ORIGINAL PAPER

Investigation of Mechanical and Microstructural Properties of Fiber-Reinforced Geopolymer Concrete with GGBFS and Metakaolin: Novel Raw Material for Geopolymerisation

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Received: 14 August 2020 / Revised: 11 October 2020 /Accepted: 12 October 2020 / Published online: 22 October 2020 \odot Springer Nature B.V. 2020

Abstract

In modern days, one or the way, development of infrastructure plays a major role that decides upon the matrix of our lifestyle. The development of geopolymer concrete (GPC) is an important step towards the production of eco-friendly concrete. The effective usage of fiber content can reduce environmental pollution issues. An attempt was made to study the effect of the blend of metakaolin (MK) and ground granulated blast furnace slag (GGBFS) with Gujcon CFR fiber as secondary reinforcement was added as source material on the performance of GPC to prevent the effects of thermal variation. Fine aggregate, coarse aggregate, sodium hydroxide, nano-silicon dioxide (nano- $SiO₂$), and cement have been used in the present work to make geopolymer mortar (GPM). GGBFS and Gujcon CFR fiber have been replaced with metakaolin an interval of 25%. The ratio of liquid to solid is 0.45 to 0.55, with a rise of 0.05. The samples are tested for split tensile and compression. To identify the surface morphology and phases SEM, XRD and EDAX have been analyzed. It is evident from the result that an equal proportion of source material with liquid to binder ratio 0.5 has yielded the highest mechanical properties.

Keywords Metakaolin \cdot Geopolymer mortar \cdot Nano-SiO₂ \cdot Gujcon CFR fiber \cdot Liquid ratio

1 Introduction

The most popular binding material for the production of concrete is Ordinary Portland Cement (OPC). The resourcefulness of cement made it ever-demanding than any other material in the construction industry. The usage of cement is ever increasing due to its ability to gain early strength and also prolonged gain in strength. Moreover, there is a need to develop and use environmentally friendly concrete. In the present study, the development of geopolymer concrete (GPC) is taken and is an important step towards the production of ecofriendly concrete. Metakaolin is a dehydroxylated form of the clay mineral that is fired under controlled conditions to form amorphous alumina silicate into a fine powder. It is widely used as cement replacement in concrete consists of smaller particle sizes approximately 1–2 μm and grater surface area compare to ordinary portland cement. Metakaolin is obtained from the calcination of kaolinitic clays at a temperature ranging from 600 to 700 °C conducted by Diffo et al. [\[1\]](#page-7-0). The metakaolin is a very fine and highly reactive material. This imparts a creamy and non-sticky texture to fresh concrete making finishing easier. By adding metakaolin material, the various properties of concrerte like workability, durability, strength, resistance to cracks and permeability can be improved. Kamseu et al. [[2\]](#page-8-0) presented a review on different aspects of alkali-activated metakaolin binder materials. It was reported that a molar ratio of 0.50 is found to be suitable for the activation of metakaolin. To minimize the composition of cement various organic materials were used such as RHA, GGBFS, bottom ash, fly ash, etc. [\[3](#page-8-0), [4](#page-8-0)]. GGBFS based GPC has the incredible compressive quality and is appropriate for structural applications. The notable factors that impact on the properties of the new concrete and the hardened concrete have been distinguished. The versatile properties of hardened concrete and the conduct and quality of strengthened basic members are like those of portland cement concrete. In this work, ground granulated blast furnace slag and fiber reinforced (Gujcon CFR) are used to concrete containing discontinuous,

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uniformly dispersed. This fibrous material increases the structural integrity of the concrete. The role of Gujcon CFR fiber is to bridge the cracks that form in concrete due to drying and plastic shrinkage and also to increase the ductility of concrete elements. Qureshi and Ghosh [[5](#page-8-0)] explored that GGBFS added to (0 to 20%) of aggregate, critical increment in strength, and some reduction in workability was seen on GPC. Atea [[6\]](#page-8-0) suggested that fiber possesses good resistance against alkalis thereby improving the durability of concrete. A highly concentrated aqueous alkali hydroxide and silicate solution are required to trigger the geopolymeric reaction. Hence, an attempt has been made in the present work to study the effect of the blend of metakaolin (MK) and GGBFS/CFR (industrial by-products) as source material on the strength and durability of GPC. Thus, the suitability of MK-GGBFS/CFR GPC has been evaluated at ambient temperature conditions.

2 Experimental Studies

2.1 Ground Granulated Blast Furnace Slag (GGBFS)

GGBFS would be off-white and has superior cement characteristics as compared to other fillers. From the blast furnaces, GGBFS is a by-product to make iron. The molten slag and molten iron were obtained when the iron ore coke and limestone melt in a furnace at 1500 °C. However, the slag is dried after heating and then taken into a ball-mill to produce a very fine powder known as GGBFS. It contains high silicates and alumina leads to improve the material properties and is designated according to ASTM C 989 [[7](#page-8-0)]. Nowadays GGBFS is used widely and is a portland cement replacing about 70%. Tables 1 and 2 shows the chemical and physical properties of GGBFS supplied by Adhipathi Minerals & Chemicals Private Limited, Hyderabad, India.

2.2 Gujcon CFR Fiber

Introducing fiber as reinforcement in concrete is considered to be the most efficient way to prevent shrinkage cracks and thereby increasing the strength and durability. Concrete with fiber as secondary reinforcement was improved structural

Table 1 Chemical Composition

Chemical Substances	Composition
Sulfate ion reported as SO_3	4%
Sulfide Sulfur (s)	2.5%

Table 2 Physical Properties

integrity and uniformly distributed in the concrete matrix. Application of fiber as secondary reinforcement is widely adopted to increase the toughness and tensile strength and also to improve the cracking deformation. Fibers in concrete will reduce the segregation of aggregates. Thus, it helps to increase homogeneity. Song et al. [\[8](#page-8-0)] suggested a significant improvement in material properties was attributed due to the addition of fibers in concrete. The physical properties of Gujcon CFR is supplied by Gokul Enterprise, Rajkot, India and is listed in Table 3.

During the batching process, GUJCON fiber is mixed with alkaline liquid and then added to the concrete for better dispersion of fiber in the concrete matrix. In GPC, GUJCON-CFR is introduced to prevent cracks due to temperature variation and to improve the strength.

2.3 Metakaolin

Metakaolin is prepared clay kaolin, ignited under closely regulated conditions to produce an amorphous aluminosilicate. To generate metakaolin, the polished kaolin is placed into a rotary kiln and compressed into a fine powder and deposited. Metakaolin is obtained from the calcination of kaolinitic clays at a temperature ranging from 600 to 700 \degree C [[9\]](#page-8-0). The metakaolin is a very fine and highly reactive material. Hence, this provides a creamy, non-sticky surface to flavorful producing concrete having completed easier. It is well-suited as concrete admixtures and cured as that of ordinary cement concrete. The metakaolin can be used as a partial or full supplementary cementitious material.

2.4 Alkali Activator

In geopolymerisation, alkaline solutions always play a significant role in synthesis. These alkaline solutions are from soluble alkali metals that are likely to have a combination of sodium-based nature. The most frequently used alkaline solution in geopolymerisation is sodium hydroxide [[10\]](#page-8-0). The sodium hydroxide flakes with 96% purity were purchased in bulk from a local supplier. While disintegration flakes in water were dissolved sodium hydroxide solution. Sodium hydroxide exhibits superior properties and strongly alkaline in nature with bitter and has a soapy feel to it. It is highly soluble in water and moderately soluble in alcohol. The density of NaOH solids in a solution differs based on the molarity (M) solvent concentration.

2.5 Nano-Silicon Dioxide

Nano-silica dioxide $(SiO₂)$ available in liquid gel form was purchased from Mk suppliers, Chennai, India. The chemical composition contains 46.89% of Silica with 52.34% of oxygen and 2.4 g/cm³ of density with a melting point of 1600 °C. This liquid is used to strengthen filler for concrete and else it is a non-toxic substrate used for biomedical applications including medication distribution and biosensors.

2.6 Fine Aggregate

The river sand was used as a fine aggregate in the present study and is collected from a local source. The physical properties of river sand are represented in Table 4. River sand is conventional to grade Zone-III as per BIS 383–1970 [[11](#page-8-0)].

2.7 Coarse Aggregate

Table 4 Physic Properties

Locally available coarse aggregate was used in the present work. The physical properties of aggregates are tested as per the procedure 49 prescribed by BIS 2386–1963 and results are presented in Table $5 \lfloor 12 \rfloor$. The result shows that the characteristics of aggregates conform to the requirements prescribed by BIS 383–1970.

3 Investigation Experimental Programme

In the present study, the ingredients such as metakaolin, bottom ash, fine aggregate, sodium silicate, and sodium hydroxide are used to make MK-GGBFS/CFR GPM. Totally twenty mixes have been formulated for the present work. The amount of MK and GGBFS/CFR varied in the range of zero to 100% by the total weight of source material with an interval of 25%. The ratio is taken as 1:4 for the source material such as MK-GGBFS/GFR to river sand and varying liquid ratio in the range of 0.45–0.55 with 0.05 increments. Since the liquid ratio of nano-silica dioxide was varied from 1.5 to 2.4. Further, the 8 M sodium hydroxide was used to activate the geopolymerization process. The detailed composition of MK-GGBFS/CFR was examined and presented in Table [6](#page-3-0). Hence, cube size specimens 70.5 mm X 70.5 mm X 70.5 mm were used to measure the compression and flexural strength at the age of 1, 3, 5, and 28 days for all investigations [\[13\]](#page-8-0). In this work, the following mix codes are used to understand the mix composition. Also, suffix numerals specify the amount metakaolin in the total quantity of source material (MK + GGBFS/CFR). Further, small letters such as a, b, and c in the mix code represent 0.45, 0.50, and 0.55 liquid to binder ratio respectively.

4 Results and Discussion

4.1 Compression Strength of aGGBFC/CFR-GPM with Liquid of 0.45

The compression strength of aGGBFS/CFR-GPM was mixed with a liquid ratio of 0.45 and is represented in Table [6.](#page-3-0) It is evident from the figure that aGGBFS/CFR-M-GPM mixes have exhibited a wide range of compression strength (i.e. age of one day). It varies from 3.21 MPa to 12.45 MPa depending on the proportion of MK and bottom ash. The aGGBFS/CFR-M100 has yielded a minimum strength of 3.21 MPa and aGGBFS/CFR-M25 has demonstrated maximum strength of 12.75 MPa at the age of one day. However, aGGBFS/CFR-M100 mortar mix has demonstrated significant improvement in compression strength; at the age of 28 days. But, the aGGBFS/CFR-M25 mortar mix has failed

Table 6 Composition of Metakaolin with GGBFS/CFR geopolymer binder

to gain the highest strength at the age of 28 days. Among all mixes, aGGBFS/CFR-M75 and aGGBFS/CFR-M0 mix exhibited a minimum strength of 9.22 MPa and maximum strength of 22.06 MPa respectively at the age of 28 days. Moreover, the geopolymeric mortar mixes produced with the various extent of source material and not observed any unique pattern in the compression strength at the age of 28 days; the rate of strength gain. Similar findings were made by Wardhono et al. $[14]$ $[14]$. This is due to the mixing of various compositions, degree of polymerization, crystalline phases over time, and also curing method explained by Rovnaník [\[15\]](#page-8-0). However, the geopolymeric gel is indistinct, brittle, and thus takes enough time to transform into a crystalline form. Hence, there is a dropin rate of strength development of geopolymer mixes over a period of time. Furthermore, a regression analysis was made (at the age of 28 days) to establish a relationship between the compression strength of GPC and percentage of MK (Fig. 1). The empirical relationship established was found to fit with fourth-order polynomial expression and is identified by using Eq. (1).

$$
f_c = 151.7866mp4 - 210.4mp3 + 70.43mp2 - 15.63mp
$$

+ 22.06 (1)

Where, mp^2 - square meter, mp^3 - cubic meter, f_c - polynomial expression.

4.2 Compression Strength of bGGBFC/CFR-GPM with Liquid of 0.50

The compression strength of bGGBFS/CFR-GPM was mixed with a liquid ratio of 0.50 and is presented in Table 6. The figure shows that bGGBFS/CFR-GPM mixes also have exhibited a very wide range of compression strength at the age of one day. It varies from 4.01 MPa to 40.72 MPa depending on the proportion of MK and GGBFS/CFR in mortar. However, the range of strength is extensively high than the mixer made

Fig. 1 Relation between percentage of MK and compression strength of aGGBFC/CFR-GPM with liquid of 0.45

with a liquid ratio is a ratio of 0.45 to 0.55 [[16](#page-8-0)]. The bGGBFS/ CFR0 GPM has yielded the least strength of 4.01 MPa and bGGBFS/CFR-M50 has demonstrated the highest strength of 40.72 MPa at the age of one day. Also, bGGBFS/CFR-M0 and bGGBFS/CFR-M50 GPM mixes have exhibited the minimum strength of 36.71 MPa and highest strength of 78.24 MPa respectively at the age of 28 days. bGGBFS/CFR-M50 has yielded the highest compression strength than other mortar mixes made with different liquid to binder ratios. The strength of bGGBFS/CFR-M-GPM mixes has gradually enhanced with a decrease of MK quantity from 100 to 50%. Moreover, a further drop in the amount of MK reduces the compression strength. An empirical relationship has been established between the compression strength of GPC at the age of 28 days and percentage of MK (Fig. 2). It is found to fit with fourthorder polynomial and is expressed by using Eq. (2).

$$
f_c = 1834.346 \text{mp}4 - 3787.34 \text{mp}3
$$

$+2307.033$ mp2–382.93mp + 36.06 (2)

4.3 Compression Strength of cGGBFC/CFR-GPM with Liquid of 0.55

The compression strength of cGGBFS/CFR-GPM was mixed with a liquid ratio of 0.50 and is labelled in Table [6.](#page-3-0) The cGGBFS/CFR-M-GPM mixes have exhibited a wide range of compression strength at the age of one day. It varies from 4.81 to 36.51 MPa depending on the proportion of MK and GGBFS/CFR in mortar. The cGGBFS/CFR-M100 mix has exhibited the highest strength, whereas cGGBFS/CFR-M0 demonstrates minimum strength at one day age. But, at the age of 28 days, cGGBFS/CFR-M75 has yielded the highest strength of 60.79 MPa among cGGBFS/CFR-M-GPM mixes [\[17\]](#page-8-0). However, this is remarkably less than the strength of the bGGBFS/CFR-M50 GPM mix. Moreover, the relationship

Fig. 2 Relation between percentage of MK and compression strength of bGGBFC/CFR-GPM with liquid of 0.50

has been developed between the compression strength of GPC at the age of 28 days and percentage of MK as shown in Fig. 3. It is found to fit with fourth-order polynomial expression and is expressed in Eq. 3.

$$
f_c = -1564.36mp4 + 2487.33mp3 - 1405.13mp2
$$

+ 198.93mp + 38.36 (3)

4.4 Water Absorption

Water absorption test is one of the important properties which influence the strength and durability of the geopolymer concrete. The water absorption characteristics of MK-GGBFS/ CFR of M 30, M 35, M 40, M 50, and M 55 grade were determined according to the BIS 15658:2006 at the age of 5 and 28 days. The sample was thoroughly immersed in water for one day at room temperature. Later then samples are taken from the water and allowed to dry for five minutes by placing coarser wire mesh. Each sample was weighed and recorded. The concrete with higher water absorption indicates the presence of a porous microstructure. The higher porosity in the concrete reduces the strength and also durability. Hatungimana et al. [[18](#page-8-0)] has specified that the water absorption of paver blocks shall not be more than 6% by mass. It is evident from the result that the paver blocks of various grades have demonstrated water absorption less than 2.83% by mass. Also, the water absorption of paver blocks reduced with an increase in the grade of geopolymer concrete and is represented in Table [7.](#page-5-0) However, it is a higher grade of geopolymer concrete shows higher compression strength and thereby the water absorption must be relatively less. Zhang et al. [\[19](#page-8-0)] reported 2.6% of water absorption in the cement concrete. It is interesting to note in the present work that the geopolymeric

Fig. 3 Relation between percentage of MK and compression strength of cGGBFC/CFR-GPM with liquid of 0.55

Table 7 Water absorption test of MK-GGBFS/CFR

concrete paver block of M 50 grade has exhibited 50% less water absorption than the cement concrete.

4.5 Abrasion Resistance

The abrasion resistance of MK-GGBFS/CFR GPC at the age of 5 days and 28 days was determined in accordance with BIS 15658:2006. Square shaped specimens measuring 75 mm with a weight of each sample is 0.1 g. It was cut from the block specimens and contact face with opposite face of the specimen was determined as parallel and flat. The cutting route of the abrasion testing system disks was uniformly spread with 20 g of the regular abrasion material. The specimen was fixed so that each sample was centrally loaded at 295 N with a speed of 20 rpm on the testing surface facing the grinding disk. The test cycle was repeated sixteen times on each sample and disc was stopped after 20 revolutions to identify abrasion resistance. The concrete used in the construction of pavements, industrial floors, hydraulic structures, etc. is likely to be subjected to rubbing, scrapping, and skidding forces. Hence, the concrete should have required ability to resist such forces to last for a longer time. Primarily, the abrasion resistance depends on strength of concrete. In turn, the strength of the concrete depends on mix proportion and surface finish [[20](#page-8-0)]. Further, the type of aggregate used in concrete will also profoundly influence the abrasion resistance. In general, the aggregates with high stiffness such as basalt, quartzite, granite, etc. used in concrete influence the abrasion resistance substantially. The abrasion resistance of MK-GGBFS/CFR of various grades is represented in Table 8 and it is evident from the result that the abrasion

resistance increased with the grade of the GGBFS/CFR. It is due to the increase in strength of the paver blocks with an increase in grades. The concrete with higher compression strength usually exhibits relatively higher abrasion resistance well agreed by Sahdeo et al. [\[21](#page-8-0)].

4.6 Microstructural and EDAX Analysis

The SEM and EDAX analysis was investigated on MK-GGBFS/CFR GPC at the age of 1, 3, 5, and 28 days to evaluate morphological characteristics of concrete.

4.6.1 SEM Analysis at 1st Day

The SEM analysis was taken on MK-GGBFS/CFR geopolymer (at the age of 1 day) and is shown in Fig. [4a.](#page-6-0) The well-developed crystalline phases noticed in the micrograph indicate the high degree of geopolymeric reactions between source material and alkaline activator. The Na-Al-Si is the principal reaction product, which contributes the early strength to the MK with GGBFS/CFR geopolymer matrix. The fractured surface along with macro cracks are distinctly observed in the micrograph. EDAX analysis was performed on MK-GGBFS/CFR geopolymer and the spectrum is presented in Fig. [4b](#page-6-0). The result shows that the alumina, silica, and iron are the main elements present in the geopolymer. The EDAX spectrum shows the presence of a small amount of calcium in GPC as the source material contains a low percentage of calcium.

4.6.2 SEM Analysis at 3rd Day

The presence of zeolite and sodium silicate hydrate gel was observed in the micrograph. Further, the presence of microcracks is observed in GPC at the age of 3 days. However, geopolymer reactions progress, the crack portion starts to fill up by geopolymer products and resulting in increased strength (Fig. [5a](#page-6-0)). The EDAX spectrum of GPC is shown in Fig. [5b.](#page-6-0) The result shows that most predominant elements present in geopolymer are silica, alumina, and sodium [[22](#page-8-0)]. Among these elements, silica is a more dominant element present in geopolymer at the age of 3 days. Also, the proportions of all the other elements significantly increased in geopolymers. The higher percentages of a mass of Na, Al, Si, and Ca have imparted remarkable strength gain at the age of 3 days of geopolymer. The gain in strength is almost 100% than the strength at the age of 3rd day.

4.6.3 SEM Analysis at 5th Day

The surface morphology of MK-GGBFS/CFR geopolymer (at the age of 5 days) presented a more dense and compact microstructure (Fig. [6a](#page-6-0)). Very few hydrated bottom ash needle-

Fig. 4 (a) SEM micrograph and (b) EDAX of MK-GGBFS/CFR geopolymer (at the age of 1st day)

Fig. 5 (a) SEM micrograph and (b) EDAX of MK-GGBFS/CFR geopolymer (at the age of 3rd day)

Fig. 6 (a) SEM micrograph and (b) EDAX of MK-GGBFS/CFR geopolymer (at the age of 5th day)

Fig. 7 (a) SEM micrograph and (b) EDAX of MK-GGBFS/CFR geopolymer (at the age of 28th day)

like products are noticed in geopolymer [[23](#page-8-0)]. Also, the geopolymer contains a glassy structure with very few microcracks. Further, the formation of sodium silicate hydrate gel improved the strength of GPC significantly at the age of 5 days. Figure [6b](#page-6-0) represents the EDAX spectrum of MK-GGBFS/CFR geopolymer. The result confirms that elements such as Si, Fe, and alumina are the predominant elements present in GPC.

4.6.4 SEM Analysis at 28th Day

It is evident from the figure that the 97 microstructure of geopolymer is much denser than a geopolymer at the age of 28 days (Fig. $7a$). Also, the geopolymer is completely free from micro-cracks. Further, geopolymeric compounds such as sodium-based geopolymeric gel and alumina silicate products are visible in micrograph [[24](#page-8-0)]. More so, bottom ash hydrated needle-shaped products have grown in size at the age of 28 days. Figure 7b represents the EDAX spectrum of MK-GGBFS/CFR geopolymer. The silica, alumina, and sodium are predominant elements present in geopolymer. The SEM and EDAX analysis confirm the presence of sodium alumina silicate as an additional product in geopolymer.

5 Conclusion

An attempt was made to study the effect of the blend of MK and GGBFS/CFR as source material on the performance GPC by taking nano-silicon dioxide with fine and coarse aggregate.

- 1. The optimum liquid/binder ratio exhibits higher compression strength to MK and GGBFS/CFR GPM made equal proportions is 0.50.
- 2. Among all mortar mixes, the bGGBFS/CFR-M50 has yielded the highest compression strength of 79.32 MPa at the age of 28 days in ambient curing. Further, the

compression strength of bGGBFS/CFR-M50 mortar is about 52% higher than the mortar made with 53 grade OPC. Further, the early strength gain of GPM is remarkably higher than the cement mortar.

- 3. The SEM micrographs indicate that the GPC at an early age has demonstrated a relatively porous microstructure. Also, it has shown the presence of macro and micro cracks. Further, it is evident from the micrographs that more and more geopolymeric gel has been formed in GPC with age, resulting in the densification of microstructure and remarkable improvement in the strength.
- 4. Moreover, the macro and micro-cracks have vanished with the presence of GGBFS/CFR and nano $SiO₂$. A similar trend was observed in water absorption and abrasive resistance. The strength development has been substantiated by the presence of dominant elements such as Si, Fe, and alumina that are noticed in EDAX analysis.
- 5. Both GGBFS and Gujcon CFR are free from cracks, crumbling, softening, and also dimensionally quite intact even after the exposure to nano silica dioxide solution over a period of time.

Acknowledgments I wish to thank the Department of Civil Engineering, Siddharth Institute of Engineering & Technology, Puttur, India, to provide facilities and necessary support in conducting experiments for providing facilities in the research work.

Compliance with Ethical Standards

Conflict of Interest The author declare that there is no conflict of interest.

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