Mechanical Properties of High Calcium Flyash-GGBFS Geopolymer Paste, Mortar and the Effect of Glass Fibre Mesh on the Strength of Geopolymer Mortar Tile

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ABSTRACT:In this paper, a combination of high-calcium fly ash (HCFA) and ground granulated blast furnace slag (GGBFS) was used along with a combination of sodium hydroxide (NaOH) and sodium silicate(Na_2SiO_3) as alkaline activators (AAs) to produce geopolymer paste and mortar. The alkaline activator ratio (AAR) was maintained at 1.5 apart from their molarity at 10 for the study. A rational method, namely minimum voids approach was used for the mix design. A commercially available glass fibre mesh was used as reinforcement in the geopolymer mortar produced above, to assess its potential for use as a flooring tile. The influence of W/S (water-to-solids) ratio and the influence of various fine aggregates, namely, river sand (R), manufactured sand (M) and construction demolition waste (D) on the various geopolymer system (GP) and on the strength characteristics, are highlighted. A maximum transverse strength (TS) of 6.25 N/mm^2 could be attained by the geopolymer tile, using three layers of glass fibre mesh and GP mortar developed. The study indicates that a combination of FA and GGBFS helps us to attain substantial strength under ambient temperature in geopolymer mortar.

Keywords:Geopolymer paste and mortar, Flyash(FA), GGBFS, Alkali-activators(AAs), Glass fibre mesh reinforcement, Compressive strength(CS), Flexural strength(FS), Flooring tile.

I. INTRODUCTION

All over the world the need for production of an alternate binder material to the conventional and well established binder, namely, Portland cement(PC) was realized as PC is energy-intensive, contributes to global warming and causes rapid depletion of natural resources (which form the raw material for production of PC). (Turner and Collins, 2013; Ke et al. 2017). The best alternate to the above material, was developed and introduced by Joseph Davidovits during 1978, which is a 'cement-free' material and was referred to by him as 'geopolymer' (GP). GP can be considered to belong to a broad class of material knows as 'alkaline – activated binders' (AAB). GP is an 'inorganic polymer' produced by the alkali-activation of 'alumina-silicate rich materials' sourced either from nature or from various industrial waste materials such as: GGBFS, fly ash (FA), silica fume (SF), meta kaolin (MK), rice-husk ash (RHA) etc. For the past four decades, there has been an explosion in the number of publications/research reports/Ph.D Thesis etc, on every aspect of development of GP based system (paste, mortar, concrete, composites), which are summarized in some of the excellent works, authored by renowned researchers [Davidovits (2015); Provis and Van Denventer (2004, 2009); Pachego-Torgal et al. (2015)]. In spite of such developments, commercial production and use is not widespread globally, leave alone India, wherein, it is in a nascent stage. Among the various reasons, the challenge is production and use at ambient temperature.

The best 'base material' for production of GP is flyash, due to various reasons such as : ease of availability in large quantities, advantageous than GGBFS from field applications etc.[Basheer and Antharjanam (2019)]. In the Indian context, it is all the more relevant and need of the hour, as the flyash is under - utilized in spite of availability of larger quantities.

During the last two decades there has been a very great interest in the use of woven fabrics (also referred to as textile reinforcement-TR) as the main reinforcement in cement-based composites, and are characterized by carbon, glass, steel, aramid or basalt fibres. A textile reinforcing mortar (TRM) is a composite comprising of high-strength fibres made of carbon, basalt or glass, in the form of textiles embedded into inorganic materials such as: cement/cementitious based materials(Raoof, 2017).Research studies on TR systems are being conducted all over the world, with the scope extending to wide range of applications such as: strengthening/structural retrofitting applications, façade panel solutions, shell structures,

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construction of new prefabricated structures and corrosion protection [Caggegi et al. (2017); Rambo et al. (2017); Koutas et al. (2019)]. However, in India the interest in the above area is of recent origin only, even in the cement/cementitious systems.

Microfibers were incorporated into fly ash - based geo polymer composite for improving the post cracking behaviour (Alomayri, 2017). Woven mesh, apart from other metallic meshes were used to study the flexural behavior of fly ash - based geopolymer ferrocement element/(s) [Srividya et al. (2012)].It can be seen that studies on the use of TR in geopolymer is rather rare even internationally, not to mention the status in India, where, it is yet to be recognized for potential applications. Thus, the attempt made is this study to use TR in the form of glass fibre mesh in geopolymer mortar for development of a flooring, tile is unique and may be first of its kind.

In view of the above, a combination of high - calcium FA and GGBFS was used to produce GP paste and mortar at ambient temperature, using a combination two widely used alkali activators (AAs), namely, sodium hydroxide and sodium silicate, to study salient strength characteristics. Further, glass fibre mesh was used as a TR in the GP mortar to produce tiles and their strength characteristics were evaluated to assess its potential as a flooring tile.

II. EXPERIMENTAL

A Alumina Silicate Materials (ASM)

Flyash was sourced from the lignite – based thermal power plant at Neyveli, Tamil Nadu state, India. GGBFS was obtained from a local steel plant in Tamil Nadu state, India. The salient physical and chemical properties of the above two ASMs are summarized in Tables 1 and 2. The median particle size of flyash is 9.68 µm, whereas it is 12.85 µm for GGBFS. This shows that the flyash used in this study is much finer than the GGBFS. Further, it can be seen that based on Blaine's specific surface, the flyash used in this study is nearly double than that of GGBS. The CaO content in flyash is about 19% which is very high. Further, the sum of (SiO_2 + Al_2O_3 + Fe_2O_3) is greater than 50%. Based on the above two criteria, the flyash used in this study is classified as Class C, according to ASTM C 618: 2003 and cementitious according to IS: 3812 - 2003. This kind of fineness exhibited by GGBFS and fineness and cementitious nature of flyash chosen in this study is expected to a positive role in the strength development in a geopolymer system. Moreover, a combination of flyash and GGBFS, such as the one considered in this study, is also expected to overcome some of the inherent difficulties of using GGBFS alone, as has been very well reported in published literature. In this study, both the precursors (Flyash + GGBFS) passing through 63 µm alone were used.

BAlkali Activators (AAs)

Sodium hydroxide (NaOH) and sodium silicate (Na_2SiO_3) are the most widely used alkali activators in the production of geopolymer. In this study, a combination of the above two AAs were used. Accordingly, NaOH (flakes) were dissolved in water and a 10 M (molar) solution was prepared, based on a rational method recommended by Rajamane and Jayalakhmi (2015), for several of their studies and extensively reported in reputed journals. Salient physical and chemical properties of the above two AAs are summarized in Table 3 and 4. Based on earlier studies of the authors the ratio of Na_2SiO_3 to NaOH [referred to herein as: 'alkali activator ratio' (AAR).]

C. Fine Aggregate (FAg)

For preparing the geopolymer mortar, three types of fine aggregates were used: river sand (R); M-sand (M), which is referred to as a' manufacture sand' in literature and by professional in India, and construction demolition waste (D), obtained especially from crushed demolition waste concrete.

Very recently, the Supreme Court of India (the apex court in India) has banned mining of river sand from river beds, so as to prevent further ecological damage caused by indiscriminate mining of river sand, spread over several decades. This ban has almost crippled the construction industry in India at the moment, impacting not only the life of several crores of unskilled/skilled work force in the unorganized sector, but also, the economy of India. This has forced the construction industry to look for an 'alternate material' to 'river sand', which has been the traditional fine aggregate in the Indian context.

Further, there is indiscriminate dumping of construction demolition waste (CDW) along with municipal solid wastes (MSW) during the last few decades, causing environmental and other problems, without appreciating and exploiting the potential use of CDW, as is the case in some of the well developed countries, like in: Japan, Singapore.

For the above reasons, and considering 'sustainability', which has become a universal phenomenon, the above three types of fine aggregates where used in geopolymer mortar whereas, 'R' is used independently, and 'D' was used in combination with 'R' and 'M', in the mortar. Salient properties of the above fine aggregates, determined by standard methods (as per relevant IS Codes) are summarized in the Table 5.

Table 1 Physical Properties of ASMs

Material	Physical state	Odour	Appearance	Bulk density (g/cc)	Particle size distribution(µm)			Specific gravity	Blaine specific surface (m^2/kg)
					d_{10}	d_{50}	d_{90}		
HCFA	Micronized power	Odourless	Grey colour powder	0.692	1.72	9.68	29.19	2.59	718

GGBFS	Micronized power	Odourless	white colour powder	0.95	1.69	12.85	46.85	2.90	358

Note: (i) HCFA - high calcium fly ash; GGBFS – ground granulated blast furnace slag.

Table 2 Chemical Composition of ASMs (in percentage by weight)

Materials	SiO_2	Al_2O_3	Fe_2O_3	$SiO_2+Al_2O_3+Fe_2O_3$	Ca O	MgO	Na_2O	K_2O	SO_3	TiO_2	LOI	Others	SiO_2/Al_2O_3	Na_2O/SiO_2
HCFA	23.76	29.24	12.01	65.03	19.17	5.40	0.19	0.09	3.29	0.77	5.74	0.23	0.81	0.008
GGBS	32.47	18.00	1.16	51.63	35.69	8.08	0.20	0.22	1.63	0.59	0.72	1.86	1.80	0.006

Note: (i) LOI – Loss of Ignition

(ii) The total percentage of ($SiO_2 + Al_2O_3 + Fe_2O_3$) is greater than 50% in the above HCFA Sample. Further, CaO content is greater than 10%. Hence, it is classified as class C flyash, according to ASTM:C 618 –(2003) or cementitious FA according to IS: 3812 (part I): (2013).

(iii) The above results are obtained from the laboratory tests conducted at a R & D centre of a reputed cement manufacturing centre in India.

Table 3 Chemical and Physical Properties of Sodium Hydroxide (NaOH)

Appearance /colour	Boiling point	Molecular weight	Specific gravity	Assay	Carbonate (Na_2CO_3)	Chloride (Cl)	Sulphate SO_4	Lead (Pb)	Iron (Fe)	Potassium (K)	Zinc(Zn)
Light yellow liquid (gel)	102ºC for 40% Aqueous Solution	184.04	1.6	97%	2%	0.01%	0.05%	0.001%	0.001%	0.1%	0.02%

Note: Properties are as given by the manufacturer.

Table 4 Chemical and Physical Properties of Sodium Silicate (Na_2SiO_3)

Chemical formula	Na_2O (%)	SiO_2 (%)	H_2O (%)	Appearance	Colour	Boiling point	Molecular weight	Specific gravity
Na_2SiO_3 (colourless)	**10.90**	36.10	53.00	Liquid(gel)	Light yellow	102ºC for 40% Aqueous Solution	184.04	1.53

Note: Properties given above areas furnished by manufacture.

Table 5 Properties of Fine Aggregate (passing through 4.75mm sieve)

Fine Aggregate	Void Content (%)	Void Ratio	Specific Gravity	Fineness Modulus(FM)	Average Particle Size
River sand	33.65	0.507	2.60	1.97	150 – 300 μm
M sand	31.71	0.460	2.60	3.23	600 μm – 1.18mm
Demolition Waste	34.08	0.517	2.30	3.48	300 - 600 μm

D Glass Fiber Mesh

In order to develop a potential application of geopolymer mortar considered in this study, a commercially available 'glass fiber mesh' was considered and used to prepare flooring tiles. The salient properties of the glass fiber mesh, as furnished by the manufacture are summarized in Table 6. The aperture size is 10×10 mm to facilitate easy application of mortar and ensure adequate bonding. Further, special coating of 'alkali – resistant' on the mesh will ensure durability of mesh in the alkaline environment of the geopolymer mortar. The mesh size of the glass fibre mesh chosen and weight (440g/m2) are within this range (8 to 30 mm mesh size, 150 to 600 g/m2 – weight) of typical commercially available non-metallic textiles that are reported in literature for strengthening applications, internationally (Koutas et al. 2019).

As (clay) flooring tiles have to satisfy the characteristics of water absorption, flexural strength and impact strength. (IS 1478:1992) use of glass fibre mesh reinforcement in geopolymer based tile is expected to improve / contribute to the tensile strength, ductility and energy absorption significantly, so as to fulfil the requirement of a typical flooring tile.

Table 6 Salient Properties of Glass Fiber Mesh

Description	Units	Details
Tensile Strength (MD*CMD)	kN/m >=	25 x 25
Tensile Elongation	% (≤)	4
Melting Point coating	Celsius (°C)	>250
Aperture Size	mm	10 x 10
Weight	g/m^2	≥ 140
Material	Alkaline resistance glass fibre mesh coated with a proprietary chemical for use in concrete reinforcement.	

Note: (i) Properties given above are as furnished by manufacturer.
(ii) MD – Machine direction; CMD – Cross machine direction.

E Preparation, Casting and Curing of Geopolymer Specimens

In the case of geopolymer paste, the amount of AA required is based on the void content of the precursor. By this approach, the initial FA$^+$ / AA required (for a constant replacement of FA by 20% of GGBFS) was obtained (0.901) (Table 7 and 8) and consequently water/solids ratio was obtained (0.47). However, the above mix was found to be highly unworkable. Therefore, a few more trails were carried out to obtain two more paste mixes corresponding to w/s ratios of 0.56 and 0.61 (Table10), which were found to be very workable. Paste cube specimens of size 50 × 50 × 50 mm were cast, cured at ambient conditions and tested at the end of 7 days for determining the compressive strength according to the relevant IS code.

The mix of the geopolymer paste which has shown the best workability (based on manual approach) was selected for the geopolymer mortar studies, using various fine aggregates. The mix proportion of geopolymer mortar using various fine aggregates and HCFA by minimum void approach is listed in the Table 9. Various mix proportions of geopolymer mortar containing F_{Ag} / paste ratios (ranging from 1.6 to 1.2) were prepared to ascertain their workability (without the addition of extra water). It was found that a good workable mix was obtained when F_{Ag} / paste ratio is 1.2. Using the above F_{Ag} / pasteratio, mix proportions of geopolymer mortar for the different fine aggregate considered in this study were obtained. Cube specimens of size 70.6 × 70.6 × 70.6 mm were cast. (Table 11). For determining the flexural strength of mortar, a potential application, based on geopolymer mortar, flooring tiles were cast, using the mortar mix which has yielded the highest strength in the mortar. The chosen glass fiber mesh used as reinforcement in different layers (1to 3) and the tile specimens of size 300 × 300 × 20 mm (l × b × t) were cast and cured at ambient conditions. The specimens were tested at the end of 7 days for their transverse strength, according to the relevant IS code.

Table 7 Void Content and Void Ratio of Various Precursors (Fineness ≤ 63µm)

Precursor	Void Content	Void Ratio
HCFA	61.50	1.16
GGBS	37.50	0.60

Table 8 Details of Proportion of Precursor in Paste (By Volume and by Weight)

Precursor	By Volume		By Weight		HCFA/AA
	HCFA	AA	HCFA	AA	AA
HCFA	38.5	61.5	47.41	52.69	0.9

Table 9 Details of Proportion of Various Fine Aggregates in the Mortar using HCFA (by Volume and by Weight)

Fine aggregate	By Volume			By Weight			HCFA/AA	FAg/Paste
	FAg	HCFA	AA	FAg	HCFA	AA		
River sand	66.36	16.17	17.47	72.0	17.0	11.0	1.00	2.56
M sand	68.29	15.25	16.47	73.0	16.0	10.0	1.00	2.80
Demolition waste	65.92	16.38	17.70	69.0	19.0	12.0	1.00	2.22

Note: FAg – Fine Aggregate

III RESULTS AND DISCUSSION

E Geopolymer Paste

For a constant AAR (1.5) and molarity (10M), as the water to solids (w/s) ratio decreases from 0.61 to 0.47, the compressive strength (CS) of the paste increases, with the highest CS attained corresponding to the lowest w/s ratio used in this study (Table 10). As the w/s ratio decreases from 0.61 to 0.51 (which is a reduction of 0.05 from the initial level of 0.61), the increase in CS of geopolymer paste is only marginal is about 16%. A further reduction in w/s ratio to 0.47(which is a reduction of 0.14 from the initial level of 0.61), the increase in compressive strength is very high, and attains the maximum CS of 8.04 N/mm^2, which is 50% higher than the CS corresponding to the initial w/s ratio of 0.61. This shows that w/s ratio below 0.50 has a greater influence on the CS of geopolymer paste.The influence of w/s ratio seems to have insufficient influence on the density of the geopolymer paste. The average density of the geopolymer paste attained is 1.86 g/cc.

FGeopolymer Mortar

For a constant (AAR 1.5), molarity (10M) and W/S ratio (0.61), the CS of geopolymer mortar is the highest (14.67 N/mm2), when 'R' alone is used as the fine aggregate in the above mortar, whereas, the CS is the lowest(9.15N/mm^2), when 'M' is used as the fine aggregate in the above mortar. When 'D' is used to replace partially 'R' and 'M' (replacement level of 50%), the CS of the above mortar lies between the above maximum and minimum levels. (Table11). The type of fine aggregate used seem to have no influence on the density of the mortar and that average density of the above mortar is 1.94 g/cc. The above value of average density of mortar is only about 5% higher than the density of the paste (evaluated under identical conditions), which is very marginal / not significant.

The FS of geopolymer mortar using 'R' is slightly higher than the FS of the mortar, where 'M' is used as the fine aggregate in the mortar. However, the variation in the FS is not significant. The above trend is similar to the trend exhibits by CS of mortar, for the above two types of fine aggregates. However, when 'D' is used as fine aggregate to partially replace 'R' and 'M', the behavior of the FS of the mortar is different from the CS of the mortar (Table 12). FS of the mortar is the maximum (3.51 N/mm^2) when a combination of 'R' and 'D 'is used as fine aggregate in the mortar. The above value of FS is about 25% higher than the corresponding CS of the mortar, which is substantial.

Table 10 Mix Proportion for Geopolymer Paste using 'HCFA' for a constant AAR (M=10; FN=63μm; AAR=1.5; GGBFS=20%)

Specimen I D	FA$^+$	FA$^+$		AA	FA$^+$/ AA	Total Water	Total Solids	Water /Solids	Density (gm/cc)	Compressive Strength (N/mm^2) (at 7 days)
		HCFA	GGBS							
10.1	93.00	74.40	18.60	155.00	0.600	94.18	153.82	0.61	1.82	5.36
10.2	102.30	81.84	20.46	146.20	0.700	88.83	159.67	0.56	1.90	6.22
10.3	117.80	94.24	23.56	130.80	0.901	79.47	169.13	0.47	1.85	8.04

Note: ***(i) 'M' refers to the molarity; (ii)'FN' refers to the fineness of both HCFA and GGBFS; (iii) FA+ = (HCFA+ GGBF); (iv) In the specimen ID, the first digit refers to the 'molarity' and the second digit refers to the serial no. allotted to the specimen at the time of casting and the same is the used in the tables for easy identification of the specimen and the corresponding results; (v)sum of all precursors and AAs=250 g, which is constant for all vi)The above mix proportion is for casting 'mortar cube', specimen of size 50X50X50mm (Indian Standard)***

Table 11 Mix Proportion and Compressive Strength (CS) of Geopolymer Mortar using Various Fine Aggregates
(M=10; FN=63m; FA$^+$/AA=0.6; F_{Ag} /Paste=1.2; GGBS=20%; AAR=1.5; water/solids=0.61)

Specimen ID	F_{Ag} used	F_{Ag}	FA$^+$		Total Water	Total Solids	Density (g/cc)	Compressive Strength (N/mm2)
			HCFA	GGBS				
10.2	River S	409.09	102.27	25.57	129.46	211.45	1.97	14.67
10.1	M Sand	409.09	102.27	25.57	129.46	211.45	1.92	9.15
10.3	M + D	409.09	102.27	25.57	129.46	211.45	1.95	12.96
10.4	R + D	409.09	102.27	25.57	129.46	211.45	1.92	13.10

Note: (i) sum of all precursors and AA_s=750 g, which is constant for all. (ii) (The above mix proportion is for casting 'mortar cube', specimen of size 70.60X70.60X70.60mm (Indian Standard) (iii)Total quantity of AA used=213.07g; (iv)Total quantity of FA$^+$=127.84g;(v) M –refers to M-sand or manufactured sand; R-refers to river sand and D – refers to demolition waste.

Table 12 Mix Proportion and Flexural Strength (FS) of Geopolymer Mortar using Various Fine Aggregates (M=10; FN=63m; FA$^+$/AA=0.6; FAg/Paste=1.2; GGBS=20%; AAR=1.5)

Specimen ID	Aggregate used	F_{Ag}	FA$^+$		Total Water	Total Solids	Water /Solid	Density (g/cc)	Flexural Strength (N/mm^2)
			Fly Ash	GGBS					
10.2	River Sand	300.00	75.00	18.75	94.94	155.06	0.61	1.42	3.16
10.1	M Sand	300.00	75.00	18.75	94.94	155.06	0.61	1.38	2.92
10.3	M + D	300.00	75.00	18.75	94.94	155.06	0.61	1.39	2.92
10.4	R + D	300.00	75.00	18.75	94.94	155.06	0.61	1.40	3.51

Note: (i)sum of all precursors and AA_s=550g, which is constant for alL(ii)The above mix proportion is for casting 'mortar beam', specimen of size 40X40X160mm (Indian Standard) ;(iii) Total quantity of FA$^+$used is 93.75g;(IV) Total quantity of AA used =156.25g.

G Geopolymer Mortar Tiles

Up to two layers of the mesh, there seems to be no influence on the TS (transverse strength) of the geopolymer mortar tiles. However, when the number of layers of the mesh is increased to three, in the higher than that of the strength of the tile with one mortar tile, the TS reaches the highest value (that is 6.25 N/mm2) (Table 13). The above strength is 25% layer of the mesh. The above increase is very high and may be attributed to the higher number of layers of mesh used as the reinforcement.

Table 13 Mix Proportion and Transverse Strength (TS) of Geopolymer Mortar Tiles using Glass Fibre Mesh and River Sand as Fine Aggregate
(M=10; FN=63m; FA$^+$/AA=0.6; FAg/Paste=1.2; GGBS=20%; AAR=1.5)

Specimen ID	No. of layers	F_{Ag}	FA$^+$		Total Water	Total Solids	Water /Solid	Density (g/cc)	Transverse Strength (N/mm^2)
			Fly Ash	GGBS					
10.1	-	2100.00	525.00	131.25	664.56	1085.44	0.61	1.87	5.00
10.2	1	2100.00	525.00	131.25	664.56	1085.44	0.61	1.91	5.00
10.3	2	2100.00	525.00	131.25	664.56	1085.44	0.61	1.94	5.00
10.4	3	2100.00	525.00	131.25	664.56	1085.44	0.61	1.89	6.25

Note: (i) sum of all precursors and AA_s=3850g, which is constant for all;(ii) The above mix proportion is for casting 'tile', specimen of size 300X300X20mm;(iii) Total quantity of FA$^+$used is 656.25g;(IV) Total quantity of AA used =1093.75g.

IV CONCLUSIONS

Based on the extensive experimental investigations carried out on geopolymer paste and using high calcium flyash (80%) and ground granulated blast furnace slag (20%) considering various types of fine aggregates, ranges of W/S ratio at a constant molarity of 10, and their application to the development of a geopolymer tile reinforced with a commercial glass fibre mesh, following are the salient conclusions:

I. The range of w/s ratio considered (0.47 to 0.61), seems to have insufficient influence on the density of the geopolymer paste and that the average density of the paste attained is 1.86g/cc.

II. The type of fine aggregate used also have insufficient influence on the density of the geopolymer paste, and that the average density of the geopolymer mortar attained is 1.94g/cc. The density of the paste and the mortar differ only by 5%, which is not significant.

III. The compressive strength (CS) of the mortar is the highest (14.67 N/mm^2) at a w/s ratio of 0.61, when river sand (R) is used as aggregate, the flexural strength (FS) of the mortar is the maximum (3.51N/mm^2 at a w/s ratio of 0.61), when a contribution of river sand (R) and demolition waste (D) is used as fine aggregate in the mortar. The FS attained is about 25% of CS attained by mortar, which is substantial.

IV. A maximum transverse strength (TS) of 6.25N/mm^2 could be attained by the geopolymer mortar tile, when it is reinforced with three layers of glass fibre mesh. The above strength attained may be attributed to the higher number of layers of mesh used as reinforcement in the manufacture of the above tile.

V. The study indicates that it is possible to attain substantial CS and FS of geopolymer mortar using a combination of high calcium fly ash (80%) and GGBFS (20%) as precursors and curing at ambient conditions. Further, by using a commercial glass fibre mesh as reinforcement in the above mortar, it has the potential for the development of flooring tiles, after extensive studies and specific evaluation, considering end - user requirement/(s) and relevant codal provisions.

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