VOL. 11, NO. 21, NOVEMBER 2016 ISSN 1819-6608
ARPN Journal of Engineering and Applied Sciences

www.arpnjournals.com

EXPERIMENTAL STUDY ON STRENGTH PROPERTIES OF METAKAOLIN AND GGBS BASED GEOPOLYMER CONCRETE

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ABSTRACT

Energy saving in building technology is among the most critical problems in the world. Thus it is a need to develop sustainable alternative to conventional concrete utilizing more environmental friendly materials. One of the possibilities to work out is the massive usage of geopolymer concrete to turn them to useful environmental friendly and technologically advantages cementitious materials. In the present study metakaolin and Ground Granulated Blast furnace slag (GGBS) is used to produce geopolymer concrete. Geopolymer concrete is prepared by using alkaline solution of sodium silicate mixed with sodium hydroxide in the ratio of sodium silicate to sodium hydroxide is 2.5 and the concentration of sodium hydroxide is 10M is considered in this experimental investigation. The geo polymer concrete specimens with different proportions of Metakaolin and GGBS were cast and tested for compressive strength, Split Tensile Strength and Flexural Strength for 3, 7 and 28 days and cured at ambient temperature.

Keywords: geo-polymer, metakaolin, ground granulated blast furnace slag, alkali activator, ambient curing.

1. INTRODUCTION

Concrete is the most widely used man made construction material in civil engineering world. As the demand for concrete as a construction material increased, the world production of cement has greatly increased since 1990. The global warming is caused by the emission of greenhouse gases such as CO_2 to the atmosphere by human activities. Among the greenhouse gases, CO_2 contributes about 65% to global warming. The cement industry is responsible for about 6% of all CO_2 emissions, because the production of one tonne of Portland cement emits approximately 0.9 tonne of CO_2 into the atmosphere. Although the use of Portland cement is still unavoidable until the anticipated future, many efforts are being made in order to reduce the use of Portland cement in concrete. These efforts include the utilization of supplementary cementitious materials such as fly ash, silica fume, granulated blast furnace slag, rice-husk ash and Metakaolin, and finding alternative binders to Portland cement [1].

Pozzolanic materials including silica fumes, fly ash, slag, Rice Husk Ash and Metakaolin have been used in recent years as cement replacement material for developing High strength Concrete with improved workability, strength and durability with reduced permeability. Metakaolin, which is a relatively new material in the concrete industry, is effective in increasing strength, reducing sulphate attack and improving air-void network. Pozzolanic reactions change the microstructure of concrete and chemistry of hydration products by consuming the released calcium hydroxide (CH) and production of additional calcium silicate hydrate (C-S-H), resulting in an increased strength and reduced porosity and therefore improved durability. The use of Metakaolin in High Strength Concrete is discussed in this paper. Use of Metakaolin in construction industry as partial replacement of cement started in the 1960's and the interest in this material has considerably increased in recent years. Metakaolin has pozzolanic properties bringing positive effects on resulting properties of concrete. Pozzolanic properties cause chemical reaction of active components with calcium hydroxide (portlandite), which is formed as a product of cement hydration. This reaction leads to formation of binding phases of following types: [2] secondary C-S-H gel, C4AH13, C3AH6, and C2ASH8 thereby increasing strength [2].

Davidovits (1988) proposed that an alkaline liquid could be used to react with the silicon (Si) and the aluminium (Al) in a source material of geological origin or in by-product materials such as fly ash and rice husk ash to produce binders. Because the chemical reaction that takes place in this case is a polymerization process, he coined the term Geopolymer to represent these binders. Geopolymer concrete is concrete which does not utilize any Portland cement in its production. Geopolymer concrete is being studied extensively and shows promise as a substitute to Portland cement concrete. Research is shifting from the chemistry domain to engineering applications and commercial production of geopolymer concrete.

There are two main constituents of geo polymers, namely the source materials and the alkaline liquids. The source materials for geo polymers based on alumina-silicate should be rich in silicon (Si) and aluminium (Al). These could be natural minerals such as kaolinite, clays, etc. Alternatively, by-product materials such as fly ash, silica fume, slag, rice-husk ash, red mud, etc could be used as source materials. The choice of the source materials for making geo polymers depends on factors such as availability, cost, type of application, and specific demand of the end users. The alkaline liquids are from soluble alkali metals that are usually sodium or potassium based.

VOL. 11, NO. 21, NOVEMBER 2016 ISSN 1819-6608
ARPN Journal of Engineering and Applied Sciences

The most common alkaline liquid used in geo polymerization is a combination of sodium hydroxide (NaOH) or potassium hydroxide (KOH) and sodium silicate or potassium silicate. [3]

2. METAKAOLIN

General the raw material in the manufacture of Metakaoline is kaolin clay. Kaolin is a fine, white, clay mineral that has been traditionally used in the manufacture of porcelain. Kaolins are classifications of clay minerals, which like all clays, are phyllosilicates, i.e. a layer silicate mineral. The Meta prefix in the term is used to denote change. In case of Metakaolin, the change that is taking place is dehydroxylization, brought on by the application of heat over a defined period of time. Dehydroxylation is a reaction of decomposition of kaolinite crystals to a partially disordered structure. The results of isothermal firing shows that the dehydroxylation begins at 4200C. [4] At about 100 - 200^{0}C clay minerals lose most of their adsorbed water. The temperature at which kaolite loses water by dehydroxilization is in the range 500-800^{0}C. This thermal actvation of a mineral is also referred to as calcining. Beyond the temperature of dehydroxylization, kaolinite retains two dimensional order in the crystal structure and the product is termed Metakaolin. Metakaolin is neither the by-product of an industrial process nor is it entirely natural. It is derived from naturally occurring mineral and is manufactured specially for cementing applications. Metakaolin is produced under carefully controlled conditions to refine its colour, remove inert impurities, and tailor particle size such, a much high degree of purity and pozzolanic reactivity can be obtained. Metakaolin is white, amorphous, highly reactive aluminium silicate pozzolan forming stabile hydrates after mixing with lime stone in water and providing mortar with hydraulic properties. Heating up of clay with kaolinite Al2O3.2SiO2.2H2O as the basic mineral component to the temperature of 500 - 600°C causes loss of structural water with the result of deformation of crystalline structure of kaolinite and formation of an unhydrated reactive form - so-called metakaolinite. The chemical equations describing this process is [2]

Table-1. Physical properties of Metakaolin.

Colour	Pink / Off-white
Pozzolan Reactivity mg Ca (OH)2 / gm	900
Average Particle size	1.4 micron
Brightness (ISO)	75 ± 2
Bulk Density (Gms / Ltr)	320 to 370
Specific Gravity	2.5

Table-2. Chemical Properties of Metakaolin.

Al_2O_3	>39.0 %
Fe_2O_3	<0.8%

3. GGBS

Ground granulated blast furnace slag (GGBS) is a byproduct of the manufacturing of iron in a blast furnace where iron ore, limestone and coke are heated up to 1500◦C.When these materials melt in the blast furnace, two products are produced i.e molten iron, and molten slag. The molten slag is lighter and floats on the top of the molten iron. The molten slag comprises mostly silicates and alumina from the original iron ore, combined with some oxides from the limestone. The process of granulating the slag involves cooling the molten slag through high pressure water jets. This rapidly quenches the slag and forms granular particles generally not larger than 5mm in diameter. The rapid cooling prevents the formation of larger crystals, and the resulting granular material comprises some 95% non-crystalline calcium-aluminosilicates. The granulated slag is further processed by drying and then ground to a very fine powder, which is GGBS (ground granulated blast furnace slag) cement it is another excellent cementitious material. [4]

Wainwright and Ait-Aider (1995) examined the influence of the composition of OPC and the addition of up to 70% GGBS on the bleed characteristics of concrete and conclude that the partial replacement of OPC with 40% and 70% of GGBS. GGBS led to increases in the bleeding of the concretes, like fly ash, also GGBS can improve many mechanical and durability properties of concrete and it generates less heat of hydration. [5]

Babu and Kumar (2000) determined the cementitious efficiency of GGBS in concrete at various replacement percentages (10-80%) through the efficiency concept by establishing the variation of strength to water-to-cementitious materials ratio relations of the GGBS concretes from the normal concretes at the age of 28 days. The 28-day compressive strength of concretes containing GGBS up to 30% replacement were all slightly above that of normal concretes, and at all other percentages, the relationships were below that of normal concretes. It was also observed that the variations due to the different percentages of slag replacement were smaller than the corresponding variations in the case of fly ash. The result showed that the slag concretes based on overall efficiency factor (k), will need an increase of 8.6% for 50% replacement and 19.5% for 65% replacement in the total cementitious materials for achieving strength equivalent to that of normal concrete at 28 days. [6 - 7]

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ARPN Journal of Engineering and Applied Sciences

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Table-3. Physical properties of GGBS.

Parameter	GGBS	IS : 12089 – 1987
CaO	37.34%	---
Al_2O_3	14.42%	---
Fe_2O_3	1.11%	---
SiO_2	37.73%	---
MgO	8.71%	Max. 17%
MnO	0.02%	Max. 5.5%
Sulphide Sulphur	0.39%	Max. 2%
Loss of Ignition	1.41%	---
Insoluble Residue	1.59%	Max. 5%
Glass Content (%)	92%	Min. 85%

4. COARSE AGGREGATE

Coarse aggregate are a broad category particulate inert materials used in construction. Hard stones are crushed to the required size and are used as coarse aggregate. The material that is retained on as IS sieve of size 4.75 is called coarse aggregate. Aggregate of essentially the same nominal maximum size and grading will produce concrete of satisfactory workability. These aggregates are bound together by the cement and fine aggregate in the presence of water to form concrete. Coarse aggregates of sizes 12mm and 20mm and fine aggregate taken from a local supplier are used in the present study and the properties as shown in Table-4.

Table-4. Physical properties of course aggregate.

Sieve size (mm)	20 mm		12 mm	
	Requirement as per IS: 383-1970	Percentage passing	Requirement as per IS:383-1970	Percentage passing
80.00	----	----	----	----
63.00	----	----	----	----
40.00	100 %	100 %	----	----
20.00	85 – 100 %	94.60 %	----	----
16.00	----	----	100 %	100 %
12.50	----	----	85 -100 %	93.4 %
10.00	0 – 20 %	14.30 %	0 – 45 %	39.23 %
4.75	0 – 05 %	2.85 %	0 – 10 %	6 %
2.36	----	----	----	----
Specific gravity		2.82	-	2.79
Water Absorption %		0.40	-	0.48
Aggregate Impact Value		12 %	-	13 %
Bulk Density (kg/m^3)		1660	-	1655
Flakiness		14 %	-	15 %
Elongation		15 %	-	16 %

5. FINE AGGREGATE

Fine aggregate should consist of natural sand or crushed stone sand. It should be hard, durable and clean and be free from organic matter etc. fine aggregate should not contain any appreciable amount of clay balls and harmful impurities such as alkalis, salts, coal, decayed vegetation etc. The silt contents should not exceed 4%.

Table-5. Physical Properties of Fine Aggregate.

I.S. Sieve (mm)	**Percentage passing through I.S. Sieve**	**Percentage passing I.S. Sieve as per IS 383**	
10	100	100	Fineness modulus = **2.2** Specific Gravity = **2.50** Bulk Density =**1625 Kg/m^3** Bulking of Sand = **23%** Silt Content = **0.25 %**
4.75	99.6	90-100	
2.36	99	75-100	
1.18	92.6	55-90	
600 micron	48.6	35-50	
300 micron	8.2	8-30	
150 micron	2	0-10	
Zone	**II**		

6. SODIUM HYDROXIDE SOLUTION

The most common alkaline activator used in geopolymerisation is a combination of sodium hydroxide (NaOH) or potassium hydroxide (KOH) and sodium silicate or potassium silicate [3]. The type and concentration of alkali solution affect the dissolution of fly ash. Leaching of Al3+ and Si4+ ions are generally high with sodium hydroxide solution compared to potassium hydroxide solution.

Therefore, alkali concentration is a significant factor in controlling the leaching of alumina and silica from fly ash particles, subsequent geo polymerization and mechanical properties of hardened geopolymer. Duchesne *et al* (2010) confirmed that in presence of NaOH in the activating solution the reaction proceeds more rapidly and the gel is less smooth. The gel composition analysed in the sample activated with the mixture of sodium silicate and NaOH is enriched in Na and Al [8].

7. SODIUM SILICATE SOLUTION

Palomo *et al* (1999) concluded that the type of activator plays an important role in the polymerization process. Reactions occur at a high rate when the alkaline activator contains soluble silicate, either sodium or potassium silicate, compared to the use of only alkaline hydroxides. A study conducted by Xu and Van Deventer (2000) showed that the addition of sodium silicate solution to the sodium hydroxide solution as the alkaline activator enhanced the reaction between the source material and the solution. Tempest *et al* (2009) state that the sodium silicate activator dissolves rapidly and begins to bond fly ash particles. Open porosity can be observed and is rapidly filled with gel as soon as the liquid phase is able to reach the ash particles. The liquid phase is important as a fluid transport medium permitting the activator to reach and react with the fly ash particles [9-10].

8. CASTING PROCEDURE

Generally the fine aggregate, coarse aggregate and fly ash are weighed to the required quantities and then they are mixed in dry condition foe 2-3 minutes and then the alkaline solutions prepared(combination of sodium hydroxide and sodium silicate) are to be taken to required quantity is added to the dry mix. This mixing is done for 5-7 minutes in the mixer for proper bonding of all the materials. After the mixing is done the mix is filled in the cube moulds of size 150mm x 150mm 150 mm in 3 layers with equal compacting an the these cubes are kept on a vibrating table so that no voids are formed.

9. EXPERIMENTAL PROGRAM

9.1 Mix design of geopolymer concrete

In the design of geopolymer concrete (GPC mix), coarse and fine aggregates together were taken as 75% of entire mixture by mass. This value is similar to that used in OPC concrete in which it will be in the range of 75% to 80% of the entire mixture by mass. Fine aggregate was taken as 30.8% of the total aggregates. From the past literatures it is clear that the average density of Cementitious materials based geopolymer concrete is similar to that of OPC concrete (2400kg/m3). Knowing the density of concrete, the combined mass of alkaline liquid and Cementitious materials can be arrived. By assuming the ratios of alkaline liquid to Cementitious materials as 0.45, mass of metakaolin and mass of alkaline liquid was found out. To obtain mass of sodium hydroxide and sodium silicate solutions, the ratio of sodium silicate solution to sodium hydroxide solution was fixed as 2.5. Extra water (other than the water used for the preparation of alkaline solutions) used respectively to achieve workable concrete. The mix proportion for geopolymer concrete is given after several trial mixes in Table-6 and the different combinations of GGBS and Metakaolin investigated are given in Table-7.

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Table-6. Mix proportions.

Materials used	Cementitious materials	Fine aggregate	Coarse aggregate	Sodium Hydroxide	Sodium silicate
Quantity of materials in kg/m3	414	660	1136	53	133

Table-7. Combinations of GGBS and Metakaolin.

Mix ID	Metkaolin	GGBS
M1	100%	0%
M2	90%	10%
M3	80%	20%
M4	70%	30%
M5	60%	40%
M6	50%	50%
M7	40%	60%
M8	30%	70%
M9	20%	80%
M10	10%	90%
M11	0%	100%

9.2 Preparation of geopolymer concrete

To prepare 10 molarity concentration of sodium hydroxide solution, 400 grams (molarity x molecular weight) of sodium hydroxide flakes was dissolved in distilled water and makeup to one litre. The sodium hydroxide solution thus prepared is mixed with sodium silicate solution one day before mixing the concrete to get the desired alkaline solution. The solids constituents of the GPCC mix i.e. kaolinite clay, GGBS and the aggregates were dry mixed in the mixer for about three minutes. After dry mixing, alkaline solution was added to the dry mix and wet mixing was done for 4 minutes. Finally extra water was added to achieve workable GPCC mix.

In this experimental work a total of 297 numbers of Geopolymer concrete specimens were cast with various mix ratios as shown in Figure-1. The specimens are of 150 mm side. Before casting machine oil was smeared on the inner surfaces of the cast iron mould. Concrete was poured into the moulds and compacted thoroughly using a tamping rod. The top surface was finished using a trowel. The GPC specimens were removed from the mould after 1or 2 days based on setting of specimens. The specimens were left at room temperature till the day of testing. Compressive strength test was conducted using a 3000kN Compression testing machine. The test was conducted as per the relevant Indian standard specifications.

10. RESULTS AND DISCUSSIONS

10.1 Compressive strength

The compressive strength of Metakaolin based geopolymer concrete at the age of 3 days 7 days and 28 days are presented in the Figure-1.

Table-8. Compressive strength.

Mix ID	Compressive strength N/mm^2		
	3 days	7days	28 days
M1	53.62	54.64	61.03
M2	51.3	53.91	56.71
M3	44.91	46.94	50.28
M4	42.14	44.32	48.13
M5	34.20	35.72	39.29
M6	31.23	34.12	35.23
M7	33.67	35.61	38.34
M8	41.23	43.75	47.35
M9	43.65	45.07	49.94
M10	50.32	52.32	55.5
M11	52.12	53.8	60.03

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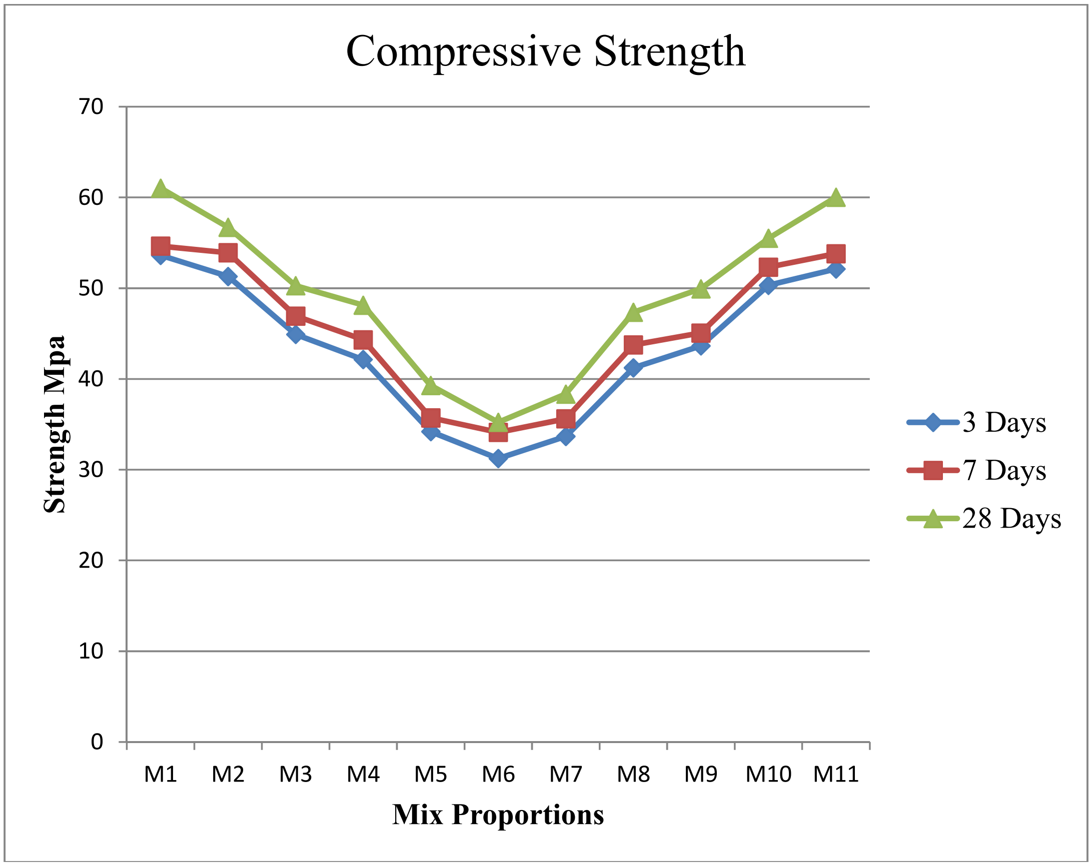

Figure-1. Compressive strength.

Compressive strength test on cubes is the most common test conducted on hardened concrete because it is an easy test to perform and most of the desirable properties of concrete are comparatively related to its compressive strength. The compression test was carried out on cubical specimen of size 150mm in a compression testing machine of capacity 3000 kN [2]. The strength is determined at 3, 7 and 28 days of casting.

The compressive strength of GGBS based kaolinite clay geopolymer concrete at the age of 28 days is presented in Figure-1. At 3 days, the mix proportion M6 has the lowest compressive strength of 31.23 N/mm^2 and the mix proportion of M1 gave the highest compressive strength of 53.62 N/mm^2 at 3 days. At 7 days, the mix proportion M1 gives the highest compressive strength of 54.64 N/mm^2 and the mix proportion M6 gives the lowest compressive strength of 34.12 N/mm^2. At 28 days, M1 cube specimens attained a compressive strength of 61.03 N/mm^2 and the mix proportion M6 gives the lowest compressive strength of 35.23 N/mm^2. It is seen from the results that when the percentage of GGBS and Matakaolin is increased the compressive strength of the geopolymer concrete. Though addition of GGBS and Matakaolin, increases the compressive strength in geopolymer concrete, GGBS and Matakaolin based geopolymer concrete shows the high compressive strength.

10.2 Split tensile strength

Split Tensile Strength of concrete cylinders 150mm diameter and 300mm long were tested as per the procedure explained in IS 5816.

Table-9. Split tensile strength.

Mix	Split tensile strength N/mm^2		
	3 days	7 days	28 days
M1	6.22	6.53	6.73
M2	5.26	5.87	5.81
M3	4.21	4.65	4.71
M4	3.92	3.98	4.23
M5	3.70	3.81	4.14
M6	3.12	3.72	3.9
M7	3.60	3.71	4.10
M8	3.71	3.79	4.20
M9	4.19	4.50	4.61
M10	5.19	5.77	5.72
M11	6.12	6.37	6.65

VOL. 11, NO. 21, NOVEMBER 2016 ISSN 1819-6608
ARPN Journal of Engineering and Applied Sciences

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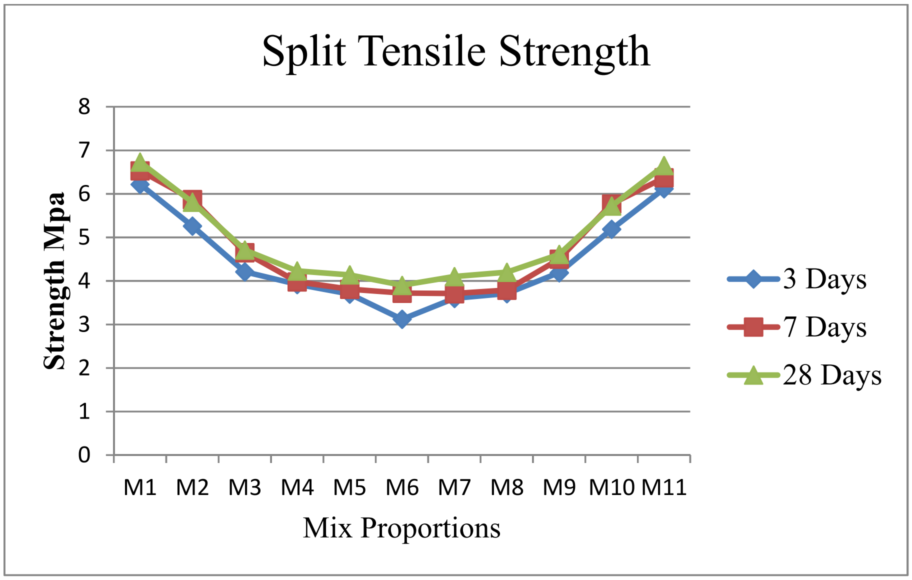

Figure-2. Split tensile strength.

The values of split tensile strength of cylindrical specimens subjected to ambient curing conditions. It is seen that control specimen M1 have higher split tensile strength. Also, the split tensile strength decreases with increase in Metakaoline.

With reference to the Table-8 and Figure-2, at 3 days, the mix proportion M6 has the lowest split tensile strength of 3.12 N/mm^2 and the mix proportion of M1 gave the highest split tensile strength of 6.11 N/mm^2 at 3 days. At 7 days, the mix proportion M1 gives the highest split tensile strength of 6.53 N/mm^2 and the mix proportion M6 gives the lowest split tensile strength of 3.72 N/mm^2. At 28 days, M1 cube specimens attained a split tensile strength of 6.73 N/mm^2 and the mix proportion M6 gives the lowest split tensile strength of 3.9 N/mm^2. It is seen from the results that when the percentage of GGBS and Matakaoline is increased the split tensile strength of the geopolymer concrete. Though addition of GGBS and Matakaoline, increases the split tensile in geopolymer concrete, GGBS and Matakaoline based geopolymer concrete shows the high split tensile strength.

Table-10. Flexural strength.

Mix	Flexural strength N/mm^2		
	3 days	7 days	28 days
M1	3.2	3.41	3.54
M2	1.96	2.57	2.81
M3	1.63	1.67	1.71
M4	1.11	1.58	1.75
M5	0.85	0.9	1.2
M6	0.75	0.822	0.87
M7	0.80	0.90	1.0
M8	1.3	1.1	1.2
M9	1.52	1.56	1.61
M10	1.86	2.46	2.71
M11	3.0	3.31	3.43

ISSN 1819-6608
ARPN Journal of Engineering and Applied Sciences

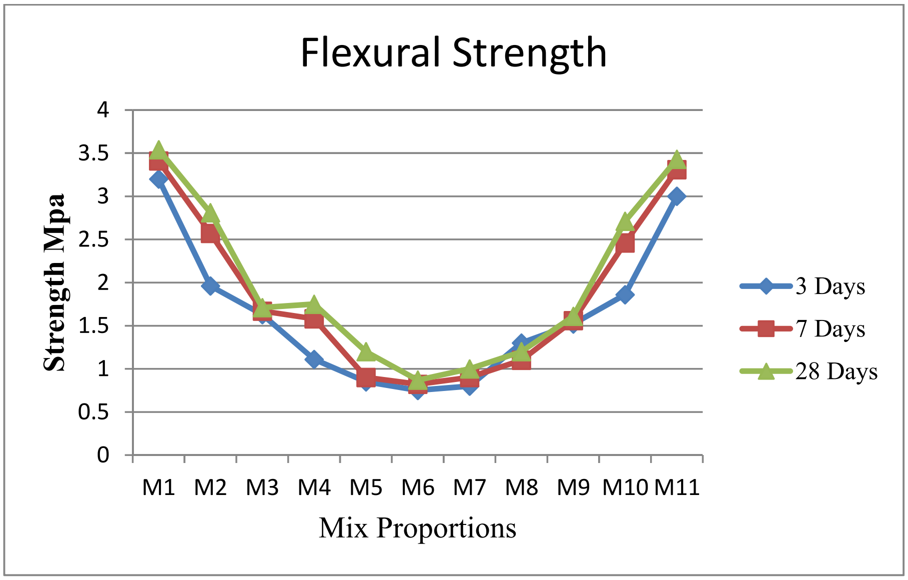

Figure-3. Flexural strength.

With reference to the Table-9 and Figure-3, at 3 days, the mix proportion M6 has the Lowest Flexural strength of 0.75 N/mm^2 and the mix proportion of M1 gave the highest Flexural strength of 3.2 N/mm^2 at 3 days. At 7 days, the mix proportion M1 gives the highest Flexural strength of 3.41 N/mm^2 and the mix proportion M6 gives the lowest Flexural strength of 0.822 N/mm^2. At 28 days, M1 cube specimens attained a Flexural strength of 3.54 N/mm^2 and the mix proportion M6 gives the lowest Flexural strength of 0.87 N/mm^2. It is seen from the results that when the percentage of GGBS and Matakaoline is increased the Flexural strength of the geopolymer concrete. Though addition of GGBS and Matakaoline, increases the Flexural strength in geopolymer concrete, GGBS and Matakaoline based geopolymer concrete shows the high Flexural strength.

11. CONCLUSIONS

The conclusions based on the limited observations from the present investigation on properties of fresh and hardened metakaolin and GGBS based concrete are:

a) Workability of geopolymer concrete decreased as the metakaolin content increases with GGBS. But increase in GGBS does not affect the workability.
b) Mechanical properties such as compressive strength split tensile strength and flexural strength shows increasing trend with the addition of metakaolin.
c) Mix with 50% of metakaolin and 50% of GGBs and seems to have good compressive split and flexural strength this may be due to increase in alkaline reaction between GGBS particles and calcium in Metakaoline.
d) Nearly 90% of total strength of GPC is achieved within age of 7days.
e) The improvement in 7 Days compressive strength over 3days is in the range of 5% to 9% over 3 days and it is 12% to 15 % for 28 Days.
f) The % improvement in strength of 7 Days is 9.25% for the mix M6.
g) Then increase in strength of GPC between 7days and 28days appeared to be high when compared with 3days and 7days.It shows that even after 7days geopolymer reaction is taking place but at a higher rate.
h) Based on the test results, the following three combinations of source material are recommended for making geopolymer concrete used in structural applications

- 100% GGBS
- 70% GGBS – 30% Metakaoline
- 50% Metakaoline - 50% GGBS

ACKNOWLEDGEMENTS

The authors wish to gratefully acknowledge the support of KaoMin Industries regarding Metakaolin supply. The help of the concrete laboratory staff of K L University is gratefully acknowledged.

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