



Influence of textile effluent on the Reaction, Structure and Properties of Fly Ash Concrete

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ABSTRACT

The transformation of industrial wastes into useful products attracts more researches to work upon for sustainable of natural resources. In this paper, industrial wastes such as treated textile effluent and fly ash were used in the preparation of plain cement concrete with the intention of reducing the environmental pollution caused by these materials. The partially treated textile effluent was used as mixing water and fly ash was used as 30% partial replacement by weight of cement in concrete. The textile effluents were collected after anaerobic treatment and tertiary treatment from textile industry. Class F Fly ash was collected from Mettur thermal power plant. The physical and chemical properties of treated textile effluent were studied. The control concrete was prepared with potable water available in the laboratory. Experimental test was performed for compressive strength of concrete at 28 days. The powdered concrete samples were examined through infrared spectroscopy, X-ray diffraction and scanning electron microscopy to study the microstructure of concrete. The compressive strength test results revealed that anaerobic effluent water (AAE) concrete accomplished higher compressive strength than control concrete. This was also in affirmation with microscopic analysis in which the formations of hydration products were well established when compared to control concrete.

Keywords: Treated textile effluent, Fly ash, Portlandite, CSH gel

Academic Discipline and Sub-Disciplines: Civil Engineering, Chemistry

Subject Classification: Environment, Waste management

Type: Experimental analysis

1. INTRODUCTION

Industrialization refers to the process of change in technology and economic development that has the integral growth of all industrial and agricultural sectors which can be attained by making use of all our natural resources. The constant utilization of raw materials leads to depletion of natural resources. In order to attain sustainability, several new techniques have to be developed to reuse waste materials in industries. The textile units use a variety of dyes, chemicals and other hazardous materials to impart desired feature to the fabric. The significant quantity of effluents generated by these industries are mostly unsuitable for further use and if disposed of into natural streams without proper treatment, causes many environmental problems and affects aquatic life. In India about 2324 textile industries were established and out of which 31% of units were situated in Tamilnadu. The effluent treatment process incorporated by several industries includes primary treatment, secondary treatment, tertiary treatment and some advanced methods such as adsorption, ion exchange, reverse osmosis, ultrafiltration, nanofiltration and ozonation [1]. In spite of strict rules and regulations laid by state government, several industries were letting their effluents in natural streams which acting as major reason for water pollution.

The Industrialization leads to the dynamic growth in construction industry. The construction sector carries out a high consumption of resources such as materials, energy and water [2]. Therefore, it is important to analyze the reuse of materials and different types of waste in building construction without affecting their technical aspects in order to attain sustainability. Many researches had attempted to integrate the textile industry waste with construction industry. Aspiras et al, used textile cutting in cement as binder and reported that this can be used for walls, ceilings or as an economical alternative concrete block [3]. Youjiang Wang used recycled fibers from textile waste in concrete and soil reinforcement and reported that the performance of concrete had been increased and there was a significant improvement in soil behavior under triaxial loading condition [4]. Many researchers studied the properties of concrete made with waste water. Concrete specimens were made with effluents from heavy industry, palm oil mill and domestic sewage. It was observed from the results that a sample attained 9.4% increase in compressive strength than the normal concrete [5]. Fresh water was totally replaced by waste water released from polyvinyl acetate resin manufacturing plant in concrete and it was reported that there is a slight to moderate increase in compressive strength than the control concrete [6]. The excessive amount of impurities present in the mixing water may not only affect the strength of concrete but also affects the setting time, corrosion of reinforcing steel, volume instability, reduced durability and may cause efflorescence. Therefore, optimal limits may be set on chlorides, sulphates, alkalis and solids in mixing water [7]. Some impurities may have adverse effect of durability properties even though they have no impact on strength and setting time [8].

The use of fly ash as a partial replacement of cement not only utilizes waste produced by the thermal power plants, but most importantly reduces carbon dioxide emissions arising from cement making. Saraswathy et al [9] conducted different activation techniques to improve the reactivity of fly ash and confirmed that 20-30% replacement of chemically activated fly ash showed better strength. For most structural applications except mass concrete, the fly ash content is often reduced to



20-30% [10]. With this background, in this research 30% replacement of cement by fly ash is adopted for the enhancement of mechanical and durability properties of concrete.

Several authors studied the microscopic aspects of concrete and the products of hydration with and without admixtures [11, -15]. Ping Yu et al [16] studied the structure of calcium silicate hydrate and reported that, the near, mid, and far-IR spectroscopic data for a series of single-phase C-S-H samples confirmed the similarity of the structure of C-S-H with Tobermorite. Portlandite (calcium hydroxide) is a major phase of cement hydration and occurs in variable sizes and shapes including platy hexagonal crystals and sheet-like masses, depending on the orientation. Ettringite is a primary product of the reactions between calcium aluminates and the sulfate phases in cement. It has a characteristic acicular shape [17]. The presence of calcite crystals by the appearance of crystalline matrix was viewed through XRD techniques in the microstructural analysis of concrete [18]. The microstructure of hydrated Portland cement pastes varies with factors such as the chemistry and fineness of the cement, the water to cement ratio, the use of admixtures, variations in mixing procedures and variations in hydration conditions. The mixing water involves in the chemical reaction and it has been estimated that on an average 23% of water by weight of cement is required for chemical reaction with Portland cement [13]. Therefore, the quality of water used in the mixing of concrete also plays a vital role in the mechanical and durability properties of concrete.

Thus, the primary aim of this work is to investigate the effect of partially treated textile effluent as mixing water in concrete. The techniques such as IR, XRD and SEM were used in this study. The microstructural aspects of concrete with the incorporation of fly ash in this concrete were also studied.

2. MATERIALS AND METHODS

2.1 Materials

The ordinary Portland cement of 53 grade conforming to IS 12269-1987 was used [19]. The class F fly ash used in the experiment was obtained from Mettur Thermal Power Plant. The locally available clean river sand conforming to IS 383-1970 [20] confining to Zone II was used. Crushed granite stones of 20 mm size graded coarse aggregate were used. The effluents were collected after anaerobic treatment (AAE) and tertiary treatment (TTE) from a textile unit located in Karur. The characteristics of effluents were given in Table (1).

The concrete was made with potable water (PW), AAE and TTE. The other groups of samples were made with these effluents and 30% flyash as partial replacement for cement (PWF, AAEF and TTEF). The mix design adopted for casting concrete was 1:1.49:2.73, W/C = 0.5.

Table 1. Characteristics of the potable water and treated effluents

Parameter	Concentration			Tolerable limits	Reference
	PW	AAE	TTE		
pH at 30 deg C	7.46	7.5	11.81	≥6.0	IS 456:2000
Total solids, ppm	600	10816	11992	50,000	ASTM C1602
Sulphates as (SO ₄),ppm	200	1070	1250	3000	ASTM C1602
Chlorides as Cl ⁻ , ppm	666	4900	5450	1000	ASTM C1602
Total Alkalinity	30	330	260	600	ASTM C1602
Calcium as Ca , ppm	-	230	530	2000	BSI
Nitrates as NO ₃ ⁻ , mg/l	-	3.5	12.7	500	BSI
Manganese (Mn),mg/l	-	1.07	0.13	500	Mindness (1981)

2.2 Methods

The concrete cubes of size 150 mm x 150 mm x 150 mm were used for determining the compressive strength at the age of 28 days. The tested concrete samples were powdered and subjected to different microscopic studies such as FT-IR, XRD and SEM.

FT-IR is one of the powerful techniques normally used for molecular characterization and also it has been found to be very useful in delineating the complex chemistry involved in the hydration of cement [21]. In the present study, mid IR radiation (4000-400 cm⁻¹) is used to study the fundamental vibrations and associated rotational vibrational structure of the concrete samples. At the age of 28 days, all the concrete samples were powdered and subjected to FT-IR analysis and the spectrum was taken in the range of 4000-450 cm⁻¹.

X-ray diffraction (XRD) is used to identify the polycrystalline phases of cement and hardened cement paste through the recognition of the X-ray patterns for each of the crystalline phases [22]. The crystalline hydrates such as



1 ettringite, monosulphate hydrate, portlandite ($\text{Ca}(\text{OH})_2$), calcium carbonate, and calcium silicate hydrate, as well as the
2 various calcium silicate, calcium aluminate and aluminosilicate phases present in the sample can be detected by XRD.
3 The present work aims to identify the crystalline phases present in the samples PW, AAE, TTE, PWF, AAEF and TTEF. X-
4 ray diffraction meter (XRD-6000 SHIMADZU) was used in this research.

5 The Scanning Electron Microscope (SEM) is a powerful instrument which permits the characterization of
6 heterogeneous materials and surfaces. Samples were completely dried at room temperature, and then examined at
7 accelerating voltages ranging from 30 to 35 kV by a SEM (JSM-6390 JEOL).

8

9 3. RESULTS AND DISCUSSION

10 3.1 Compressive strength

11 The compressive strength results were given in Table (2). From the compressive strength results it can be understood that
12 the AAE and AAEF samples exhibits better strength than control concrete made with potable water in both type of
13 samples.

14

Table 2. Compressive strength of concrete

Mix Type	Compressive strength of concrete at 28 days
PW	39.8
AAE	54.2
TTE	46.2
PWF	38.6
AAEF	53.4
TTEF	46.8

15

16 3.2 Infrared Spectroscopy (IR)

17 Table (3) reports the assignments of IR active bands present in the spectra based on the previously published works. Fig
18 (1) shows the IR spectrum of PW, AAE, TTE, PWF, AAEF and TTEF. The region in the range $400\text{--}1500\text{ cm}^{-1}$ represents
19 the silicate region [14]. The presence of peak at 458 cm^{-1} (approx) indicates the deformation of SiO_4 tetrahedra. Also, the
20 presence of anhydrous calcium silicate is confirmed by the presence of presence of peak at 458 cm^{-1} [23]. PW and TTE
21 samples shows a peak at 686 (approx) which is absent in the sample AAE. The band in the range of $800\text{--}1200\text{ cm}^{-1}$
22 represents the asymmetric and symmetric stretching vibrations of Si-O bond. The bands at $994, 1006$ and 1008 cm^{-1} are
23 due to the silicate SiO stretch /sulphate SO stretch, which mainly represents the C-S-H gel and other silicate phases
24 [14,24]. The bands in the range of $1400\text{--}1500\text{ cm}^{-1}$ correspond to the asymmetric stretching of CO_3^{2-} . From Fig (1) it can
25 be shown that all the samples exhibit bands at 1422 cm^{-1} which corresponds to the asymmetric stretching of CO_3^{2-} .

26 The bands appeared above 1600 cm^{-1} indicates the water region [6]. The band at 1640 cm^{-1} approximately
27 is due to H-O-H bending vibration of molecular H_2O which is observed in all the three samples. The observation of band
28 at $2800\text{--}3700\text{ cm}^{-1}$ is due to the stretching vibrations of O-H groups in H_2O or hydroxyls with a wide range of hydrogen
29 bond strengths [16]. The acute band observed at 3637 cm^{-1} in PW and AAE indicates the formation of calcium hydroxide.
30 The comparisons of spectrum obtained for cement pastes without fly ash and with fly ash are given in table 4 and 5
31 respectively. In comparison to the spectrum of PW (Table 4), almost all of the bands in the investigated spectra (AAE and
32 TTE), are shifted to the lower frequency region. This may be due to the formation of silicates of different stoichiometry.
33 The strong band with a maximum at 776 cm^{-1} could indicate the presence of cristobalite with a well-formed crystal lattice
34 [16].

35 The spectra obtained for the samples containing fly ash exhibits almost same values of peak wave number but
36 with different intensities. The intensities of different wave numbers were less for AAE and TTE when compared to PW in
37 the samples without fly ash. But, in contrary to this trend, the observed bands of AAEF and TTEF shows higher intensity in
38 comparison to PWF. The wave number corresponding to ettringite is $3420\text{--}3635\text{ cm}^{-1}$ [25]. The presence of ettringite for
39 AAEF and TTEF is observed to be twice intense as that of PWF. When compared to PWF, the observed band for C-S-H
40 gel is three and four times as intense for AAEF and TTEF respectively. The blending materials such as fly ash, silica fume
41 and other pozzolanic materials are added to cement to overcome the bad effects of $\text{Ca}(\text{OH})_2$ [26]. This can be rightly
42 justified from the spectra that the peak corresponding to calcium hydroxide has less intensity in the flyash samples. The
43 intensity and nature of spectra obtained in the samples without flyash and with flyash (Table 4 and 5) can be compared. It
44 can be clearly understood that the decreasing nature of frequency is observed in the almost all the samples of the group
45 with flyash. This may be due to the fact that the pozzolanic reaction will be slower in concrete with flyash than the concrete
46 without flyash.

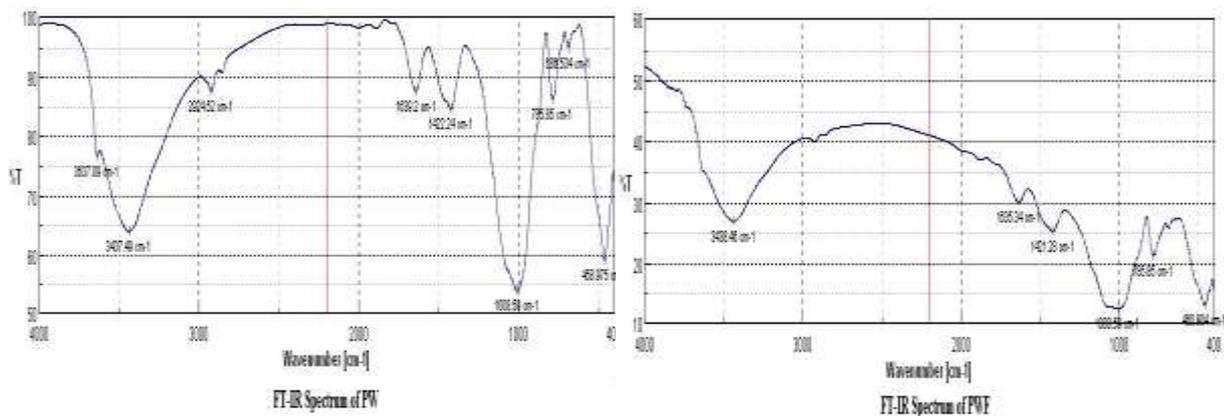
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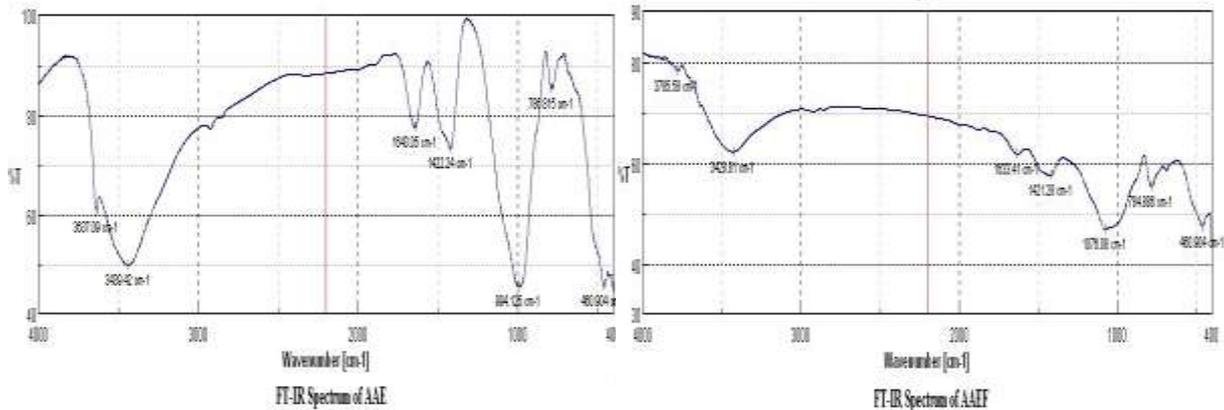
1 **Table 3. Assignment of bands**

Wave number cm^{-1}	Assignment	References
800-1200	Asymmetric and symmetric stretching vibrations of Si-O bond	[16,14]
660 (Approx)	Si-O-Si bending vibration	[16]
400-500	Deformation of SiO_4 tetrahedra	[16]
458-464	Anhydrous calcium silicate	[24]
1400-1500	Asymmetric stretching of CO_3^{2-}	[16]
875 (Approx)	Out of plane bending of CO_3^{2-}	[16,14]
1640,1622.1	H-O-H bending vibration of molecular H_2O	[16,14,24]
2954, 2952, 2970, 2966	Bound water HOH bend	[24]
2800-3700	Stretching vibration of O-H groups in H_2O or hydroxyls with a wide range of hydrogen- bond strength	[16,14]
3642 (Approx)	Calcium Hydroxide	[23]
3420 and 3635	Ettringite	[25]

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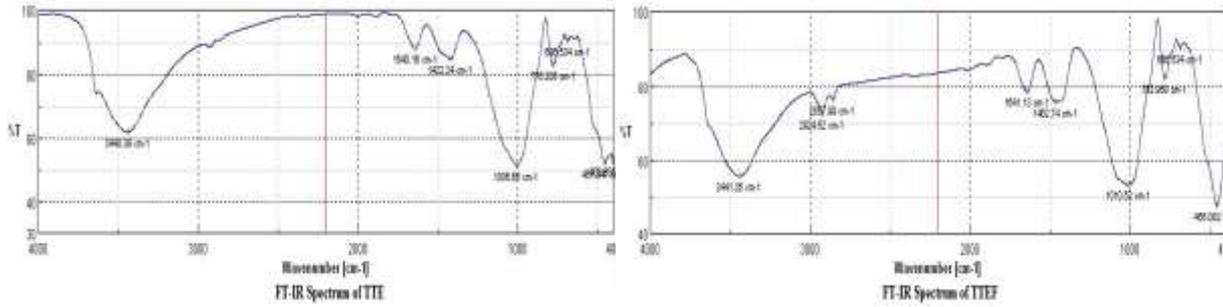


Fig 1 – FT-IR Spectra of concrete samples

Table 4 - Comparison of spectrum of cement pastes without flyash

PW			AAE			TTE		
Wave number cm ⁻¹	Intensity		Wave number cm ⁻¹	Intensity		Wave number cm ⁻¹	Intensity	
	%	Nature		%	Nature		%	Nature
-	-	-	-	-	-	407.87	53.38	M
458.975	59.12	M	460.904	45.70	M	457.04	52.10	M
686.534	94.94	VS	-	-	-	686.534	90.51	VS
785.85	86.28	S	786.815	85.42	S	776.208	83.03	S
-	-	-	994.125	45.68	M	-	-	-
1008.59	53.96	M	-	-	-	1066.66	56.19	M
1422.24	84.63	S	1422.24	73.42	S	1422.24	84.83	S
1639.2	87.70	S	1643.05	77.73	S	1640.16	88.17	S
2924.52	87.55	S	-	-	-	-	-	-
3437.49	64.04	S	3439.42	49.99	M	3440.39	62.12	S
3637.09	76.48	S	3637.09	60.68	S	-	-	-

Table 5- Comparison of spectrum of cement pastes with fly ash

PWF			AAEF			TTEF		
Wave number cm ⁻¹	Intensity		Wave number cm ⁻¹	Intensity		Wave number cm ⁻¹	Intensity	
	%	Nature		%	Nature		%	Nature
460.904	13.15	W	460.904	47.46	M	456.082	47.80	M
-	-	-	-	-	-	686.534	90.32	VS
785.85	21.27	W	784.886	55.52	M	782.958	82.23	S
1000.59	12.63	W	1076.08	46.97	M	1010.52	53.38	M
1421.28	25.27	W	1421.28	57.59	M	1462.74	75.86	S
1635.34	30.11	M	1633.41	61.78	S	1641.13	78.59	S
-	-	-	-	-	-	2857.99	76.59	S
-	-	-	-	-	-	2924.52	72.68	S
3438.46	26.97	W	3428.81	62.37	S	3441.35	55.74	M
			3785.58	78.42	S			

Note: W-Weak, S- Strong, M-Medium, VS- Very strong



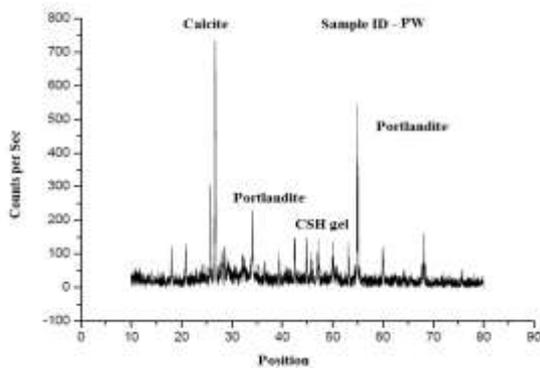
1 3.3 X-ray diffraction (XRD)

2 The XRD spectra obtained for the samples PW, AAE and TTE are given in Fig (2). The main phases observed for
3 PW (Control concrete) are calcite (CaCO_3), portlandite (Ca(OH)_2) and CSH gel. For AAE sample similar phases were
4 found, however Ca(OH)_2 and calcite were found in less quantity. The CSH gel was detected in XRD, whereas this is also
5 in accordance with IR results. In addition to the above mentioned products, calcium sulphate dihydrate was observed in
6 AAE. In similar to PW and AAE, calcite was observed in TTE. In TTE, C_4AH_x and calcium aluminate was found to be
7 different from the sample PW. The absence of portlandite in TTE is in agreement with IR results. It can also be noted that
8 the intensity of portlandite is less in AAE when compared with PW. Calcium langbeinite is found to be a major component
9 in all the fly ash samples. Calcium langbeinite is considered to be the major sulfate-containing phase to crystallise from the
10 flux and acts as an integral retarder. This can be formed due to the presence of sufficient sulphate in the concrete where
11 the alkalis are present as sulphates [27].

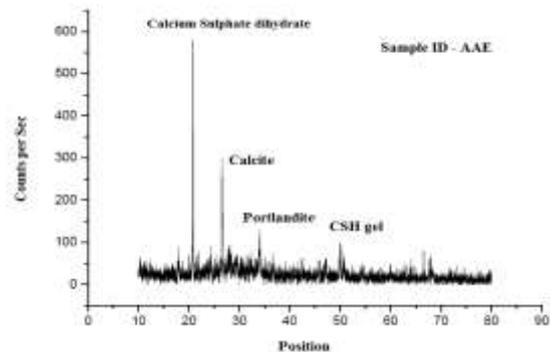
12 The 2-theta value corresponding to Ettringite is 32.4 [24] and it was found to be very less in PWF when compared
13 to AAEF and TTEF. This is found to be in agreement with the results obtained from IR. The 2-Theta value corresponding
14 to calcium carbonate is 21.01 [28] which were found more in PWF and TTEF and it was found less in AAEF which clearly
15 indicates that most of calcium hydroxide is converted in PWF and TTEF. The 2-Theta value for alite is 32.56 which were
16 found to be in major proportion in PWF and it is minor and trace in AAEF and TTEF respectively. Calcium hydroxide
17 whose 2-Theta value was 18.1 [24] decreases much more rapidly in the C_3A pastes containing pozzolana but the reaction
18 cannot be related to the lime-pozzolana reaction which is much slower [29]. This agrees with the fact that the quantity of
19 portlandite is less in the samples containing fly ash when compared with the samples without fly ash.

20 3.4 Scanning Electron Microscope (SEM)

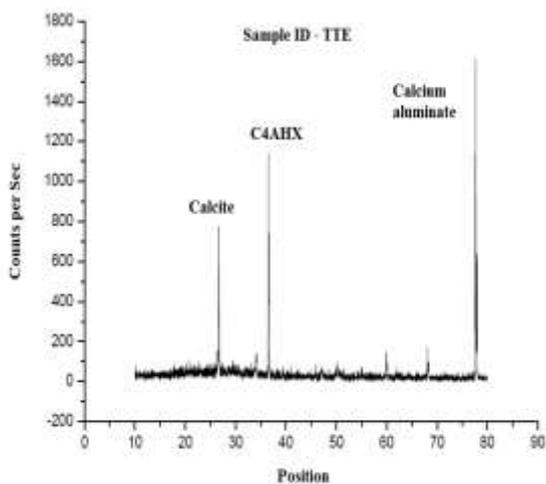
21 The powdered concrete samples were analyzed using this technique and the images are shown in Fig (3). As noticed
22 in XRD, the formation of CSH gel is predominant in PW and AAE samples whereas this is less appeared in all the other
23 samples. This can be well recognized by the mechanical properties of the concrete where the compressive strength was
24 better for PW and AAE. The morphology of ettringite in the hydrated pastes containing calcium aluminate compounds and
25 sulphates depends on the water-solids ratio or the total available space [28]. Slender, needles or spherulites of ettringite
26 was formed in all the samples except PWF. This was supported by the results obtained in XRD. The SEM analysis
27 discovered the presence of distinct calcite crystals in the concrete samples except AAEF. The absence of calcite is
28 noticed in AAEF as observed in XRD. The fly ash particles which were still participated in the process are retaining their
29 smooth and spherical shape while others were surrounded by products of hydration.



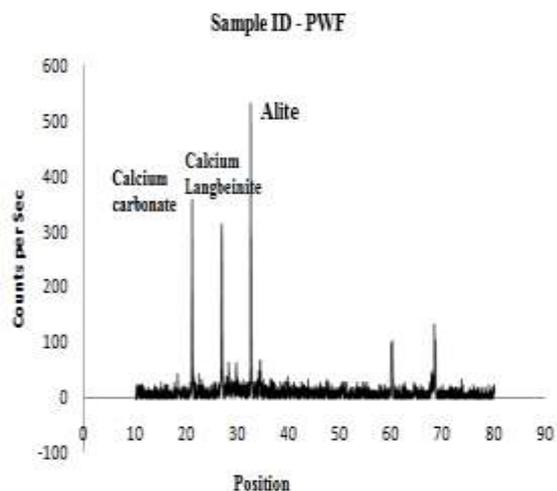
30 (a)



31 (b)



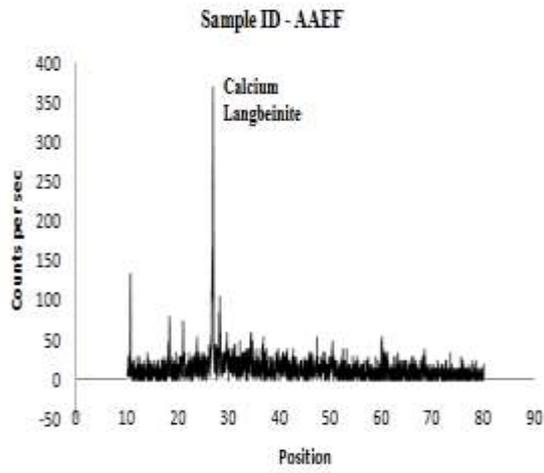
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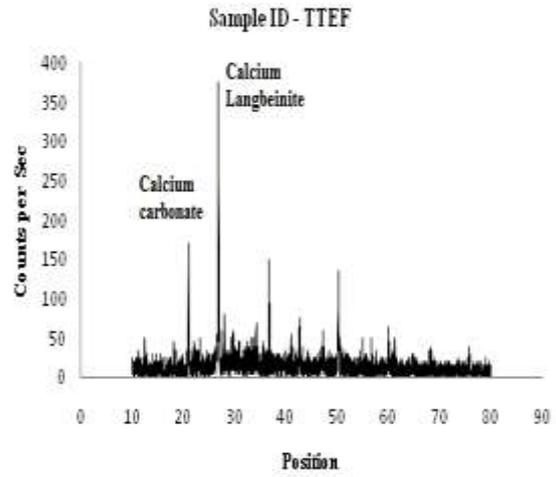


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(c)



(d)



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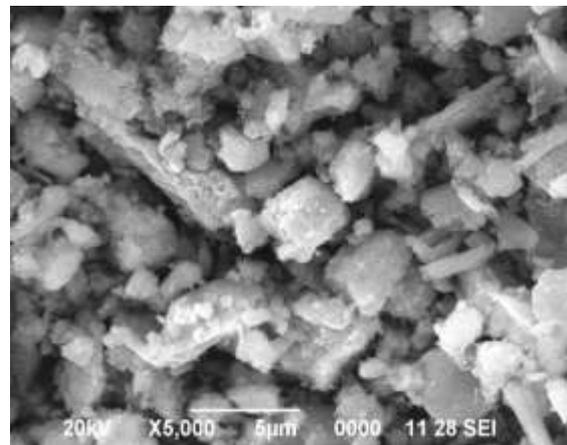
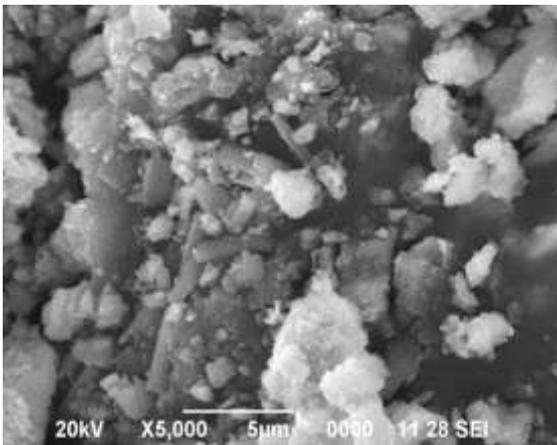
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(e)

(f)

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Fig 2 – XRD – (a) PW (b) AAE (c) TTE (d) PWF (e) AAEF (f) TTEF

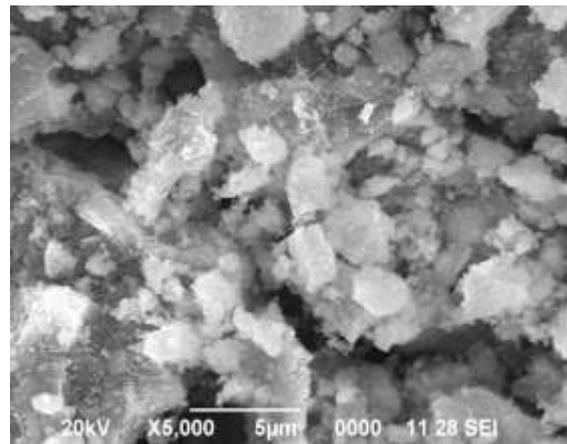
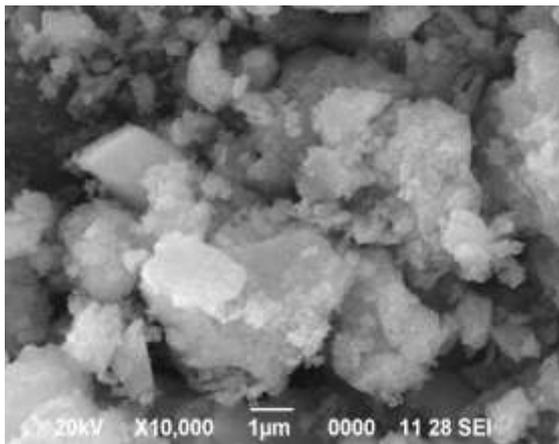


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(a)

(b)

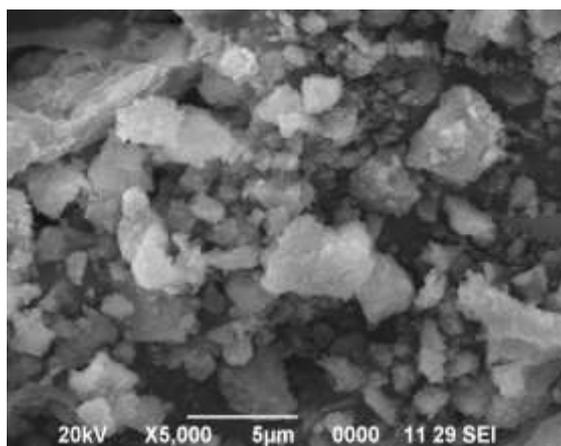


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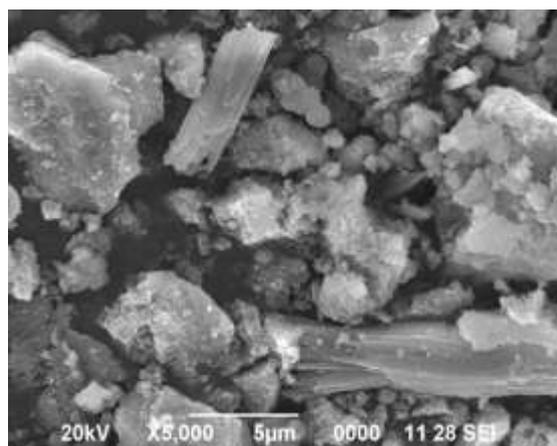
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(c)

(d)



(e)



(f)

Figure (3) SEM images of concrete samples
(a) PW (b) AAE (c) TTE (d) PWF (e) AAEF (f) TTEF

4. CONCLUSIONS

The following conclusions were drawn from the above experimental investigation.

1. The chloride content of the effluents was observed to be higher than the permissible limits.
2. The compressive strength was increased by 36.18% for AAE at 28 days when compared to PW. Similarly an increase of 38.34% for AAEF at 28 days when compared to PWF. The increase in compressive strength was slightly low for TTE and TTEF when compared with AAE and AAEF respectively.
3. The acute band observed at 3637 cm^{-1} in PW and AAE indicates the formation of calcium hydroxide. Similarly, the presence of portlandite is clearly depicted in XRD results.
4. From IR, it can be stated that the formation of CSH gel for AAEF is thrice higher than that of PWF and it was four times higher for TTEF. This can also be justified by the XRD.
5. The formation of ettringite and CSH gel is clearly indicated in SEM observation for the samples AAE and AAEF. Thus, it can be concluded that the anaerobic effluent and fly ash can be potentially used as mixing water in concrete.

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