete.

Keywords: Citric Acid, Admixture, Initial and final setting time, Compressive strength, Splitting tensile strength, Modulus of rupture.

استعمال حامض ألستريك كمضاف ودراسة تأثيره على بعض خواص الخرسانة

الخلاصة

الغرض من هذا البحث هو لمعرفة تأثير حامض ألستريك كأحد المضيافات المبطئة المؤثرة. تشير النتائج العملية إلى إن الرزمة المثالية من حامض ألستريك هي ٠,٠٢ ٪ من وزن الاسمنت, وإن هذه الرزمة سببت تأخير في وقت التجمد الابتدائي والنهائي بمقدار ٤٢: ١ و ١٨: ٣ دقيقة: ساعة على التوالي ونقصان في نسبة الماء- الاسمنت بمقدار ١٣٪ بالمقارنة مع خلطة الخرسانة المرجعية, وبذلك فأن حامض ألستريك يمكن تصنيفه حسب المواصفة الأمريكية ASTM-C494 كمضاف كيميائي مقلل للماء ومؤخر للتجمد نوع د (D).

شمل البحث أيضا تقييم تأثير استخدام حامض ألستريك على خواص الخرسانة, مقاومة الانضغاط, مقاومة الشد الانشطاري, معاير الكسر ومعامل المرونة الديناميكي في الظروف الاعتيادية وبعد التعرض لمحاليل الأملاح. أظهرت بصورة عامة النتائج بأن استخدام حامض ألستريك في الخرسانة يحسن من خواصها في الظروف الاعتيادية, حيث كانت نسبة الزيادة في مقاومة الانضغاط, مقاومة الشد الانشطاري, معاير الكسر ومعامل المرونة الديناميكي بعمر ١٨٠يوم ٥٦٪ , ١٣٪ , ٢٤٪ و ٨ ٪على التوالي مقارنة بالخرسانة المرجعية. كذلك يكون الأداء جيدا بعد التعرض لمحلول الأملاح لفترة ١٨٠ يوم بالمقارنة مع الخرسانة المرجعية.

1-Introduction

Admixtures have long been recognized as important component of concrete used to improve it's performance. ACI committee 212.3R reported that "chemical admixtures are used to enhance the properties of concrete and mortar in the plastic and hardened state"[1].

Chemical admixtures are the essential component of self compacting concrete (SCC) which is originally developed in Japan, then the use of SCC is spreading world wide because of it's

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very attractive properties in the fresh state as well as after hardening [2].

The chemicals used as admixtures can broadly be divided into two groups:

1- Surface-active chemicals: these chemicals begin to act on the cement-water system instantaneously by influencing the surface tension of water and by absorbing on the surface of cement particles.

2-Set-controlling chemicals: these chemicals break up into their ionic constituents and affect the chemical reaction between cement compounds and water, so these chemicals either retarding or accelerating the setting time of concrete [3].

Set-controlling admixtures can be divided into several categories, based on their chemical composition:

a-Lignosulfonic acids and their salts.
b-Hydroxycarboxylic acids and their salts.
c-Sugars and their derivatives.
d-Inorganic salts.

Lignosulfonic and hydroxycarboxylic acids also posses water-reducing properties and these admixtures can be classified under both groups [4].

Efforts have been made to study the effect of various carboxylic acids on the hydration of cement.[5,6], but still more researches are needed to understand the effect of using carboxylic acids on durability and properties of concrete. In this investigation many efforts have been made to study the effect of using citric acid on properties and durability of concrete.

2-Experimental Work

2-1 Materials

2-1-1 Cement

Ordinary Portland cement manufactured by Kubayisa cement factory was used throughout this investigation. It's chemical composition and physical properties are given in Table (1). The results conform to the provisions of Iraqi specification No. (5)-1984.

2-1-2 Fine Aggregate

Natural sand of maximum size 4.75mm brought from Al-Ukhaider region was used in this investigation. It's gradation lies in zone (3). The grading test results conform to Iraqi specification No. (45)-1984.

2-1-3 Coarse Aggregate

Coarse aggregate of maximum size 19mm from Al-Nibaie region was used, it conforms to the provisions of Iraqi specification No.(45)-1984.Table (2) shows the properties of fine and coarse aggregate.

2-1-4 Mixing Water

Potable water was used throughout this investigation for mixing and curing.

2-1-5 Citric Acid

Citric acid is hydroxyted carboxylic acid. At room temperature, citric acid is a white crystalline powder exists either in anhydrous form, or as amonohydrate that contains one water molecule for every molecule of citric acid. It is very soluble in water [7].

Table (3) indicates the properties of citric acid, while Figure (1) shows the Infra-Red spectrum (IR spectra) for citric acid.

2-2 Mix Proportions

The reference mix was designed according to British method BRE [8]. The mix proportion was 1: 1.62: 3.14 by weight (cement: sand: coarse aggregate) with water-cement ratio of 0.515 which was quantified to obtain workability (slump of 100±5mm). The 28-day compressive strength was about 25N/mm^2. The reduction in water-cement ratio as a result of using different dosages of citric acid while maintaining the desired workability (slump 100±5mm) was then determined.

2-3 Optimum Dosage Determination of Citric Acid

The optimum dosage of citric acid was determined as a percent of weight of cement by measuring the initial and final setting time of concrete with and without citric acid. This has been made according to ASTM-C403 using penetration strength test of cement and sand mortar taken from concrete mix with different periods.

2-4 Test Specimens

Specimens were prepared from the reference concrete mix and concrete mix with optimum dosage of citric acid. Accordingly the details of specimens were as below:

1- Compressive strength of concrete at different ages (three cubes of 100mm for each age).
2- Splitting tensile strength of concrete at different ages (three cylinders of 100×200mm for each age).
3- Modulus of rupture of concrete at different ages (three prisms of 100×100×400mm for each age).
4- Dynamic modulus of elasticity of concrete at different ages (two prisms of 100×100×400mm).
5- Water absorption of concrete (three cubes of 100mm).

2-5 Mixing

Cement, sand and coarse aggregate were mixed dry for one minute, the required dosage of citric acid was mixed with water, then water mixed with dry materials for two minutes until the mix became homogeneous.

2-6 Preparation of Specimens and Curing

The oiled moulds were filled with concrete mixture according to specifications. The compaction of the specimens was carried out by using vibrating table. After casting, the moulds were covered with polyethylene sheet for about 24 hours and then demoulded for curing continuously by water for different periods of time (3, 7 ,14 ,28 ,60 ,90 and 180 days)and then tested. Other specimens were cured continuously by water for 28 days and then they were immersed in salt solution for different periods in order to study the behaviour of concrete with and without citric acid after exposure to salts solution. The test solution consisted of three types of salts, magnesium sulfate ($MgSO_4.7H_2O$), calcium chloride ($CaCl_2$) and sodium chloride ($NaCl$) with concentrations of as that found in the underground water in the southern part of Iraq [9].

The salt solution was replenished until the time of test. Table (4) shows the concentration of the previous salts.

2-7 Measurement of Dynamic Modulus of Elasticity of Concrete

Dynamic modulus of elasticity of concrete was measured according to B.S.1881:Part209:1990 using resonant frequency method. The dynamic modulus of elasticity (E_d) in GPa was calculated by applying the following equation:

$$E_d=4n^2 L^2 \rho\times10^{-15}$$

Where:
n: resonance frequency in Hz.
L: length of specimen in mm.
ρ: density of concrete in Kg/mm^3.

3-Results and Discussions

3-1 Setting Time

First trial was made using dosage of citric acid 2% by weight of cement. It was found that the delay in the initial and final setting time relative to reference mix is not within ASTM-C494 specifications. Then many trials were made using dosages of citric acid 1%, 0.5% and 0.1% and 0.02% by weight of cement until the optimum dosage was obtained.

The initial and final setting time of reference concrete mix and concrete mix with different dosages of citric acid are shown in Table (5). It can be observed that the optimum dosage of citric acid is 0.02% by weight of cement. Table (5) shows also that the optimum dosage of citric acid cause a delay in initial and final setting time of (1:42) and (3:18) hour: minute respectively relative to reference mix. These results are conform to ASTM-C494 (2003) requirements which specified that the delay in initial and final setting time must not be less than (1) hour and not more than (3:30) hours relative to reference mix.

It is noticed that using over dosages of citric acid (0.1% and greater) retards the initial and final setting time for longer time which made it not conform to ASTM-C494 specifications. Similar behaviour was found when using other retarders [10]. This may be attributing to the fact that retarders slow down the rate of early hydration of C_3S by extending the length of dormant period (stage 2). The extension of the dormant period is proportional to the amount of retarding admixture that is used, and when the dosage exceeds a certain critical point, the C_3S hydration will never proceed beyond stage 2 and the cement paste will never set. Retarders also tend to retard the hydration of C_3A and related aluminate phases. Thus, hydroxycar-boxylic acids are used to control the handling time of regulated set cement [4].

3-2 Water Reduction

Table (6) shows the effect of different dosages of citric acid on water content of the concrete mix. The wkater-cement ratio was adjusted to obtain the same workability of reference mix. Generally, the results indicate that citric acid allows a reduction in water-cement ratio relative to the reference mix. The reduction in water-cement ratio was 13% for concrete with optimum dosage of citric acid (0.02% by weight of cement). This may be due to the mode of action of plasticizers whose molecules absorbed on the surface of the cement particles giving them a negative charge. So, they repel each other resulting in deflocculating and dispersion of cement particles. This will greatly enhancing the fluidity of the system and consequently lowering the amount of water required to attain a certain workability [10,11,12].

3-3 Effect of Citric Acid on Mechanical Properties of Concrete

Figures (2-5) and Table (7) show some of the mechanical properties at different ages for reference concrete and concrete with optimum dosage of citric acid. It can be seen that the compressive strength, splitting tensile strength, modulus of rupture (ultimate flexure strength) and dynamic modulus of elasticity increase with age for both concrete mixes. The rate of increase in strength is rapid at early ages (3 to 14 days) and up to 28 days. Thereafter, the rate is slow at 60, 90 and 180 days. This is due to the hydration process which slows down at later ages with the completion of concrete inner structure.

Generally it can be noted from the table and figures that compressive strength, splitting tensile strength, modulus of rupture and dynamic modulus of elasticity of concrete containing citric acid increase relative to reference concrete at all ages. The percentage increase in compressive strength, splitting tensile strength, modulus of rupture and dynamic modulus of elasticity at 28-day was about 60% ,32% ,20% and 3%, while at 180-day the percentage increase was about 56% ,13% ,24% and 8% respectively relative to the reference concrete. This is because of citric acid is both retarding and water reducing admixture which used to produce concrete with greater strength due to the reduction in water-cement ratio [1,12].

3-4 Effect of Citric Acid on Properties of Concrete after Exposure to Salt Solutions

3-4-1 Total Water Absorption

Absorption test on several 100mm concrete cubes was carried out according to ASTM-C642. The results of absorption of reference concrete and concrete with citric acid at different exposure ages are shown in Table (8). Generally it can be observed that the total water absorption of reference concrete and concrete with citric acid decreases up to exposure age 90 days. This reduction may be due to the partial filling of pores by the hydration products which reduce capillary porosity. Also the results show increase in total water absorption with the increase in the exposure age up to 150 days. This behaviour is as a result of the gradual disintegration of concrete which results from the internal reaction between the salts and cement hydration products. The table indicates also that water absorption of concrete containing citric acid is lower than that without citric acid. The value of decrease at exposure age of 3 ,7 ,28 ,60 ,90 and 150 days are about 41% ,31% ,36% ,28% ,28% and 38% respectively.

3-4-2 Compressive, Splitting Tensile and Flexural Strengths

Properties of reference concrete and concrete containing citric acid after exposure to compensation effect of salt solutions at different periods are shown in Figures (6-8), Table (9) and Table (10). Generally the results show that the compressive strength, splitting tensile strength and modulus of rupture of both concrete after exposure to salt solutions decrease with the time of exposure in comparison with that of specimens not exposed to salt solutions at age 28 days. This may be attributed to the fact that both chlorides and sulphates salts when present in solution can react with the hardened cement paste. The chloride react with aluminate phase of cement forming tricalcium chloroaluminat $C_3A_3CaCl_2.nH_2O$, while the sulphates react with monosulfoaluminate producing ettringite. These two products cause an increase in the solid volume of concrete which in turn is responsible for expansion and cracking of concrete leading to loss in strength and disruption [13,14].

Results show also that the compressive strength, splitting tensile strength and modulus of rupture of specimens containing citric acid after exposure to salt solutions demonstrated a slight decrease when compared with reference specimens. The decrease in compressive strength, splitting tensile strength and modulus of rupture for reference concrete after 180 days exposure to salt solutions was about 11%, 18% and 8.5%, while for specimens containing citric acid was 2.5%, 6% and 5% respectively relative to specimens not exposed to salt solutions at age 28 days. This behaviour is probably due to the water reduction caused by the action of citric acid leading to lower porosity and permeability of the concrete, therefore the penetration of the sulphate and chloride ions decreases.

4-Conclusions

From the experimental results, the following conclusions can be drawn:

1- Citric acid is found to be one of the effective retarders. The optimum dosage of citric acid is 0.02% by weight of cement. This dosage causes a reduction in water-cement ratio of 13% relative to reference mix.

2 -Over dosages of citric acid 0.1% and greater) retards the initial and final setting time for very long time.

3- The optimum dosage of citric acid causes a delay in initial and final setting time of 1:42 and 3:18 hour: minute respectively in comparison with reference mix.

4- It is possible to use citric acid as a chemical admixture and can be classified according to ASTM-C494 as water-reducing and retarding admixture (Type D).

5- The incorporation of citric acid in concrete increases the compressive strength, splitting tensile strength, modulus of rupture and dynamic modulus of elasticity at all ages. The percentage increase at age 180 days was about 56%, 13%, 24% and 8% respectively relative to reference concrete.

6- Concrete containing citric acid has good performance after 180 days exposure to salt solutions as compared with reference concrete. The decrease in compressive strength, splitting tensile strength and modulus of rupture for reference concrete (not containing citric acid) was about 11%, 18% and 8.5%, while for concrete containing citric acid was about 2.5%, 6% and 5% respectively relative to concrete specimen not exposed to salt solution at age 28 days.

5- References

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Table (1) Cement Characteristics

Chemical Analysis		Compound		Physical properties
Oxide	**% by weight**	**Compositions (%)**		
CaO	62.60	C_3S	51.05	Fineness (Blaine) 366 m^2/Kg (Iraqi standard ≥ 230 m2/Kg)
SiO_2	20.40			
Al_2O_3	5.94	C_2S	20.05	Initial setting time 105 minutes (Iraqi standard ≥ 45 minutes)
Fe_2O_3	2.76			
MgO	3.33	C_3A	11.08	Final setting time 231 minutes (Iraqi standard ≤ 10 hours)
SO_3	1.69			
Na_2O K_2O	0.80	C_4AF	8.39	Compressive strength (N/mm^2) (Iraqi standard ≥ 15) 3days 22.3 (Iraqi standard ≤ 23) 7days 28.4
L.O.I	1.90			
Insoluble Residue	0.50			
Specification requirements according to Iraqi standard				
MgO≤5.0 SO_3≤ 2.8				Loss of ignition≤ 4.0 Insoluble residue ≤1.5

Table (2) Aggregate Properties

Sieve Size (mm)	Gradation (% Passing)			
	Gravel		Sand	
	Typical Sample	Limit of Iraqi Specification	Typical Sample	Limit of Iraqi Specification
37.50	100	100		
20.00	97.30	95-100		
14.00	-----	-----		
10.00	43.50	30-60		
4.75	5.70	0-10	100	90-100
2.36			92.9	85-100
1.18			82.6	75-100
0.600			62.4	60-79
0.300			19.2	12-40
0.150			3.0	0-10

SO_3 content in sand = 0.07% (Specification requirement 0.5% Max.)
SO_3 content in gravel = 0.08% (Specification requirement 0.1% Max.)
Clay content in sand = 0.1% (Specification requirement 1% Max.)
Absorption of sand = 2%
Absorption of gravel = 1.4%
Specific gravity of sand = 2.65
Specific gravity of gravel = 2.68

Table (3) Properties of citric acid*

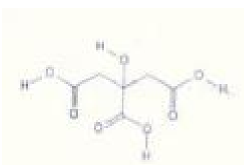
<table>
<tr><td>1- Physical properties:
a-Physical state: solid
-Color: white
-Odor: acidity
d-Ignition: aliphatic saturated</td><td>7-Acidity:
Pka₁ = 3.15   Pka₂ = 4.77   Pka₃ = 6.4</td></tr>
<tr><td>2- Physical Constant
a- Melting point: 150 C°</td><td>8- Solubility in:
H2O: +ve; NaOH: +ve; NaHCO3: +ve; HCL (dil): -ve; H2SO4: -ve</td></tr>
<tr><td>3- Molar mass: 192 g/mol</td><td>9- Sodium fusion test:
N: -ve; S: -ve; I: -ve; Br: -ve; Cl: -ve; P: -ve</td></tr>
<tr><td>4- Density: 1663 kg/ m3</td><td>6- Primary classification tests:
Reagent | Result | Inference
NaHCO3 | +ve | There is carboxylic acid
H2SO4 / heat | +ve | There is carboxylic acid</td></tr>
<tr><td>5- Chemical formula: C6H8O7
6- Structural formula
</td><td>The functional group indicated by above test is carboxylic acid group (COOH)</td></tr>
</table>

Table (4) Salt solution compositions [9]

Salt type	Concentrations	
	gm/l	Ppm
$MgSO_4.7H_2O$	17.9	17900
$CaCl_2$	5.5	5513
NaCl	45.1	45100

Table (5) Effect of citric acid (C.A.) on the initial and final setting time of concrete mix

Sample No.	Dosage of C.A. (%by weight of cement)	Setting time(hrs)		Delay in setting time relative to reference mix (hrs)	
		Initial	Final	Initial	Final
1	0.00	8:18	13:30	-	-
2	0.02	10:00	16:12	1:42	3:18
3	0.10	20:00	No setting	11:42	-
4	0.50	27:00	No setting	18:42	-
5	1.00	35:18	No setting	27:00	-
6	2.00	48:00	No setting	39:42	-

Table (6) Reduction in water- cement ratio for concrete mixes with different dosages of citric acid (C.A.)

Sample No.	Dosage of C.A. (%by weight of cement)	Water-cement ratio	Slump(mm)	Reduction in water-cement ratio (%)
1	0.00	0.515	102	0
2	0.02	0.448	100	13
3	0.10	0.424	105	17.67
4	0.50	0.422	95	18.06
5	1.00	0.442	105	14.17
6	2.00	0.455	100	11.65

Table (7) Effect of citric acid on mechanical properties of concrete

Age (days)	Compressive strength (N/mm^2)			Splitting tensile strength (N/mm^2)			Modulus of rupture (N/mm2)			Dynamic modulus (Gpa)		
	Reference concrete	Concrete with C.A.	% Increase	Reference concrete	Concrete with C.A.	% Increase	Reference concrete	Concrete with C.A.	% Increase	Reference concrete	Concrete with C.A.	% Increase
3	1.26	10.77	754.8	0.17	1.08	535.3	0.28	2.31	725.0	41.46	44.31	6.9
7	5.22	17.00	225.7	0.58	1.62	179.3	1.05	2.89	175.2	42.50	45.71	7.6
14	13.40	22.55	68.3	1.42	2.22	56.3	2.59	3.54	36.7	43.37	46.38	6.9
28	27.28	43.56	59.7	2.26	2.98	31.9	4.68	5.61	19.9	45.83	47.36	3.3
60	29.79	47.36	59.0	3.10	3.91	26.1	4.82	5.87	21.8	46.99	49.73	5.8
90	31.16	49.30	58.2	3.70	4.20	13.5	4.95	6.12	23.6	47.72	50.23	5.3
180	32.09	50.10	56.1	3.84	4.35	13.3	5.10	6.32	23.9	47.99	51.73	7.8

Table (8) Total water absorption of concrete after exposure to salt solution

Concrete type	Total water absorption after exposure to salt solution (%)					
	Age (days)					
	3	7	28	60	90	150
Reference concrete	1.91	0.96	0.81	0.43	0.36	0.48
Concrete with C.A.	1.12	0.66	0.52	0.31	0.26	0.30
% Decrease	41.4	31.3	36.0	28.0	28.0	37.5

Table (9) Mechanical properties of concrete after exposure to salt solution

Exposure age (days)	Compressive strength (N/mm^2)			Splitting tensile strength (N/mm^2)			Modulus of rupture (N/mm^2)		
	Reference concrete	Concrete with C.A.	% Increase	Reference concrete	Concrete with C.A.	% Increase	Reference concrete	Concrete with C.A.	% Increase
0*	27.28	43.56	59.7	2.26	2.98	31.9	4.68	5.61	19.9
7	27.15	43.50	60.2	2.24	2.97	32.6	4.65	5.60	20.4
28	26.60	43.00	61.7	2.17	2.90	33.6	4.58	5.55	21.2
60	26.35	42.70	62.0	2.12	2.86	34.9	4.52	5.50	21.7
90	25.00	42.53	70.1	2.00	2.82	41.0	4.44	5.42	22.1
180	24.28	42.47	74.9	1.85	2.79	50.8	4.28	5.33	24.5

* Concrete properties at age 28 days (before exposure to salt solution).

Table (10) The percentage decrease in mechanical properties of concrete after exposure to salt solution in comparison with that at 28days (before exposure)

Exposure age (days)	Decrease in compressive strength (%)		Decrease in Splitting tensile strength (%)		Decrease in Modulus of rupture (%)	
	Reference concrete	Concrete with C.A.	Reference concrete	Concrete with C.A.	Reference concrete	Concrete with C.A.
7	0.50	0.10	0.90	0.34	0.64	0.18
28	2.50	1.30	4.00	2.70	2.10	1.10
60	3.40	2.00	6.20	4.00	3.40	2.00
90	8.40	2.40	11.50	5.40	5.10	3.40
180	11.00	2.50	18.20	6.40	8.60	5.00

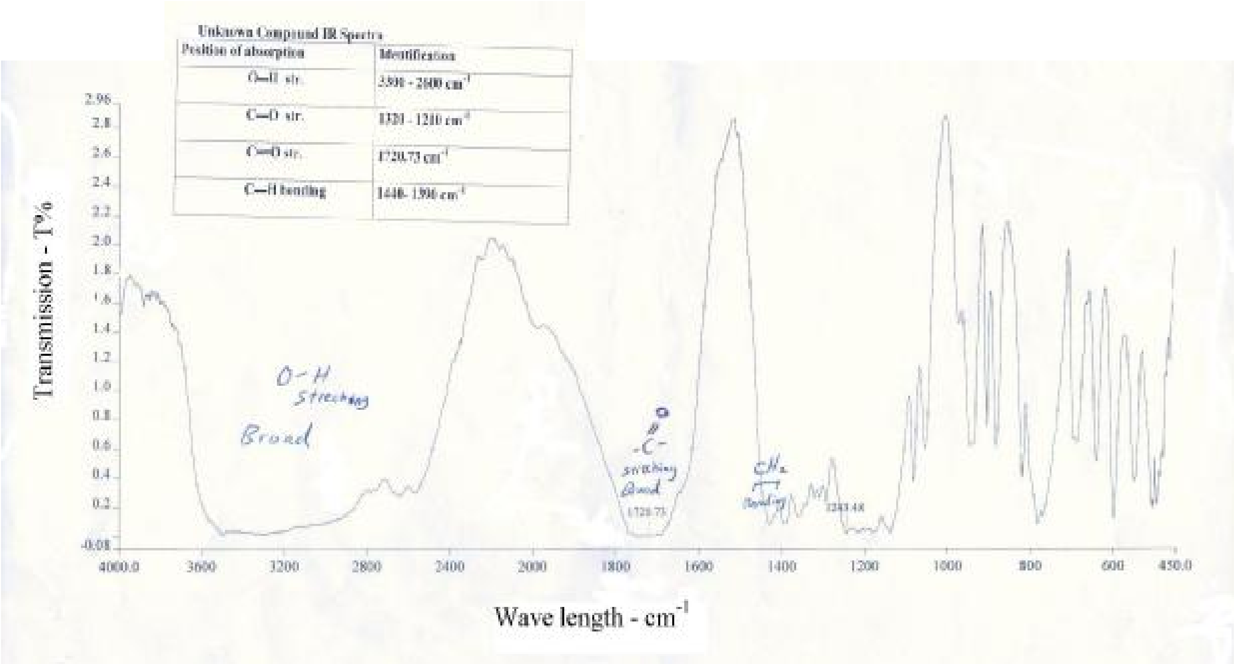


Fig. (1) Infra-red spectrum for citric acid

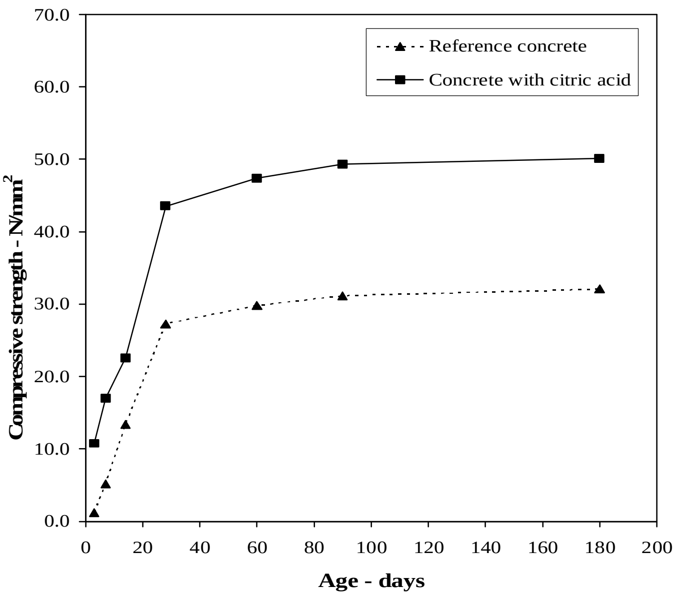


Fig.(2) Compressive strength of reference concrete and concrete with citric acid

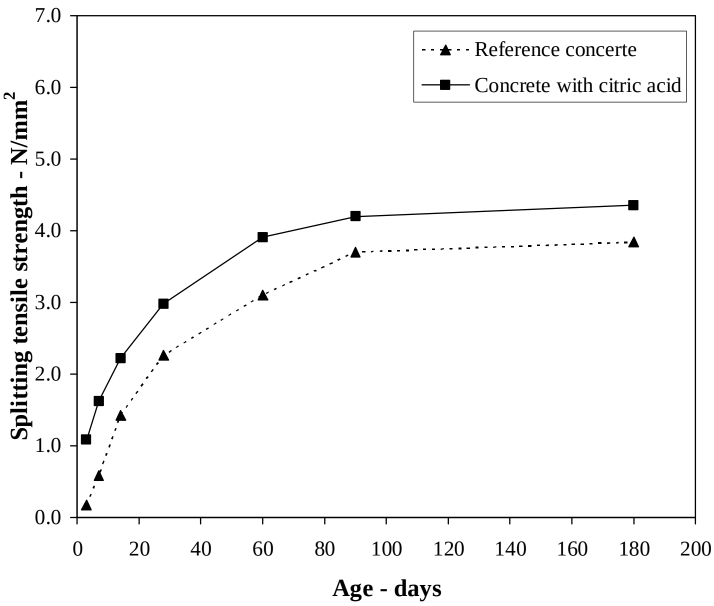


Fig. (3) Splitting tensile strength of reference and concrete with citric acid

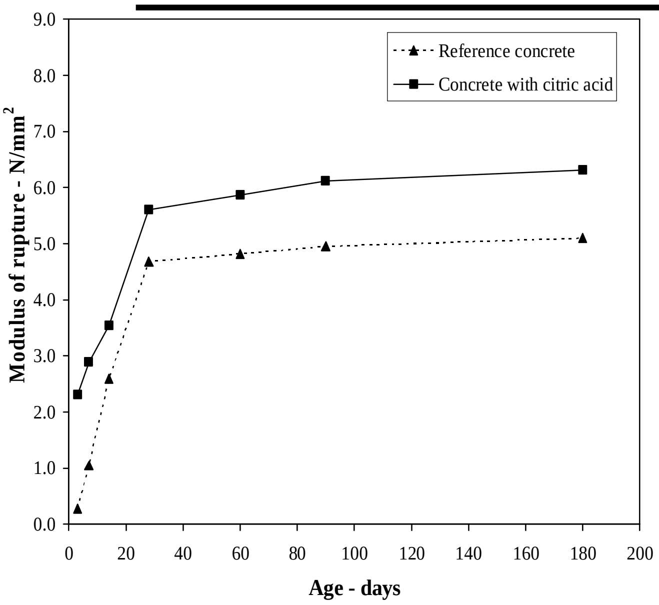


Fig.(4) Modulus of rupture of reference concrete and concrete with citric acid

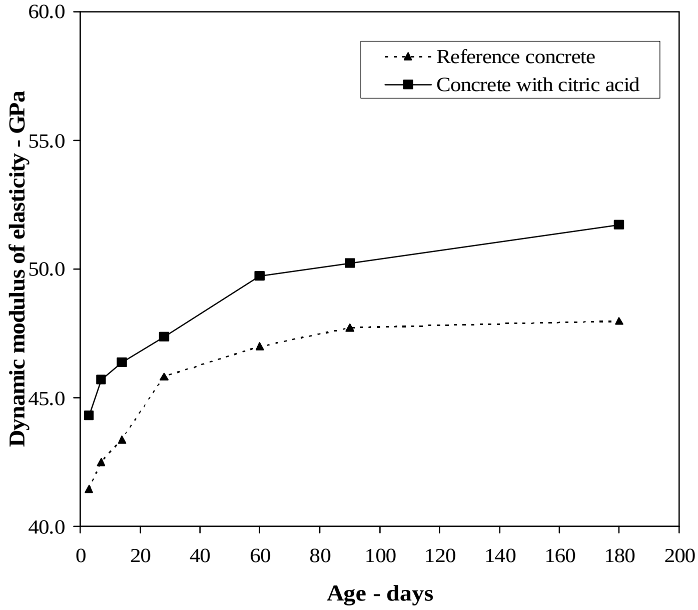


Fig.(5) Dynamic modulus of elasticity of reference concrete and concrete with citric acid

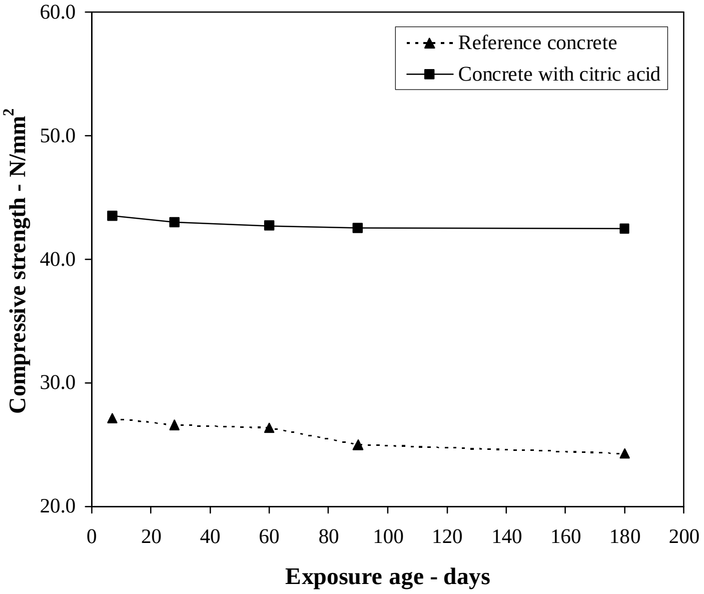


Fig.(6) Compressive strength of reference concrete and concrete with citric acid after exposure to salt solutions

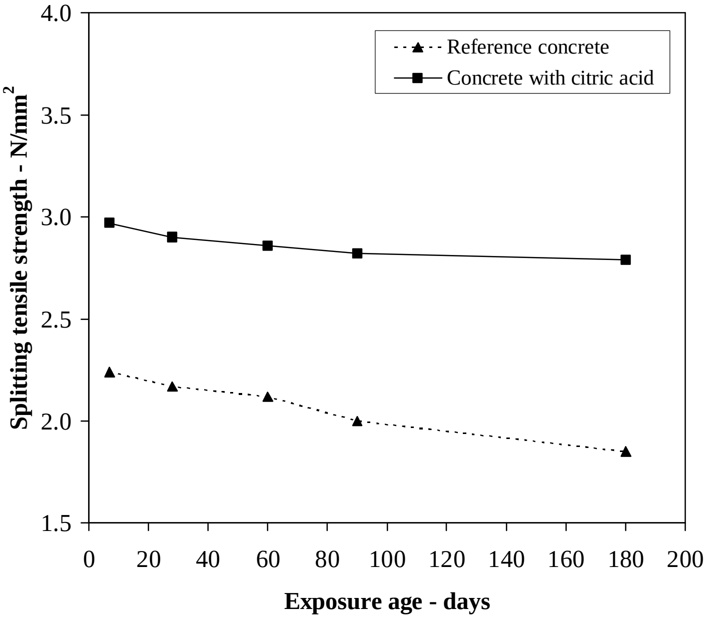


Fig.(7) Splitting tensile strength of reference concrete and concrete with citric acid after exposure to salt solutions

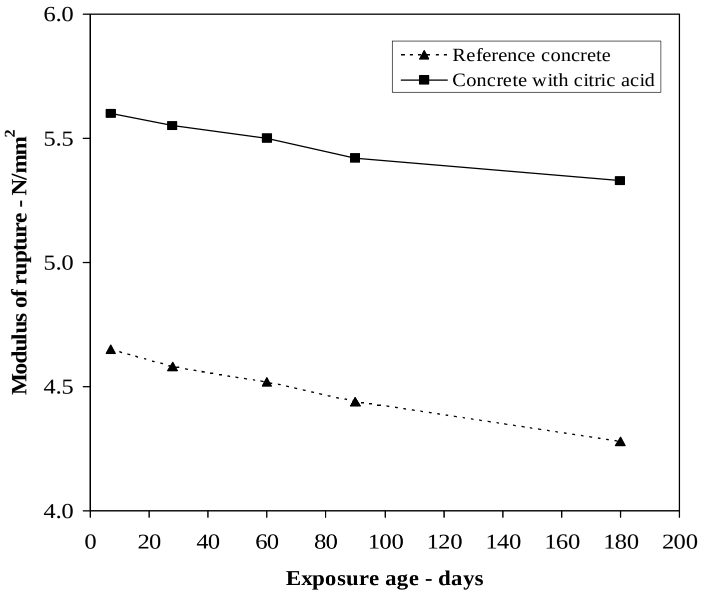


Fig.(8) Modulus of rupture of reference concrete and concrete with citric acid after exposure to salt solution