Effects of SF and Ilmenite on the Chemical, Mechanical and Radiation Behavior of Matrices Used as Solidification of Wastes"

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Abstract: Radioactive waste, arising from civilian nuclear activities as well as from defence-related nuclear-weapon activities, possesses a formidable problem for handling and protecting the environment to be safe is the present and in the future generations. Cement-based waste forms are among the most commonly used waste disposal and site remediation options. However, concrete knowledge of the processes controlling long-term performance of the waste forms is lacking. The aim of this work is to obtain solidified waste form products in compliance with the interim disposal regulations of radioactive waste. Concrete is commonly used as a biological shield against nuclear radiation. Concrete is an inorganic material consisting of cement, water, and aggregates. The quality of any concrete depends on the quality of the paste and aggregate and the bond between them. Different concrete mixes were designed of different concentrations of ilemenite(10% to 60% volume)as fine and coarse aggregate mixed with silica fume (from 5% to 15%) and water-to-cement ratio of 40% . We have studied effects of different concentrations on compressive strength, bulk density, the effect of dose gamma ray to investigate on resistance of concrete to radiation, and the leaching of radioactive wastes (Cs-137). Test results revealed the compressive strength and shielding of the concrete to increase with the growth of coarse ilemenit increase; while, the leaching of waste appears decrease as silica fume use as mineral admixture .

[A. El- Dakroury and Mona S. Gasser. **Effects of SF and Ilmenite on the Chemical, Mechanical and Radiation Behavior of Matrices Used as Solidification of Wastes.** Nature and Science 2012;10(5):92-99]. (ISSN: 1545-0740). http://www.sciencepub.net. 10

Keywords: Heavy weight high strength Concrete, Ilemenite, aggregates ,radiation shielding , silica fume , gamma dose , concrete radiation shielding.

1. Introduction

As the human population and industrial demands grow, nuclear technology has oriented humanity towards using synthetic energy to complement conventional energy sources, which are running out. Constructions of nuclear power plants have been increased for many purposes, especially for energy supply all around the world. However, the issue of the potential impact of nuclear leakage on the environment, and a potential crisis is attracting much interest and stimulating many discussions Concrete is considered to be an excellent and versatile shielding material; it is widely used for shielding nuclear power plants, particle accelerators, research reactors, laboratory hot cells and medical facilities and concrete is used as a structural and shielding material for the storage and disposal of radioactive wastes[1]. Heavyweight concretes have been produced for a long time. These types of concretes were first used to enhance the safety of particular buildings having the tendency of sliding and which had no safety against overturning. Concrete is a relatively inexpensive material, it can be easily handled and cast into complex shapes and of its low price and good shielding performance. With the development of nuclear energy and particularly for protection from fatal rays such as neutron and γ, which have the ability to penetrate objects, heavyweight concrete began to be used as a shield. Heavyweight concretes are produced for their resistance to nuclear radiation, To choice of a shielding materials the linear attenuation coefficients (μ) and mass attenuation coefficients ($\mu\backslash\rho$) which are defined as the probability of a radiation interacting with a materials per unit path length, is important quantity and this has to be known [2,3]. Concrete is an inorganic material and primarily consists of water, cement and aggregate. As water reacts with cement to form hydration products (cement paste), concrete gradually develops its strength. Behaviors of cement paste and aggregate are known to differ under irradiation conditions: cement paste tends to contract slightly and aggregates tend to expand. The behavior of concrete, the heterogeneous mixture of cement past and aggregates, depends on the kind of aggregates in the concrete.

2. Experimental

Materials

Detailed information about the materials used and their characteristics are given.

Cement Portland: cement has been found to be adequate for production of high strength concrete. Experience has indicated that PC is, in most cases, entirely satisfactory and adequate for constructing concrete shielding.

Table 1 properties of Portland cement (PC) used in the study

Property	**PC**
Oxide composition : %	
SiO_2	21.23
Al_2O_3	4.25
Fe_2O_3	1.98
CaO	63.21
MgO	1.97
SiO_3	2.87
Na_2O	.14
K2O	-
TiO_2	-
$Fe_2 O_3$	-
FeO	-
P	-
S	-
LOI	2.3
Mineral Composition : %	
C_3S	63.0
C_2S	10.98
C_3A	6.93
C_4AF	7.1
Physical properties	
Colour	gray
Specific gravity	3.11: 3.77
Bulk density: Kg/ m^3	2300
Specific surface :m^2/Kg	308

Ilemnite ores: these ores consist of crystalline ilmenite with either magnetite or hematite and constituents of the associated gabbroic or anorthositic rocks. Massive ilmenite ores can form coarsely crystalline massive tough rocks but vary, from deposit to deposit, and within a deposit, in specific gravity, composition, hardness, and suitability for use as concrete aggregate. Many ilmenite ores consist of ilmenite disseminated in rock rather than concentrated as a major rock-forming mineral. Ilmenite concentrated from beach sands is usually altered to a variable degree, and its mechanical properties probably differ from those of unaltered ilmenite. One of the most widely used types of heavy aggregates is ilmenite ore.

Chemical Formula**:** Fe++TiO_3 , **Composition:** Molecular Weight = 151.73 gm**,** Mineral class: oxides**,** Crystal system: hexagonal.

Aggregate**:**_Generally, concrete mixes designed today will be composed of 70 to 80% aggregate by volume. The aggregate will usually consist of both a fine and coarse component. Aggregate types and sizes play an essential role in modifying concrete properties. We have produced plain concrete (PC) using ilimenite –based aggregates with five different grain sizes

Table 2: Properties of Ilemnite (IL)used in the study

Property	**Ilemnite (IL)**
Oxide composition : %	
Ti	31.56
TiO_2	52.65
Fe	36.81
FeO	47.35
O	31.63
Physical properties:	
Colors	**Black**
Bulk density: g/ m^3	**4.68**

Table 3: Grading of fine and coarse aggregates of ilmnite

Coarse aggregate, Sieve size (mm)	Fine aggregate, Sieve size (mm)
75mm (CA1)	600 µm (FA1)
50mm (CA2)	150 µm (FA2)
25mm (CA3)	

Silica Fume (SF) used in the study **:**Silica fume, also referred to as microsilica or condensed silica fume, is a byproduct material that is used as a pozzolan This byproduct is a result of the reduction of high-purity quartz with coal in an electric arc furnace in the manufacture of silicon or ferrosilicon alloy. Silica fume rises as an oxidized vapor from the 2000°C (3630°F) furnaces. When it cools it condenses and is collected in huge cloth bags. The condensed silica fume is then processed to remove impurities and to control particle size. Condensed silica fume is essentially silicon dioxide (usually more than 85%) in noncrystalline (amorphorous) form. Since it is an airborne material like fly ash, it has a spherical shape It is extremely fine with particles less than 1 µm in diameter and with an average diameter of about 0.1 µm, about 100 times smaller than average cement particles.Silica fume is sold in powder form but is more commonly available in a liquid. Silica fume is used in amounts between 5% and 10% by mass of the total cementitious material. It is used in applications where a high degree of impermeability is needed Silica fume is sold in powder form but is more commonly available in a liquid. Silica fume is used in amounts between 5% and 10% by mass of the total cementitious material. It is used in applications where a high degree of impermeability is needed and in highstrength concrete. Silica fume must meet ASTM C 1240. provide an extensive review of silica fume [4,5] .

Table 4: Properties of Silica Fume (SF) used in the study

Property	**Silica Fume (SF)**
Oxide composition : %	
SiO_2	90
Al_2O_3	0.4
Fe_2O_3	0.4
CaO	1.6
SiO_3	0.4
Na_2O	0.5
K_2O	2.2
Na	1.9
LOI	3.3
Physical properties :	
Colour	Gray
Specific gravity	2.22
Bulk density: Kg/ m^3	2.4
Specific surface :m^2/Kg	20,000
Particle Size	< 1µm

Preparation of Specimens for Test

In this study, series of specimens were prepared and tested in the laboratories of hot laboratories.

The factors that can affect the strength of concrete, density, water absorption, leach-ability, effect of gamma on the compressive strength and shielding can be classified into these categories:

Effect of Silica Fume content, effect of fine aggregate, coarse aggregate, and companied of silica fume ,fine and coarse aggregate

The specimens used in the study take three shapes:

1-Stan dared cubes (15x15x15) cm for compressive strength

2-Stan dared cylinders (10x2) cm for shielding

3- Stan dared cylinders (2x2) cm for leach-ability

The mixture with different ratio for different materials with water / cement ratio are given in table 5.

Table 5. Mixture Proportions (%)

Batch	Batches	Cement	SF	FA1	FA2	CA1	CA2	CA3
1	100/0/0/0/0/0/0	100	0	0	0	0	0	0
2	**90/10/0/0/0/0/0**	**90**	**10**	**0**	**0**	**0**	**0**	**0**
3	85/15/0/0/0/0/0	85	15	0	0	0	0	0
4	80/10/10/0/0/0/0	80	10	10	0	0	0	0
5	70/10/10/10/0/0/0	70	10	10	10	0	0	0
6	**65/10/15/10/0/0/0**	**65**	**10**	**15**	**10**	**0**	**0**	**0**
7	65/10/10/15/0/0/0	65	10	10	15	0	0	0
8	55/10/10/15/10/0/0	55	10	10	15	10	0	0
9	45/10/10/15/10/0/0	45	10	10	15	10	10	0
10	35/10/10/15/10/10/0	35	10	10	15	10	10	10
11	30/10/10/15/15/10/10	30	10	10	15	15	10	10
12	30/10/10/15/15/10/10	30	10	10	15	10	15	10
13	**30/10/10/15/15/10/10**	**30**	**10**	**10**	**15**	**10**	**10**	**15**

SF : silica fume , FA1: Fine aggregate 1, FA2 : Fine aggregate 2, CA1: coarse aggregate 1,CA2: coarse aggregate 2, CA3: coarse aggregate 3

3. Results and Discussion

Compressive strength tests

A total of samples are prepared and tested at three different SF content (5,12,17) of cement and six different curing times (3,28,60 and 90 days). the results are presented in Fig 1 , where each point on graph represents the average of three tests. The effect of Fine Aggregate (FA) on the compressive strength are presented in Fig. 2, the effect of Coarse Aggregate (FA) on the compressive strength are presented in Fig 3 and the comparison between Silica Fume (SF), Fine Aggregate (FA) and Coarse

Aggregate (CA) on the compressive strength are presented in Fig. 4. From Figure 1 it can be seen that use of 10% silica fume results in a long term increases in strength with 10% silica fume and decrease compressive strength with15 % silica fume. From Figs. 2,3,4 it can be seen that the compressive strength increases are apparent when the silica fume content with 15 % from (FA2) more than 15% (FA1). It is also clear that these increases begin to develop with mixed with 15% from (CA3) more than 15% from (CA2) and (CA1). The results show that the higher coarse aggregate ratio of 0.35 gave higher compressive strength than for the fine aggregate. However, in the mixes containing silica fume with 35% coarse aggregate and 25 % fine aggregate in dependent of coarse aggregate types at all ages [6,9,10] .

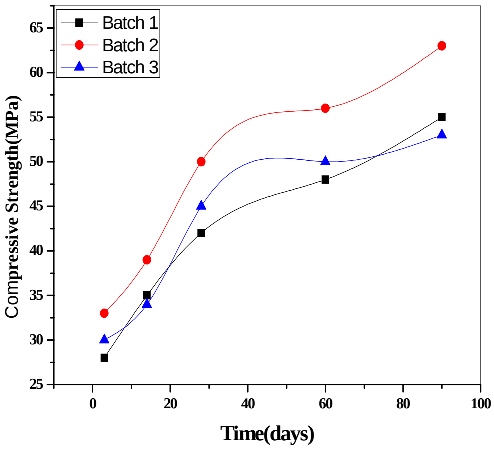

Fig.1: Effect of Silica Fume (SF) on the compressive strength

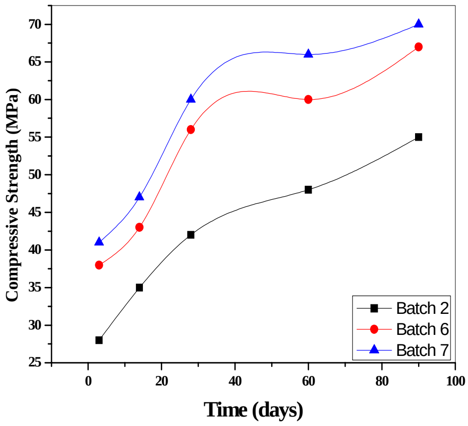

Fig.2: Effect of Fine Aggregate (FA) on the compressive strength

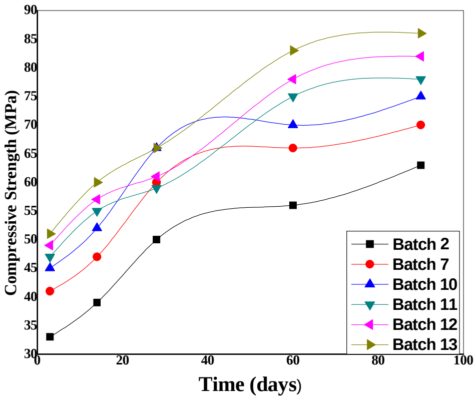

Fig.3: Effect of Coarse Aggregate (FA) on the compressive strength

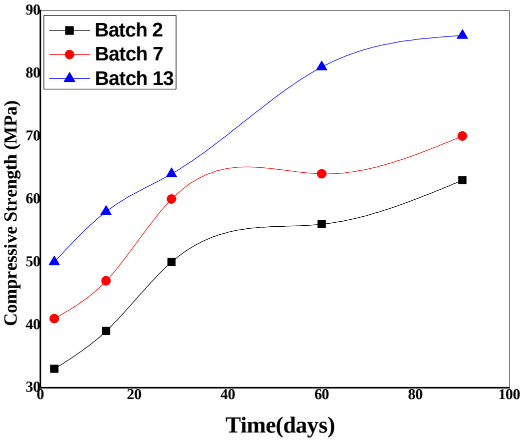

Fig.4: Comparison between Silica Fume (SF), Fine Aggregate (FA) and Coarse Aggregate (CA) on the compressive strength

-Density and Water Absorption for different Mixtures test

The measured density and water absorption as 28 days are given in table 6. The highest density and lowest water absorption for the sample 13 are 4897 kg /m^3 and 11.8 respectively.

Table 6. The Density and Water Absorption for different Mixtures

Batch	**Batches**	**Density(kg/m^3)**	**Water Absorption**
1	100/0/0/0/0/0/0	2500	21.8
2	**90/10/0/0/0/0/0**	**2550**	**16.9**
3	85/15/0/0/0/0/0	2430	20.4
4	80/10/10/0/0/0/0	2562	16.0
5	70/10/10/10/0/0/0	2597	15.7
6	65/10/15/10/0/0/0	2759	15.5
7	**65/10/10/15/0/0/0**	**2986**	**15.1**
8	55/10/10/15/10/0/0	3127	14.5
9	45/10/10/15/10/10/0	3685	14.0
10	35/10/10/15/10/10/10	3985	13.5
11	30/10/10/15/15/10/10	4334	13.2
12	30/10/10/15/10/15/10	4521	12.4
13	**30/10/10/15/10/10/15**	**4897**	**11.8**

Leaching

Upon completion of the curing process, the cement control and different specimens were demolded and leached in accordance to the specifications and procedures outlined in ANSI/ANS 16.1.18 [9]. This standard was specifically designed to measure the release of radionuclides from solidified low-level radioactive waste forms. It permits the accumulation of leaching data in a relatively short period of time using test specimens of simple shapes and finite dimensions. The procedure involves sampling and replacing leachants at designated time intervals of 2 , 7 hrs, 1, 2, 3, 4, 5, 19, 47, and 90 days. It also requires a rinse of the test specimen prior to the leaching regimen to determine the total activity of the (Cs-137) at the beginning of the first leaching interval. This value was designated as American National Standards Institute ANSI/ANS 16.1 standard test that is designed to examine the leaching rate of contaminants in concrete and grout specimens to determine the cumulative fractions leached and effective diffusion coefficients. This test is conducted according to protocol specified by ANSI [9]. The ANSI 16.1 leach test is a regulatory test that requires leaching the test specimens; at the end of a defined period all the solution is replaced with fresh deionized distilled water. The Composition of Ground water leachant .used for the test was in Table 7.

Effective Diffusivity Calculations

Based on a number of leach studies, **Serne *et al.*** [9,10] concluded that leaching from a semiinfinite solid source would be the most appropriate model to describe the release of trace contaminants from grout and cement specimens. Leaching of semi-infinite solids has been described mathematically using either incremental leach rates or cumulative leach rates [8-10]. Using incremental leach rates, the effective diffusion coefficient for each leach interval (Dei) for a species of interest is expressed as:

$$D_{ei} = \pi\left[\left(\frac{a_n}{A_o}\right)/\Delta t_n\right]^2\left[\frac{V}{S}\right]^2[T]$$

Using cumulative leaching rates, the effective diffusion coefficient (Dec) is expressed as:

$$D_{ec} = \pi/4\left[\left(\frac{\Sigma a_n}{A_o}\right)/\Delta t_n\right]^2\left[\frac{V}{S}\right]^2\left[\frac{1}{t}\right]$$

Where

D_{ei} = effective diffusivity coefficient (cm^2/s) for the leaching interval, t_n – tn-$_1$

D_{ec} = effective diffusivity coefficient (cm2/s) for the cumulative leach interval, t_n – to

a_n = activity of radionuclide leached during the leaching interval, t_n – tn-$_1$

Σa_n = total activity of radionuclide cumulatively leached during the interval, t_n – to

A_o = total initial activity of radionuclide in the specimen

$\sum a_n/A_o$ = fraction of radionuclide leached during interval, t_n – tn-$_1$

Δtn = duration of the nth leaching interval, $t_n - t_{n-1}$ in seconds

V = volume of the specimen, cm^3

S = geometric surface area of the specimen, cm^2

T = mean leaching time = $\left[\frac{1}{2\left(\sqrt{t_n}+\sqrt{t_{n-1}}\right)}\right]^2$

t = total elapsed time from leaching initiation in seconds.

Table 7: Composition of Ground water leachant .TDS: total dissolved solids

Soluble cations (mg/L)						Soluble anions(mg/L0		
TDS	PH	K^+	Na^+	Mg^+	Ca^{2+}	Cl^-	SO^{4-}	HCO^{3-}
1.04	7.4	22	147	14	73	138	319	274

3. Results and Discussion

The leaching data from static tests are plotted as cumulative leach as a function of leaching time the cumulative fraction release (CFR) values for each formulation at leaching intervals as specified in the ANSI/ANS 16.1 procedure. ANSI/ANS 16.1 defines cumulative fractional release as the sum of all fractions leached during all previous leaching intervals, plus the fraction leached during the last leaching interval($\sum$ an / A_0) using the initial amount of the species of interest present in the specimen as unity. All CFR values were corrected for radioactive decay back to the beginning of the first leaching interval. The variation of cumulative leach fractions of Cs-137 are found in Figure 5 and the calculated average effective diffusion coefficients are listed in Table 8.

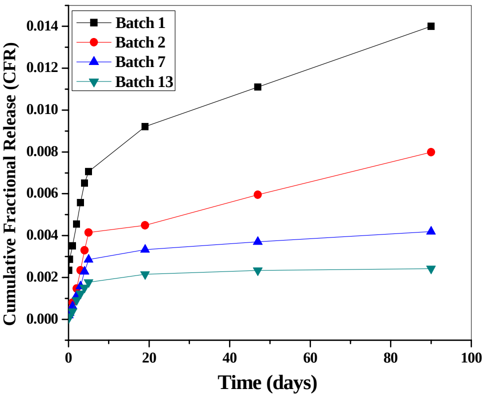

Fig. 5: Compression between Cumulative Fractional Release (CFR) for the Batches 1, 2, 7, and 13 for (Cs-137)

Table 8 :Calculated effective diffusion coefficients(D_{ec}) for Cs-137 from static leach tests

Batch	Batches	Calculated effective diffusion coefficient Dec
1	100/0/0/0/0/0/0	$4.65x10^{-2}$
2	**90/10/0/0/0/0/0**	**$5.18x10^{-8}$**
3	85/15/0/0/0/0/0	$4.94x10^{-7}$
4	80/10/10/0/0/0/0	$3.15x10^{-8}$
5	70/10/10/10/0/0/0	$2.87x10^{-8}$
6	65/10/15/10/0/0/0	$3.4x10^{-8}$
7	**65/10/10/15/0/0/0**	**$3.12x10^{-10}$**
8	55/10/10/15/10/0/0	$2.97x10^{-10}$
9	45/10/10/15/10/10/0	$2.5x10^{-10}$
10	35/10/10/15/10/10/10	$7.87x10^{-11}$
11	30/10/10/15/15/10/10	$8.2x10^{-11}$
12	30/10/10/15/10/15/10	$7.2x10^{-11}$
13	**30/10/10/15/10/10/15**	**$5.77x10^{-11}$**

The sample 1 results for the cement control samples 2 and 3 results were obtained from leach tests conducted on cement mixed with 10 % and 15 % (mass fraction), respectively, silica fume. The fineness and high surface area of silica fume causes alteration of the pore structure of cement paste, which can act to change the cumulative fraction release (CFR) values leaching rates and average effective diffusion coefficients of cesium . The smaller leach fractions obtained for samples 2 and 3 indicate that addition of 10 % silica fume reduces leach fractions of Cs-137, which can be attributed to the reduction in volume of large pores and capillaries that led to reduced permeability of the cement paste. The 15 % (mass fraction) silica fume addition (sample 3) did not significantly alter the leaching rates of these nuclides. This is due to the tendency of the high surface area of silica fume particles to absorb water. If too much water is adsorbed, gel formation in the cement paste is inhibited causing void formation to increase, there by increasing the permeability of the cement form. Furthermore, the addition of ilmentite at both the 10 % and 15 % (mass fraction) with different aggregate were found to decrease the leach rates of Cs-137. The mechanism that causes the reduction in leaching is the reaction of titanium oxide (TiO2) with water, which forms a gelatinous material that fills the pores and reduces the permeability of the cement.

Together, the C-S-H gel and the $Ca(OH)_2$

comprise more than 80 % of the cement paste volume and, therefore, control the chemical properties of the aqueous phase of Portland cement. This is due to the fact that these compounds buffer the calcium concentration and contribute to the high pH . C-S-H is the principal buffering agent that maintains a high pH for the very long-term and is largely responsible for the stability of the cement form. C-S-H is a colloidal, amorphous compound with a high surface area, C-S- H therefore has the ability to immobilize metal ions by addition and substitution reactions as follows [13]

Addition Reaction:

$$C-S-H+M\rightarrow M-C-S-H$$

Substitution Reaction:

$$C-S-H+M\rightarrow M-C-S-H+Ca^{2+}$$

As stated in the previous section, the lower the calcium to silica ratio in the C-S-H, the more cation incorporation can occur. Addition of certain pozzolanic materials can be used in these circumstances to free Ca(OH)2, lower the calcium/silicate ratio without compromising the integrity of the matrix, and effectively increase the metal fixation ability of the cement. [13].

Addition of silica fume increases early strength while reduces permeability , Because of their small size, silica fumes can improve packing by filling the spaces between the cement particles. In addition, they also react with calcium hydroxide during cement hydration, which may reduce bleeding and porosity [14] .

Shielding test

Radiation shielding tests were performed using Cobalt-60 as the source for gamma rays. Cobalt-60 has a half-life of 5.27 years, and its gamma rays have energies of 1171 keV and 1332 keV.
relationship is found:

$$I = I_o \times e^{-\mu x}$$

Where:
I = is the gamma ray intensity after the shield material,
I_o = is the gamma ray intensity before the shield material,
μ = is the attenuation coefficient factor and x is the shield material thickness

Half and Tenth Thickness

The half value layer (or half thickness) is the thickness of any particular material necessary to reduce the intensity of an X-ray or gamma-ray beam to one-half its original value. In similar fashion, one can define a tenth thickness as the depth required to reduce the photon intensity by a factor of ten.

$$\frac{I(x_{1/2})}{I(0)}=\frac{1}{2}=e^{-\mu x_{1/2}} \quad \Rightarrow \quad x_{1/2}\equiv\frac{\ln(2)}{\mu}$$

The relationship between the tenth value thickness (TVT), The attenuation coefficient and the half-value layer (HVL) are shown in table 7,and the relation between half and tenth thickness with attenuation coefficient are shown in fig7results show that the higher the density the smaller the TVT thickness. The TVT of silica fume concrete was smaller than concrete without silica fume by 10 % but 15 % silica fume as decrease . ilminte has a fine aggregate has increase density and decrease (TVT) and (HVL) but the coarse aggregate more effect on density [15-17].

Table 7: The relationship between the tenth value thickness (TVT), The attenuation coefficient and the half-value layer (HVL)

Batch	Batches	Density(kg/m^3)	μ	HVT(mm)	TVT(mm)
1	100/0/0/0/0/0/0	2500	0.15231	33.54	11.12
2	**90/10/0/0/0/0/0**	**2550**	**0.14792**	**31.06**	**10.25**
3	85/15/0/0/0/0/0	2430	0.13852	29.62	9.94
4	80/10/10/0/0/0/0	2562	0.13521	28.03	8.45
5	70/10/10/10/0/0/0	2597	0.12975	26.55	8.21
6	65/10/15/10/0/0/0	2759	0.12483	23.28	7.85
7	**65/10/10/15/0/0/0**	**2986**	**0.12101**	**19.60**	**7.13**
8	55/10/10/15/10/0/0	3127	0.1175	19.03	6.90
9	45/10/10/15/10/10/0	3685	0.1123	18.45	6.17
10	35/10/10/15/10/10/10	3985	0.0985	17.76	5.87
11	30/10/10/15/15/10/10	4334	0.0876	16.62	5.34
12	30/10/10/15/10/15/10	4521	0.07659	15.57	4.98
13	**30/10/10/15/10/10/15**	**4897**	**0.06864**	**15.13**	**4.43**

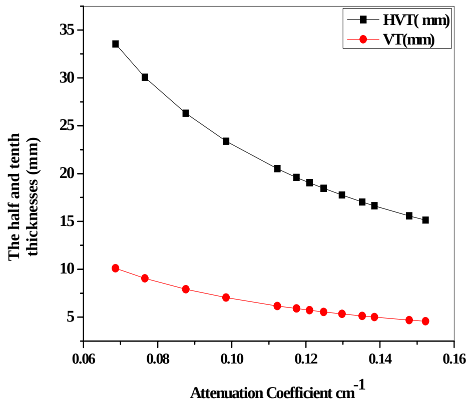

Fig. 7: Relation between half and tenth thickness with attenuation coefficient

5. Conclusions

Concrete is a heterogeneous, multiphase material, it is a mixture of cement paste, fine and coarse aggregates in a range of sizes and shapes, large aggregates in a range of gradations, and various types of void spaces. the concrete is heterogeneous when the paste is observed to be a mixture of different types of crystalline structures, at varying degrees of hydration, which form an amorphous gel. We have found that effect of iliment added to concrete on its essential properties. Mechanical properties, especially the compressive strength of concrete with ilemtite , there was a significant relationship between the increase of ilminte ratio, density and radiation permeability of the concretes and unit weight of concrete for shielding increases with replacement ratio of ilmenit to concrete and Increased ilminte ratio had a positive effect on linear attenuation coefficient Increased ilminte ratio had a positive effect on linear attenuation coefficient. In all concrete series, there was a significant relationship between the increase of silca fum ratio, density and radiation permeability of the concretes.

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4/2/2012