*Gazi University Journal of Science*
*GU J Sci*
*24(2):299-307(2011)*

*www.gujs.org*

# The Effect Of Antifreeze Admixtures On Compressive Strength Of Concretes Subjected To Frost Action

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*Received: 28.07.2010 Accepted: 28.10.2010*

**ABSTRACT**

This study aims to determine the effect of antifreeze admixture used in the concretes subjected to frost action on the concrete compressive strength. Three groups of concrete samples of C30 class were prepared with antifreeze admixtures by using the mixture of 30% calcium nitrate and 5% hydroxy ethoxy amin (A), calcium nitrate (B), Polyhydroxy amine (C). Concrete samples are put in the freezer in a fresh state 15 min after the mixing. Samples are subjected to frost action in 0, -5, -10, -15 and -20°C degrees for 48 hours. On the concrete samples water cured after 48 hours, compressive strength tests of 28 day are conducted. Consequently, it is seen that with the increase of the set temperature that concrete is cured, compressive strength of concrete is decreased and among all types of antifreezes the maximum decrease is seen between 0 and -5°C while the decrease in compressive strength is relatively less for -10, -15 and -20°C.

***Keywords:*** *Antifreeze, concrete production in cold weather, compressive strength.*

## 1. INTRODUCTION

The fundamental operations such as preparation, mixing, transportation, pouring, consolidation of concrete and curing of concrete besides material properties of aggregates, cement, water and admixtures and concrete mixture ratios are the most significant criteria that affect the concrete strength [1, 2].

The hydration of Portland cement contains the reaction of calcium silicate and aluminate phases to produce hydrated products [3]. The high temperature during the cement hydration increases the hydration time. Similarly, the opposite situation, i.e. slow hydration temperature also increases the hydration time. American Concrete Institute (ACI), based on a study, stated that under the condition of the air temperature lower than 5 °C more than 3 days and less than 10°C for more than half of the 24hr period, concrete set process slows down and without some thermo protection measures concrete does not harden[5,6]. Some protective measures are necessary during concrete hydration and these can be summarized as the increase of concrete inner temperature to a degree above 5°C by isolation materials or some other methods [7].

Companies do stay very distant to the protection of concrete with isolation materials in cold weathers depending on the structure type since the cost of application is very high. On the other hand, studies on the use chemical admixtures to prevent the freeze of the water inside the concrete have been continuing [8]. Chemical admixtures are defined as the materials added to the concrete during the mixing process in relatively small amount compared with the mass of cement in order to change some properties of fresh or hardened concrete [9]. Chemical admixtures, in liquid or solid state added to mortar or fresh concrete, which increase the initial hydration temperature and protect the concrete or mortar

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from frost action by decreasing the freezing temperature of water, are called concrete antifreezes [10].

There are various ways to protect concrete from cold weather. The protection by warming the concrete during casting or during setting by structural element heater, by covering the structure and by applying vapor cure to accelerate the hardening are among the common applications. However, these applications are expensive and the use of antifreeze to retard the freezing can be 1.2-1.5 times more economical than the above-mentioned ways of protection. Antifreeze admixtures lower the freezing point of water under $0^oC$ and accelerate the hydration of cement however, the long term effects of antifreeze admixtures on concrete are not known [7].

In the literature, it has been found that voids in aggregates, cement paste and interfaces of aggregates are effective in the freeze of concrete. Concrete's affection from the frost action depends on the amount of voids that water can penetrate in the materials composing concrete [12-14]. In cold weather, water inside the voids in concrete freezes and the volume increases approximately 9%. Considering the fact that 91% of void volume of concrete is filled with water, great hydraulic pressure is created by the dilatation of water thus resulting in the cracking of hardened cement paste around aggregates [12, 15].

In the study of Topçu et al, the effect of antifreeze on hardened concrete was investigated. They found that in different groups of concretes with 0,17% antifreeze and without antifreeze produced by the use of NA3k chemical admixture with 0,5 % ratio, the positive effects of the use of antifreeze in concrete production in cold weathers on compressive strength are prominent when compared with the strength values without any antifreeze admixture. With the use of antifreeze there observed 3MPa (16%) increase in the compressive strength of 7-day [16].

Yıldırım et al studied the effect of antifreeze admixtures in cold weather conditions on the compressive strength of concrete experimentally. The extent of the effect of antifreeze admixtures on different types of cements (CEM I 42.5, CEM II/A-S and CEM II/B-S) used for concrete production was investigated. The study revealed that the use of concrete antifreeze had positive effects on the strength of concrete. In the study the samples were subjected to frost action between -4 and -7 for a day. They drew the conclusion that the applicability of the antifreeze is to be tested for lower temperatures [17].

The aim of the study is to investigate the effect of three different types of antifreeze admixture (30% calcium nitrate+5% hydroxy ethoxy amine mixtures, calcium nitrate, polyhydroxy amine) added in four different ratios (0%, 1% 1,5% and 2%) on the compressive strength of concrete under five different cold weather conditions (0 °C, -5°C, -10°C, -15°C and -20°C).

## 2. MATERIALS AND METHODS

### 2.1. Materials

In the study, there used the aggregates obtained from Kırşehir Obruk quarry with specific bulk density of 2.69, 2.71 and 2.74 $g/cm^3$ and grain size of 0-4, 4-11.2, 11.2-22.4 mm. In the mixtures CEM I 42.5 R, a product of Set Ankara cement factory, high amount of water reducer, super plasticizer that result in early high strength and water from city network were used. The physical, chemical and mechanical properties of the cement used in concrete productions are given in Table 1.

Table 1. The properties of CEM I 42.5R chemical, physical and mechanical

| Component | % | Physical properties | | |
|---|---|---|---|---|
| $SiO_2$ (%) | 20,32 | Setting time, Inıtial (min) | | 118 |
| $Al_2O_3$ (%) | 5,59 | Setting time, Final (min) | | 177 |
| $Fe_2O_3$ (%) | 3,09 | Hacim sabitliği (mm ) | | 2 |
| CaO (%) | 62,50 | Specific surface (blaine), ($cm^2/g$) | | 3172 |
| MgO (%) | 1,74 | Specific gravity ($g/cm^3$) | | 3,09 |
| $SO_3$ (%) | 3,29 | | | |
| $Na_2O$ (%) | 0,34 | Mechanical properties | | Comp. strength (MPa) |
| $K_2O$ (%) | 0,91 | | 2. days | 30,8 |
| LOI, (%) | 1,18 | | 7. days | 39,5 |
| Insoluble residue, (%) | 0,31 | | 28.days | 56,0 |

Furthermore, in the mixture there used three different types of antifreeze as 30% calcium nitrate + 5% hydroxy ethoxy amine mixture (A), calcium nitrate (B) and polyhydroxy amine (C). Some properties regarding the antifreezes are given in Table 2.

Table 2. The antifreeze type and properties

| Additive no | Additive code | Chemical content | Properties | |
|---|---|---|---|---|
| 1 | A | 30% calcium nitrate+ 5% hydroxy ethoxy amine mixture | Density | : 1.25 ± 0.03 kg/L |
| | | | pH | : 6,00 – 6,50 |
| | | | Chloride | : ≤ %0,1 (TS EN 480–10), [18] |
| | | | Alkali content | : ≤ %10 (TS EN 480–12), [19] |
| 2 | B | Calcium nitrate | Density | : 1.25 ± 0.03 kg/L |
| | | | pH | : 6,00 – 8,00 |
| | | | Chloride | : ≤ %0,1 (TS EN 480–10) , [18] |
| | | | Alkali content | : ≤ %10 (TS EN 480–12) , [19] |
| 3 | C | Polyhydroxy amine | Density | : 1.25 ± 0.03 kg/L |
| | | | pH | : 6,00 – 6,50 |
| | | | Chloride | :≤ %0,1 (TS EN 480–10) , [18] |
| | | | Alkali content | : ≤ %10 (TS EN 480–12) , [19] |

**2.2. Methods**

The study was conducted in two stages. In the first one after mixing concrete samples casted into 100x200 mm formworks were put into freezer within 15 minutes. In 2-4 hours the temperature inside the freezers lowered to the temperature that the samples will be subjected to. The samples were kept in the freezers with 0°C, -5°C, -10°C, -15°C, -20°C for 2 days. In the second stage, they were cured in the water with room temperature for 26 days.

***2.2.1. Test of aggregate***

Grain size distribution of aggregates complies with TS 3530 EN 933-1/A1 [20] while specific bulk density with TS 3529 [21] and specific gravity and water absorption test with TS EN 1097–6 [22].

***2.2.2. Preparation of concrete mixture***

The mixture of concrete was prepared according to TS 802 standard [23]. Concrete class of the samples is C30, water/cement ratio (w/c) is 0.52 and super plasticizers (SP) are 1% of cement weight. SP admixture was used by complying ASTM – C 494 Type F [24] and TS EN 934-2 [25] standards while antifreeze admixtures by complying TS 11746 [26]. Material amounts in $m^3$ are given in Table3.

Table 3. The mixture ratios of concrete samples

| Materials | Type | Specific gravity ($gr/cm^3$) | Weight, (Kg) | Volume, ($m^3$) | Aggregates ratio, (%) |
|---|---|---|---|---|---|
| Crushed sand | 0-5 | 2,69 | 867 | 0,322 | 46,0% |
| Crushed sand-1 | 5-12 | 2,71 | 380 | 0,141 | 20,0% |
| Crushed sand-2 | 12-22 | 2,74 | 650 | 0,237 | 34,0% |
| Total Aggregates (kg) | | | 1897 | 0,700 | 100,0 |
| Cement | OPC 42,5 | 3,09 | 330 | 0,107 | |
| Chemical admixture (1%) | SP | 1,19 | 3,3 | 0,003 | |
| Air | | | | 0,02 | |
| Water | Network water | 1 | 170 | 0,170 | |
| Total materials (kg) | | | 2400,3 | 1,000 | |

***2.2.3. Experiments of fresh concrete***

The specific bulk density test, slump test, ve-be test and air content tests of freshly mixed concrete samples are conducted by complying with TS 2941 [27], TS EN 12350-2 [28], TS EN 12350-3 [29] and TS EN 12350-7 [30], respectively.

***2.2.4. Experiments of hardened concrete***

Twelve different types of concrete were produced by using three different antifreeze admixtures with different amounts (0,5% - 1% - 1,5% - %2). By preparing three samples from each group and using five different temperatures (0, -5, -10, -15 and -20°C) to simulate the frost action for 48 hours in total 180 samples were prepared for this study. Concrete samples complied with TS EN 12390–2 standard [31].

After kept in different temperatures for 48 hours, concrete samples were cured in a laboratory environment with 20±2 °C and 50-60% relative humidity in a curing pool for 26 days.

***2.2.5. Statistical evaluation***

The results obtained from the samples with three antifreeze types (A,B and C), four different admixture ratios (0,5% , 1% ,1,5% ,2%) and five different set temperature (0, -5, -10, -15 and -20°C) were evaluated by three factor variance analysis technique with repeated measures. In the variance analysis technique to identify the differences among antifreeze type, admixture ratios and set temperatures Duncan multiple range test was used. Furthermore, nonlinear regression analyses were conducted to determine the changes in compressive strength by admixture ratio and set temperature. Nonlinear mathematical model equation is given in Eq.1.

$$Y = P_1(U,V) / P_2(U,V) \quad (1)$$

$$P_1(U,V) = B_0+B_1U+B_2U^2+B_3V+B_4UV+B_5V^2$$
$$P_2(U,V) = 1+B_6U+B_7U^2+B_8V+B_9UV+B_{10}V^2$$

Here;
Y = The estimated values of compressive strength,
U = The value % of admixture amount
V = The value of setting temperatures,
$B_n$= Equation coefficients.

## 3. RESULTS AND DISCUSSION

### 3.1. Results of Aggregates

According to sieve analysis values, $A_{22.4}$, $B_{22.4}$, $C_{22.4}$ curves and granulometric curves depending on the maximum grain size are shown in Figure 1. The granulometry curve of the aggregate used in the mixture is seen between $A_{22\ 4}$ and $C_{22.}$ curves. The physical properties of crushed aggregate and limit values are given in Table 4.

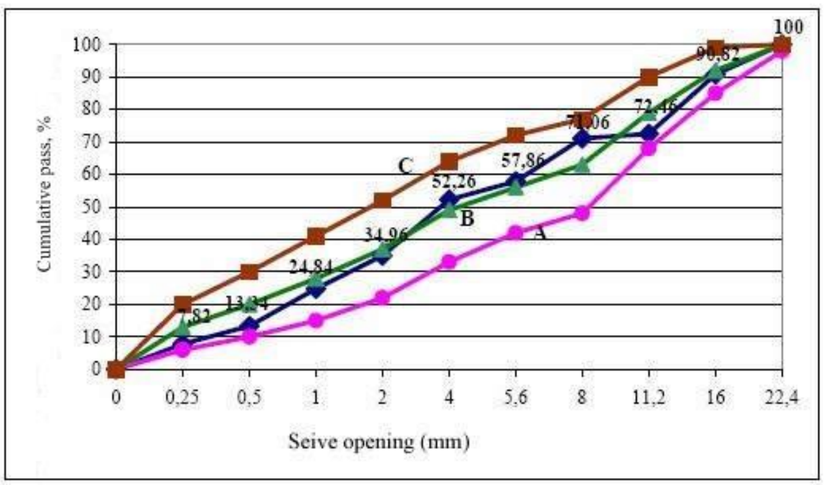


Figure 1. Aggregate granulmetric boundary curves depending on the maximum grain

Table 4. The properties of coarse aggregate

| Aggregate tests | Aggregate type | | | Limits values |
|---|---|---|---|---|
| | 0-4. | 4-11.2 | 11.2-22.4 | |
| Loose unit weight, (g/cm$^3$) | 4,14 | 4,325 | 4,10 | -- |
| Dense unit weight, (g/cm$^3$) | 5,16 | 4,80 | 4,75 | -- |
| Unit weight, (kg/dm$^3$) | 2,61 | 2,71 | 2,74 | 2,5-2,8 [33] |
| Water Absorption, (%) | 1,0 | 0,42 | 0,37 | 1,00 [33] |

As seen in Table 4, bulk density, unit weight and water absorption values show compliance with the literature.

### 3.2. Results of Fresh Concrete

The results obtained from fresh concrete tests are given in Table 5. The consistency of the freshly mixed concrete was determined as plastic.

Table 5. The properties of fresh concrete

| Antifreeze Type | Antifreeze Ratio, (%) | Unit weight, (kg/dm$^3$) | Slump, (mm) | Ve-Be, (sn) | Air content, (%) |
|---|---|---|---|---|---|
| A | 0,5 | 2,384 | 80 | 10 | 1,9 |
| | 1 | 2,390 | 95 | 84 | 2,1 |
| | 1,5 | 2,395 | 100 | 8 | 2,3 |
| | 2 | 2,399 | 115 | 3 | 2,4 |
| B | 0,5 | 2,383 | 70 | 12 | 2,1 |
| | 1 | 2,384 | 80 | 10 | 2,2 |
| | 1,5 | 2,386 | 95 | 5 | 2,4 |
| | 2 | 2,391 | 100 | 4 | 2,6 |
| C | 0,5 | 2,384 | 80 | 11 | 2 |
| | 1 | 2,389 | 90 | 10 | 2,2 |
| | 1,5 | 2,394 | 105 | 5 | 2,3 |
| | 2 | 2,397 | 110 | 3 | 2,4 |

The unit weight of concretes that contain normal aggregates ranges between 2, 2-2,4 kg/dm$^3$ [34, 35]. As seen in Table 5, the unit weights of fresh concretes had a range of 2,383- 2,399 kg/m$^3$. With the increase in the ratio of antifreeze in the mixture, slump values and air content of the concrete increases while ve-be values decrease. This can be explained by the effect of the type of antifreezes.

### 3.3. Results of hardened Concrete

#### *3.3.1. Compressive strength*

Explanatory statistics regarding the compressive strength of C30 concrete samples on 28th day are given in Table 6.

Table 6. The descriptive statistics of compressive strength values

| Temperature, (°C) | Antifreeze ratio (%) | N | Antifreeze type A | | B | | C | |
|---|---|---|---|---|---|---|---|---|
| | | | Mean (MPa) | Std. Deviation | Ort, (MPa) | Std. Deviation | Ort, (MPa) | Std. Deviation |
| 0 | 0.5 | 3 | 19,67 | 0,92 | 16,12 | 0,20 | 18,93 | 0,46 |
| | 1 | 3 | 28,42 | 0,93 | 23,24 | 0,40 | 25,53 | 0,15 |
| | 1.5 | 3 | 22,2 | 0,38 | 19,14 | 0,39 | 22,07 | 0,30 |
| | 2 | 3 | 20,16 | 0,20 | 18,89 | 0,23 | 21,83 | 0,15 |
| -5 | 0.5 | 3 | 17,53 | 0,23 | 15,09 | 0,25 | 16,71 | 0,14 |
| | 1 | 3 | 18,64 | 0,75 | 16,23 | 0,12 | 18,13 | 0,12 |
| | 1.5 | 3 | 18,46 | 0,22 | 15,85 | 0,32 | 17,08 | 0,50 |
| | 2 | 3 | 18,01 | 0,30 | 15,63 | 0,43 | 16,87 | 0,12 |
| -10 | 0.5 | 3 | 17,29 | 0,17 | 14,95 | 0,14 | 16,23 | 0,13 |
| | 1 | 3 | 17,64 | 0,19 | 15,39 | 0,07 | 16,81 | 0,19 |
| | 1.5 | 3 | 17,56 | 0,15 | 15,42 | 0,14 | 16,73 | 0,21 |
| | 2 | 3 | 17,52 | 0,30 | 15,52 | 0,26 | 16,55 | 0,18 |
| -15 | 0.5 | 3 | 17,23 | 0,19 | 14,82 | 0,12 | 15,93 | 0,09 |
| | 1 | 3 | 17,59 | 0,19 | 14,94 | 0,14 | 16,42 | 0,22 |
| | 1.5 | 3 | 17,45 | 0,28 | 15,24 | 0,14 | 16,09 | 0,49 |
| | 2 | 3 | 17,22 | 0,05 | 15,47 | 0,22 | 16,1 | 0,06 |
| -20 | 0.5 | 3 | 17 | 0,22 | 14,75 | 0,55 | 15,06 | 0,10 |
| | 1 | 3 | 17,27 | 0,83 | 14,8 | 0,39 | 15,98 | 0,19 |
| | 1.5 | 3 | 17,13 | 0,15 | 15,04 | 0,33 | 15,49 | 0,20 |
| | 2 | 3 | 17,1 | 0,16 | 15,25 | 0,13 | 15,51 | 0,12 |

Average compressive strength values regarding each antifreeze type and temperature are provided in Figure 2 (A, B, C).

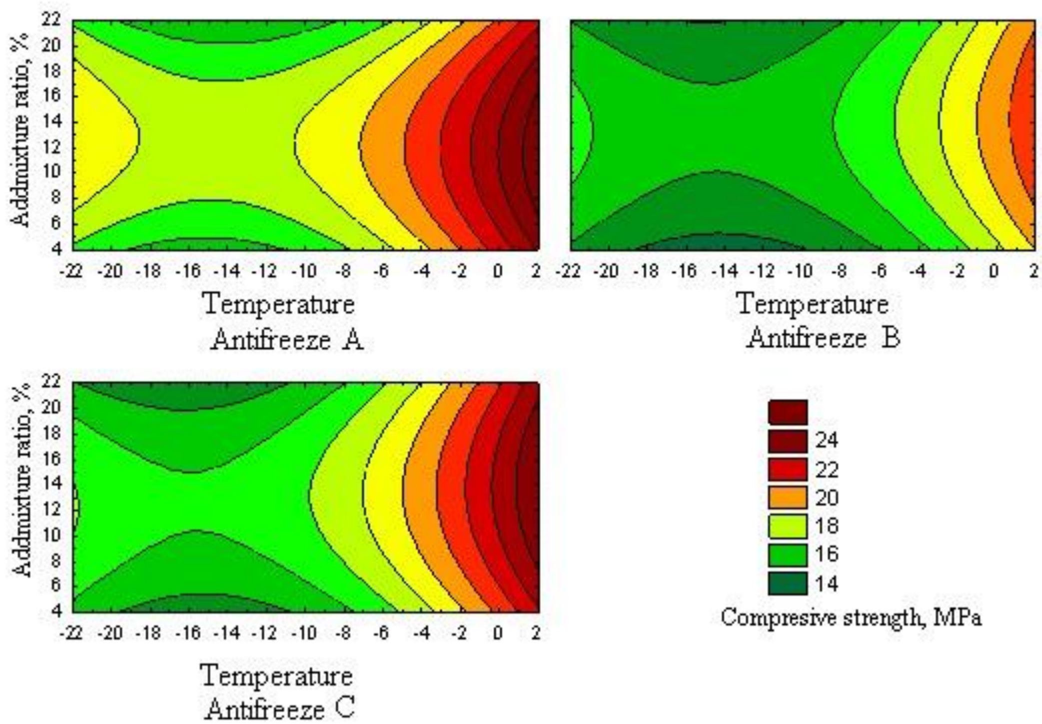


Figure 2. The value of compressive strength of additive A, B and C in concrete samples

In Figure 2, y axis is the admixture ratio while x-axis is the temperature of initial setting. For three types of antifreezes a color can be obtained by intersecting x and y-axes and from the scale on the right side of the figure color based compressive strength value can be found. The maximum compressive strength values are seen for all different admixture types in the samples that were subjected to $0^{o}C$ (Fig.2). Besides, it is observed that with the decrease in temperature the compressive strength values decreased although until an optimum point the strength increased with increasing antifreeze ratio. However, it is seen that in high antifreeze ratio strength values started to decrease.

With the strength values obtained, three factor variance analyses were conducted regarding antifreeze type, admixture ratio and temperature. The analysis revealed that antifreeze, temperature, admixture ratio, antifreeze-temperature and temperature-admixture ratio interactions are statistically significant ($\alpha<0.05$). In other words, it is seen that the compressive strength values change both with temperature and admixture ratio and these changes are significant. Furthermore, modal equations obtained through nonlinear regression analysis are given in Table 7.

Table 7. Non-linear regression models

| Test | Regression coeff., (r2) | Equation | | Model equation |
|---|---|---|---|---|
| | | Antifreeze | Degree | |
| Compressive strength | 0,966 | A | 2 | $Y=((17,58)-(0,242)*KM-(0,108)*KM^2-(3,096)*PS-(0,108)*KM*PS+(0,167)*PS^2) / (1-(0,0195)*KM-(0,0066)*KM^2-(0,1836)*PS-(0,0067)*KM*PS+(0,00953))*PS^2$ |
| | 0,999 | B | 3 | $Y=((15,2)+(1,5)*KM-(0,11)*KM^2-(0,005)*KM^3-(3,78)*PS-(0,275)*KM*PS+(0,001)*KM^2*PS+(0,323)*PS^2+(0,00785)*KM*PS^2-(0,00835)*PS^3)/(1+(0,1)*KM-(0,008)*KM^2-(0,00035)*KM^3-(0,25)*PS-(0,02)*KM*PS+(0,00007)*KM^2*PS+(0,02)*PS^2+(0,0005)*KM*PS^2-(0,00053)*PS^3)$ |
| | 0,986 | C | 3 | $Y=((16,89)-(1,234)*KM-(0,067)*KM^2-(0,000064)*KM^3-(3,212)*PS+(0,115)*KM*PS+(0,0062)*KM^2*PS+(0,218)*PS^2+(0,0012)*KM*PS^2-(0,005)*PS^3) / (1-(0,087)*KM-(0,0047)*KM^2-(0,00002)*KM^3-(0,203)*PS+(0,0074)*KM*PS+(0,000376)*KM^2*PS+(0,014)*PS^2+(0,00007)*KM*PS^2-(0,00032)*PS^3)$ |

Y: The value compressive strength, KM: Ratio of admixture, PS: Setting temperature

The regression analysis shows that in all types of antifreeze there is a strong relationship between admixture ratio/ temperature and compressive strength.

## 4. RESULTS

For cold weather concrete casting there prepared samples with admixtures containing 30% Calcium nitrate and 5% hydroxy ethoxy amine mixture (A), Calcium nitrate (B), Polyhydroxy amine (C). After 2 days of frost action at 0, -5, -10, -15 and -20°C, the evaluation of the data obtained from compressive strength tests of 28 day revealed that

- For all temperatures and admixture ratios compressive strength of 28 day of the samples did not reach 30 MPa,
- The concrete samples subjected to $0^oC$ had better strength values in all admixture ratios.
- For the antifreeze type A, the maximum compressive strength, 28,42 MPa, was observed at $0^oC$ and at the concrete samples that had 1% antifreeze admixture.
- For the samples with antifreeze type A, there observed 40,18% decrease in compressive strength between the samples of $0^oC$ and $-20^oC$. 85,64% of this decrease occurred between $0^oC$ and $-5^oC$ while 14,36% of it was seen between $-5^oC$ and $-20^oC$.
- For the antifreeze type B, the maximum compressive strength, 23,24 MPa, was observed at $0^oC$ and at the concrete samples that had 1% antifreeze admixture.
- For the samples with antifreeze type B, there observed 36,53% decrease in compressive strength between the samples of $0^oC$ and $-20^oC$. 88,46% of this decrease occurred between $0^oC$ and $-5^oC$ while 11,54% of it was seen between $-5^oC$ and $-20^oC$.
- For the antifreeze type C, the maximum compressive strength, 25,53 MPa, was observed at $0^oC$ and at the concrete samples that had 1% antifreeze admixture.
- For the samples with antifreeze type C, there observed 41,01% decrease in compressive strength between the samples of $0^oC$ and $-20^oC$. 70,68% of this decrease occurred between $0^oC$ and $-5^oC$ while 29,32% of it was seen between $-5^oC$ and $-20^oC$.
- In the samples containing antifreeze type A for all temperatures, the samples with 1% admixture ratio gave the maximum compressive strength,
- In the samples containing antifreeze type B, for 0°C and -5°C the samples with 1% admixture ratio, for -10°C, -15°C and -20°C the ones with 2% admixture ratio gave the maximum compressive strength,
- In the samples containing antifreeze type C, for all temperatures, the samples with 1% admixture ratio gave the maximum compressive strength,
- For all types of antifreeze, the decrease in the compressive strength was the maximum between 0°C and -5°C  the decrease was relatively less among 5°C, -10°C, -15°C and -20°C,
- It is seen that there observed 1% increase in the compressive strength of the samples subjected to 0°C and -5°C and similarly 2 % increase in

that of the samples subjected to -10°C, -15°C and -20°C for the cases of the admixture with calcium nitrate.

As a conclusion, it is seen that in the concrete production in cold weathers, the use of antifreeze is effective but not sufficient to protect the concrete from frost action. Beside the use of antifreeze some other protective measures should be used.

**ACKNOWLEDGEMENTS**

In this study, project 07/2008–14 code "Determination Effect of Antifreeze Admixture on Durability Of Concrete" that support the work of the Scientific Research Projects of Gazi University (BAP), I offer my thanks.

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