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Strength and microstructural properties of geopolymer mortar using rice husk ash

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Abstract
With increase in industrialization the use of concrete has increased the production of cement which is not environment friendly as it has increased damages to the nature in search of limestone and energy involved. Rice Husk Ash is a waste product which is rich in silica and improper disposal of rice hush ash (RHA) leads to air pollution and land fill problem. The Rice Husk Ash in presence of Silica – Alkali reactor acts as a binding material and can be used effectively in formation of geopolymer mortar. This study was as such carried out to investigate the properties of geopolymer concrete made with rice husk ash to check the suitability as a construction material. In order to achieve the objectives, the geopolymer mortar is being casted with mixture of Sodium Hydroxide and Sodium Silicate as silica alkali reactor with variable proportion of Alccofine i.e. 10%, 30% and 50% The compression strength was also compared with different molarity of Sodium Hydroxide i.e. at 8 Molar, 12 Molar and 16 Molar solutions. This strength was further studied by varying temperature of curing at 27^0C and elevated temperature curing i.e. 90^0C. The chemical composition and microstructure of sample were studied through Energy Dispersive X-ray spectroscopy test (EDX) and Scanning Electron Microscopy test (SEM).

Keywords: rice husk ash, strength, geopolymer mortar, microstructure

Introduction
Due to industrialization the use of cement in construction industry has increased which results in rise of prices of cement in India. The cement also has another disadvantage from environmental point of view as manufacturing of cement produces 5-7% of worldwide CO_2 and due to production of these greenhouse gases it also results in climate change. Due to such concerns various efforts and research are carried out to use waste material in construction. In India rice husk is major waste material as the production of rice in India is in large amount. Every year 20 million ton of rice is produced in India which results in 24 million tons of rice husk and 4.4 million tons of rice husk ash. The production of rice husk is carried out in controlled burning of rice husk to produce rice husk ash having silica and small amount of carbon content. In 1996 Zhang and Malhotra referred a comparative study of engineering characteristics of silica fumes and rice husk ash as pozzolanic additives for high performance concrete. With revelation to high carbonation the resistance of mortar having rice husk ash toward chloride is decreased. In 2000 Mehta and Pearth carried out a series of experiments and proposed that rice husk ash can be used effectively for lowering temperature of mass concrete as compared to Ordinary Portland Cement (OPC). Later 2004 Mehta and Malhotra gives statement after carrying out various researches that rice husk ash with particle finer than ordinary Portland cement enhances the concrete properties i.e. higher replacement amount results in reducing the value of water absorption and increases its compressive strength.

Materials and Experimental Procedure
Material
Alccofine 1203 is being used for research work. Particle size of alccofine is 4-6 micron which is much smaller as compared to cement which is 50-90 micron. Specific gravity of alccofine is 2.86 and that of cement is 3.3 and bulk density is 600-700 kg/m^3 Rice husk ash was obtained from open burning in a small heap of rice husk with maximum temperature of burning of 650°C

Mix Proportion
Rice Husk Ash is being replaced by using alccofine 1203 in proportion 90-10, 70-30, 50-50 by weight. For geopolymer mortar mixing and geopolymer mortar activation sodium hydroxide (NaOH) and sodium silicate (Na_2SiO_3) is used. The experiment is carried at consistency of 0.4 of solid. The experiment is carried out at 8 molarity, 12 molarity and 16 molarity of sodium hydroxide with equal quantity of sodium silicate. A total of 9 samples and 27 specimens were prepared of different proportion and at different molarity whose compressive strength is being checked at 7 days and 28 days. The cubic specimen of mortar of size 70.6 x 70.6 x70.6 mm were cast for determination of all measurement 9 samples of each molarity was casted with 3 of each proportion making the total 27 samples. The average compressive strength is considered.
In presence of sodium hydroxide and sodium silicate reaction in geopolymer mortar takes place. The main reaction component in geopolymer mortar sodium aluminosilicate

hydrate gel (NASH) which is alkali activated material. As we know the Rice Husk Ash is rich in silica so it can be used for making geopolymer mortar.

Table 1: Mix Proportion of geopolymer mortar

Mix Designation	Fine Aggregate (kg/m^3)	Rice Husk Ash (kg/m^3)	Alccofine (kg/m^3)	Activator solution (kg/m^3)	Molarity
GPM1 (10%AF)	1141.54	342.46	38.05	608.82	8
GPM2 (30%AF)	1141.54	266.36	114.15	608.82	8
GPM3 (50%AF)	1141.54	190.25	190.25	608.82	8
GPM4(10%AF)	1141.54	342.46	38.05	608.82	12
GPM5 (30%AF)	1141.54	266.36	114.15	608.82	12
GPM6 (50%AF)	1141.54	190.25	190.25	608.82	12
GPM7(10%AF)	1141.54	342.46	38.05	608.82	16
GPM8 (30%AF)	1141.54	266.36	114.15	608.82	16
GPM9 (50%AF)	1141.54	190.25	190.25	608.82	16

Preparation, casting, and curing of Samples

Before the mixing of the mortar ingredients, aggregate were prepared to the saturated surface dry condition. Sodium hydroxide was prepared 24 hours prior to final mixing. In this study, NaOH and Na_2SiO_3 were mixed about 1 hour before the mixing of dry components used in GPC. All the dry ingredients such as RHA, aggregates, and Alccofine were dry mixed with the pan mixture. Then alkaline activator solution is added at a slow rate to the dry materials. The mixing was done for about 5 minutes to produce Alccofine activated fresh mortar. After mixing all the ingredients in pan mixture, the mix was poured into cube mould in three layers and put on the vibrating table for proper compaction. The compaction was done for 1 to 2 minutes. 70.7 mm cubes were cast for compressive strength tests. A rest period of one day is given to all the specimens. The samples were then cured at the ambient and heat condition at 27°C and 90°C.

Compressive Strength Test

The geopolymer mortar being cast in 70.6 x70.6 x 70.6 mm is being cured at 27 ± 2'C and at high temperature (90'C) The is being tested at 7 days and 28 days. The following is the strength obtained in experiment at various temoerature and at different molarity.

Table 2: Molarity table

Sr. No	Molarity	Mass of NaOH (gm)	Mass of water (gm)
1	8 M	262	738
2	12 M	361	639
3	16 M	444	556

Following results are being obtained from Experiments at room temperature

Table 3: Compressive Strength of 8M solution specimen at ambient temperature

	Compressive Strength (N/mm^2)		
	3 Days	7 Days	28 Days
GPM1	11.72	18.78	28.45
GPM2	15.05	22.71	33.30
GPM3	12.57	19.12	28.50

From the results it can be seen that, the compressive strength of the specimen increased from 11.70 MPa to 28.45 MPa and from 15 MPa to 33.30 MPa at the ages of 3 days and 28 days for 10% and 30% Alccofine, respectively. The strength of 30% Alccofine specimen is about 20% more than that of 10% Alccofine. After 30% strength started decreasing as can be seen in GP3 specimen. Also, the rate of gain of strength increased after 7 days as shown in Fig 4.1

Table 4: Compressive Strength of 8M solution specimen at elevated temperature

	Compressive Strength (N/mm^2)		
	3 Days	7 Days	28 Days
GPM1	25.41	46.20	47.81
GPM2	30.45	52.50	54.60
GPM3	22.38	43.05	44.38

The result showed a maximum compressive strength of 54.60 MPa with 30% Alccofine at 28 days in heat curing. There is an increase in strength of 13.60% as the Alccofine proportion increases from 10% to 30%. On the other hand, it showed a significant decrease in strength of about 18% when the proportion was increased from 30% to 50%. Unlike the ambient cured sample in this case the rate of gain of strength was lowered after period of seven days as described in above figure.

Table 5: Compressive Strength of 12M solution specimen at ambient temperature

	Compressive Strength (N/mm^2)		
	3 Days	7 Days	28 Days
GPM4	12.60	19.68	29.03
GPM5	14.31	22.91	34.40
GPM6	12.56	19.88	29.07

From the results, it can be depicted that compressive strength of mortar when prepared with 12M alkali activator solution increased to about 4-7% than the mortar which was prepared using the 8M solution. Also, similar to above ambient cured sample this graph also showed rise in slope of line which clearly showed that there is gain in rate of gain of strength after a period of 7 days.\

Table 6: Compressive Strength of 12M solution specimen at elevated temperature

	Compressive Strength (N/mm^2)		
	3 Days	**7 Days**	**28 Days**
GPM4	26.12	46.23	48.00
GPM5	32.80	53.76	55.48
GPM6	25.63	43.81	45.35

For heat cured samples it can be concluded that the compressive strength of mortar when prepared with 12M alkali activator solution increased to about 1-3% than the mortar which was prepared using the 8M solution. The strength of heat cued sample was more than 12M ambient cured sample by 38% The behavior of rate of strength gain was similar as in above case of heat cured sample.

Table 7: Compressive strength of 16M sample at ambient temperature

	Compressive Strength (N/mm^2)		
	3 Days	**7 Days**	**28 Days**
GPM7	13.00	21.48	31.18
GPM8	17.06	26.54	38.24
GPM9	13.23	20.97	30.13

From the results, it can be depicted that compressive strength of mortar when prepared with 16M alkali activator solution increased to about 14-16% and 10-12% than the mortar which was prepared using the 8M and 12M solution, respectively. The strength of GPM7 and GPM9 showed nearly same strength at age of 3 days

Table 8: Compressive Strength of 16M sample at Elevated Temperature

	Compressive Strength (N/mm^2)		
	3 Days	**7 Days**	**28 Days**
GPM7	28.58	49.15	50.94
GPM8	29.38	56.18	58.30
GPM9	25.00	46.51	48.21

For heat cured samples it can be concluded that the compressive strength of mortar when prepared with 16M alkali activator solution increased to about 6-8% and 8-9%than the mortar which was prepared using the 8M and 12M solution, respectively. The 28 days strength of 16M elevated cured sample was higher than that of ambient cured sample by 34.5%

EDX Test

It stands for Energy-dispersive X-ray spectroscopy (EDS, EDX, EDXS or XEDS). It is a technique of analyzing chemical charaterstic of a sample. It depends on interaction of some source of X-ray excitation and a sample. It is done by exciting the sample and focusing an electron beam on it. This excitation causes emission of X-RAY from the sample. These individual X-RAYs are picked up by X-RAY detector and converted into electrical voltage signals. The signal produced in these samples act as a basic for element analysis and allows to elemental analysis to heat to be conducted in samples. In this when electron beam is focused on sample it strikes on electron and forcefully take place of that electron this state become very unstable so in order to have stability element release energy in form of X-RAY.EDX report of sample with 50% Alccofine 1203 ambient temperature:

Standards in sample are silica oxide, alumininum oxixde, potassium in form of MAD-10 felspar, calcium in form of wollastonite. The weight percentage and their atomic percentage is given below.

Following is the Edx report of sample with 50% Alccofine1203 at ambient tempertature of 8 Molar specimen.

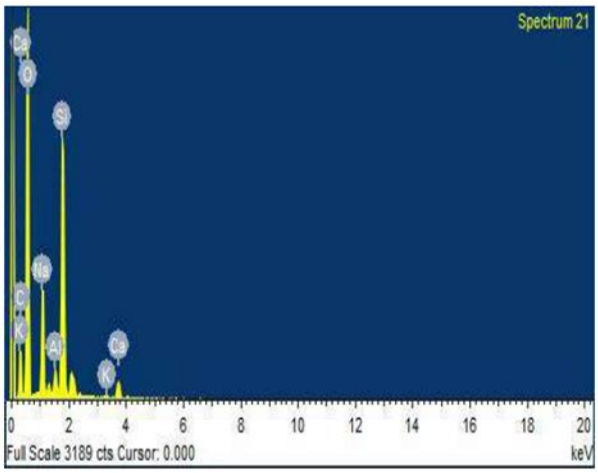

Fig 1

Table 9

Sample	**Ambient cured sample with 50% alccofine**	
Element	**Weight %**	**Atomic %**
O K	53.26	68.05
NA K	6.59	5.86
AL K	1.52	1.15
SI K	24.03	17.49
K K	1.02	0.53
CA K	13.58	6.93
Total	100	

Following is the Edx report of sample with 50% Alccofine1203 at elevated tempertature of 8 Molar specimens.

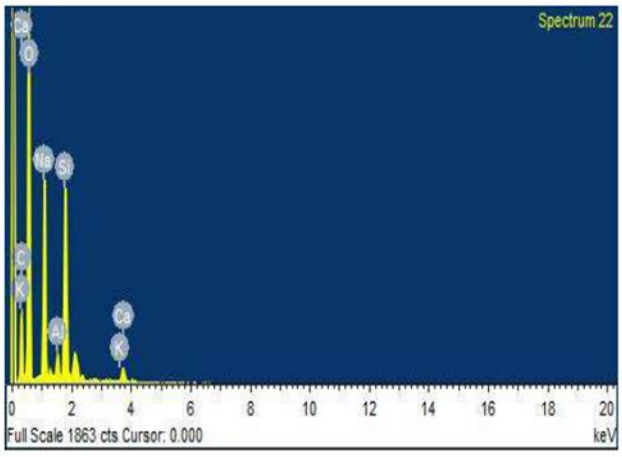

Fig 2

Table 10

Sample	Heat cured sample with 50% arccosine	
Element	**Weight %**	**Atomic %**
O K	53.40	68.10
NA K	13.23	11.57
AL K	1.76	1.31
SI K	19.39	13.88
K K	0.66	0.34
CA K	11.57	5.80
Total	100	

The above report shows that the percentage of present elements is different in elevated temperature cured and ambient temperature cured specimens. The major change in change in chemical composition is shown in silica, sodium and potassium sample. As it is shown from compressive strength sample table the strength of heat cured sample is high than that of ambient cured sample. One can conclude that strength gain is rapid in elevated temperature samples that mean that the hydration and polymerization process is fast and better when temperature rises at time of curing period.

There are two figures shown above of EDX report i.e. Fig 4.6 & Fig 4.7 with different curing conditions of 8 Molarity sample. Observations are showing that there is a difference in percentage of both the specimens i.e. 6.59% and 13.23% respectively. Also the observations are showing that there are difference in other element compounds too i.e. Potassium, Silica Oxygen etc. and this is due to change in temperature at time of curing, geopolymerization and hydration process of sample.

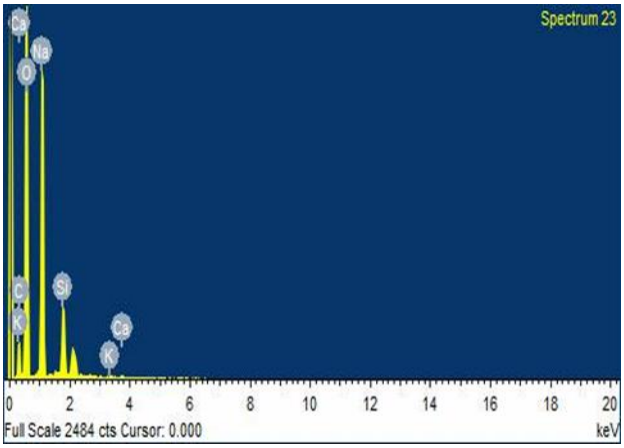

Fig 3

Table 11

Sample	Ambient cured sample with 10% arccosine	
Element	**Weight %**	**Atomic %**
O K	61.53	71.13
Na K	26.89	21.63
Si K	9.61	6.33
K K	0.19	0.09
Ca K	1.78	0.82
Total	100	

Following is the Edx report of sample with 10% Alccofine1203 at elevated tempertature of 8 Molar specimens.

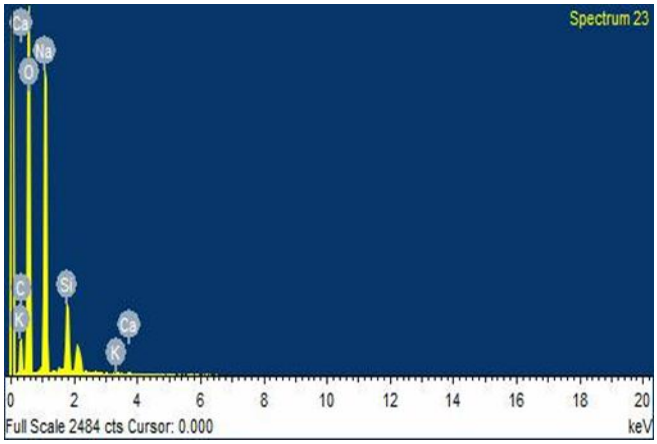

Fig 4

Table 12

Sample	Ambient cured sample with 10% alccofine	
Element	**Weight %**	**Atomic %**
O K	64.05	74.93
Na K	15.46	12.63
Si K	15.03	9.87
K K	1.05	0.50
Ca K	4.41	2.07
Total	100	

After complete observations of two different samples i.e. 10% Alccofine and 50% Alccofine. The readings are noted in table 4.9 and 4.8 above. The tables are showing that there was a difference in percentage of oxygen compound i.e. 64.05% in 10% sample and 53.4% in 50% Alccofine sample respectively. It is also observed that there is an increase in sodium compound if we compare both the specimen i.e. 13.23% in 50% Alccofine sample and 15.46% in 10% Alccofine sample respectively. But the observations are showing that unlike Sodium and oxygen, potassium compound percentage is decreasing i.e. 1.05% in 10% Alccofine specimen and 0.66% in 50% Alccofine specimen process. The strength of 10% Alccofine sample is more than that of 50% Alccofine sample. It can be concluded from these tables that, in 10% Alccofine sample silica is taking part in hydration or geopolymerzation process due to which the percentage of silica compound is increasing. On other hand in 50% Alccofine sample the silica is not taking any part in hydration or in geopolymerization process. Thus the strength of 10% Alccofine sample is higher than that of 50% Alccofine sample.

Scanning Electron Microscopy (SEM)

It is a type of electron microscope in which an electron bean is passed and focused on specimen. This beam when strikes on specimen release energy and displace the electron from compound and the vacant place of electron in compound is taken by electron of electron beam. For example if it focused on sodium than sodium has two electron in its outermost shell So, beam replace electron first from outer most shell and then from inner shell These free electrons are highly unstable and are sensed by sensors which collectively produce enlarged images of sample.

SEM images of samples

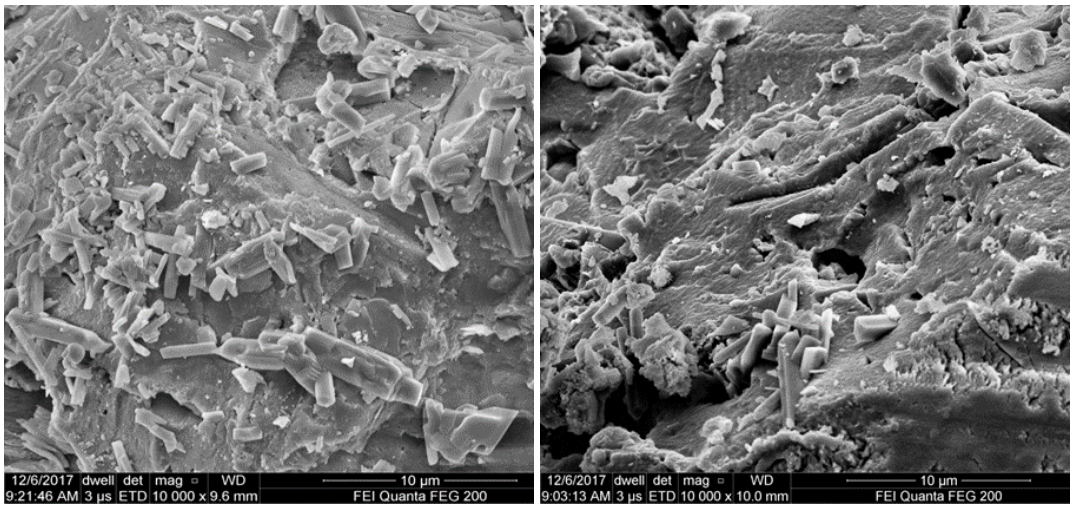

Fig 5: (a) 8 Molar specimen heat cured, (b) 8 Molar specimen ambient cured with 50% Alccofine

The both the above images shows the microstructure of same sample but treated differently i.e. one is cured at room temperature and other at elevated temperature. This creates the difference in their micro structure. The first difference one can make from these images is the difference of color the color of ambient cured specimen is dark as compared to that of heat cured. The heat cured sample shows low percentage of flaky minute structure that shows the geopolymerization of specimen in hydration process. The higher the structure lower is the strength gain in sample.

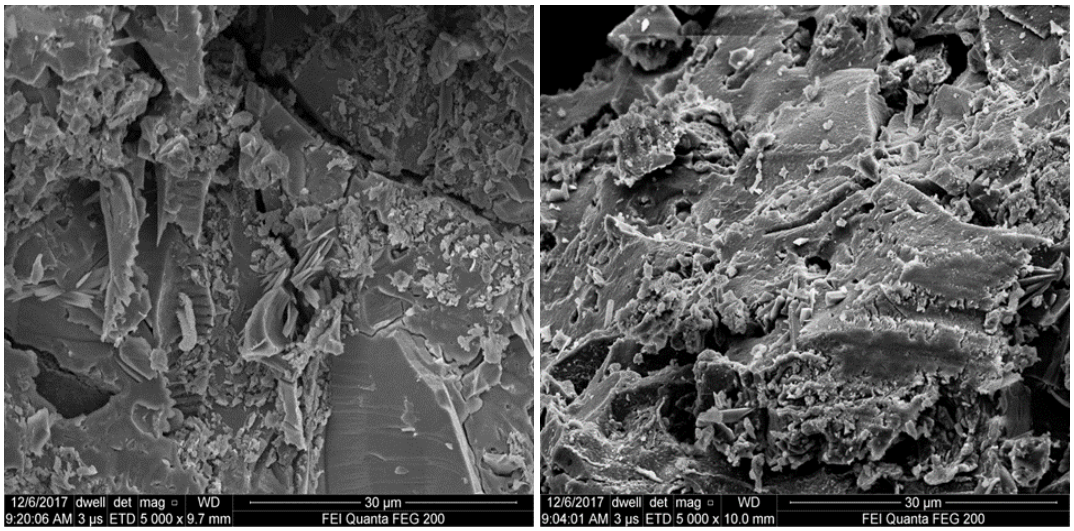

Fig 6: (a) 8 Molar specimen heat cured, (b) 8 Molar specimen ambient cured with 50% Alccofine

The above images show the formation of cracks in specimen. These cracks are produced with strength gain in specimen through geopolymerization of specimens. The strength gain is carried out on formation of NASH gel and CSH gel of Alccofine and geopolymerization of Alccofine with Rice Husk Ash. The formation of crack are also the result of shrinkage in hydration process and formation of capillary porosity but being fine in nature the porosity and size of compared to that in cement mortar is much small in nature. The small is size of void and its percentage higher is its strength gain.

In following images these are the voids left behind in the specimen cured at room temperature. The voids are small as in scale less than 10 micron meter but it adversely affect the strength of specimen. These voids resemble the shape of a hole in sample and may be due to improper mixing of ingredients of may be any other reasons

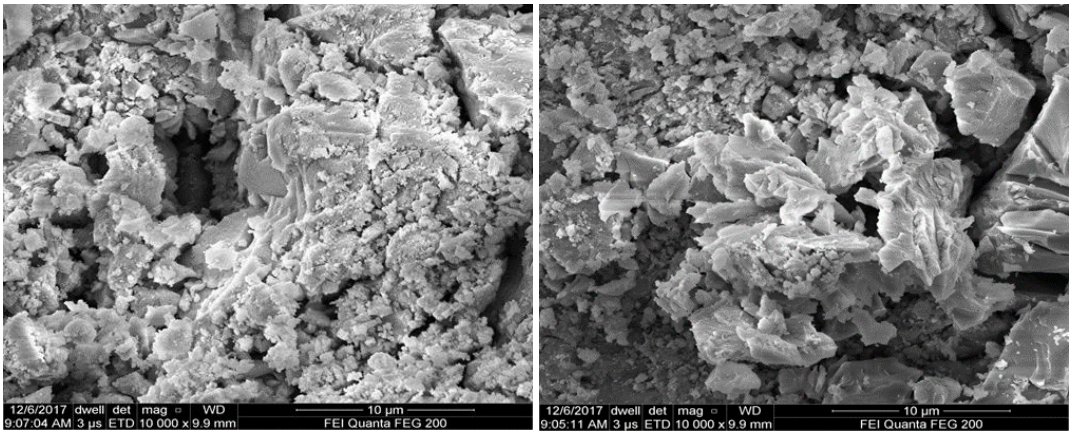

Fig 7: Voids formed in 50% Alccofine specimen ambient cured

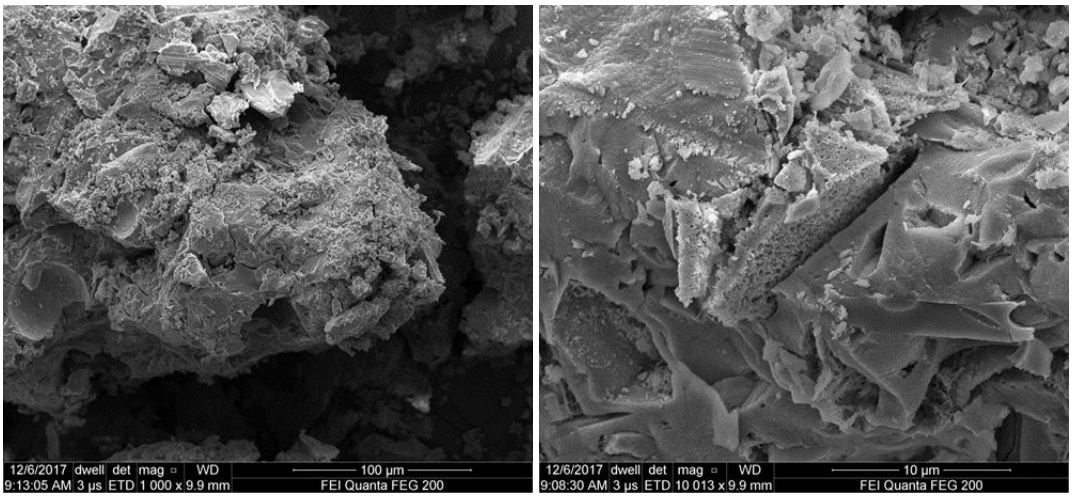

Fig 8: (a) Unreacted RHA particle (b) unreacted Alccofine particle

These above two figures are of specimen having 50% Alccofine and cured at room temperature. The figure (b) resembles the unreacted particle of Alccofine which forms bread like structure in specimen. The formation of such structure is highly undesirable as it directly affect the strength of specimen. The figure in right (a) represents a spherical unreacted Rice Husk Ash particle. Similarly to Alccofine case it too effect the strength and undesirable.

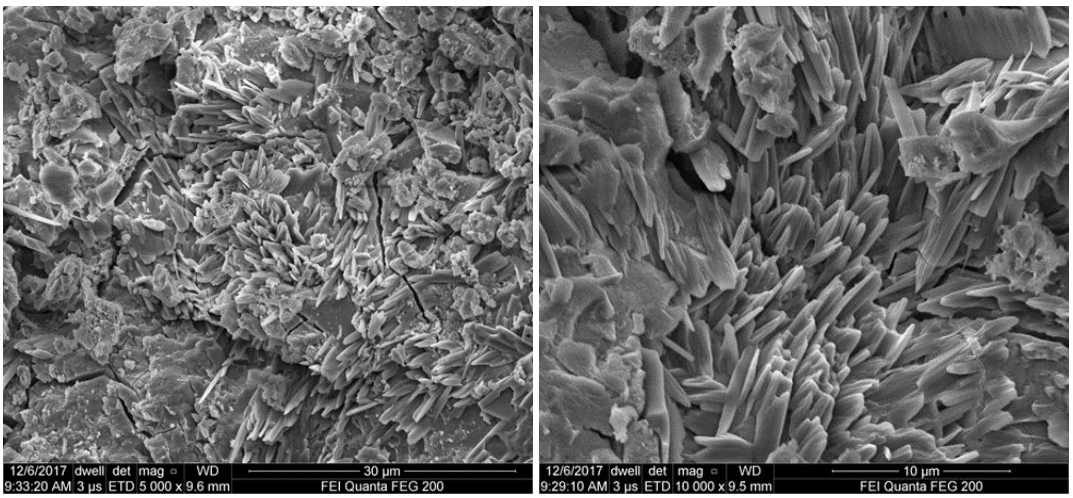

Fig 9: SEM images of 10% Alccofine specimen at room temperature

The above figure shows large number of flaky structure as observed in above cases. This represents the presence of NASH gel and CSH gel. The small no of micro cracks in it shows that the strength gain of sample is not complete totally.

When strength gain of sample is complete i.e. hydration process is complete in it than the amount of flaky structure decreases which decreases the percentage of sodium compound in sample as it decreases with heat curing sample when compared to that of specimen cured at room temperature.

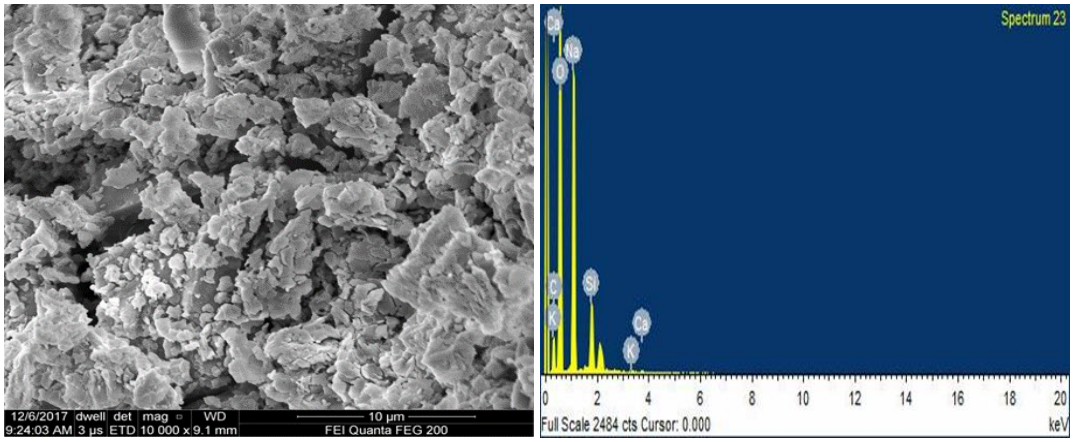

Fig 10: SEM images and EDX graph of 10% Alccofine specimen at room temperature

The above image on comparing with EDX shows a high percentage of Sodium in form of Sodium Albite and silica dioxide. So, it shows the presence of Sodium Aluminosilicate Hydrate gel having large amount of void in it which form structure close to pallets of sodium. The dark grey in back ground is carbon tape on which sample is stick for SEM and EDX tests.

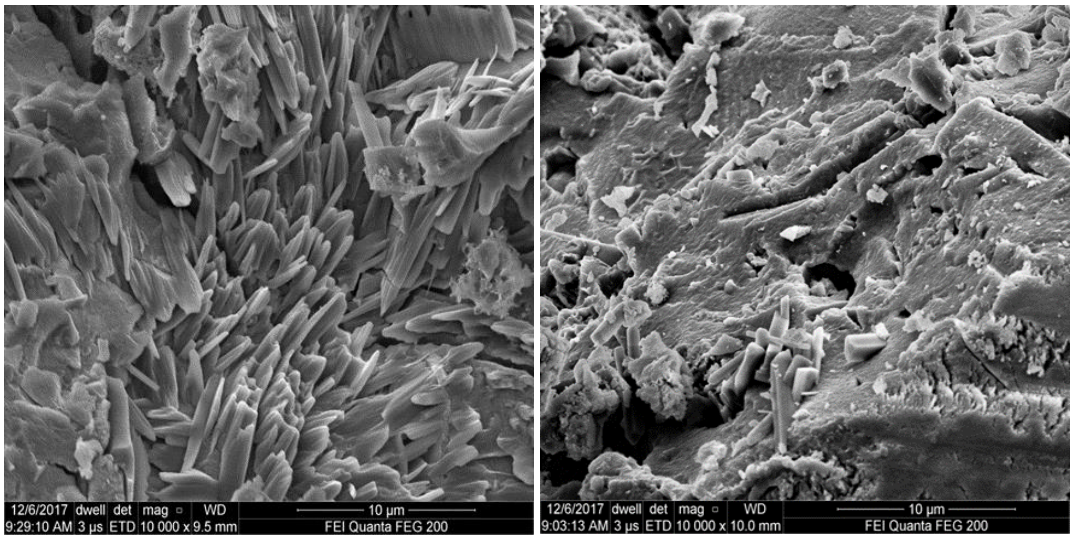

Fig 11: SEM images of 10% and 50% Alccofine specimen at room temperature

The above images show the microstructure of ambient cured sample with 10% and 50% Alccofine. The Alccofine variation causes variation in Aluminosilicate also. In 10% sample the Aluminosilicate was collective and at near to each other but when Alccofine percentage is increased there is dispersion in Aluminosilicate. So, from these images and compressive strength table it can be concluded that maximum strength is obtained when Aluminisilicate is more scattered nor when Alumiosilicate is concentrated at one place. The maximum strength as seen in strength table is obtained at optimum quantity of Alccofine i.e. 30% (GP2) which also effect the position of Aluminosilicate. It will scatter the Aluminosilicate as compared to its concentration in Mix1 and it will be less scattered than its concentration in GPM3

Analysis of Result

From the above findings it can be seen that highest compressive strength is obtained at elevated temperature of highest molarity sample i.e. 16 Molar sample. It can also be seen that with increase in percentage of Alccofine the strength of sample first increases and then decreases. Now the maximum strength of sample was obtained with optimum quantity of Alccofine i.e. 30%

From the above results it can be seen that compressive strength of 16 Molar Heat cured sample at 30% Alccofine is 4.84% higher than 12 Molar sample and 6.34% than 8 Molar sample. Also, the strength of heat cured sample was up to 52.45% higher than that of ambient cured sample. It is also observed in above results that the strength gain of ambient

cured sample is approximately liner after 7 days (a little reduction in slope) but that of heat cured sample the strength gain is quite rapid in first seven days and then the rate strength gain of sample reduces which cause reduction in slope of curve. This is due to rise in rate of hydration and silica alkali reaction initiated by silica alkali reactor.

Also, when Molarity increase from 8 Molar to 12 Molar and 12 Molar to 16 Molar the increase in strength takes place i.e. 11.09% between 8 Molar samples and 16 Molar sample in 3 days and 8.6% between 12 Molar and 16 Molar in 3 days. Similarly 6% and 5.9% respectively in 7 days and 5.14% and 5.8% in 28 days respectively in heat cured 10% Alccofine samples.

The another results obtained from above samples is that the compressive strength of geopolymer mortar is attributed by formation of sodium Aluminosilicate hydrate gel and 3-D network of silicoaluminate structure higher the concentration of sodium hydroxide higher is formation of aluminosilicate gel.as a result of it a stable silicoaluminate is produced to provide higher strength to mortar. The strength of sample increases with production Aluminosilicate gel. Moreover when aluminosilicate gel is well dispersed throughout the sample or concentrated at one place the strength gain will be lower. The maximum strength will be obtained at optimum dispersion of aluminosilicate gel.

Conclusion

Based on the observations and discussion made in the study following conclusion can be derived upon

- Maximum strength was observed with 16M NaOH, 30% Alccofine and at heat curing.
- Temperature significantly affected the strength of the RHA based geopolymer mortar. Strength of 38.24 MPa for ambient cured and 58.3 MPa for heat cured sample was noticed, respectively.
- The percentage increase in strength increased when Alccofine content changed from 10 to 30% of RHA. However, opposite trends were observed when Alccofine content changed from 30 to 50% of RHA in all the cases.
- The strength of geopolymer mortar increased with increase in molarity of samples. The strength of the specimens with same percentage of Alccofine (30%) and heat curing were 54MPa, 55MPa and 58.3MPa with 8M, 12M and 16M NaOH solution, respectively.

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