

HIGH STRENGTH CONCRETE USING ULTRA-FINE TiO₂ AND BASALT FIBER- A STUDY ON MECHANICAL AND DURABILITY CHARACTERISTICS

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This paper reports the experimental investigation of strength and durability characteristics of fiber reinforced concretes made with silica fume (SF) and ultra-fine TiO₂ (UFTiO₂) as partial substitution for cement and basalt fibers added to it. The size of the ultra-fine TiO₂ used was obtained using zeta analyzer. M50 grade of concrete was used and the specimens were cast using cement blended with silica fume in a uniform proportion of 10% and ultra-fine TiO₂ added in 1%, 2%, 3% and 4%, by weight of cement. Basalt fibers were added in different levels at 0.5% and 1% by volume of concrete. The specimens were subjected to various mechanical tests such as compression, split tensile strength, test for modulus of rupture and impact strength. In addition, durability parameters such as deterioration, sorptivity, porosity and corrosion of rebar using half-cell potential were also performed and the results are reported. By comparing the results it was observed that the mechanical properties showed improvement with addition of basalt fibers and for an optimum use of UFTiO₂ up to 2% after which there was reduction in strength. Also, it was noted that, all the specimens with UFTiO₂ and basalt fiber added to them, possessed better strength characteristics.

Keywords: Ultra-fine TiO₂, Basalt fibres, Drophammer test, Deterioration, porosity, sorptivity, Half-cell potential method

1. Introduction

Concrete, though widely got appreciated for possessing various attractive characteristics, their weakness in resisting tension and strain are also to be noted. Methods to limit their brittleness are still under research and use of proper fibers in concrete can help improving such properties. Such fiber incorporated concrete have wide spread applications in civil engineering and military field [1, 2]. But since there are chances that the technical and economic efficiency of fiber reinforced concrete are affected by various parameters such as use of mineral admixtures and the physical characteristics of fibers, a proper case is required while choosing a suitable supplementary cementitious material and fibers. Also it has to be noted that though it is beneficial to use fibers in concrete, incorporating them in concrete can make the mixing process difficult which may result in voids paving way for permeation of water and other harmful weathering agents into it when the concrete is exposed to aggressive environment [3]. So, it is better to check the durability characteristics of the fiber reinforced concrete before using in field. Steel, glass, polypropylene and basalt fibers are the different accessible fibers that are used in a wide range [4, 5]. Afroughsabet and Ozbakkaloglu [6] have discussed the mechanical and durability characteristics of high-strength concrete with hybrid fibers using steel and polypropylene fibers. Sakthivel *et al* [7] have investigated the impact performance of Hybrid steel mesh and fiber reinforced cementitious composites and reported

that specimens involving hybrid components performed better than those with only steel mesh. Nia *et al* [8] had done experimental investigations using steel fibers and from their investigations it was noted that the compressive strength and tensile strength increased by 14.4% and 62% when steel fibers were used. Also, they mentioned that use of steel fibers assures better strength improvement than polypropylene fibers in most of the strength tests. Nili and Afroughsabet [9] reported that a maximum increase of 74% and 33% was attained in compressive and tensile strength respectively for the concrete with silica fume and 1% steel fiber than the specimens without silica fume addition. The results suggested that combined use of silica fume with steel fibers in concrete had shown significant improvement in flexural strength. In current research, in addition to strengthening concrete with fibers, mineral admixtures of ultra-fine size were also used. Use of nano sized mineral admixtures is common when high strength concrete is to be made and, the only drawback in such using of nano sized mineral admixtures is their higher cost. This can be reduced by using ultra-fine mineral admixtures. Karthikeyan and Dhinakaran [10-13] have done works with silica fume, metakaolin and ceramic powder in their ultra-fine state and also with UFTiO₂.

Norhasriet *al* [14] had mentioned in their report that the ultra-fine particles of nanosized mineral help in reducing the formation of micro pores and act as filling agents and are more useful in developing of ultra high performance

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Han *et al* [15] have discussed the mechanism lying behind the use of engineered cementitious composites. The above literature review reveals that more scope is available in using of nano sized mineral admixtures in concrete and many research works are being done to improve the strength of the concrete in an economic way. Also it is understood that concrete in addition to poses a higher strength should also exhibit some ductile properties. The present research aims to connect these two parameters by using ultra-fine mineral admixtures and fibers together in the concrete. Rather than using costly nano sized mineral admixtures, a high strength blended concrete mix was developed using silica fume, ultra-fine mineral admixtures and fibers. Ultra-fine TiO₂ was used in this regard in varying percentages ranging from 1% to 4%. The use of nano-TiO₂ in the construction field is noted from earlier research works, nano-TiO₂ is considered as an effective agent for developing self cleaning concrete [16]. Researchers have reported few favorable influences of nano-TiO₂ such as, improving of compressive and flexural strengths, enhancing of abrasion resistances [17, 18] which are beneficial for the construction industry. Han *et al* [19] reported that nano-SiO₂ coated TiO₂ (NSCT) possessed pozzolanic effect, helping the nano fillers in the cement matrix to achieve proper dispersion and leading to an increased flexural strength with 87% more than the reactive powder concrete without NSCT. Nazari and Riahi [20] used TiO₂ nano particles together with ground granulated blast furnace slag to study the physical, thermal and mechanical properties of self compacting concrete. The size of TiO₂ used in present research was not up to the limits specified for nano TiO₂, but the size was less than that of the other mineral admixtures used in this research, so it mentioned as ultra-fine in present work. To improve the pozzolanic property, silica fume was added in a constant percentage. Also, to improve the ductile properties, fibers were added. As more research resources were available using steel fibers, basalt fibers were chosen for the present work and were added in 0.5% and 1% of the volume of concrete.

2. Materials and Methods

2.1 Materials used

Ordinary Portland cement was used for this research. River sand and broken stones of size 12 mm were used as fine and coarse aggregate respectively. Basalt fibers in two percentages of 0.5% and 1% were added to concrete.

The silica fume used for the research was obtained from Oriental Exporters, Navi Mumbai, Maharashtra, India. As fly ash and silica fume

concrete.

were considered the most popular mineral admixtures among the pozzolanic materials for using in high strength concrete, it was decided to use silica fume for the present research. The size of TiO₂ was obtained using a zeta analyzer, it was found to be 794.5 nm. It is designated in this paper as ultra-fine TiO₂. The density was 3.97 g/cm³ which is reported as mentioned by the supplier. To obtain the workable concrete mix a super plasticizer, CONPLAST SP 430 was used.

2.2 Mix-proportion

A high strength concrete possessing a compressive strength of 50 MPa was designed and the mix proportion arrived was 1 : 1.04 : 2.13. ACI 211 [21] guide lines were adopted for the present work. A total of 15 mixes were made. M1, M2, M3 and M4 represent the mixes in which UFTIO₂ was used as replacement for cement in 1%, 2%, 3% and 4%. BAF0, BAF0.5 and BAF1 represent specimens with basalt fiber added in 0, 0.5 and 1% respectively thus making up a total of 12 mixes. In addition, two more mixes (MF0.5, and MF1) were made with basalt fiber added in 0.5% and 1% without adding any mineral admixture and including the control concrete, the total number of mixes used were 15. Except of Control, MF0.5 and MF1 all the combinations were made by partially replacing of cement with ultra-fine TiO₂ powder and silica fume, with basalt fibers added in 0.5% and 1% by volume of concrete. Table 1 shows the mix proportion details.

2.3 Testing methods

Table 2 shows the tests performed and the codal provisions adopted for casting and testing the specimens. The impact resistance of a specimen can be adopted by several methods such as: i) drop weight method, ii) instrumented impact, iii) explosive impact and iv) pendulum charpy/izod impact. Among the methods adopted for testing the impact resistance, drop weight method is preferred by many researchers due to its simplicity [29].

2.3.1 Durability characteristics

Meson *et al* [30] have presented a detailed review on corrosion of steel fiber reinforced concrete. In the present research, few methods which are simple for measuring corrosion and calculate deterioration and which are commonly used were adopted. The specimens were cured in aggressive acid (1% H₂SO₄) and base (5% NaCl) environment for 60 days after completion of their prescribed curing period of 28 days in normal water and the corresponding tests were carried out. The corrosion of rebar in the specimens was estimated using half-cell potential method as specified in ASTM C876 [25]. Other tests related to durability are listed in Table 2.

Table 1

Mix Id	C	M1	M2	M3	M4
Silica fume, replacement percentage	-	10			
UFTiO ₂ , replacement percentage	-	1	2	3	4
Basalt fiber, replacement percentage		0.5 and 1			
Fine aggregate, kg/m ³	544.18				
Coarse aggregate, kg/m ³	1113.84				
Water, l/m ³	182.9				
Superplasticizer	1.5 l per 100kg of cement				
w/c	0.32				
Mix ratio	1:1.04:1.3				

Table 2

Testing Methods				
Tests Performed	Nature of test	Code adopted	Specimen type	Size mm
Strength characteristics				
Compression	Strength	BS 1881-108 ^[22]	Cube	100x 100 x 100
Split-tensile		ASTM C496 ^[23]	Cylinder	100(d)x200 (h)
Impact		ACI 544 ^[24]	Cylinder	152(d) x 63.5 (h)
Durability				
Acid /Chloride attack	Deterioration	BS 1881 ^[22]	Cube	100 x 100 x 100
Half-cell potential	Corrosion	ASTM C876 ^[25]	Cylinder	60 (d) x 120 (h)
Sorptivity	Water absorption	ASTM C1585 ^[26]	Cylinder	100 (d) x 50 (h)
Porosity	Volume of pores	ASTM C642 ^[27]		
Structural Behaviors				
Flexure	Modulus of Rupture	ASTM C78 ^[28]	Prism	100 x 100 x 500

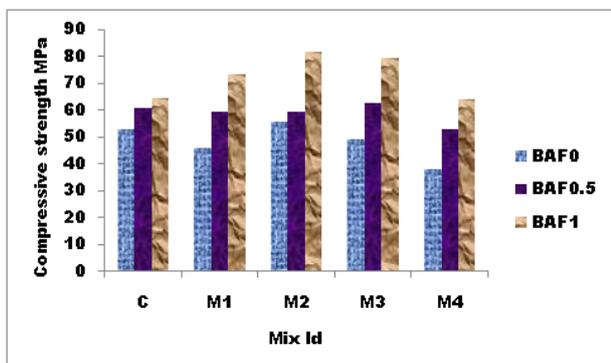


Fig. 1 - Compressive strength test .

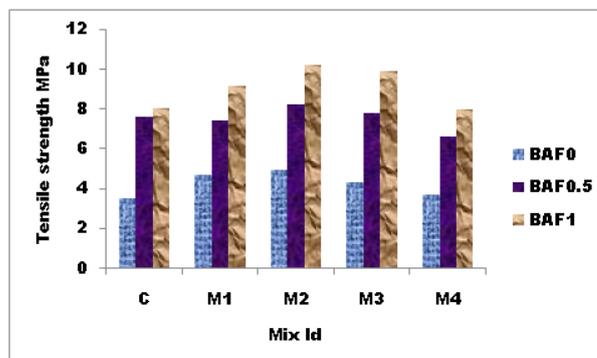


Fig. 2 -Split tensile strength.

3. Results and Discussion

3.1 Compressive and Tensile Strength in Normal Water Curing

Figures 1 and 2 illustrate the compressive and tensile strength results of the mixes compared with the control concrete. M2 has shown the highest compressive strength among all combinations and M4 has registered the least strength. Except M4 all specimens have shown strength either equal or more than the mean target

strength required for M50. M1 and M2 possessed gradual strength increase after which the strength got decreased reaching drastically for M4. Though there is a strength decrease after M2 in both compressive and tensile strength for specimens with basalt fiber, all were found to be more than the desired design strength of M50. Addition of UFTiO₂ more than the required quantity affected the results badly. It could be noticed that the strength reduction has started from 3%

replacement of UFTiO₂. M3 specimens containing basalt fibers added in 0%, 0.5% and 1% all

possessed less values than the specimens categorized under M1 and M2 and for 4% replacement the specimens faced further reduction. The reason for less strength of this concrete is due to the addition of UFTiO₂ in a larger replacement level of 4% which might have dissolved. So, it is understood once again that a small increase in the quantity of UFTiO₂ cannot help in filling up pores but may end up in dissolving thereby affecting the binding properties of the concrete leading to less strength. M4 which had the larger replacement level of 4% UFTiO₂ was affected by this and showed less strength both in compression and split tensile tests; however the arresting of cracks by fibers have taken care that the strength has not reached below the target mean strength. Nazari and Riahi [20] in their report had discussed the effect of nano-TiO₂ using in self compacting concrete with ground granulated blast furnace slag and reported that the compressive strength of the specimens increased by nano TiO₂ using up to 3% and then decreased strength when higher levels were used. The authors stated that the reason for such decrease in strength may be due to the over dosing of nano TiO₂ than the required amount which did not combine with the liberated lime during hydration process and had lead to the excess silica leaching out weakening the concrete. This was found to be true with the present research as the strength values increased for a replacement level of 2% and later started showing signs of decreased strength.

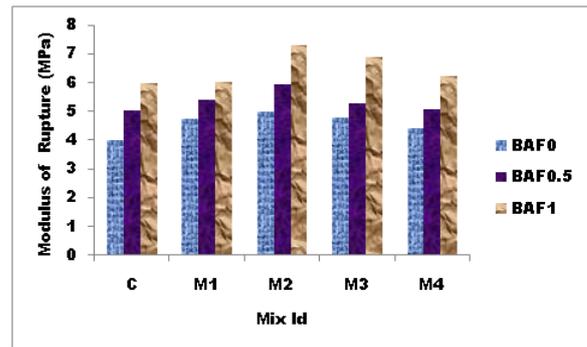


Fig. 3 - Flexural strength.

3.2 Flexural Strength

Figure 3 shows the flexural strength developed by the specimens for different combinations with UFTiO₂ and basalt fibers. Control specimens possessed the lowest modulus of rupture value. Much difference was not noticed in the flexural strength values of other non-fibered specimens, they withstood up to the breaking load and avoided sudden brittle failure as the UFTiO₂ has provided a better packing up of pores in the concrete. All the fibered specimens have shown a reasonable increase in the flexural strength when compared with the non-fibered specimens. From Figures 1 and 2 it was apparent that M2 specimens have shown better compressive and tensile strength characteristics. It is once again proved in flexural strength results too. The fibered M2 specimens, namely BAF0.5 and BAF1 have registered a maximum flexural strength of 5.94 MPa and 7.3 MPa respectively. By comparing of

Table 3

		Impact strength				
Parameters studied	Addition of fibers	Mix id				
		C	M1	M2	M3	M4
No. of blows required for I crack	0	25	21	29	26	20
Energy kN-m		1218	1023	1412	1266	974
No of blows required for failure		30	31	37	34	26
Energy kN-m	0.5%	1461	1510	1802	1656	1266
No. of blows required for I crack		28	36	42	39	34
Energy kN-m		1264	1753	2045	1899	1656
No of blows required for failure	1%	34	45	54	48	41
Energy kN-m		1656	2192	2630	2338	1997
No. of blows required for I crack		35	41	54	50	38
Energy kN-m		1705	1997	2630	2435	1851
No of blows required for failure		44	52	66	64	49
Energy kN-m		2143	2532	3214	3117	2386

the modulus of rupture results with the control specimen it was found that the flexural strength

mix. Control concrete registered a higher deterioration with the strength values being 30.69

has increased to a maximum of 48.5 % for 0.5% basalt fiber added specimens and 82.5% for 1% basalt fiber added specimens.

3.3 Impact Strength

Table 3 shows the impact strength results. A considerable increase in strength was observed when fibers were used in the mix. Control specimens have crushed and failed badly with minimum number of blows. Non-fibered specimens, tightly packed with SF and UFTiO₂ resisted the impact to some extent and later started showing development of cracks and failed by crushing. The specimens mixed with fibers have shown better resistance to impact. In fact the resistance increased at increase of the fibers addition from 0.5% to 1%. But the addition of fibers restricted with 1% as excess of fibers addition will affect the compaction, fresh properties and also will add unnecessary weight to the specimens. Though all fibers mixed specimens have performed well compared to the non fibered specimens, it is the case of M2, which had consistently shown good compressive, tensile and flexural strength, showed better resistance to impact resistance also.

3.4. Deterioration of Non Fibered Specimens

Specimens exposed to acid environments are attacked by SO₂ and other components formed in the atmosphere damp conditions. More significantly, the sulphur compounds present are more responsible for deterioration as they react with both calcium hydroxide and C₃A forming CaSO₄ and Calcium sulpho aluminate (ettringite), both will usually occupy a larger volume than the parent compounds leading to disruption of concrete making it weak and porous.

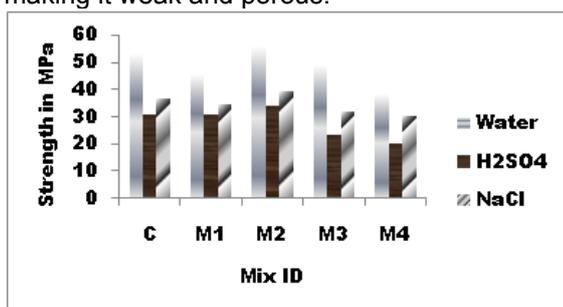


Fig.4 -Strength of specimens with 0% Basalt fiber in aggressive environment.

Figure 4 shows the compressive strength results which indicate that among the various mixes exposed to normal water curing and aggressive environment, M2 has shown a higher compressive strength of 55.55 MPa irrespective of the curing environment i.e normal or aggressive. The strength later showed a gradual decrease for M3 and reached a lower value of 37.98 MPa for M4

MPa and 36.35MPa in H₂SO₄ and NaCl environment respectively. M1 mix showed a deterioration differing from the control mix only marginally indicating that the small replacement level of 1% UFTiO₂ though used with 10% silica fume was insufficient to improve strength characteristics. The deterioration rate of M2 mix was less in both acid and base environment indicating that the replacement level was sufficient to improve the mechanical properties. The mixes made with higher UFTiO₂ content registered lower values with, M4 showing a higher deterioration both in acid and base environment. The reason for this high deterioration is that part of the UFTiO₂ particles have dissolved in the aggressive acid environment. Silica fume sustained their strength to some extent but as a major quantity of cement (3% and 4%) was replaced by UFTiO₂ in M3 and M4 mixes, the required binding properties were lacked leading to a more deteriorated stage. Results of Karthikeyan and Dhinakaran [10] indicate that using ultra-fine silica fume in aggressive environment had helped to improve the strength but here due to combined use of SF and UFTiO₂ the response was mixed with strength gain in some mixes and strength loss in other. Also here a uniform replacement level of 10% was adopted for all the mixes and so the variation of strength in the non-fibered specimens depends fully on the replacement levels by UFTiO₂ only. Replacement by UFTiO₂ in a very small level as 1% was not sufficient to fill the pores in concrete and a slightly larger increase was also not provided satisfactory results as they started dissolving especially when exposed to aggressive environment. From the results it is understood that a moderate replacement level could fetch better improvement in strength.

3.5 Deterioration of Specimens with 0.5% and 1% Basalt Fiber

Figures 5 and 6 illustrate that addition of basalt fibers indeed improved the strength properties. All the specimens possessed strength almost equal to or higher than the control specimen proving that addition of basalt fibers has some impact. MF0.5% and MF1% specimens cast without any mineral admixture and added only with basalt fibers, cured in water, developed strengths of 60.8 MPa and 64.32 MPa respectively. M2 has registered a higher compressive strength of 65.6 MPa and 81.84 MPa with both 0.5% and 1% basalt fibers added. M4, which has shown a less strength of 37.98 MPa without any addition of fibers, has shown a strength of 52.64 MPa and 63.88 MPa when basalt fibers were added to it.

From the results it is understood that the deterioration percentage of specimens exposed to bases is less in general when compared with the

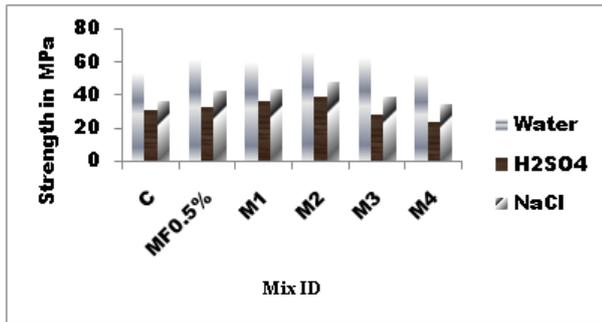


Fig.5 -Strength of 0.5% Basalt fiber added specimens in aggressive environment.

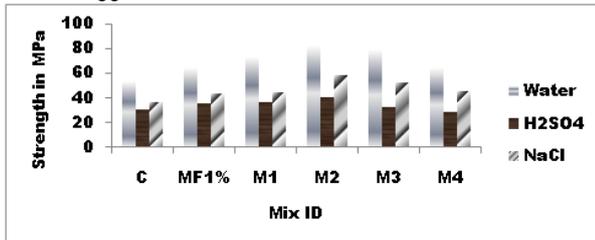


Fig.6 - Strength of 1% Basalt fiber added specimen in aggressive environment.

specimens exposed to acid. The reason being the calcium hydroxide in cement, on reacting with salts or other bases, can lead to the formation of gypsum, brucite and aragonite. They are capable of acting as a temporary barrier or a protective layer and can prevent further ingress of the salt water [31]. However on continuous exposure the protective layer may degrade leading to penetration of salt and the concrete becomes porous. In the present work also, the same trend followed. Control specimens showed an assured strength of 30.69 MPa and 36.35 MPa in the acid and base medium respectively. It can be observed that the addition of 0.5% of basalt fibers improved the strength, to 32.65 MPa and 42.36 MPa respectively. Similar trend was observed when 1% basalt fiber was added as the strength of MF1% specimens has improved to 35.4MPa and 43.59 MPa. A minimum deterioration of 8% and 11.73% was observed for M2 in acid and base medium when the fibers were added in 0.5%. M4 specimens possessed higher deterioration rate of 17.16% and 11.26% after subjecting to curing in H_2SO_4 and NaCl respectively for 60 days. The variations in the results were mainly due to the changes made in the replacement levels of $UFTiO_2$ as SF was added in a constant replacement level of 10%. At higher replacement levels, $UFTiO_2$ was unable to fill the pores properly due to which the deterioration of the specimens M3 and M4 increased gradually. Since basalt fibers were used, the specimens have not faced the problem of corrosion of fibers in aggressive environment.

3.6 Corrosion resistance

On observing Figure 7, it is understood that the rebar of control specimen has corroded

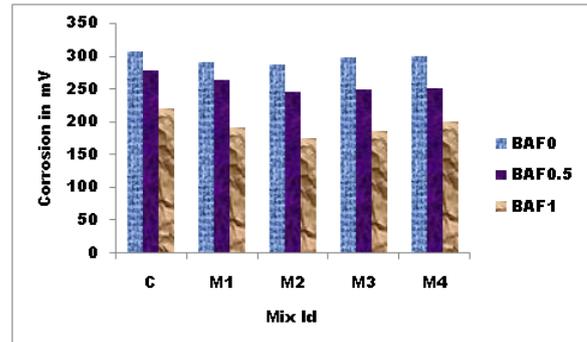


Fig.7 - Corrosion of the specimens in aggressive acid medium.

most, registering the highest potential measurement of -307 mV among all specimens. ASTM C876 [26] guide lines interpret the above range of potential as a high risk corrosion state. The value of the measured potential between the reinforced bar and the reference electrode depends on the type of reference electrode used. In the current research, saturated calomel electrode was used as reference electrode. The corrosion values of all specimens thout fibers shown in Figure 7 indicate that use of a minimum quantity of ultra-fine material failed to fill the pores completely when the specimens were exposed to aggressive environment. Even M2 which has possessed higher strength values in normal water have shown poor corrosion resistance. Among the non reinforced specimens, M2 had shown a slight improvement in corrosion resistance.

The corrosion behaviour of the BF0.5 specimens with 10% SF, 0.5% basalt fibers and $UFTiO_2$ varying from 1% to 4% proved that a very small addition of 0.5% basalt fibers, have improved the corrosion resistance of the specimen. The results indicate that by use of $UFTiO_2$ in a nominal level and with the help of fibers and also with the support of a pozzolanic agent, the corrosion of rebar in specimens can be controlled. The fibers particles make the concrete denser which results in better corrosion resistance. This is confirmed on comparing with other researchers reports; Karthikeyan and Dhinakaran [13] had stated by their experimental investigations that use of ultra-fine mineral admixtures in higher dosages affected the strength and durability.

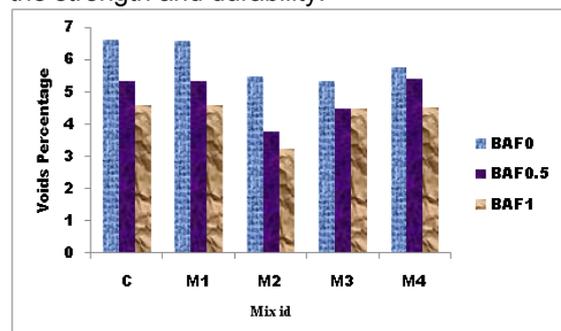


Fig.8 - Percentage of voids.

Table 4

Specimen Replacement levels	Sorption Coefficients		
	Specimens with 0% Basalt fiber	0.5% Basalt fiber added specimen	1% Basalt fiber added specimen
CONTROL	0.025	-	-
MF0.5	-	0.022	-
MF1	-	-	0.024
M1	0.023	0.02	0.021
M2	0.018	0.015	0.015
M3	0.023	0.027	0.025
M4	0.023	0.029	0.026

3.7. Sorptivity

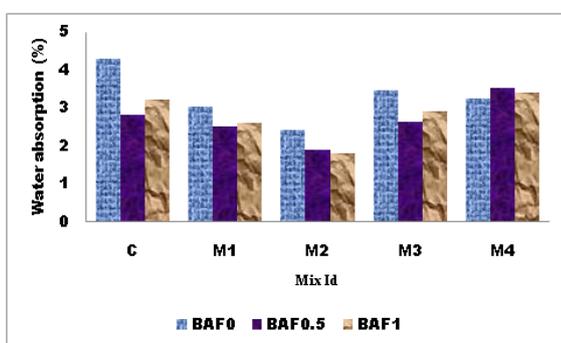


Fig.9 - Water absorption.

Figure 9 illustrates the water absorption characteristics. On all the three cases, it was observed that the control specimen registered high water absorption of 4.28%. M2 specimens possessed low water absorption values of 2.4%, 1.9% and 1.79%. Increasing the fibers percentage in concrete helped it to become denser and improved the resistance towards water penetration. The water absorption showed a gradual decrease up to 2% replacement of cement with UFTiO₂ and then showed increased values. Increasing the quantity of ultra-fine mineral admixtures found no use in improving the durability properties. Razak *et al* [32] in their experimental investigations have reported that water absorption of 2.9% and 4.8% can be obtained when SF was used as mineral admixture. Karthikeyan and Dhinakaran in their report [10] mentioned that water absorption less than 1.5% was observed for specimens with 9.5% silica fume, 0.5% UFTiO₂ and 0.5% steel fiber. Mohsaniet *al.* in their report [33] mentioned that the use of nano TiO₂ together with rice husk ash showed improved durability properties. The authors further reported that the use of such nano materials yielded a more packed pore structure and the permeability decreased with increase of nano TiO₂ and rice husk ash content.

In the present work, lowest water absorption of 1.79% was observed for specimens with 10% silica fume, 2% UFTiO₂ and 1% basalt fibers. The sorptivity values shown in Table 4 also reflected the water absorption characteristics illustrated in Figure 9. Here too, M2 showed less sorption coefficient of 0.018, 0.015 and 0.015 for different fiber reinforcement levels namely 0% (BAF0), 0.5% (BAF0.5) and 1% (BAF1) respectively. The sorption values are inversely proportional to the strength values and it is proved here as M2 has registered higher compressive strengths.

3.8. Porosity

The apparent porosity values of the specimens shown in Figure 8 reflect the water absorption and sorptivity results. Control specimen shown high porosity values of 6.6%. The voids percentage of all non-fibered specimens were found to be higher and were nearer to the value of control specimen indicating that using ultra-fine minerals alone is not sufficient for filling up the pores developed in concrete, but the values did not show any drastic difference. Less voids percentage was observed for M3 specimen among all the non fibered specimens. A drastic difference can be observed among the voids percentage of non fibered specimens and those with fibers. Khotbehsaraet *al* [34] performed experimental investigations for studying the durability characteristics of self-compacting cement mortars using nano particles of ZrO₂, SnO₂, CaCO₃ in different dosages together with fly ash and reported that use of the nano particles improved the durability properties, diminishing the porosity and chloride permeability. Sharmila and Dhinakaran [35] in their experimental works reported that using ultra-fine GGBS had improved the sorptivity characteristics of high strength concrete. In the present work also the durability properties were better when ultra-fine mineral

admixtures were used. M2 concrete with fibers and 2% UFTiO₂ have shown less percentage of voids of both 0.5% and 1% fiber content specimens. It is understood from the results that when the concrete is made denser less will be the voids percentage.

4. Conclusions

The following were the conclusions arrived from the experiments conducted:

- All specimens have crossed the mean target strength in fibers reinforced concrete specimens, in particular, M2 mixes performed well in all normal water and aggressive acid and base environments; they registered higher strengths both in non-fibered and fiber reinforced concrete.
- The strength was improved gradually when UFTiO₂ was used in 1% and 2% and started decreasing for 3% and 4% irrespective of fibers addition; excess replacement of cement with UFTiO₂ failed to provide proper bonding.
- The combined action of the pozzolanic reaction by silica fume and filling up of pores both by silica fume and UFTiO₂ and the arresting of cracks by fibers, all helped in improving of the strength.

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