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Changing Mechanicals Characteristiques of Cementitious Materials Using Titanium Dioxide

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Abstract

Since many years ago, the substitution of cement by other cementitious supplementary elements has being a purpose for many researchers. This is to reduce the impact of producing cement on our environment. In this article, we are interested in the possibility of substituting cement with titanium dioxide and titanium dioxide + fly ash. To achieve this purpose, we have manufactured mortars and cement pastes specimens with different rates of replacement of cement by titanium dioxide (0%, 0.1%, 1%) on the one hand and titanium dioxide + fly ash on the overhand. The flexural and compressive strength of each specimen has been determined.

Keywords

Silicates, Flexural Strength, Compressive Strength, Cement, Titanium Dioxide

1. Introduction

Cement can be described as a crystalline compound of calcium silicates and other calcium compounds having hydraulic properties [1]. The four major compounds that constitute cement (Bogue's Compounds) are Tricalcium silicate, abbreviated as C_3S, Dicalcium silicate (C_2S), Tricalcium aluminate (C_3A), Tetracalcium aluminoferrite (C_4AF) where C stands for CaO, S stands for SiO_2, A stands for Al_2O_3 and F for Fe_2O_3 [2] [3]. Cement is the main constituent of the concrete, the widely used in structures, from buildings to factories, from bridges to airports. This makes concrete to be one of the most investigated materials of the 21st century.

The increased use of cement is essential in attaining a higher compressive strength. But, cement is a major source of pollution [4] [5]; It is for this reason that several authors have been interested in finding new materials to limit the use of cement [6]. It is in this sense that [7] designed concrete based on volcanic ash and found mechanical properties similar to those of ordinary cement concrete.

At the same time, nanomaterials are showing their interest, with a large number of researchers looking to them as an alternative.

Moreover, Hossain and al. have shown that the use of nanomaterials by replacement of a proportion of cement can lead to a rise in the compressive strength by developing supplementary chemical reactions [8] of the concrete as well as a check to pollution.

In this article, we are interested in improving the crack resistance, improving the mechanical properties of concrete using titanium dioxide and fly ash.

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 C_{30}, C_{40}, grade of concrete is cement, fly ash grade II, sand, coarse aggregate, water, and Titanium dioxide (TiO_2) admixture. 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 showed that fly ash is a potential source of construction material and soil stabilizer. Although it is one of the lowest

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

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. In this study, we used fly ash grade II.

2.1.5. 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.

2.1.6. Properties of Titanium Oxide, Anatase

The average size of Titanium Oxide was 25 nm with 99.8% metals basis from Particle Size Analyzer.

2.1.7. Properties of Cement Paste and Mortar

Cement paste and mortar are prepared with a water/cement ratio (w/c) of 0.32, using a blade-type high shear blender. Before mixing, polycarboxylate superplasticizers (PCE) solution was prepared with deionized water. With the addition of polycarboxylate superplasticizers (PCE) solution dosage 0.3% b.w.c., Cement paste was mixed for 2 min at low speed and then 2 min at high speed. The table below showed the proportion and quantities of material used by following Chinese standard.

2.2. Methods

2.2.1. Mix Calculations for Cement Paste

The design of each mix began with 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 sand. Superplasticizer and air entraining agent were added based on experience and trial mixing prior to beginning the test program. Table 2, Table 3 below detail the actual weights of the mixture components.

2.2.2. Mix Calculations for Mortar

The design of each mix began with constant paste content (water + cement + supplementary cementitious materials) of 0.32 by weight of the total mix. The

Table 2. Mixture proportions with W/C ratio 0.32 for cement paste without fly ash.

Cement Paste type		water (g)	Cement (g)	Titanium dioxide TiO_2	Water reducing agent (g)	Test pieces
Pure Cement Paste	PO	1664	5200		5.4	12
Nano Titanium dioxide TiO_2	T01	1664	5200	5.2	5.4	12
	T1	1664	5200	52	5.4	12
Total dosage		4992	15,600	57.2	16.2	36

Table 3. Mixture proportions with W/C ratio 0.32 for cement paste content fly ash.

Cement Paste type		water (g)	Cement (g)	Fly Ash	TiO_2	Water reducing agent (g)	Test pieces
Pure Cement Paste	P0	1664	4160	1040		5.4	12
Nano Titanium dioxide TiO_2	T01	1664	4160	1040	5.2	5.4	12
	T1	1664	4160	1040	52	5.4	12
Total dosage		4992	12,480	3120	57.2	16.2	36

weight of cement and water was adjusted based on the specified water to binder ratio. The remainder of the mixture consisted of sand. Superplasticizer and air entraining agent were added based on experience and trial mixing prior to beginning the test program. Table 4, Table 5 below detail the actual weights of the mixture components.

2.2.3. Test Procedures

Curing Regimens

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 3 days, 7 days and 28 days.

2.2.4. Testing

Testing procedures used to evaluate compressive strength, flexural strength and interatomic behaviors between cement and titanium dioxide are presented in this section.

2.2.5. SEM Test

Scanning Electron Microscope (SEM) test is performed by technical experts, and thus it is not explained here and only the results are presented in the result and discussion section.

2.2.6. Flexural Strength Test for Mortar and Cement Paste

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.

2.2.7. Compressive Strength Test for Mortar and Cement Paste

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.

Table 4. Mixture proportions with W/C ratio 0.32 for mortar discontent fly ash.

Mortar type		Water (g)	Cement (g)	Sand (g)	TiO_2	Water reducing agent (g)	Test pieces
Pure Mortar	PO	576	1800	5400	None	5.4	12
Nano Titanium dioxide TiO_2	T01	576	1800	5400	1.8	5.4	12
	T1	576	1800	5400	18	5.4	12
Total dosage		1728	5400	16,200	19.8	16.2	36

Table 5. Mixture proportions with W/C ratio 0.32 for mortar content fly ash.

Mortar type		Water (g)	Cement (g)	Fly Ash (g)	Sand (g)	TiO_2 (g)	Water reducing agent (g)	Test pieces
Pure Mortar	PO	576	1440	360	5400	None	5.4	12
Nano Titanium dioxide TiO_2	T01	576	1440	360	5400	1.8	5.4	12
	T1	576	1440	360	5400	18	5.4	12
Total dosage		1728	4320	1080	16,200	19.8	16.2	36

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. Scanning Electron Microscope (SEM) Images and EDS Results

Take a small piece of the sample to after full salt soaked in ethanol termination of hydration, then 50˚C drying in the oven for 24 h. The surface morphology and element distribution of cement were analyzed by SEM and EDS energy spectrum analysis. Through SEM, it can be seen that the surface morphology of the sample is shown in Figure 1 and Figure 2 after substituting cement by different sizes of Nano-materials in the case of an investigation.

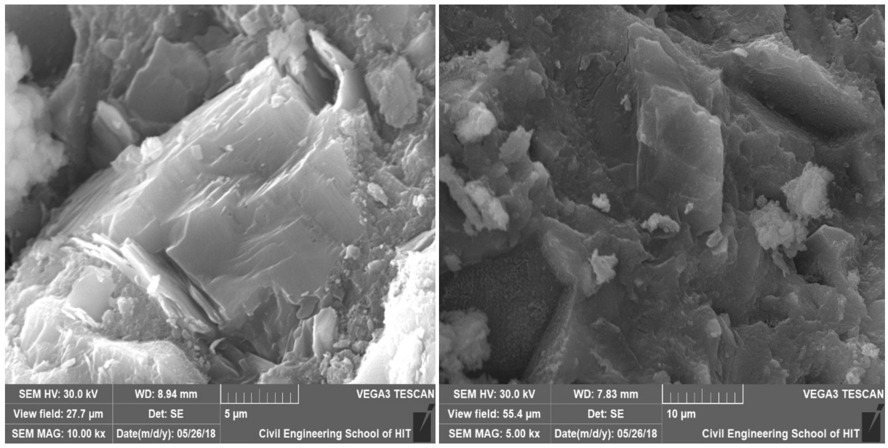
3.1.1. Comparison of SEM Micrographs

Figure 1 shows the FESEM micrograph of control mortar specimen. In this figure, it can be clearly seen that the C-S-H gel is distributed with lots of empty spaces between the lumps. The lumps can be $Ca(OH)_2$ which declines the Interfacial Transition Zone (ITZ) [9]. The microstructure looks to contain mainly formless substances.

Figure 2 shows the FESEM micrograph of the mortar specimen with Titanium Dioxide 0.1% b.w.c. A uniform microstructure with very little void can be seen. The absence of $Ca(OH)_2$ crystals indicates that CNT has reacted with $Ca(OH)_2$ [10] and converted it into C-S-H gel [11].

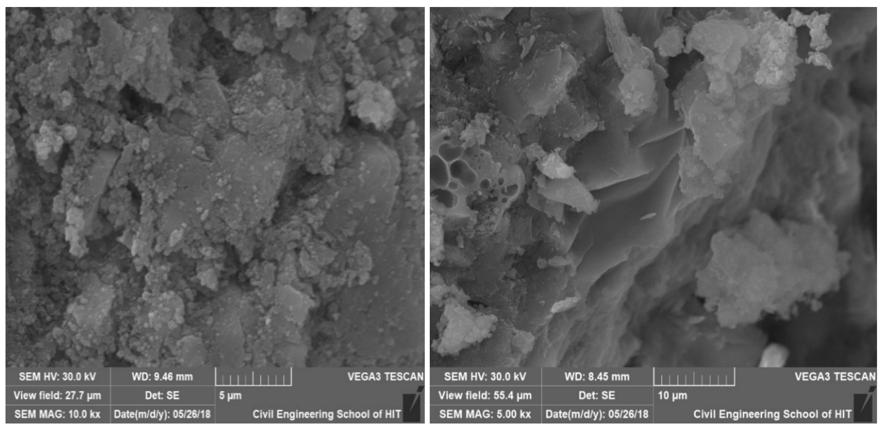
3.1.2. Comparison of Chemical Composition of the Specimen

Figure 3 and Table 6 show the comparative chemical arrangement of mortar specimen deprived of Titanium Dioxide. High concentration of calcium is due to the formation of $Ca(OH)_2$ crystals which declines the ITZ [9].



(a) (b)

Figure 1. SEM image of control specimen with different magnification. (a) at magnification 5 nm; (b) at magnification 10 nm.



(a) (b)

Figure 2. SEM image of Tiatanium Dioxide specimen with different magnification. (a) at magnification 5 nm; (b) at magnification 10 nm.

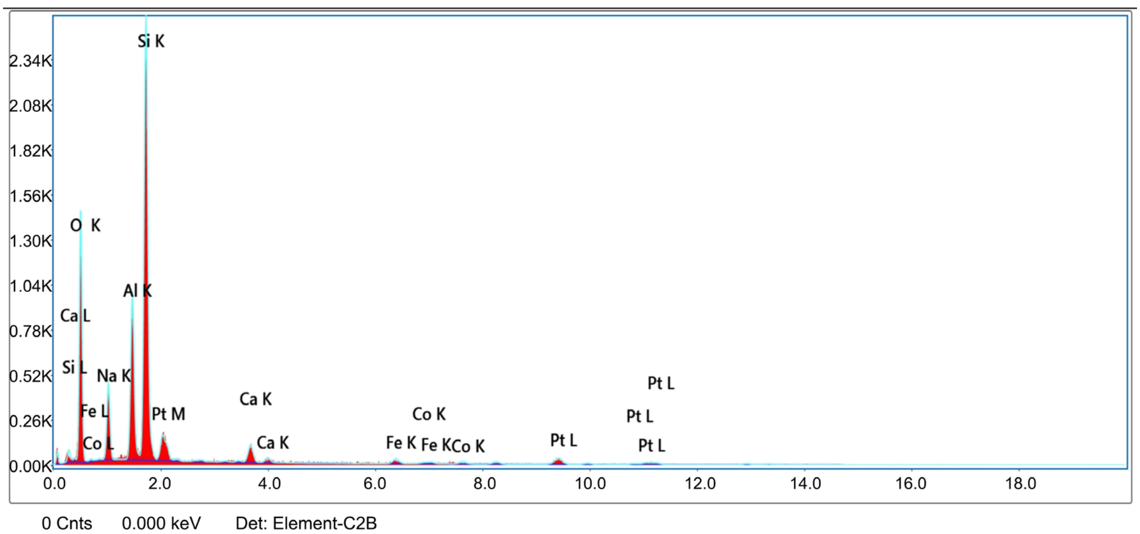


Figure 3. Relative chemical composition for the control specimen.

Table 6. Relative smart quant results for control specimen.

Element	Weight (%)	Atomic (%)	Net Int.	Error (%)	K ratio	Z	A	F
O K	42.65	56.97	289.36	9.26	0.1162	1.0578	0.2576	1.0000
NaK	11.16	10.37	102.86	10.70	0.0272	0.9703	0.2505	1.0014
AlK	11.52	9.13	251.72	7.92	0.0465	0.9552	0.4215	1.0035
SiK	29.13	22.17	718.44	7.08	0.1239	0.9784	0.4344	1.0011
CaK	1.35	0.72	37.36	9.89	0.0101	0.9339	0.7916	1.0164
FeK	0.51	0.20	12.86	24.39	0.0047	0.8423	0.9891	1.1015
CoK	0.15	0.05	3.48	35.73	0.0014	0.8248	0.9992	1.1310
PtL	3.54	0.39	22.05	19.71	0.0254	0.6380	1.1118	1.0117

Figure 4 shows the relative chemical composition of the concrete specimen with NS 0.6% b.w.c. This figure looks contradicting due to the high percentage of silica and a low percentage of calcium. A good percentage of oxides can be due to the reaction of silica with $Ca(OH)_2$ which produces C-S-H gel [11] [12] (Table 7).

3.2. Comparison Results and Analysis of Mechanical Test

The change in compressive strength and flexural strength for the blended sample (in %) for 3, 7 and 28 days is shown respectively in the Table below. A graphical representation of this result is shown respectively in Figure 5 below.

Tables 8-10 show a better increase of compressive strength when we use T01 (without Fly Ash). These observations may be explained by the lower activity factor of T1 [13]. In fact, the reaction of amorphous silica and alumina phases with $Ca(OH)_2$ leads to the formation of more CSH [11] [14]. Contrary to compressive strength, we have a loss of flexural strength when we use either T01 or T1 (without Fly Ash). This agrees with the general trend in the literature. In fact concrete is much stronger in compression than it is in tension [15].

The diagrams Figures 5-7 show the real evolution of the mortar compressive strength.

Figure 8 shows that from 3 days to 28 days, the *Mortar* compressive strenght evolution curve when we use T01 is up all the overs. Then, the appropriate rate of substitution of cement by Titanium Dioxide Nanotube to increase the *Mortar* compressive strength (without fly ash) is the T01.

Tables 11-13 show the best increase of compressive strength when we use T01 (with Fly Ash). These observations may be explained by the lower activity factor of [13]. In fact, the reaction of amorphous silica and alumina phases with $Ca(OH)_2$ leads to the formation of more CSH [11] [14].

It is noticed that all the fly ash samples present lower mechanical properties than the discontent fly ash samples. These results are obviously due to a lesser amount of cement in all mixes containing both fly ash and carbon nanotubes [11] [12].

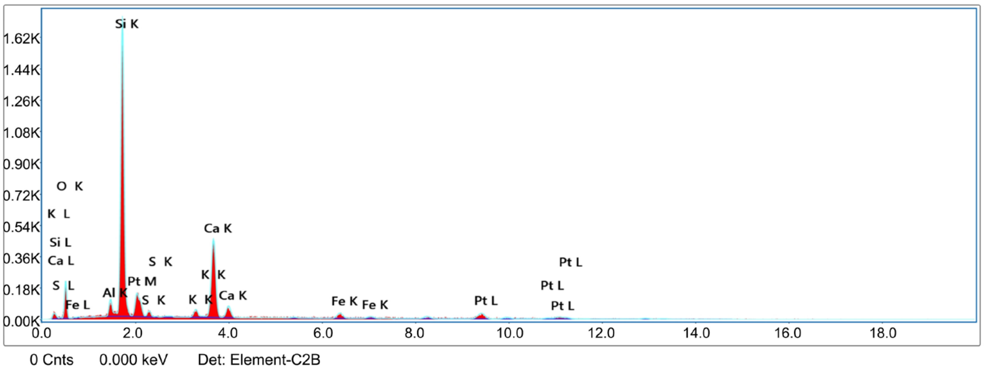


Figure 4. Relative chemical composition for the titanium dioxide specimen.

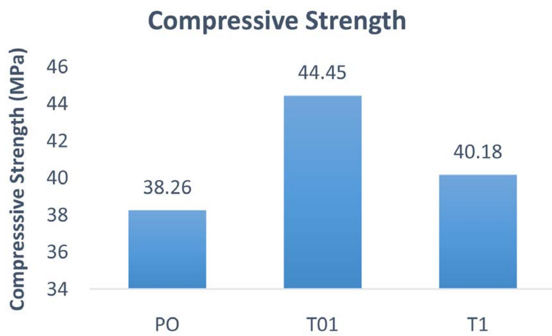


Figure 5. 3-day test mortar specimen without fly ash.

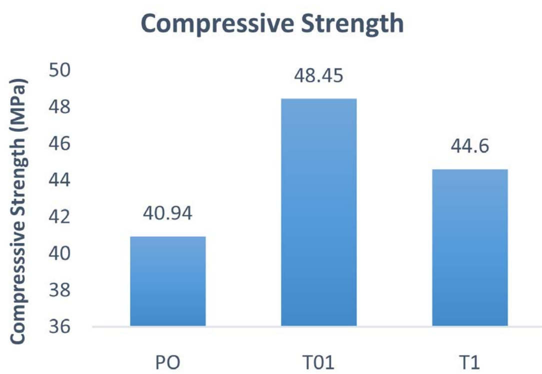


Figure 6. 7-day test mortar specimen without fly ash.

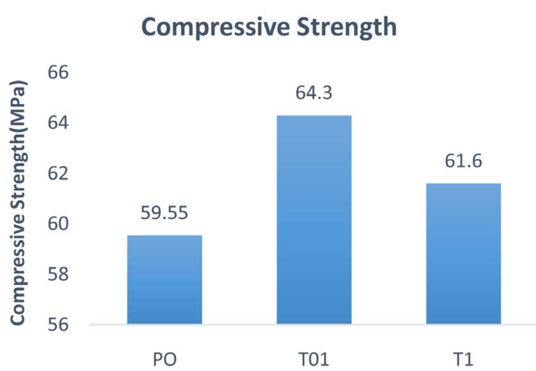


Figure 7. 28-day test mortar specimen without fly ash.

Table 7. Relative smart quant results for the titanium dioxide specimen.

Element	Weight (%)	Atomic (%)	Net Int.	Error (%)	K ratio	Z	A	F
O K	20.10	40.19	31.24	14.88	0.0220	1.1301	0.0969	1.0000
MgK	1.08	1.42	7.67	24.95	0.0027	1.0618	0.2374	1.0013
AlK	8.67	10.28	91.83	9.78	0.0304	1.0259	0.3415	1.0021
SiK	4.44	5.06	60.12	11.26	0.0186	1.0517	0.3982	1.0032
K K	0.39	0.32	7.07	34.85	0.0030	0.9878	0.7610	1.0317
CaK	40.22	32.11	687.10	3.34	0.3380	1.0082	0.8244	1.0109
TiK	0.99	0.66	13.36	20.53	0.0065	0.9181	0.6966	1.0236
FeK	14.69	8.41	185.25	4.15	0.1228	0.9126	0.8828	1.0376
PtL	9.42	1.54	32.69	17.90	0.0684	0.6951	1.0517	0.9933

Table 8. Comparison mechanical strength of mortar specimen without fly ash at 3-day test.

Type	Flexural	Increase in Strength (%)	Compressive	Increase in Strength (%)
PO	8.4	-	38.26	-
T01	6.9	−17.85	44.45	16.19
T1	6.27	−25.36	40.18	5.03

Table 9. Comparison mechanical strength of mortar specimen without fly ash at 7-day test.

Type	Flexural	Increase in Strength (%)	Compressive	Increase in Strength (%)
PO	9.67	-	40.94	-
T01	8.1	−16.23	48.45	18.34
T1	7.37	−23.78	44.6	8.94

Table 10. Comparison mechanical strength of mortar specimen without fly ash at 28-day test.

Type	Flexural	Increase in Strength (%)	Compressive	Increase in Strength (%)
PO	10.1	-	59.55	-
T01	10.8	6.93	64.3	7.98
T1	9.6	−4.95	61.6	3.44

Table 11. Comparison mechanical strength of mortar specimen with fly ash at 3-day test.

Type	Flexural	Increase in Strength (%)	Compressive	Increase in Strength (%)
PO	6.4	-	28.38	-
T01	6.03	−5.78	33.17	16.88
T1	5.97	−6.72	29.99	5.66

Table 12. Comparison mechanical strength of mortar specimen with fly ash at 7-day test.

Type	Flexural	Increase in Strength (%)	Compressive	Increase in Strength (%)
PO	7.53	-	31.98	-
T01	7.43	−1.33	37.85	18.36
T1	7.36	−2.26	34.84	8.96

Table 13. Comparison mechanical strength of mortar specimen with fly ash at 28-day test.

Type	Flexural	Increase in Strength (%)	Compressive	Increase in Strength (%)
PO	9.5	-	54.14	-
T01	9.2	−3.16	58.45	7.98
T1	9.23	−2.84	56.51	4.39

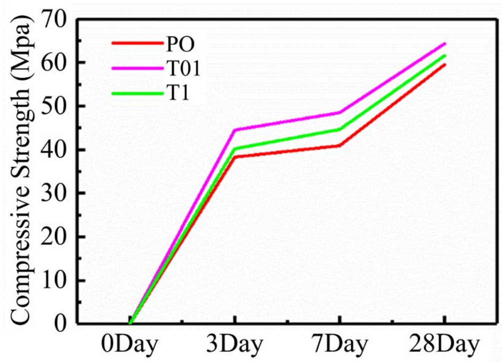


Figure 8. Change in compressive strength of mortar specimen without fly ash from 3 days to 28 days.

Contrary to compressive strength, we have a loss of flexural strength when we use either T01 or T1 (with Fly Ash). This agrees with the general trend in the literature. In fact concrete is much stronger in compression than it is in tension [15].

The diagrams **Figures 9-11** show the real evolution of the *Mortar* compressive strength.

Figure 12 shows that, from 3 days to 28 days, the *Mortar* compressive strength evolution curve when we use T01 is up to all the overs. Then, the appropriate rate of substitution of cement by Titanium Dioxide Nanotube to increase the *Mortar* compressive strength (with fly ash) is the T01. We remark that Mortar specimens are gaining in mechanical characteristics while gaining in age. This agrees with the literature.

Tables 14-16 show a better increase of compressive strength when we use T01 (without Fly Ash). These observations may be explained by the lower activity factor of T1 [13]. In fact, the reaction of amorphous silica and alumina phases with $Ca(OH)_2$ leads to the formation of more CSH [10] [13]. Contrary to compressive strength, we have a loss of flexural strength when we use either T01 or T1 (without Fly Ash). This agrees with the general trend in the literature [15].

The diagrams **Figures 13-15** show the real evolution of the *Cement Paste* compressive strength.

Figure 16 shows that, from 3 days to 28 days, the *Cement Paste* compressive strength evolution curve when we use T01 is up to all the overs. Then, the appropriate rate of substitution of cement by Titanium Dioxide Nanotube to increase the *Cement Paste* compressive strength (without fly ash) is the T01.

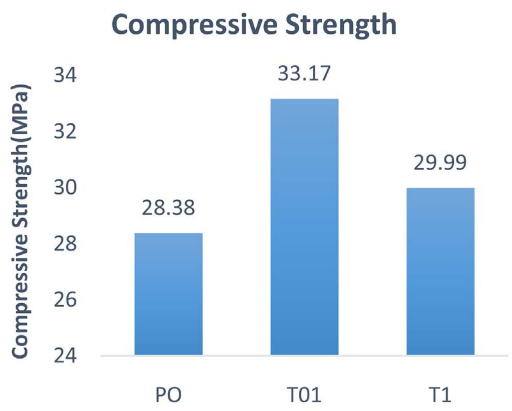


Figure 9. 3-day test mortar specimen with fly ash.

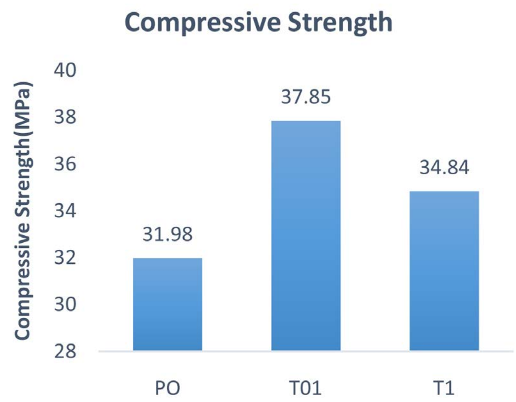


Figure 10. 7-day test mortar specimen with fly ash.

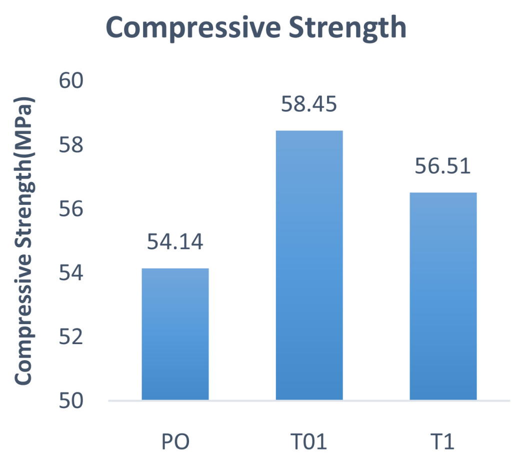


Figure 11. 28-day test mortar specimen with fly ash.

Table 14. Comparison mechanical strength of cement paste specimen without fly ash at 3-day test.

Type	Flexural	Increase in Strength (%)	Compressive	Increase in Strength (%)
PO	7.5	-	38.6	-
T01	9.6	28.00	47.35	22.67
T1	8.2	9.33	42.7	10.62

Table 15. Comparison mechanical strength of cement paste specimen without fly ash at 7-day test.

Type	Flexural	Increase in Strength (%)	Compressive	Increase in Strength (%)
PO	8.4	-	41.68	-
T01	10.5	25.00	51.74	24.15
T1	9.5	13.10	47.52	14.02

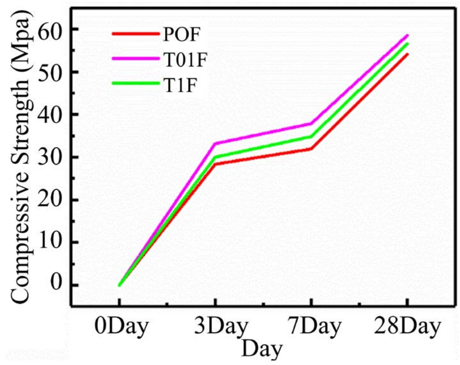


Figure 12. Change in compressive strength of mortar specimen with fly ash from 3 days to 28 days.

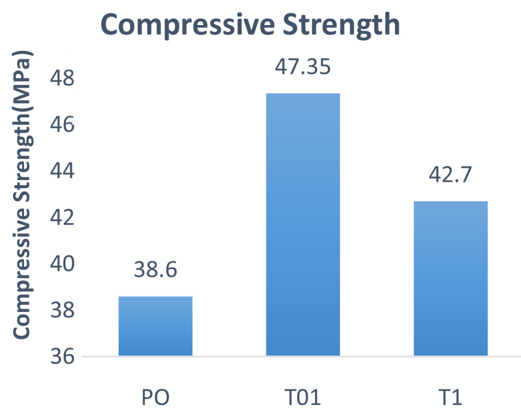


Figure 13. 3-day test cement paste specimen without fly ash.

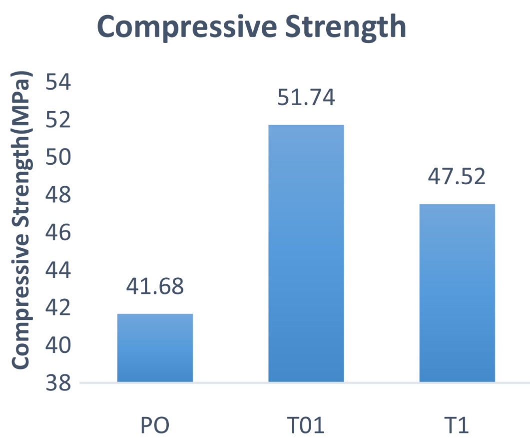


Figure 14. 7-day test cement paste specimen without fly ash.

Table 16. Comparison mechanical strength of cement paste specimen without fly ash at 28-day test.

Type	Flexural	Increase in Strength (%)	Compressive	Increase in Strength (%)
PO	9.9	-	63.6	-
T01	12.2	23.23	68.2	7.17
T1	11.4	15.15	65.3	2.67

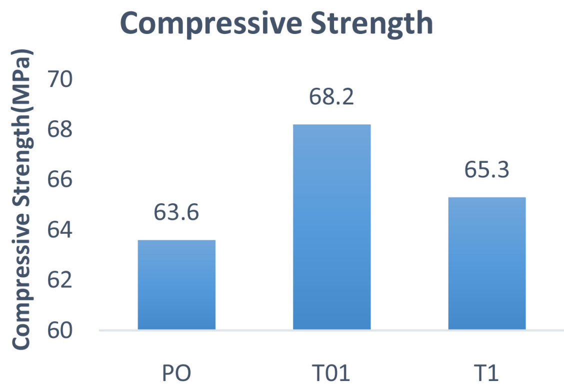


Figure 15. 28-day test cement paste specimen without fly ash.

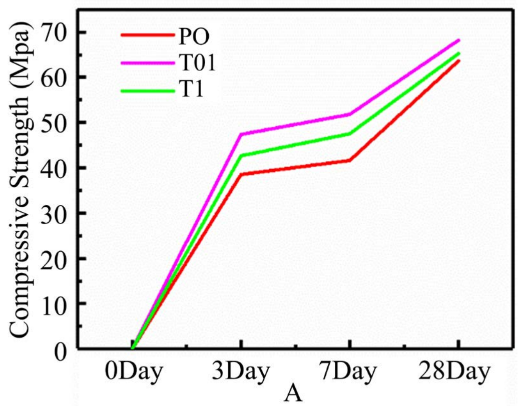


Figure 16. Change in compressive strength of cement paste specimen without fly ash from 3 days to 28 days.

Tables 17-19 show the best increase of compressive strength when we use T01 (with Fly Ash). These observations may be explained by the lower activity factor of T1 [13]. In fact, the reaction of amorphous silica and alumina phases with $Ca(OH)_2$ leads to the formation of more CSH [11] [14].

It is noticed that all the fly ash samples present lower mechanical properties than the discontent fly ash samples. These results are obviously due to a lesser amount of cement in all mixes containing both fly ash and carbon nanotubes [11] [12].

Contrary to compressive strength, we have a loss of flexural strength when we use either T01 or T1 (with Fly Ash). This agrees with the general trend in the literature because cementitious materials are much stronger in compression than they are in tension [15].

The diagrams Figures 17-19 show the real evolution of the Cement Paste compressive strength.

Figure 20 shows that, from 3 days to 28 days, the *Cement Paste* compressive strength evolution curve when we use T01 is up all the overs. Then, the appropriate rate of substitution of cement by Titanium Dioxide Nanotube to increase the *Cement Paste* compressive strength (with fly ash) is the T01.

Table 17. Comparison mechanical strength of cement paste specimen with fly ash at 3-day test.

Type	Flexural	Increase in Strength (%)	Compressive	Increase in Strength (%)
PO	6.5	-	28.9	-
T01	8	23.08	33.8	17.11
T1	7.2	10.77	30.5	5.66

Table 18. Comparison mechanical strength of cement paste specimen with fly ash at 7-day test.

Type	Flexural	Increase in Strength (%)	Compressive	Increase in Strength (%)
PO	7.9	-	43.2	-
T01	8.6	8.86	51.1	18.36
T1	8.9	12.66	47.0	8.96

Table 19. Comparison mechanical strength of cement paste specimen with fly ash at 28-day test.

Type	Flexural	Increase in Strength (%)	Compressive	Increase in Strength (%)
PO	9	-	55.8	-
T01	11.2	24.44	60.2	7.98
T1	10.5	16.67	58.2	4.39

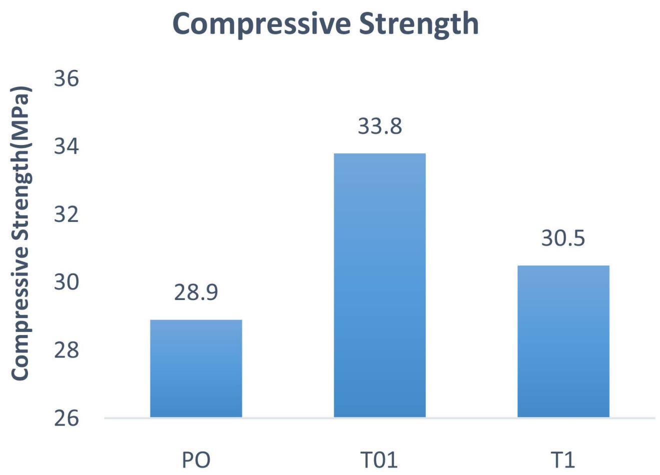


Figure 17. 3-day test cement paste specimen with fly ash.

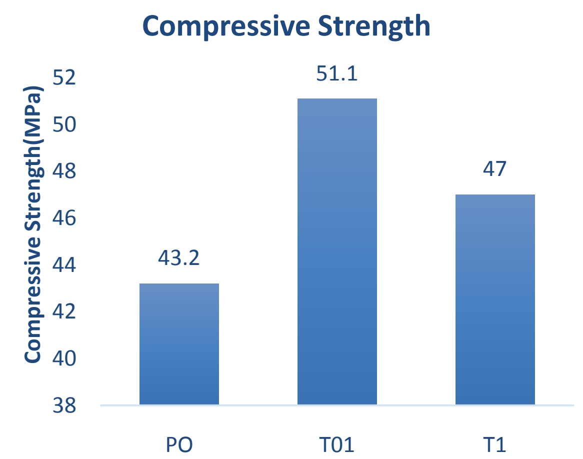


Figure 18. 7-day test cement paste specimen with fly ash.

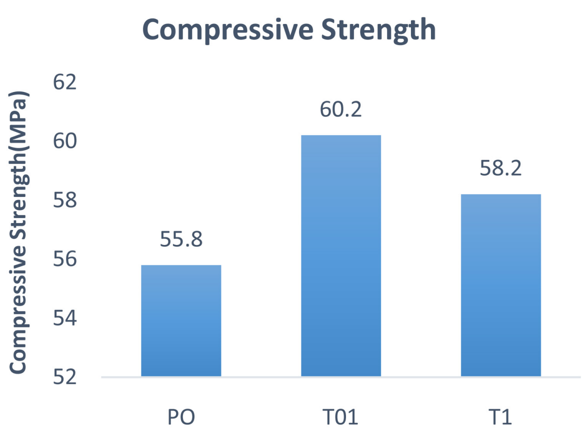


Figure 19. 28 days test cement paste specimen with fly ash.

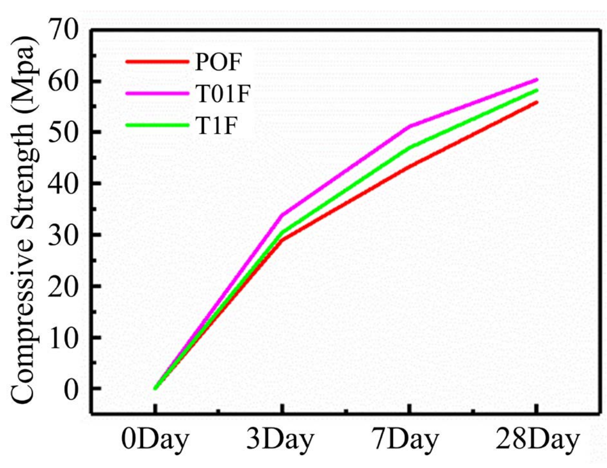


Figure 20. Change in compressive strength of cement paste specimen with fly ash from 3 days to 28 days.

4. Conclusion

The use of Titanium Dioxides offers interesting results when the purpose is to increase the compressive strength of the cementitious materials; specially the use of the T01 gives very good results in compressive strength increasing it for all the cases. However, using Titanium Dioxide Nanotubes provides a light negative effect on flexural strength. But using titanium dioxide and fly ash shows bad results either for compressive strength or flexural strength because of the lesser amount of binder comparing to the case of using titanium dioxide T01 alone.

Conflicts of Interest

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

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