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Carbon Nanotubes and Resistance to Freeze-Thaw Cycles

Sena Peace Hounkpe[1], Valéry K. Doko[2], Smith O. Kotchoni[3], Hui Li[3], Abbas T. Datchossa[2*]

[1]Laboratory of Water Technical Sciences, UAC, Cotonou, Benin
[2]Laboratory of Applied Energetics and Mechanics (LAEM), EPAC, UAC, Cotonou, Benin
Email: *datchossatiamboabbas@gmail.com

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Abstract

The research of materials with good properties is one of the important concerns of scientists groups, and more again in region where materials are subjected to freeze and thaw cycles. In the case of this paper, it has been a matter of evaluating of the effect of carbon nanotubes on concrete resistance to freeze and thaw cycles. Thus, it has been manufactured concretes with different rates of addition (0%, 0.1%, 0.5%, 1% bwc) of cement by carbon nanotubes. The durability factor, determined for C30 specimens at 28 days, shows that C005 provides a better resistance to freezing-thawing cycles with a 54.96 as index.

Keywords

Silicates, Flexural Strength, Compressive Strength, Cement, Carbon Nanotubes, Freeze-Thaw Cycles

1. Introduction

Concrete is a material of the world widely used in structures, from buildings to factories, from bridges to airports. The world is facing an amazing population explosion and has to cater its building materials needs. Therefore, it is urgently needed to improve the strength and durability of concrete. Several supplementary cementitious materials are added to concrete improving its properties [1] [2] while improving the mechanical reactions [3] [4]. Of the various technologies in use, Nano-technology looks to be a promising approach in improving the properties of concrete.

Several previous authors such as [5] [6] [7] research above the resistance of cementitious materials to freezing and thawing cycles.

In this article, we are interested in improving the crack resistance, improving the mechanical properties of concrete in the course of the freeze-thaw cycle by using Carbon nanotubes and fly ash together.

2. Materials and Methods

2.1. Materials

2.1.1. General

This chapter is concerned with the details of the properties of the materials used, the method followed to design the experiment and the test procedures followed. The theory is supplemented with a number of pictures to have a clear idea of the methods.

2.1.2. Materials Properties

The materials used to design the mix for C30, C40, grade of concrete is cement, fly ash grade II, sand, coarse aggregate, water, Carbon Nanotube (CNT). The properties of these materials are presented below.

2.1.3. Properties of Cement

Ordinary Portland cement (Chinese Standard GB 8076-2008) Classified as 42.5R was applied in This Study. Chemical Composition, Mineral Composition, as well as Physical Performance of the cement, are shown in Table 1. The contents of oxides were measured Through X-Ray Fluorescence. The Content of F-Cao was analyzed by the Franke Method. The mineral phases were calculated by The Bogue Method.

2.1.4. Fly Ash

The disposal of Fly ash poses increasingly difficult problems for many urbanized regions. A viable solution to the problem is reclamation of Fly ash for Civil Engineering applications. Previous researchers shown that fly ash is a potential source of construction material and soil stabilizer. Although it is one of the lowest cost and most widely used materials in the world, cement raises many concerns for the environment and human health. Many studies have been conducted with the aim of reducing the cost of cement for soil stabilization; one option is to partially replace cement with waste materials such as fly ash. This study we used Fly ash grade II.

2.1.5. Properties of Fine and Coarse Aggregate

The China ISO Standard Sand Compiling with GB/T 17671-2005 was used to prepare cement Mortar. For coarse aggregate, the parent concrete is crushed through mini jaw crusher. During crushing it is tried to maintain to produce the maximum size of aggregate in between 20 mm to 4.75 mm. The coarse aggregate particle size distribution curve is presented in Figure 1.

Table 1. Chemical and mineral compositions of cement (Wt/%).

Chemical Composition (%)										Mineral Composition (%)			
Sio_2	Fe_2O_3	Al_2O_3	Cao	MgO	SO_3	FcaO	Cl^-	Na_2Oeq	LOSS	C_3S	C_2S	C_3A	C_4AF
20.560	3.230	4.600	62.560	2.570	2.950	0.870	0.011	0.530	2.040	57.340	18.900	6.470	11.250

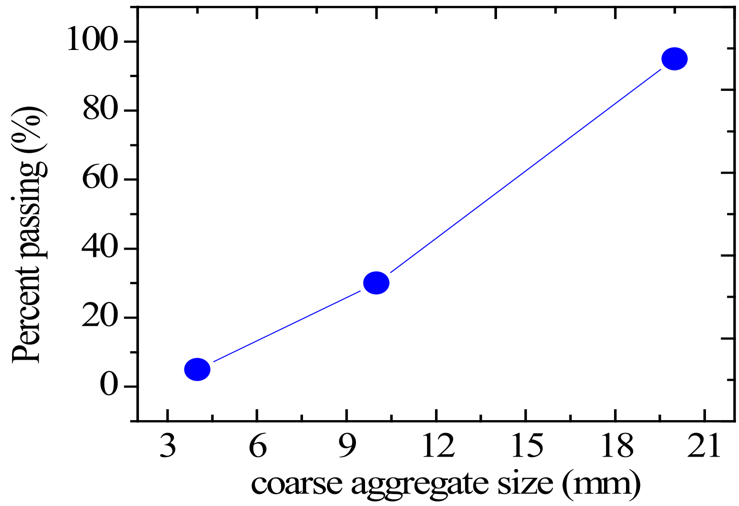

Figure 1. Curve of coarse aggregate particle size distribution.

2.1.6. Properties of Water

Tap water was used in this experiment. The properties are assumed to be same as that of normal water. Specific gravity is taken as 1.00. Pure water (deionized water) was used to make mortar specimen and cement paste.

2.1.7. Properties of Carbon Walled Nanotubes

Table 2 showed the properties of the Carbon nanotube.

2.2. Methods

2.2.1. Mix Design

Specification for mix proportion design of ordinary concrete. The mix design for C30, C40 grade of concrete is described below in accordance with Chinese Standard Code IS JGJ552011 and JGJ /T55-96.

- **Target strength for C30 Mix Proportioning:**

$$fcu,o \geq fcu,k + 1.645\sigma$$

Characteristic compressive strength at 28 days: $fcu,k = 30$ MPa

Assumed standard deviation (**Table 4.0.2** of JGJ552011): $\sigma = 5$ MPa

Target average compressive strength at 28 days: $fcu,o = fcu,k + 1.645\sigma =$ 38.225 MPa

- **Target strength for C40 Mix Proportioning**

Characteristic compressive strength at 28 days: $fcu,k = 40$ MPa

Assumed standard deviation (**Table 4.0.2** of JGJ552011): $\sigma = 5$ MPa

Target average compressive strength at 28 days: $fcu,o = fcu,k + 1.645\sigma =$ 48.225 MPa

- **Selection of water Cement ratio:**

Maximum water content per cubic meter of concrete (refer Tables 1-17 of JGJ/T55-96): Wmax = 175L (for 40 mm slump).

Since the slump was less than 40 mm, no adjustment was required. To start with let us assume a water-cement ratio of 0.42.

Table 2. Properties of carbon nanotube.

TEST ITEM	values
Weight	100 g
OD	50 nm
Length	<10 um
Purity	>98 wt%
MFG CODE	TNM8 171127

➢ **Calculation of cement content:**

Mass of water selected per cubic meter of concrete = 175 kg.

Mass of cement per cubic metre of concrete = 175/0.35 = 500 kg.

Minimum cement content = 260 kg/m^3 (for moderate exposure condition, Cl 1.7 of JGJ/T55-96). Maximum cement content = 550 kg/m^3 (Cl. 1.7 of JGJ/T55-96)

So, the selected cement content is alright.

➢ **Proportion of volume of coarse Aggregate and Fine Aggregate content:**

Volume of coarse aggregate per unit volume of total aggregate (Tables 1-17 of JGJ/T55-96) = 1278 kg

(This is corresponding to 20 mm size aggregate and Zone III fine aggregate for water-cement ratio of 0.35)

Mix calculations for cement paste

The design of each mix began with a constant paste content (water + cement + supplementary cementitious materials) of 0.32 by weight of the total mix. The weight of cement and water were adjusted based on the specified water to binder ratio. The remainder of the mixture consisted of an equal weight of fine and course aggregate. Superplasticizer and air entraining agent were added based on experience and trial mixing prior to beginning the test program. The below tables detail the actual weights of the mixture components.

The mix proportion for concrete grade C30 and C40 are resumed in the following Tables 3-7.

2.2.2. Test Procedures

1) Mixing Sequence

Before concrete mixing could begin, the concrete mix ingredients had to be weighed and placed in containers so that the ingredients could be placed into a mixer with little loss of time. Each ingredient was weighed and placed carefully into the mixer. The mixing drum had to be prepared. The purpose of this mix is to wet the mixing drum so that no water is lost during the preparation of the actual mix. The concrete ingredients were placed into the rotating drum following the order. The dry constituents of each mixture were mixed for 15 min using the mixer. Then, 75% of the water was added and mixing continued for 10 min. Next, the remaining 25% of the water was added and mixing continued for another 15 min.

2) Curing Regimens

Table 3. Proportion of materials for concrete mix design.

Designation	Water (g)	Cement (g)	Sand (g)	Aggregate (g)
C30	400	952	1138	2310
C40	350	946	1046	2558

Table 4. Mix proportion with W/C ratio 0.42 for C30 discontent fly ash.

Type of concrete	Designation	Water (g)	Cement (g)	sand (g)	Aggregate (g)	CNT (g)
Ordinary	P0	2500	5950	7112.5	14,437.5	
	C001	2500	5950	7112.5	14,437.5	0.595
CNT	C005	2500	5950	7112.5	14,437.5	2.95
	C01	2500	5950	7112.5	14,437.5	5.95
Total usage		10,000	23,800	28,450	57,750	9.495

Table 5. Mix proportion with W/C ratio 0.42 for C30 content fly ash.

Type of concrete	Designation	Water (g)	Cement (g)	Fly ash (g)	sand (g)	Aggregate (g)	CNT (g)
Ordinary	P0	2500	4760	1190	7112.5	14,437.5	
	C001	2500	4760	1190	7112.5	14,437.5	0.59
CNT	C005	2500	4760	1190	7112.5	14,437.5	2.95
	C01	2500	4760	1190	7112.5	14,437.5	5.95
Total usage		10,000	19,040	4760	28,450	57,750	9.49

Table 6. Mix proportion with W/C ratio 0.42 for C40 discontent fly ash.

Type of concrete	Designation	Water (g)	Cement (g)	sand (g)	Aggregate (g)	CNT (g)
Ordinary	P0	4550	12,298	13,624	33,254	
	C001	4550	12,298	13,624	33,254	1.2298
CNT	C005	4550	12,298	13,624	33,254	6.149
	C01	4550	12,298	13,624	33,254	12.298
Total usage		18,200	49,192	54,496	133,016	19.6768

Table 7. Mix proportion with W/C ratio 0.42 for C40 content fly ash.

Type of concrete	Designation	Water (g)	Cement (g)	Fly ash (g)	Sand (g)	Aggregate (g)	CNT (g)
Ordinary	P0	4550	9838.4	2459.6	13,624	33,254	
	C001	4550	9838.4	2459.6	13,624	33,254	1.2298
CNT	C005	4550	9838.4	2459.6	13,624	33,254	6.149
	C01	4550	9838.4	2459.6	13,624	33,254	12.298
Total usage		18,200	39,353.6	9838.4	54,496	133,016	19.6768

The specimens remained in their molds for 24 hours at room temperature, 25˚C. The Specimens tested were generally curing with air cured at 25˚C and RH

92|% for 3days, 7 days and 28 days.

3) Preparation of Test Specimen

For conducting compressive strength test on concrete cubes of size 100 × 100 × 100 mm are cast. A rotary mixture is used for thorough mixing and a vibrator is used for good compaction. After successful casting, the concrete specimens are demolded after 24 hours and put in curing room for 3 days, 7 days and 28 days maintaining 25˚C.

2.2.3. Testing

1) Testing to evaluate the durability factor

- **Freezing and Thawing**

Prismatic specimens with dimensions of 100 mm × 100 mm × 400 mm were tested for resistance to freezing and thawing according to GB/T50082-2009. In accordance with this testing method, a 5 hours-cycle was selected. One full cycle of freezing and thawing consisted of a rapid temperature decrease from 8˚C to −18˚C in approximately 2 h 20 min. The temperature was then held constant for 8 min before raising the temperature back to 3˚C in 53 min. The temperature was then held constant at 3˚C for 10 min. Testing was conducted during this 10 min window.

The specimens were subjected to 300 cycles of freezing and thawing (or less if the RDM of a specimen dropped below 60). Length, mass, and fundamental frequency (to compute dynamic elastic modulus) measurements were taken at intervals no greater than 25 cycles. Distilled water was used in the conditioning water bath and for immersion of the specimens during freezing and thawing cycles.

- **Dynamic Elastic Modulus**

Testing procedures for determining dynamic elastic modulus are specified in ASTM C 215. To determine the dynamic elastic modulus, concrete prisms are excited over a wide range of frequencies using an impact hammer or a transducer to identify the frequency at which the maximum amplitude occurs. The frequency at which the maximum amplitude occurs is the resonant frequency. The concrete specimen is generally assumed to be a single degree of freedom system, for which the resonant frequency is referred to as the fundamental frequency. The fundamental frequency is used to compute dynamic elastic modulus, E_d, with the following equation:

$$E_D = Cm\omega_r^2 \quad (1)$$

where C is a constant that accounts for Poisson's ratio and the geometry of the specimen, m is the mass of the specimen, and ω_r is the measured fundamental frequency.

When ASTM C 215 is used to monitor deteriorating concrete, it is common to present results in terms of the relative dynamic modulus, computed as follows:

$$RDM = \frac{En}{Eo}(100) \quad (2)$$

where *RDM* is the relative dynamic modulus after n cycles of freezing and thawing, *En* is the dynamic elastic modulus after n cycles, and *Eo* is the dynamic elastic modulus at zero cycles of freezing and thawing. After completion of freezing and thawing cycles, a durability factor, *DF*, can be computed as:

$$DF = \frac{RDM \cdot N}{M} \quad (3)$$

where *N* is the number of cycles imposed and *M* is the specified number of cycles (usually 300).

2) Testing procedures used to evaluate compressive strength and flexural strength are presented in this section.

➢ **Flexural Strength Test**

Flexural testing machine Reference number YAW-300 was used. Flexural strength was evaluated according to Chinese standard with the software Super Test version 8 and the load rate was 50 N/s. Prismatic specimens with dimensions of 40 mm × 40 mm × 160 mm were loaded using a third point loading setup across their strong axis. Three specimens from each batch were tested at an age of 3, 7, and 28 days and the mean Flexural strength of three specimens is considered as the Flexural strength of the specified category.

➢ **Compressive Strength Test**

Compressive testing machine Reference number YAW-300 was used after 3, 7, and 28 days of curing with surface dried condition as per Chinese Standard. The compressive strength of specimens is determined with the software Super Test version 8 and the load rate was 2.4 KN/s. Three specimens are tested for typical category and the mean compressive strength of three specimens is considered as the compressive strength of the specified category.

3. Presentation of Results and Analysis

This chapter is concerned with the presentation of results of the experiments carried out towards the objective of the article.

3.1. Comparison Results and Analysis of Mechanical Test

The change in compressive strength for the blended sample (in %) for 3, 7 and 28 days is shown respectively in the Tables below.

Tables 8-10 show a good increase of the Concrete C30 compressive strength when we use C001 and C005 (without Fly Ash) with a better result in using C005. The good results observe with C001 and C005, this rates of addition allow a better reaction of Carbon Nanotubes with portlandite [8] [9]. In fact the formed hydration products help to fill voids between aggregates [10]. Contrary to compressive strength, we have a loss of the Concrete C30 flexural strength when we use each of the three rates of Carbon nanotubes (without Fly Ash). This is in accordance with the general trend of concrete researchers.

Figure 2 shows that, from 3 days to 28 days, the Concrete C30 compressive strength evolution curve when we use C005 is up all the overs. Then, the

Table 8. Concrete C30 without fly ash at 3-day test.

Type	Compressive	Increase in Strength (%)
PO	24.4	-
C001	27.3	11.89
C005	27.4	12.30
C01	23.7	−2.87

Table 9. Concrete C30 without fly ash at 7-day test.

Type	Compressive	Increase in Strength (%)
PO	37.4	-
C001	40.4	8.02
C005	42.7	14.17
C01	35.9	−4.01

Table 10. Concrete C30 without fly ash at 28-day test.

Type	Compressive	Increase in Strength (%)
PO	40.4	-
C001	44.43	9.98
C005	47.5	17.57
C01	39.99	−1.01

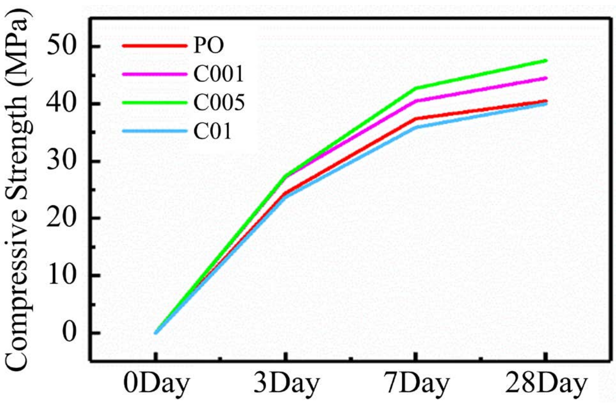

Figure 2. Change in compressive strength of concrete C30 specimen without fly ash from 3 day to 28 days.

best rate of Carbon Nanotube to increase the Concrete C30 compressive strength (without fly ash) is the C005.

Tables 11-13 show a good improvement of the Concrete C30 compressive strength when we use C001 and C005 (with Fly Ash). The good results observe with C001 and C005, this rates of addition allow a better reaction of Carbon Nanotubes with portlandite [8] [9]. In fact, the formed hydration products help to fill voids between aggregates [10]. Contrary to compressive strength, we

Table 11. Concrete C30 with fly ash at 3-day test.

Type	Compressive	Increase in Strength (%)
PO	21.7	-
C001	23.3	7.37
C005	25.5	17.51
C01	24.6	13.36

Table 12. Concrete C30 with fly ash at 7-day test.

Type	Compressive	Increase in Strength (%)
PO	28.7	-
C001	29.6	3.14
C005	30.4	5.92
C01	29.9	4.18

Table 13. Concrete C30 with fly ash at 28-day test.

Type	Compressive	Increase in Strength (%)
PO	31.6	-
C001	34.07	7.82
C005	34.35	8.70
C01	33.13	4.84

have a loss of flexural strength when we use each of the three rates of Carbon nanotubes (with Fly Ash). This agrees with the general trend of concrete researchers.

Figure 3 shows that, from 3 days to 28 days, the Concrete C30 compressive strength evolution curve when we use C001 is up all the overs. Then, the best rates of Carbon Nanotube to increase the Concrete C30 compressive strength (with fly ash) are the C005 and the C001.

It can be seen that using fly ash and carbon nanotubes at the same time in concrete provides bad results in comparison with using carbon nanotubes alone. This may be due to the fact that the binder is not enough because of the higher amount of pozzolan [11] [12].

Tables 14-16 show a good increase of the Concrete C30 compressive strength when we use C001 and C005 (without Fly Ash) with a better result in using C005. The good results observed with C001 and C005, this rates of addition allow a better reaction of Carbon Nanotubes with portlandite [8] [9]. In fact, the formed hydration products help to fill voids between aggregates [10]. Contrary to compressive strength, we have a loss of the Concrete C30 flexural strength when we use each of the three rates of Carbon nanotubes (without Fly Ash). This is in accordance with the general trend of concrete researchers. The diagrams show the real evolution of the *Concrete C40* compressive strength.

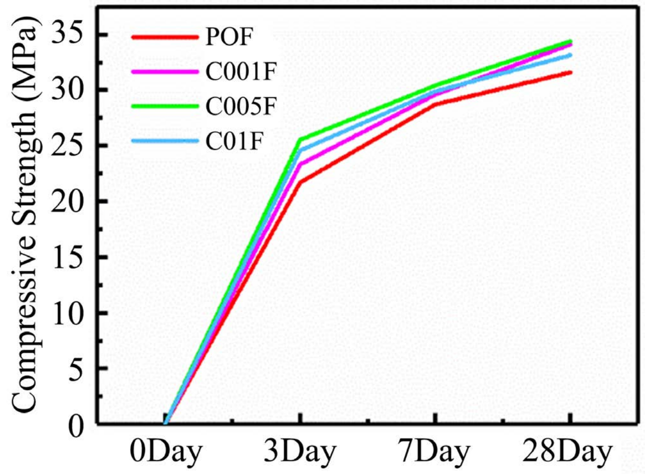

Figure 3. Change in compressive strength of concrete C30 specimen with fly ash from 3 day to 28 days.

Table 14. Concrete C40 without fly ash at 3-day test.

Type	Compressive	Increase in Strength (%)
PO	40.9	-
C001	45.6	11.35
C005	45.5	11.18
C01	39.9	−2.45

Table 15. Concrete C40 without fly ash at 7-day test.

Type	Compressive	Increase in Strength (%)
PO	43.6	-
C001	47.4	8.72
C005	48.6	11.47
C01	47.3	8.49

Table 16. Concrete C40 without fly ash at 28-day test.

Type	Compressive	Increase in Strength (%)
PO	45.6	-
C001	57.8	26.75
C005	55.6	21.93
C01	50.1	9.87

Figure 4 shows that, from 3 days to 28 days, the *Concrete C40* compressive strength evolution curve when we use C001 is up all the overs. Then, the best Carbon Nanotube to increase the *Concrete C40* compressive strength (without fly ash) is the C001 and the C005.

Tables 17-19 show a good improvement of the Concrete C30 compressive

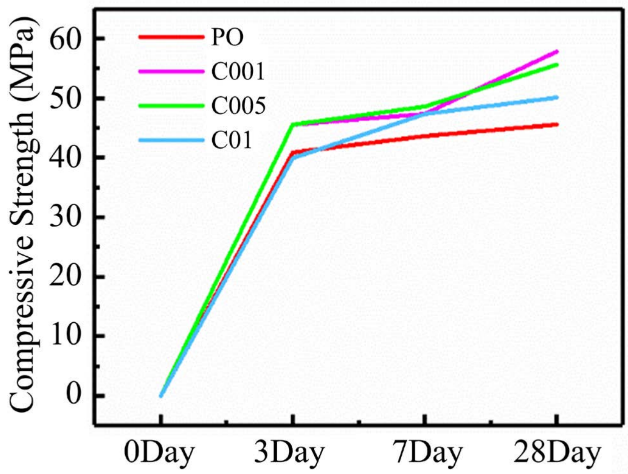

Figure 4. Change in compressive strength of concrete C40 specimen without fly ash from 3 day to 28 days.

Table 17. Concrete C40 with fly ash at 3-day test.

Type	Compressive	Increase in Strength (%)
PO	26.4	-
C001	30.4	15.15
C005	31.3	18.56
C01	26.8	1.52

Table 18. Concrete C40 with fly ash at 7-day test.

Type	Compressive	Increase in Strength (%)
PO	37	-
C001	44.7	20.81
C005	39.4	6.49
C01	32.8	−11.35

Table 19. Concrete C40 with fly ash at 28-day test.

Type	Compressive	Increase in Strength (%)
PO	43.1	-
C001	51.5	19.49
C005	51.9	20.42
C01	41.3	−4.18

strength when we use C001 and C005 (with Fly Ash). The good results observe with C001 and C005, this rates of addition allow a better reaction of Carbon Nanotubes with portlandite [8] [9]. In fact, the formed hydration products help to fill voids between aggregates [10]. Contrary to compressive strength, we have a loss of flexural strength when we use each of the three rates of Carbon nanotubes (with Fly Ash). The diagrams show the real evolution of the Concrete C40 compressive strength (with Fly Ash).

Figure 5 shows that, from 3 days to 28 days, the Concrete C40 compressive strength evolution curve when we use C001 is almost up all the overs. Then, the best Carbon Nanotube to increase the *Concrete C40* compressive strength (with fly ash) is the C005.

It can be seen that using fly ash and carbon nanotubes at the same time in concrete provides bad results in comparison with using carbon nanotubes alone. This may be due to the fact that the binder is not enough because of the higher amount of pozzolan [11] [12].

3.2. Durability Factor

The durability factor of concrete is its index of ability to resist damaging effects induced by different mechanical and environmental loadings during its service life.

Durability factor calculations

$$DF = PN/M \tag{4}$$

$$P_i = \frac{f_{ni}^2}{f_{0i}^2} \times 100 \tag{5}$$

where, DF = durability factor of the test specimen

P = relative dynamic modulus of elasticity at N cycles, %

f_{ni}^2 = the horizontal base frequency of the concrete specimen after the N Times of freezing-thawing

f_{0i}^2 = the initial value of the transverse base frequency of the concrete specimen before the N Times of freezing-thawing

N = number of cycles at which P reaches the specified minimum value for discontinuing the test or the specified number of cycles at which the exposure is to be terminated, whichever is less, and

M = specified number of cycles at which the exposure is to be terminated.

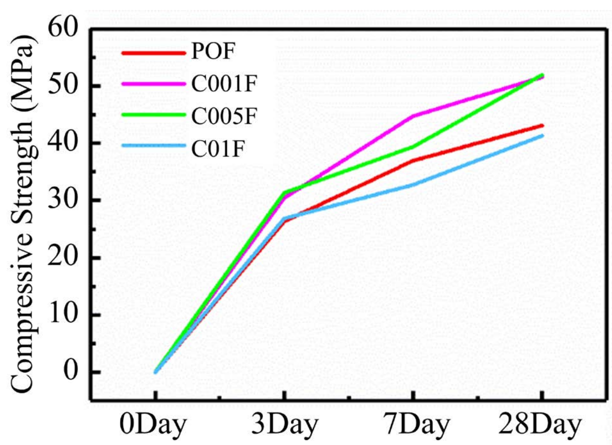

Figure 5. Change in compressive strength of concrete C40 specimen with fly ash from 3 day to 28 days.

The durability factor was determined for standard specimen and modified with Carbon Nanotube. In standard specimen, we had pure concrete and in the modified specimen we had concrete with Carbon Nanotube.

The durability factor was calculated when the dynamic modulus of elasticity of the specimen fell below 60% during the next cycling period. The durability factor is graphed in Figure 6. An acceptable durability factor is one at or above 60. The durability factor has been determined only on the concrete specimens without fly ash because of the good compressive strengths observed on it. The tables below are shown the durability factor for each specimen.

Tables 20-23 show the durability of control specimen and the modified specimen. Respectively we have 52.50, 50.99, 54.96, and 34.19 for PO, C001, C005 and C01 respectively. The gap value between standard specimen and the modified was respectively −1.51, 2.46 and 18.31.

It's clear that C005 provides a better concrete as it is shown in the sections above.

Figure 6 shows the variation of Dynamic Modulus of each specimen with the number of cycles. It can be seen that the dynamic modulus of all the modified specimens are up that of the control specimen for number cycles lesser

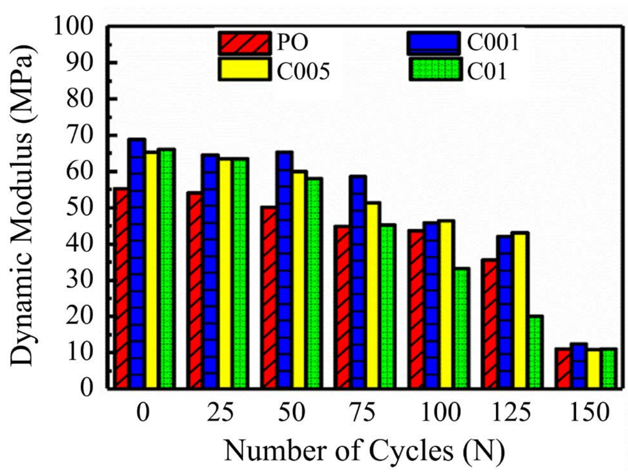

Figure 6. Dynamic modulus of the control and carbon nanotube modified specimen.

Table 20. Durability performance for the control specimen.

N	frequency	dynamic modulus	Percentage
0	2668	58,800	100.00
25	2648	58,600	99.66
50	2410	47,600	80.95
75	2342	45,000	76.53
100	2322	45,700	77.72
125	2080	37,044	63.00
150	1288	13,800	23.47
	Durability factor		52.50

Table 21. Durability performance for the C001 specimen.

N	frequency	dynamic modulus	Percentage
0	2866	68,800	100.00
25	2841	64,500	93.75
50	2773	65,200	94.77
75	2647	58,600	85.17
100	2335	45,800	66.57
125	2254	42,100	61.19
150	1225	12,500	18.17
	Durability factor		50.99

Table 22. Durability performance for the C005 specimen.

N	frequency	dynamic modulus	Percentage
0	2773	65,200	100.00
25	2737	63,500	97.39
50	2671	60,000	92.02
75	2461	51,400	78.83
100	2335	46,500	71.32
125	2278	43,000	65.95
150	1138	10,800	16.56
	Durability factor		54.96

Table 23. Durability performance for the C01 specimen.

N	frequency	dynamic modulus	Percentage
0	2809	66,100	100.00
25	2737	63,500	96.07
50	2629	58,100	87.90
75	2315	45,200	68.38
100	2002	33,300	50.38
125	1552	20,000	30.26
150	1151	11,000	16.64
	Durability factor		34.19

than 75. Above this value of number of cycles, the dynamic modulus of modified specimens start decreasing more quickly with a better value at C005-modified specimen. This proves again that 0.05% is the best rate of substitution of carbon nanotube to have concrete more resistant to freeze-thaw cycles.

4. Conclusions

After reviewing the data for test results, conclusions were drawn. The study showed that: the use of carbon nanotubes gives very interesting results when the purpose is to increase compressive strength of cementitious materials especially when we use C001 or C005. The best rate of carbon nanotube to increase compressive strength of cementitious materials recommended is the C001 except the case of concrete C30 where we recommend the using of the C005. Contrary to compressive strength, we have a loss of flexural strength when we use Carbon nanotubes. The determination of the durability factor shows that C005 is better to have concrete more resistant to freeze-thaw cycles. Respectively, we have 52.50, 50.99, 54.96, and 34.19 for PO, C001, C005 and C01 respectively.

Conflicts of Interest

The authors declare no conflicts of interest regarding the publication of this paper.

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