International Journal of Recent Technology and Engineering (IJRTE)
ISSN: 2277-3878, Volume-8 Issue-5, January 2020

Fly Ash Based Geopolymer Mortar - Carbonation and Microstructural Performance

Salmabanu Luhar, Ismail Luhar

Abstract*: Concrete is the construction material of highest exigency which, in turn, created a huge demand for ordinary Portland cement as a binder. Regrettably, its current production process is highly energy intensive, and at elevated temperature, when Calcination of Limestones takes place, it liberates embodied anthropogenic CO_2 polluting the environment. Not only that, but it is also user-hostile and comparatively costly too. Therefore, construction industries looking at concrete research sectors to invent new, environmentally- compassionate durable, sustainable and cheap construction and binding materials instead of present ones. Nowadays, Geopolymer binders and composites are synthesized by activating alkali solution with Silica and Alumina rich pozzolanic material such as Fly ash, have emerged out as a brilliant substitute to conventional binders and composites through the process of Geopolymerization. The objective of the present study is to investigate the performance of Fly ash based geopolymer mortar in whereby there is 100% substitution of cement by Fly ash, i.e., the absolute absence of cement. The present study has examined the mix composition parameters comprise Sodium hydroxide concentration and aggregate to binder ratio, whereas process parameter included curing temperature. Also, the Carbonation tests were conducted along with microstructure investigations viz., X-Ray Diffractometry (XRD) and Scanning Electron Microscopy (SEM).

***Keywords* : Geopolymer mortar; Carbonation; fly ash,; Waste tyre; Microstructure.**

I. INTRODUCTION

In earlier times, ancient Egyptians were employing mortars which were made from burned Gypsum and Sand while subsequent advancement in mortar technology applied a blend of Lime and Sand which developed their strength at a snail's pace in the course of Carbonation. The significance of mortar strength is wide-ranging. It is far and extensively recognized that strength of construction structure relies upon the strength of mortar. It is a material employed in masonry type of constructions to plug and seal the irregular spaces among the building blocks viz., bricks, stones, and concrete blocks together, etc. utilized in constructional and infrastructural works. That means the key objective of mortar is to bind together the individual masonry units adhesively.

Consequently, it protects against the penetration of air and water through the joints in a masonry assembly through bonding the non-masonry elements of an assembly like joint reinforcement and ties. A good balance of strength and bond is necessitated. This drives to both good structural performance and weather resistance. For these reasons, the durability of Geopolymer mortar is also a significant part to investigate in detail. It is a well-known fact that production of binder of conventional mortar, i.e., Ordinary Portland Cement (OPC), not only devours natural restricted resources as raw materials but also emits CO_2- a primary Green House Gas (GHG), pushing a solemn dilemma of Global warming and polluting environment too [1-8]. This high temperature obtained by burning natural confined minerals Coals reactions consumes high energy [9-12]. Hence, researchers, engineers, scientists and concrete technologists are compelled to beat the bushes for the user and eco-friendly, sustainable and at same the time economical building materials [13-16].

Quite recently, an innovative mortar of Geopolymeric origin has attracted the attention of researchers on account of its excellent attributes. Geopolymer mortar is an inorganic construction material which is found durable and sustainable by touching acceptance limits. Geopolymer construction technology has lent its hand in order to offer the same through the exhibition of their excellent strength, durability, thermal, freeze thaw, etc. attributes establishing them as novel promising sustainable construction materials.

Geopolymers are inorganic Zeolite like materials which can be produced by reaction between pozzolanic Alumina and Silica rich precursor and alkali activators through an exothermic process of Geopolymerization at low temperature in alkaline medium [17,18]. Fly Ash is such an Alumina and Silica rich pozzolanic industrial by-product found in copious quantity filling lands and posing challenges of not only health hazards but also polluting air, soils, surface and sub-surface water. As a result, to get rid of such a titanic amount wastes of Fly Ash its systematic disposal management is the need of the hour. Thus, to use it for manufacturing of Geopolymer mortar can lend a hand to solve the purpose. Durability parameters of Geopolymer mortar through carrying out laboratory tests will direct to judge its sustainability which is highly desirable statistics to prove it as a sustainable material. That is the reason why the author has gone after investigations on a significant parameter of durability in the context of Geopolymer mortar.

Manuscript published on January 30, 2020.
* Correspondence Author
Salmabanu Luhar*, Institute of Mineral Resources Engineering, National Taipei University of Technology, Taipei, Taiwan.,
Malaviya National Institute of Technology, Rajasthan, India,
Ismail Luhar, Shri Jagdishprasad Jhabarmal Tibrewala University, Rajasthan, India.

II. MATERIAL PARAMETERS

Following materials were used to making fly ash based geopolymer mortar. Table -1 shows material characterization [8].

Table-I: Material characterization

Materials	Value/Type
Fly ash	Class-F
Rice husk ash	Locally available
Fine aggregate, water absorption, fineness modulus, specific gravity	0.5%, 2.56 and 2.61, respectively [3].
Sodium silicate	Water glass
Sodium hydroxide flakes	having 98% purity in distilled water were used.

III. INVESTIGATION OF THE APPARENT POROSITY AND WATER ABSORPTION OF GEOPOLYMER MORTAR

The porosity of mortar is a vital and necessary property for predicting its durability and strength. In this study, the influence of the aggregate binder ratio and curing temperature on the apparent porosity of the geopolymer mortar was examined.

A. Test Method

The apparent porosity can be predicted indirectly by calculating the bulk density. However, being a porous matrix, such as indirect methodology may produce erroneous results. In view of the above, Montes et al. [17] proposed a novel methodology to measure the porosity of mortar and concrete using Archimedes principle. The apparent porosity “n" can be expressed regarding weights as,

$$n = \frac{(M_w - M_d)}{(M_w - M_s)} \times 100 \quad (1)$$

Where,

Mw = Weight of specimen after immersion in water for 48 h

Md = Weight of specimen after drying in an oven at 85°C for 24 h

Ms = Weight of specimen when suspended in water

In the present study, geopolymer mortar specimens were cast by mixing the various components in predefined proportions. The specimens had dimensions of 50 mm x 50 mm x 50 mm. After air curing the mortar specimens for the duration of 28 days, the samples were placed in an oven for drying. The dry weight was measured at 24 h periods until the difference between dry weights measured in two subsequent drying durations was less than 0.05%. This final dry weight is Md.

Further, the specimen was soaked in water for 48 h, and its saturated surface dry weight was measured. This process was repeated at 24 h intervals to a tolerance of 0.05%. The final saturated surface dry weight is Mw. Finally, the weight of the specimen was measured in suspension in the water, giving Mw. Finally, based on the above equation, the apparent porosity was calculated.

B. Test Programme

This test examined the effect of changes in the aggregate to binder ratio and curing temperature on the apparent porosity and water absorption capacity of the mortar. For this purpose, the test matrix in Table 1 was adopted. The average apparent porosity was calculated by measuring the apparent porosity of three specimens with identical compositions and curing conditions. The next section presents the results of all tests conducted to evaluate the apparent porosity and water absorption capacity of the specimens.

C. Result Analysis and Discussion

Fig.1 shows the variation in porosity concerning the aggregate to binder ratio and curing temperature. It can be seen that the porosity initially decreases as the aggregate to binder ratio increases; after a certain point, porosity increases with further increases in the aggregate to binder ratio. The initial decrease in porosity can be attributed to the proper compaction of the fine aggregates and binder into the mortar matrix. An increase in the curing temperature helps the mortar to achieve a densified matrix, and lower porosity, which is evident from Fig.1.

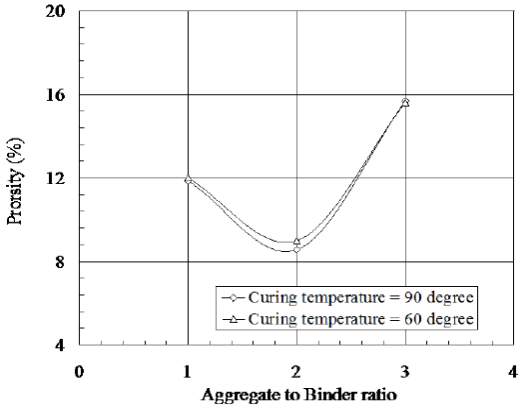

Fig.1. Variation of porosity with aggregate to binder ratio

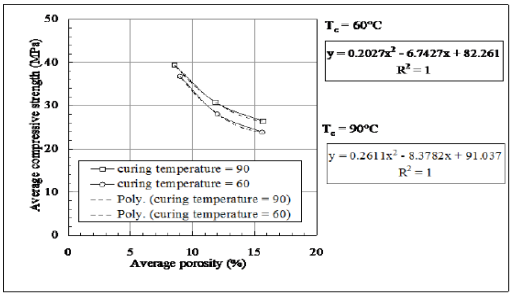

Fig.2 Relation between compressive strength and porosity

Fig.2 shows the relation between average compressive strength, average porosity, and curing temperature. Note that the compressive strength of the alkali-activated geopolymer mortar is inversely proportional to average porosity. With an increase in temperature, at approximately constant porosity, the average compressive strength increases.

The above relation can be fitted to a polynomial to obtain an empirical relation between the parameters. This relation could then be used to predict the average compressive strength of the geopolymer mortar.

IV. INVESTIGATION OF THE CARBONATION OF GEOPOLYMER MORTAR

According to the literature, the durability of the mortar is significantly affected by exposure to CO_2. To understand the process of carbonation and the effect of the mortar mix composition on the carbonation process, a series of experiments was conducted. This section describes the test method and programme adopted in the present study and presents the test results and discussion.

A. Preparation of mortar specimen

The carbonation of the mortar specimens was conducted according to the CPC18 standard [19].

Pre-defined proportions of the various components were blended manually to achieve the mix composition. The paste was poured into moulds to prepare specimens as shown in Fig.3. The beam specimens had a length of 160 mm, breadth of 40 mm, and height of 40 mm. After curing at room temperature for 300 min, the specimens were placed in an oven for 24 h to achieve curing due to heating.

B. Test procedure

Accelerated carbonation tests were carried out in a chamber under specific experimental conditions: atmospheric pressure, the temperature of 20 ±2°C, relative humidity of 65 ±5%, and a constant concentration of CO_2. The concentration of CO_2 was maintained at 5% to allow for a reasonable test duration in the laboratory. The relative humidity was maintained at 65% using an ammonium nitrate saturated solution in the carbonation chamber. Specimens were placed into the carbonation chamber.

After air curing, the specimens were painted on four faces using two layers as explained in CPC18 [7] shown in Fig. 4. The carbonation was quantified in terms of the variation in carbonation with depth. The carbonation depth can be used to compute the carbonation coefficient of the geopolymer mortar. To measure the carbonation depth, the beam specimens were split into two parts along their length after completion of the carbonation process. A phenolphthalein solution (prepared by mixing 1% phenolphthalein in 70% ethyl alcohol) was sprayed on the freshly broken surface.

This solution is used to identify the change in pH of the freshly broken surface. In the presence of this solution, non-carbonated surfaces become pink, whereas carbonated surfaces remain colourless. The difference in the colour profile was then measured according to a predefined scale. Measurements of the carbonation depth were conducted according to CPC18 [19] guidelines. These guidelines recommend a certain methodology when the profile of the carbonation is undulating or curvy.

C. Test Programme

The test series for the carbonation experiments was termed Series B. A mix composition with a NaOH concentration of 14M was selected. Further, parameters for the aggregate to binder ratio and curing temperature were varied while the values of other parameters were held constant. Table-I presents the test programme for series B and the corresponding parameter values.

Table-I. Summary of the test programme adopted for Series B in the present study

Mixes	NaOH concentration	aggregate to binder ratio	Curing temperature (°C)
C-1	14M	1:1	60°
C-2	14M	2:1	60°
C-3	14M	3:1	60°
C-4	14M	1:1	90°
C-5	14M	2:1	90°
C-6	14M	3:1	90°

D. Results and Discussion

In this study, the influence of the aggregate to binder ratio and curing temperature on the carbonation process of geopolymer mortar was studied. For this purpose, several samples with the mix compositions stated in Table-I were placed in the carbonation chamber. The samples were tested at specific times, and the carbonation depth was measured according to CPC18 [19].

V. INFLUENCE ON CARBONATION DEPTH

Figs. 3 and 4 depict the variation in carbonation depth with time and aggregate content in the geopolymer mortar. As can be seen from Fig.4, the carbonation depth increases with carbonation time. The carbonation depth was found to be the smallest in samples with an aggregate binder ratio of 2:1. Note that the compressive strength test and porosity test indicated that the geopolymer made up of an aggregate to the sand ratio of 2:1 gave the maximum compressive strength and minimum porosity. Hence, the reduced amount of carbonation can be attributed to the decreased porosity and compact state of the geopolymer matrix. Similarly, the samples with an aggregate-binder ratio of 3:1 gave the maximum carbonation depths throughout the test. The higher amount of carbonation can be attributed to the increased porosity of the sample, as evident from the porosity test results reported in previous sections.

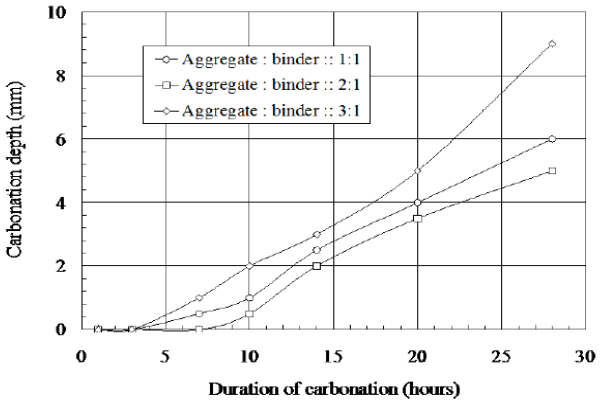

Fig.5. Variation of carbonation depth with carbonation time (h) (curing temperature 60°C)

Fig.3 demonstrates the variations in carbonation depth with carbonation time for the samples cured at 90°C. It can be seen that a marginal decrease in carbonation depth was achieved at the higher curing temperature. This can be attributed to the solidification of the gel under the elevated temperature, resulting in a decrease in the porosity of the matrix. This decrease in porosity hinders the diffusion of CO2 into the matrix of the geopolymer mortar. Based on the results of the carbonation experiments, it can be concluded that the durability of geopolymer mortar against carbonation can be improved by tuning the mix composition of the mortar. It was found that the fine aggregate to binder ratio and curing temperature are vital mix composition parameters in dictating the durability of geopolymer mortar when subjected to carbonation.

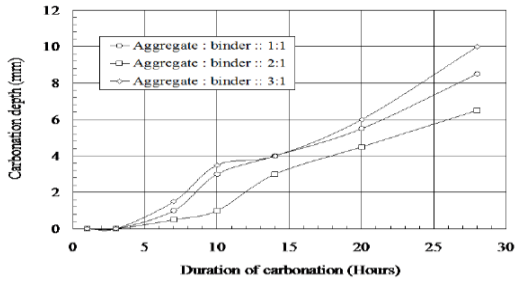

Fig.4 Variation of carbonation depth with carbonation time (h) (curing temperature 90°C)

VI. INFLUENCE ON MICROSTRUCTURE OF GEOPOLYMER MORTAR UNDER CARBONATION

Figs. 5 and 6 present SEM images of the mortar before and after the carbonation process. These allow us to compare the microstructure of the surface of the mortar during carbonation. It can be seen that small depressions in the external surface of the mortar are present before the carbonation; these may be due to uneven geopolymerization of the mortar. In Fig. 5, it is interesting to note that the depression in the surface changed during carbonation. The carbonation caused the surface to deteriorate at the micro-level. As evident from the image, the deteriorated material was found in the particulate form.

Figs. 7, 8 and 9 shows micrographs of the geopolymer mortar for aggregate to binder ratios of 1:1, 2:1, and 3:1. It can be seen that needle-type crystals form during the carbonation of the mortar. The formation of these crystals is most pronounced in the case of a 3:1 aggregate to binder ratio. The presence of different crystals can be further investigated with the help of XRD analysis.

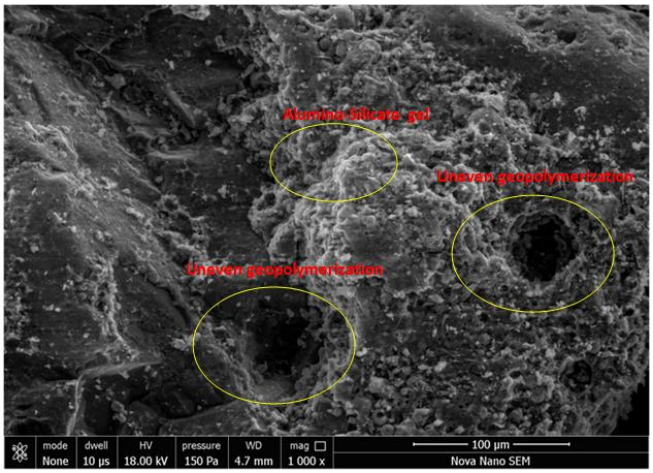

Fig.5 Micrograph of geopolymer mortar before carbonation

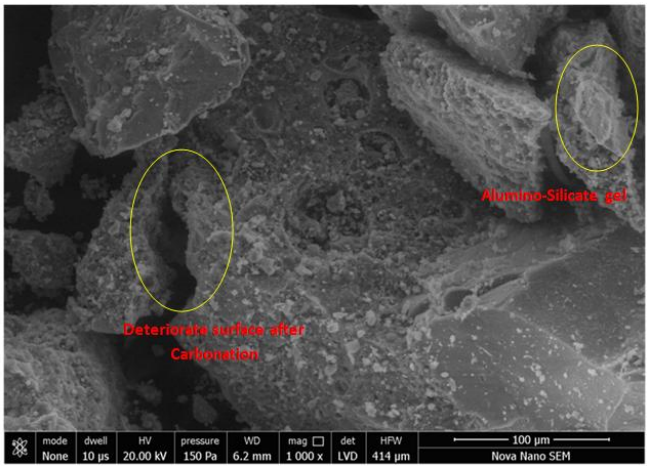

Fig.6 Micrograph of geopolymer mortar after carbonation

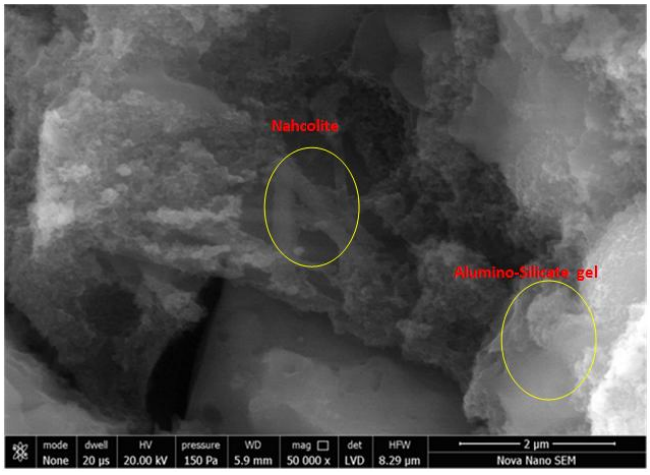

Fig. 7 Micrograph of carbonated samples with an aggregate to binder ratio of 1:1

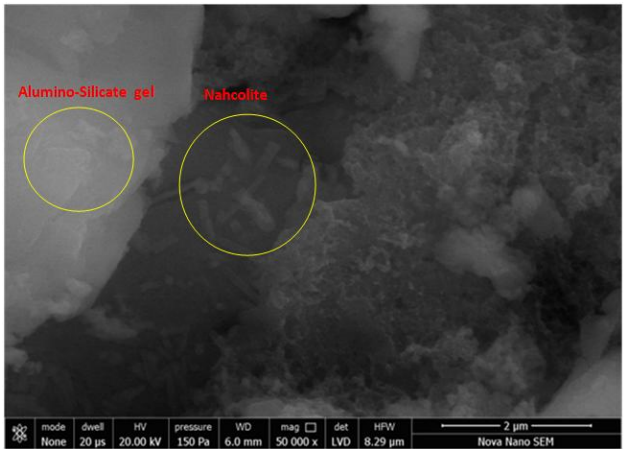

Fig.8 Micrograph of carbonated samples with an aggregate to binder ratio of 2:1

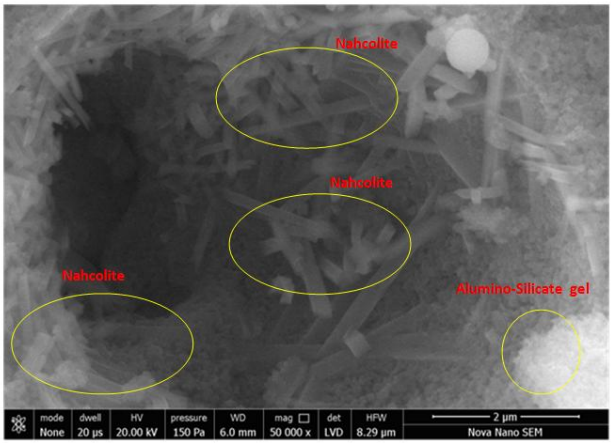

Fig.9 Micrograph of carbonated samples with an aggregate to binder ratio of 3:1

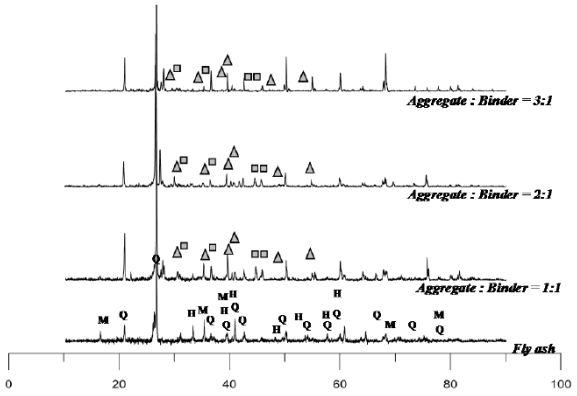

Fig. 10 XRD pattern of carbonated geopolymer mortar (curing temperature 60°C)

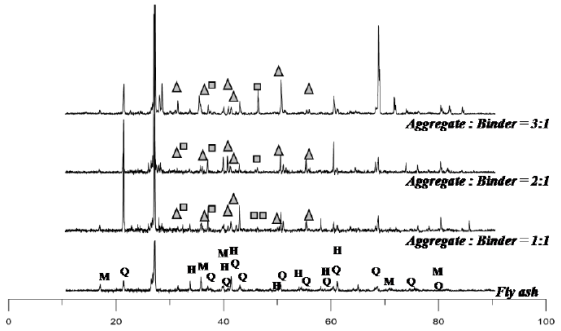

Fig. 11 XRD pattern of carbonated geopolymer mortar (curing temperature 90°C)

XRD images of carbonated geopolymer mortar were obtained for various aggregate binder ratios (1:1, 2:1, and 3:1) and curing temperatures (60°C and 90°C). The carbonation process significantly changes the pore chemistry. In this study, with the help of diffractograms, changes in pore solution chemistry could be identified. For this purpose, a mineral named nahcolite (N) was investigated. As shown in Fig.10 and 11, at the onset of carbonation, the pore solution chemistry changes and nahcolite (N) is formed. In parallel, sodium carbonate hydrate is also formed. During carbonation, under the prevalent thermodynamic and environmental conditions, nahcolite (N) is the most favourable formation product. Similar observations were observed in mortars with various aggregate-binder mix compositions and curing temperatures.

VII. CONCLUSION

From the results reported in this research paper, the following conclusions can be drawn:

1) The carbonation depth was found to increase at lower curing temperatures. This highlights the importance of the curing temperature to arrest the possible degradation of mortar when subjected to carbonation, which in turn influences the long-term performance of the mortar.
2) Similarly, an aggregate-binder ratio of 2:1 was found to give the best performance against carbonation. This indicates the importance of an appropriate mix composition to ascertain the performance of geopolymer mortar.

REFERENCES

1. Bosoga A., Masek O., Oakey, E. J.,CO2 Capture Technologies for Cement Industry, Energy Procedia,2009, Pp:133-140.
2. S. Luhar, T.-W. Cheng, D. Nicolaides, I. Luhar, D. Panias, K. Sakkas, Valorisation of glass wastes for the development of geopolymer composites – durability, thermal and microstructural properties: a review, Constr. Build. Mater. 222 (2019) 673–687, https://doi.org/10.1016/j.conbuildmat.2019.06.169.
3. S. Luhar, T.-W. Cheng, D. Nicolaides, I. Luhar, D. Panias, K. Sakkas, Valorisation of glass waste for development of Geopolymer composites – mechanical properties and rheological characteristics: a review, Constr. Build. Mater. 220 (2019) 547–564, https://doi.org/10.1016/j.conbuildmat.2019.06.041.
4. S. Luhar, T.-W. Cheng, I. Luhar, Incorporation of natural waste from agricultural and aquacultural farming as supplementary materials with green concrete: a review, Compos. B Eng. 175 (2019), https://doi.org/ 10.1016/j.compositesb.2019.107076 107076.
5. S. Luhar, P. Chaudhary, I. Luhar, Influence of Steel Crystal Powder on Performance of Recycled Aggregate Concrete 431 (2018) 102003, https://doi.org/10.1088/1757-899x/431/10/102003.
6. S. Luhar, S. Gourav, A review paper on self healing concrete, J. Civ. Eng. Res. 5 (3) (2015) 53–58, https://doi.org/10.5923/j.jce.20150503.01.
7. S.Luhar, I.Luhar, Strength improvement studies of bacterial concrete, Indian Concrete Journal 2017, 91 (10),pp. 12-16.
8. S. Luhar, Performance evaluation of rubberized geopolymer concrete and fly ash based geopolymer mortar. Thesis. 2018.
9. Xueying, L., Xinwei, M., Shoujie Z., and Enzu Z., Mechanical Properties and Microstructure of Class C Fly Ash-Based Geopolymer Paste and Mortar, Materials 2013, 6, 1485-1495.
10. S. Luhar, I. Luhar, Application of artificial neural network for predicting compressive strength of geopolymer concrete, Indian Concrete Journal, 2019, 98 (2), pp. 38-43.

11. F.U.A. Shaikh, S. Luhar , H. Arel, I. Luhar, Performance evaluation of Ultrahigh performance fibre reinforced concrete – A review, Construction and Building Materials 232 (2020) 117152, https://doi.org/10.1016/j.conbuildmat.2019.117152.
12. S. Luhar, U.V.Dave, Investigations on mechanical properties of fly ash and slag based geopolymer concrete, Indian Concrete Journal, 2016, 90 (2), pp.34-41.
13. Choudhary V., S.Luhar, Fly ash utilization: A review, International Journal of Civil Engineering and Technology, 2017, 8 (4), pp. 301-312.
14. S. Luhar, Fly, ash and Slag based geopolymer concrete: experimental facts. LAP Lambert Academic Publishing; 2016.
15. S.Luhar, I.Luhar, Fly Ash Based Geopolymer Mortar- Strength Performance, International Journal of Recent Technology and Engineering (IJRTE), 8(5), January 2020.
16. S.Luhar, I.Luhar, Application Of Seawater And Sea Sand To Develop Geopolymer Composites, International Journal of Recent Technology and Engineering (IJRTE), 8(5), January 2020.
17. Montes F., Valavala S., Haselbach M., A new test method for porosity measurements of Portland cement pervious concrete, Journal of ASTM International, 2005.
18. S.Luhar, I..Luhar, Rubberized Geopolymer Concrete: Application of Taguchi Method for Various Factors, International Journal of Recent Technology and Engineering (IJRTE), 8(5), January 2020.
19. RILEM Committee TC56, Measurement of hardened concrete carbonation depth -CPC 18, Materials and Structures, 1988, Pp:453-455.

AUTHORS PROFILE

Dr. Salmabanu Luhar, D.litt.(SAARC), D. Litt.(N. Korea), Postdoc. (Taiwan), Ph.D.eng, M.Eng, B.Eng, D.Eng, is an inventor, researcher, author, chartered engineer, approved valuer, and has worked for a research project funded by Department of Science and Technology (DST), Ministry of Science and Technology, New Delhi as Principal Investigator under "Woman Scientist" scheme. She has not only the experience as assistant professor but also well-known for her patent, European books, outstanding publications, national and global awards, records, and affiliations, etc. She has been bestowed with 'Best researcher" award. Her research expertises are advances in conventional and geopolymer construction composites technology and construction building material science.

Dr. Ismail Luhar(GOLD MEDALIST), D.Litt.(N.Korea), M.Sc., B.Sc.; inventor, researcher, author. He is a topper in all subjects at his Masters, winning three academic gold medals. He is known to have a patent, to author books (Europe) and remarkable articles. He has been bestowed with "Best Research Paper Award, Dr. Vikram Sarabhai Life Time Achievement International Award". His area of expertise is Geological structural engineering, Water resources, Geochemistry, Geopolymers and conventional Building materials technology. His research interests are structural, Petroleum, Environmental and Engineering Geology, Hydrogeology, Mineralogy, Gem Stones, Novel Construction Material science. material science.