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Production of Nano-Concrete with Fumed Silica, Metakaolin & Portland Cement Using High Energy Mixing

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Abstract – *The application of Nano-Technology in cement mortar or concrete has added new dimension to the efforts to improve its different properties. Nano-particles, because of their very small particles size can affect the properties of a cement mortar or concrete by changing its micro structures. This study concerns with the use of fumed silica as a nano particles and metakaolin to improve the compressive strength of the nano-concrete. An experimental investigation has been carried out to replace the cement with fumed silica of 2%,5%,6%,7%,8%,9% &10% and metakaolin of 10%,15% & 20%. The tests conducted on it shows a considerable increase in the compressive strength. The strength increase was observed with a certain amount of Fumed silica & metakaolin. The FSEM micrograph shows that the micro structures of hardened nano-concrete is improved on addition of fumed silica. A sample of industrial kaolin and a representative sample is taken from Swat Khyber Pukhtton Khwa Pakistan mine reservoir and tests like thermo gravimetric analysis and X-ray diffraction are performed, to find out either it is kaolin or some other mineral.*

Keywords: *Nano-technology, nano-concrete, fumed silica, metakaolin ,Compressive strength*

1. INTRODUCTION

Concrete is among the most utilized material on this planet. It is the material of present & future. The wide use of concrete in structures like buildings to airports, from roads to tunnels, from bridges to air ports makes it the most studied material. Due to the high use of concrete or cement mortar, there is an vital need to improve its durability and strength. There are different materials which can be used in nano concrete production but cement plays a key role. To improve the properties of the concrete, the mechanisms of the cement hydration has to be calculated properly and also may be studied at micro level. Concrete properties have been improved by adding different supplementary materials. Some of the supplementary materials are metakaolin, fly ash, silica fume, rice husk hash and even bacteria. There are different technology in use to improve its properties but nano-technology is the positive approach in enhancing the properties of the concrete.

Hence nano technology may have the potential to make concrete with superior properties through the optimization of material behavior and performance that significantly increase the durability & mechanical properties. To develop nano-technology in concrete or mortar, it requires a multi disciplinary approach consist of teams of concrete materials, civil engineers, physicts, chemists and also material scientists. The increased use of cement is essential in attaining high compressive strength, tensile strength and flexural strength but cement is a major source of pollution. The use of very small proportion of the nano-particles on a replacement of cement can leads to the higher compressive strength as well as check to pollution. In this research work the fumed silica has been used as nano-particles with metakaolin as a pozolanic material as modify cement replacement by high energy mixing techniques.

2.LITERATURE REVIEW

Tejas Patel (15 Sept 2014) Institute of Technology Nirma University defined the nano-concrete as "A concrete with any ingredients namely cement particles, admixtures, aggregates or other smart materials or additives having particle size less than 500 nano meter which improve the properties of the fresh and hardened concrete is known as Nano Concrete". Dr.N Bhanumathidas and N Kalidas have defined nano-concrete as, " The concrete made with pozolanic binders and micro aggregates and completely avoiding use of course and fine aggregates in order to continue the durability issues caused by occurrence of transition zone in control concrete is called Nano Concrete". There is no precise and acceptable definition for nano concrete. Many researchers have defined nano-concrete on the basis of type of materials used, the properties of concrete improved and most importantly the size of the particles are used.

Tang (2003) found that addition of nano-silica in cement mortar, a reduction in $Ca(OH)_2$ & increased in C-S-H gel is noticed through XRD & DTA testing. Sobolev (2005) & Jo (2007) experimentally studied that nano-particles fills the pores of the cement & also it reacts with calcium hydroxide and generates calcium silicates hydrate gel. Gaitro et al 2010 experimentally investigated that addition of nano-SiO_2 in cement paste, noticed that nano-particles increase the initial heat of hydration, increase the quantity of C-S-H gel and also reduction in porosity.

Li et al 2004 experimentally studied that use of nano-Fe_2O_3 & nano-Silicon dioxide in cement mortar increase the compressive & flexural strength as compared to plain cement mortar. Fan et al (2004) used ZrO_2 powder in cement paste and noticed the reduction in porosity and permeability, improvement in micro structures of the paste. Carvellet et al 2006, used nano-$CaCO_3$ as nano particles and found improvement in flexural strength in cement mortar or concrete. Qing et al (2003) investigated that there was major improvement in early age of interfacial transition zone (ITZ) structure with respect to the reduction in $Ca(OH)_2$. Zi li et al (2006) experimentally investigated that during the hydration of cement, the nano-alumina particles fill the pores at the past-aggregates interface & also creates a dense ITZ structure by decreasing the porosity & further increased elastic modulus.

Munoz & Meininger (2010) studied experimentally that microstructure of the pase is improved by addition of nano particles without improving in the Interfacial Transition Zone. But after the addition of nano-particles by NPTF on aggregates surface before concrete mixing significantly improves the ITZ of the concrete. In 2004, Kuennen studied that self healing polymers includes a micro capsulated agent and also a catalytic chemical, mainly applicable to repair the micro cracks in bridge piers & columns. These capsules when present in the structure are broken by occurring crack, works as healing agent with catalyst. G.Li et al 2007 studied that carbon nano tubes treated with a mixed solution of HNO_3 & H_2SO_4 when added to cement paste results in increase the compressive strength and also considerably lower the electrical conductivity

Chang et al 2007 & Musy et al 2008 experimentally investigated that addition of nano clay particles have shown a very good role in reducing the shrinkage of the concrete. Vaile et all 2004 experimentally studied that the concrete with nano-TiO_2 are very effective & is suitable for the outdoor structures because the concrete made with nano titanium oxide can be cleaned by self cleaning. Dham et al 2010 investigated that by replacing the fraction of conventional cement with nano cement faster the initial heat of hydration and also the induction period is reduced. It may also cause the reduction in permeability of the concrete with increase tensile & compressive strength.

3. EXPERIMENTAL

3.1. Materials

The mixes constituents were OPC, metakaolin, fumed silica, water & sand. The super plasticizer used was Ultra Super Plast 427. The fumed silica was used as nano-particles. The composition and properties of fumed silica & Ultra Super Plast 427 are shown in table 1 & table 2 respectively.

Table 1: Properties of FS

Properties	Unit	Value
Specific surface area	M2/g	175-225
PH value		3.7-4.5
Silicon dioxide	%	>99.8
Particle size		14nm
Fineness	$M^2 g^{-1}$	190

Table 2: Properties of Ultra Super Plast 427

Characteristics	Value
Appearance	Brown liquid
Specific gravity @ 20 ^oC	Nil
Air Entrainment	Less than 2%
PH	7.5-9.5

The metakaolin was obtained after heating the pure kaolin which was obtained from Ceramic industry in a kiln upto 800 ^oC. The XRD & TGA analysis were performed on MK to check its chemical compositions and its behavior with thermal changes. XRD analysis on MK shows that the sample contain rich oxide of silicon dioxide as shown in table 3..

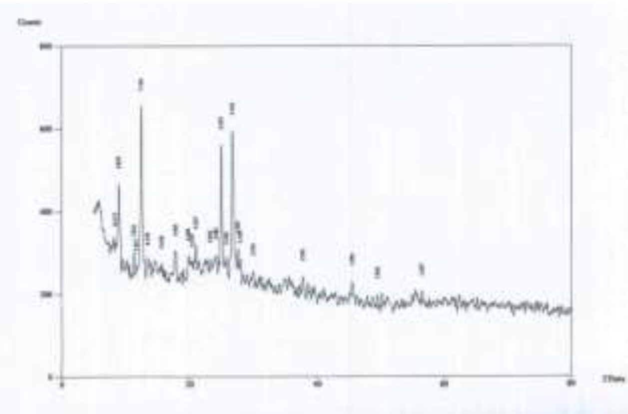


Figure 1: XRD Peak analysis of MK

Table 3: Chemical composition of Industrial Kaolin

S. No	ICDD No	Mineral Name	Material Name
1	* 291489	$Al_2 Si_2 O_5 (OH)_4.2H_2O$	HALLOYSITE
2	*501799	$Ca_4 Al_6 Si_6 O_{24} CO_3$	
3	* 150742	$Mg_3 Al_2 (Si O_4)_3$	PYROPESYN
4	D 30418	Ca-Mg-Al-Si-O	
5	D 30428	Ca-Mg-Al-Si-O	

A sample was taken from a reservoir SWAT Khyber Pukhtoon Khwa Pakistan because it was known that there are reservoirs of kaolin. After performing the XRD & TGA analysis, it was found that the reservoir is not of kaolin but that is actually pure dolomite. It is necessary to mention that many researchers have used dolomite in concrete applications and has been found very useful. So the Dolomite of Swat can be used in concrete applications. The XRD peak analysis graph is shown in figure 2 and its chemical composition is given in table 4.

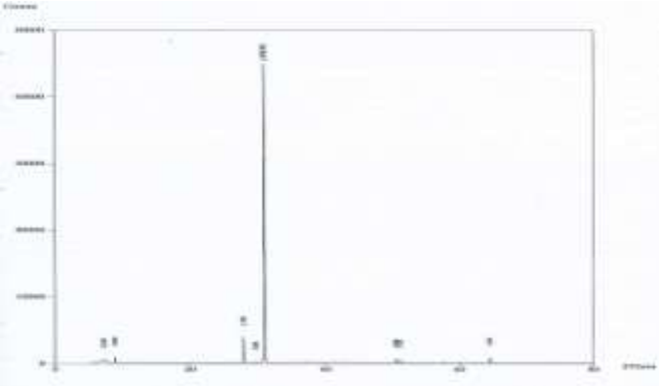


Figure 2: XRD Peak analysis of Dolomite

Table 4: Chemical composition of Dolomite Swat reservoir

No	ICDD No	Mineral Name	Material Name
1	360426	Ca Mg $(CO_3)_2$	DOLOMITE
2	110078	Ca Mg $(CO_3)_2$	DOLOMITE
3	340517	Ca (Mg,Fe) $(CO_3)_2$	DOLOMITE,FERROAN

As per TGA test data performed on industrial kaolin at CRL laboratory Peshawar, 5.971gm of the sample was taken. The sample was hold for 1minute at 40 ^{o}C and after that it was heated at constant rate i.e 10.00 ^{o}C/Min up to 1000 ^{o}C. TGA graph of the sample at figure 3 shows that at start the sample is absorbing heat and is endothermic process. After some stage the system becomes exothermic that is because of evaporating water from the sample. When the sample was heated above 600 ^{o}C, it losses most of its water and the process is known as de-hydroxilization. At this stage all the water gets evaporated & kaolin converts into metakaolin which becomes amorphous and reactive metakaolin.

$$\underset{\text{Kaolin}}{Al_2O_3.2SiO_2.2H_2O} \xrightarrow[\text{600-850 } ^{o}\text{C Heat}]{} \underset{\text{Metakaolin + water}}{Al_2O_3.2SiO_2 + 2H_2O}$$

Figure 3: Thermal Analysis of Industrial Kaolin

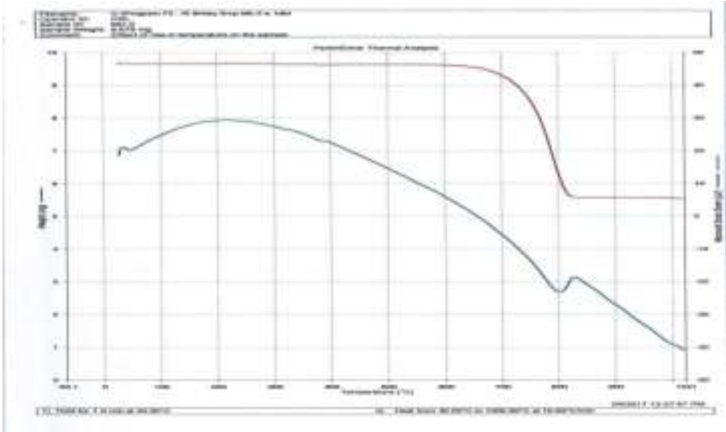


Figure 4: Thermal Analysis of Dolomite Swat

After performing XRD test on the Swat mine sample, it was confirmed that the sample is not kaolinite or kaolin clay. The chemical composition of the sample shows that it is dolomite. TGA analysis as shown in figure 4 on the sample was performed in order to check its behavior with increasing temperature. As per test data performed at CRL laboratory, 9.575gm of the sample was taken and was hold for 1minute at 40 ^{o}C and after that it was heated at constant rate i.e. 10.00 ^{o}C /Min up to 1000 ^{o}C. The figure 4 shows that thermal decomposition of the sample took place in two phases, which is shown in following two reactions.

- Ist reaction: $CaMg(CO_3)_2 \rightarrow CaCO_3 + MgO + CO_2$ (above 650 ^{o}C)
- 2nd reaction: $CaCO_3 \rightarrow CaO + CO_2$ (b/w 800 ^{o}C and 850 ^{o}C)

The observed weight loss is 0.83% mainly due to loss of water (below 600 ^{o}C) and between 650 ^{o}C & 700 ^{o}C, the sample decomposes into calcium carbonates, Magnesium oxide & carbon dioxide. Above 800 ^{o}C, there is sudden loss in mass of the sample, because of the decomposition of calcium carbonates into Calcium oxide and carbon dioxide & the here the weight loss is about 43.80% and the total weight loss is about 45%.

3.2. Mix Proportion & Compressive Strength of Nano-Concrete

Twenty four mix proportion as shown in table 5, were designed to study the effects of FS & Mk on heat of hydration & compressive strength after mixing by an electric drill mixer. The control mix had no FS & Mk. Different percentages of FS i.e 2%,5%,6%,7%,8%,9% & 10% were used with 10%,15% & 20% Mk as partial replacement by weight of cement is used. The mix proportion was 1:2.75 with water-binder ratio of 0.5 and also Ultra Super Plast 427 plasticizer is used as admixture. Super plast 427 is used because due to the use of fumed silica and its high surface are, the requirement of water is dramatically increased, so as to meet requirement of water Super plast is used.

Table 5: Mix Proportion & Compressive Strength of Nano-concrete

S.No	Mix ID	Cement (gm)	Fumed Silica (gm)	Metakaolin (gm)	Fine Aggregates (mg)	Admixture (ml)	Water (ml)	W/B	Compressive Strength (Psi)	
									7 days	8 days
1	F0M0	83.33	0	0	229.2	1.25	41.67	0.5	3023.15	4231.68
2	F2M 10	73.33	1.67	8.33	229.2	1.25	41.67	0.5	2731.12	4957.16
3	F2M15	69.16	1.67	12.5	229.2	1.25	41.67	0.5	2980.91	5098.58
4	F2M20	65	1.67	16.67	229.2	1.25	41.67	0.5	3412.52	5243.68
5	F5M10	70.833	4.17	8.33	229.2	1.25	41.67	0.5	3026.83	4327.19
6	F5M15	64.13	4.17	12.5	229.2	1.25	41.67	0.5	3337.22	4727.58
7	F5M20	62.46	4.17	16.67	229.2	1.25	41.67	0.5	3256.41	4327.18
8	F6M10	70.5	4.5	8.33	229.2	1.25	41.67	0.5	2497.87	3717.41
9	F6M15	66.3	4.5	12.5	229.2	1.25	41.67	0.5	2811.94	3924.96
10	F6M20	62.16	4.5	16.67	229.2	1.25	41.67	0.5	2764.18	3862.51
11	F7M10	69.17	5.83	8.33	229.2	1.25	41.67	0.5	2406.03	3249.06
12	F7M15	65	5.83	12.5	229.2	1.25	41.67	0.5	3402.36	3612.72
13	F7M20	60.84	5.83	16.67	229.2	1.25	41.67	0.5	2609.90	3605.38
14	F8M10	68.33	6.67	8.33	229.2	1.25	41.67	0.5	2399.60	2813.77
15	F8M15	64.21	6.67	12.5	229.2	1.25	41.67	0.5	2226.96	3294.98
16	F8M20	60	6.67	16.67	229.2	1.25	41.67	0.5	2518.07	3050.70
17	F9M10	67.55	7.45	8.33	229.2	1.25	41.67	0.5	2167.27	2479.50
18	F9M15	63.43	7.45	12.5	229.2	1.25	41.67	0.5	2106.66	3122.33
19	F9M20	59.21	7.45	16.67	229.2	1.25	41.67	0.5	2389.50	2964.38
20	F10M10	66.67	8.33	8.33	229.2	1.25	41.67	0.5	1882.58	2143.39
21	F10M15	62.55	8.33	12.5	229.2	1.25	41.67	0.5	2049.70	2940.50
22	F10M20	58.33	8.33	16.67	229.2	1.25	41.67	0.5	1759.56	2934.99
23	F0M25	62.5	0	20.83	229.2	1.25	41.67	0.5	3294.98	4852.46
24	F10M0	75	8.33	0	229.2	1.25	41.67	0.5	2563.98	3548.44

To find the effects of fumed silica as nano-particles & metakaolin as pozolanic material on the compressive strength of nano-concrete cubes of 2" * 2" are casted. First the nano-concrete constituents which are cement, sand, metakaolin, fumed silica are taken of the mix design as shown in Table5. The materials are then dry mixed in a pan until it gives us homogeneous dry mix. Then the dry mix is taken in a pot and water of the required amount is added to the dry mix and is mixed with the help of a electric drill mixer. The blades of the drill mixer are made by a skilled welder with better quality steel because during mixing the wearing & tearing of the blades may not occur. Super Plast admixture is also added in the middle. When the materials are properly mixed, the cubes are then casted.
The HEM technique can easily be followed in controlled mix. As we increase the amount of FS to the mix, lumps of the mix are seen in the concrete during mixing. As we further increase the amount of fumed silica, the lumps production were increased which makes the mixing process tougher and may be one of the reason in degrading the quality of the mix design.

3.3:Curing & Testing

The nano-concrete cubic samples of different mix design after 24 hours of casting were then kept in water upto the required days of testing. i.e.7days & 28 days compressive strength. The compressive strength of the samples were then performed by UTM machine in laboratory. For each mix design and days, 3 samples were tested and then average of these 3 samples was taken compressive strength of each mix design of nano-concrete. The mix design made with

2%,5%,6%,7%,8%,9% & 10% and 10,15% &20% metakaolin used as modify cement replacement by mass & their compressive strength for 7&28 days are shown in table5.

Figure 5 shows the compressive strength of the mix design made by 10% Mk with different percentages of FS i.e. 2%,5%,6%,7%,8%,9% & 10% as by weight of cement replacement. In the same way, figure 6 & 7 give use the compressive strength for the mix design made by15% & 20% Mk respectively with different percentages of FS as mentioned before.

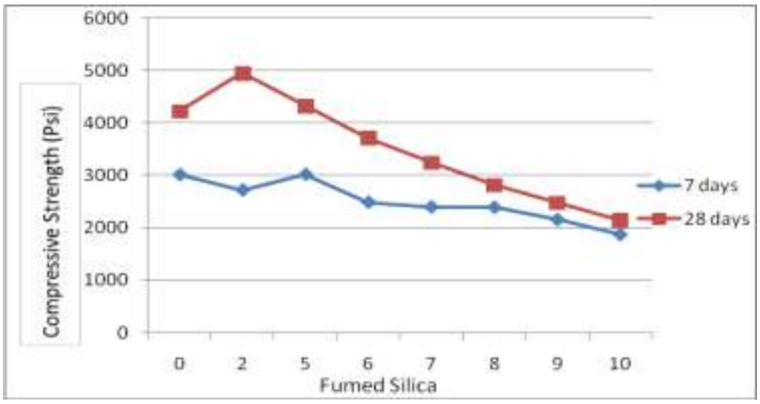


Figure 5: Nano-concrete with 10% Mk & different percentages of FS

The strength result shows that the compressive strength made 2% FS with 10%,15% & 20% Mk has shown better result as compared to samples of controlled mix and other mix desgins. As the dosage of FS was increased further, the decrease in compressive strength occurs. This shows that the increased aount of FS remains un-reactive and has caused negative effect on the mechanical property on nano-concrete because of agglomeration of FS & Mk in the mix that weakens the bond between the cement paste & aggregates.

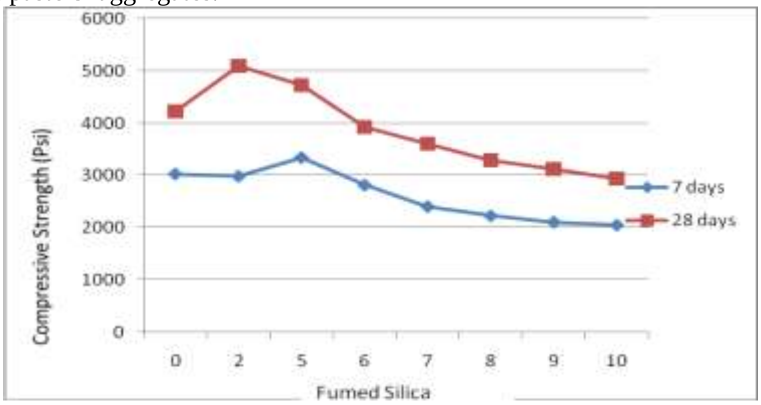


Figure 6: Nano-concrete with 15% Mk & defferent percentages of FS

The increase in compressive strength of the samples may be due to filling of voids by the nano-particles i.e Fumed silica, so as to prevent the production of $Ca(OH)_2$ crystals. The fumes silica and metakaolin also act as pozolanic material which further reacts with calcium hydroxide and produce C-S-H gell, which increases the compressive strength of the nano-concrete samples. The compressive strength results have shown that the increase in early age of strength i.e. 7 days, due to high surface area of the fumed silica as compared to 28 days strength. The amorphous FS increases the rate of heat of hydration & helps in gaining early strength of the mix

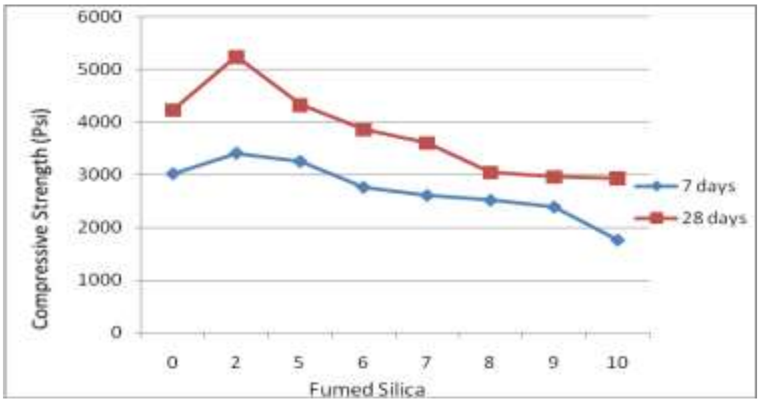
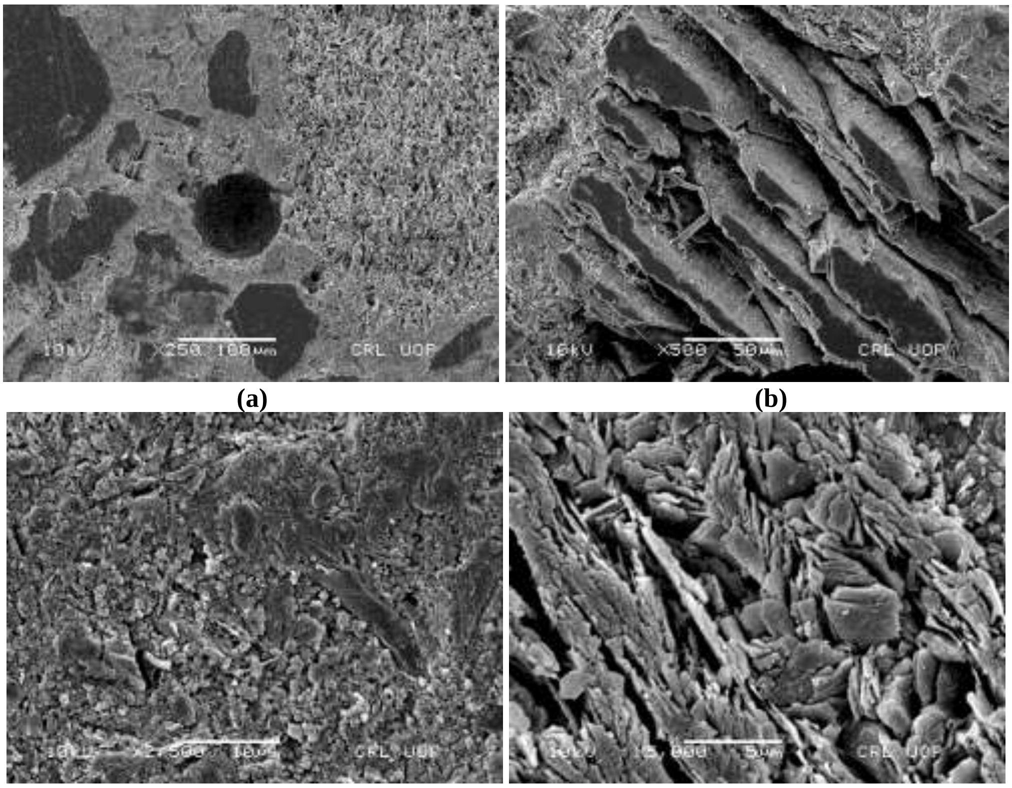


Figure 7: Nano-concrete with 20% Mk & different percentages of FS

The minimum strength among the mix design was shown by 10%FS & 10% Mk, 10% FS & 15%Mk and 10% FS & 20% Mk.It shows the increased amount of FS has weakened the compressive strength of the nano-concrete samples. From above test result, it can be predicted that the optimum amount of FS may be between the 0.5% to 2% when used with 10%,15% & 20% Mk as partial replacement by weight of cement is used.

3.4.Scanning Electron Microscopic Analysis

The nano-concrete cubic sample of the controlled mix was made by High Energy Mixing. The idea of the HEM was taken from the research work of Vladien Fridman. When a controlled mix is mixed by HEM technique, there is possibility of 5nm colloidal globules & controlled mix gets converted into nano-concrete mix. HEM technique is easy & efficient in controlled mix & homogeneity of the concrete constituents can easily be achieved. HEM technique is "bottom up" approaches in nano-technology. The SEM image of the controlled mix are shown in figure 8.



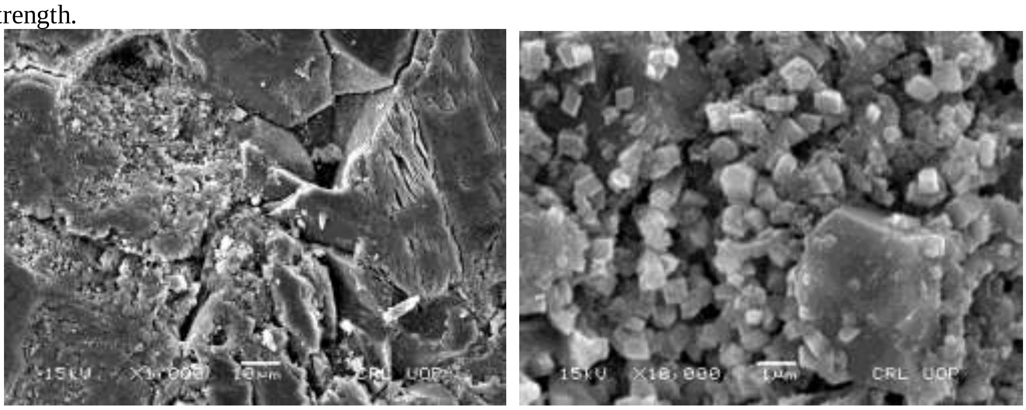
(a) (b)

(c) (d)

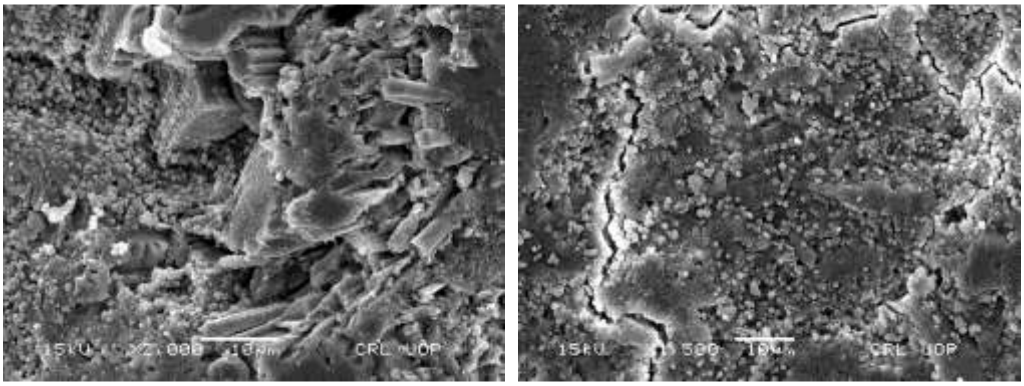
Figure8:SEM Images of Controlled Mix

The SEM analysis of the above controlled mix made by HEM has shown the sample porous which shows the sign of light weight sample, but this porosity can be overcome by using nano-particles with better strength.

SEM images shown in figure 9 are of nano-concrete sample made with 10% FS & 10% Mk mixed by high energy mixing by a electric drill mixer. The images shows the smooth surface and also porosity of the sample is decreased because of the improvement in micro-structure of nano-concrete sample. In SEM images shows the suspended particles of Mk & FS, which gives indication that the excessive FS & Mk remained un-reactive and also it is one of the reason in decreasing the sample strength.



(a) (b)



(c) (d)

Figure9: Nano-concrete with FS & Mk

The excessive C-S-H gell is formed because of pozolanic reaction between the calcium hydroxide and fumed slica as well as metakaolin. The images shows some visible particles of metakaolin and fumed silica, which means that excessive Mk & FS remained un-reactive and made the sample weaker. The SEM figure 9 (c), that there are some hydrated cement particles, which are high density C-S-H particles and also in some cased it consists of interior core of un-hydrated cement. These are individual solid particles and some time called as phenograins because it can be distinctly visible in microscope.

The SEM figure 9 (d) shows that the shrinkage cracks are appeared between the particles and the cement. It may be because of the chemical shrinkage caused by the cement hydration or it may be due to excessive use of fumed silica & metakaolin. From SEM images its is concluded that if the optimum amount of FS & Mk are used, it will gives us better result with improved strength. To find the optimum amount of FS as nano particles in production of nano-concrete, further research is need in this field.

IV.CONCLUSION & RECOMMENDATION

4.1. Conclusions

On the basis of this research work, the following conclusions were drawn:

1. Increase in compressive strength of the samples may be due to filling of voids by the nano-particles i.e Fumed silica, so as to prevent the production of $Ca(OH)_2$ crystals. The fumes silica and metakaolin also act as pozolanic material which further reacts with calcium hydroxide and produce C-S-H gell, which increases the compressive strength of the nano-concrete samples.
2. The increase in early age strength i.e. 7 days due to high surface area of the fumed silica as compared to 28 days strength.
3. SEM micrograph shows that addition of fumed silica and metakaolin to mix fill up the micro pores between the C-S-H gel and making the microstructure more uniform and compact.
4. The increase in strength is maximum for fumed silica 2% with metakaolin 10%,15% & 20%, but if we are increasing the amount of Fumed Silica, the compressive strength graph gets decreasing because the increasing amount of Fumed Silica and Metakaolin remain un-reactive and makes the nano-concrete weak.
5. After addition of fumed silica to the mix, lumps of the mix are seen in the concrete during mixing. As we further increase the amount of fumed silica, the lumps production gets increased which makes the mixing process tougher and may be one of the reason in degrading the quality of the mix design.
6. The sample from Swat mine was tested for Kaolin (China Clay) but the XRD results shows its Dolomite. Many researchers have worked on dolomite and have found that it is very useful and effective material in concrete and mortar due to its high surface hardness & density, resistance to weathering and also can be used as a filler material in asphalt and concrete applications.
7. The SEM graph shows that HEM makes the nano concrete porous, but by using nano particles, the porous problem can be overcome, which is the sign of light weight concrete.
8. SEM analysis of High Energy Mixing concrete shows improvement in microstructures which may cause low capillary action in concrete.
9. High Energy Mixing may not be economic because of high power consumption and also if course aggregates are used, it will cause wear & tear of the mixing drum and its maintenance cost will be higher.

4.2. Recommendation

1. Fumed Silica may be tried on concrete mix or cement mortar with minimum quantity. i.e. Starts from 0.1% up to 5% as a cement replacement.
2. HEM techniques may be more effective if we follow the w/b ratio upto 0.75 because of drastically increase in water demand after addition of Fumed Silica.

3. A proper understanding on the use of Fumed Silica & Metakaolin can be develop if the size effect of both can be studied in details.
4. Detail investigation of the microstructure throughout the year at different interval may be performed because it may give us a good idea of the reactions in the concrete or mortar.
5. The Same research may be repeated after adding the course aggregates in it.
6. The dolomite sources are available in abundance in Swat KPK which can be used in cement concrete or mortar for the improvement its properties.
7. The optimum quantity of Fumed Silica for cement mortar or concrete may be decided after doing many research on it.
8. The durability properties of cement mortar or concrete with fumed silica as nano particles may also be tested in future.

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