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Mechanical and microstructural properties of fly ash-based engineered geopolymer mortar incorporating waste marble powder

by

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Mechanical and microstructural properties of fly ash-based engineered geopolymer mortar incorporating waste marble powder

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Abstract

The marble processing industry produces a large volume of unmanaged waste in the form of micro-fine marble particles, usually referred as waste marble powder (WMP). Unregulated and open disposal of WMP has adverse effects on the environment. Marble is usually rich in calcium content, which can be used in geopolymer technology thereby enhancing its recycling value. This research sought to determine the viability of WMP as a supplementary binder and polymerisation potential of its high calcium content (55.96%). For this purpose, WMP was used as fly ash replacement by weight (0, 5, 10, 15 and 20%) in geopolymer mortar (GPM) while other mix proportions are kept the same. The results indicated that WMP substitution adversely affected the water absorption (WA), ultrasonic pulse velocity (UPV), compressive and flexural strengths of engineered GPM. The mechanical strength trends were supported by, scanning electron microscopy (SEM), energy dispersive X-ray spectroscopy (EDS), X-ray diffraction (XRD) and Fourier transform infrared (FTIR) spectroscopy tests, which revealed that the calcium content of WMP showed poor alkali activation. Marble particles remained unreacted in the GPM matrix and failed to form additional geopolymeric compounds as Ca/Si ratio was found to consistently decrease with higher WMP substitution. Accordingly, WMP can be used in geopolymers in combination with siliceous binder (fly ash) without significantly reducing the mortar mechanical properties and thus the resulting GPM can find broad applications in practice.

36 Keywords: Waste marble powder, Geopolymer mortar, UPV, SEM, EDS, XRD, FTIR.

37 **1. Introduction**

38 Waste marble is a by-product of marble cutting and processing industry produced in the form
39 of different sized aggregates and slurry. About 200 metric tons of marble waste are generated
40 annually on a global scale, of which China accounts for 34%, followed by Italy (19%) and
41 India (16%) (Pappu et al. 2019). Marble slurry contains micro-fine particles, commonly known
42 as waste marble powder (WMP) when dried, constituting approximately 20% of the total
43 marble waste (Khan et al. 2020). There is no systematic way to dispose of marble slurry and is
44 usually dumped in nearby open spaces, resulting in soil pollution. The fine size of WMP
45 reduces the permeability of topsoil which causes water logging. In addition, marble particles
46 increase the alkalinity of soil, thus harming its productivity and loss of local greenery. Thus,
47 there is a need to judiciously manage this non-biodegradable waste. This industrial by-product
48 has been recycled in various applications such as brick manufacturing, landfills and road
49 construction (Hebhoub et al. 2011).

50 Some researchers have shown the application of fine sized WMP in concrete manufacturing as
51 a partial or complete replacement of sand to prevent the over-dependence and depletion of
52 natural aggregates (Aliabdo et al. 2014; Ashish 2018; Ghani et al. 2020; Singh et al. 2017).
53 Kabeer and Vyas (2018) have demonstrated that WMP could be successfully used to replace
54 sand (up to 100%) in conventional cement mortar, with the optimum mix (20% WMP) showing
55 an 84% increase in compressive strength. It was found in another study by Hebhoub et al.
56 (2011) that the optimum compressive strength of 35.3 MPa can be obtained by replacing sand
57 with 50% marble waste aggregates. The performance under parameters such as dry shrinkage
58 and water absorption were found to be similar to the reference mix with natural sand.
59 Generally, WMP exhibits higher density and Blaine's fineness than sand, enabling it to
60 efficiently achieve a pore-filling effect in mortar and enhance mechanical properties such as
61 porosity and density of the matrix structure (Ashish 2019).

62 In recent years, marble powder has also been investigated as a partial replacement to OPC in
63 pastes and mortar, as WMP generally consists of high calcium oxide (30-60%) content and
64 may present hydration potential. Comprehensive research was conducted by Ashish (2018) to
65 investigate the feasibility of partial WMP replacement for OPC and sand amalgam. The author
66 reported that when WMP was used as cement replacement, the 7-days compressive strength
67 first increased by 7.17% for 10% WMP but then decreased by 6.77% for 15% WMP. Further,

68 EDX examination found a reduction in the elemental content of cementitious C_3S and C_2S
69 compounds, which discredited WMP of any evident role in the hydration process. However,
70 considering the substantial improvement in carbonation resistance, the author concluded that
71 WMP could be used as a suitable additive in concrete. Vardhan et al. (2015) observed that the
72 presence of WMP had a detrimental effect on the early hydration process, resulting in an
73 increase in both the initial and final setting time. The observed decrease in compressive strength
74 was attributed to the increase in number of voids with increase in WMP percentage from 10 to
75 50%, as determined through SEM images. Moreover, XRD investigation did not find any
76 new compositions of cementitious phases. Instead, an increase in the intensity of crystalline
77 peaks corresponding to calcite and Portlandite was detected. Other studies have also reported
78 findings showing a decrease in mechanical strength when using more than 10 % WMP as a
79 cement replacement in concrete or mortar (Wang et al. 2021; Lezzerini et al. 2022). However,
80 some studies also revealed positive effects of WMP as cement paste replacement on early age
81 (7-days) compressive strength of mortar. These effects were attributed to fluctuations in CaO
82 and Fe_2O_3 content of WMP (Vardhan et al. 2019). According to Kumar et al. (2020), the
83 observed densification of the concrete matrix in SEM pictures can be attributed to the filling
84 effect of WMP particles, rather than any modification in pozzolanic activity. According to Arel
85 (2016), the substitution of 5-10% of cement with marble dust would result in a 12% reduction
86 in CO_2 emissions.

87 Geopolymer is a sustainable technology that harnesses the binding properties of industrial by-
88 products, otherwise treated as waste and dumped, to produce a valuable construction material.
89 This method requires activation of silicates and aluminates with an acidic or alkaline solution
90 to form an inorganic polymeric chain (Palomo et al. 1999). Geopolymer mortar is an
91 environmentally friendly construction material that is used as an alternative to greenhouse gas-
92 emitting OPC-based products (Lee et al. 2016; Zhao et al. 2021). Despite its promising
93 potential, active research is being conducted to assess the feasibility of other waste materials
94 such as recycled aggregates that could enhance the mechanical strength and durability of the
95 geopolymer composite structure (Gill et al. 2023, 2023).

96 There is a sufficient number of research studies which experimented on hydration potential of
97 WMP in OPC-based mortar, but very limited studies have been done to explore the alkali
98 activation potential of WMP in geopolymer-based mortar. Wang et al. (2011) indicated that
99 dissolution of marble powder used as a replacement for natural aggregates could introduce

100 calcium-based compounds in geopolymer gel resulting in enhancement of the matrix strength.
101 Saloni et al. (2021) examined employing utility of waste marble aggregates (WMA) as a partial
102 substitute to natural coarse and fine aggregates (NA) in fly ash-based alkali activated concrete.
103 In their study, the addition of 50% WMA increased the strength by formation of additional
104 CASH gel, but pore microstructure deteriorated as reflected by deteriorating durability
105 properties. Some researchers have attempted various ways to valorize marble powder as a
106 potential precursor in rice husk ash and kaolin based geopolymers by combining it with cement,
107 clay, gypsum and blast furnace slag, with limited success (Lee et al. 2020; Komnitsas et al.
108 2021; Kamseu et al. 2022; Kaya et al., 2022;).

109 Considering the high calcium content and lower percentages of silica and alumina (2 to 5%) in
110 marble powder, a primary binder rich in amorphous silica and alumina content is necessary to
111 support the development of inorganic C-A-S-H polymeric chain, based on geopolymer reaction
112 mechanism (Duxson et al. 2007). Fly ash (FA) is a by-product of coal-fired thermal power
113 plants and an established geopolymer precursor which contains high percentages of Al_2O_3 and
114 SiO_2 (Saloma et al. 2016). Almost 370 million tonnes (MT) of FA is generated per year around
115 the world (Dwivedi and Jain 2014). India and China, being the biggest producers of FA,
116 produce about 112 MT and 100 MT of FA per year (Dwivedi and Jain 2014), respectively.
117 However, geopolymer made from Indian fly ash, classified as low-calcium with less than 10%
118 CaO, shows slow setting and low early-age strength (Chatterjee 2010; Rangan 2014; Jindal et
119 al. 2017). This type of geopolymer requires heat curing to expedite the polymerisation process,
120 which increases the overall production cost (Nath et al. 2015; Nikvar-Hassani et al. 2022).
121 Incorporation of calcium-based products such as slag and OPC in FA based GPC has shown to
122 significantly improve its mechanical and durability properties (Nath and Sarker 2015; Mehta
123 and Siddique 2017, 2018). This study attempts to utilize the calcareous property of WMP in
124 FA-GPM to gain similar benefits. Furthermore, fly ash availability for concrete sector is
125 declining as it has shown advantageous application and cheap consumption in other
126 construction fields such as road base construction, soil modification and structural fills among
127 others (Alam and Akhtar 2014; Surabhi 2017; Yousuf et al. 2020).

128 Therefore, efforts to make use of other by-products such as WMP as contributing geopolymer
129 binder are justified and indeed sought. Re-use of WMP in GPM would reduce material cost
130 and enhance its sustainability measures. The purpose of this study is to cover the knowledge
131 gap in this area and help identify WMP as a plausible precursor in geopolymer development.

132 This study investigates the polymerising potential of different MP-FA combinations (0, 5, 10,
 133 15 and 20%) with Na₂SiO₃-NaOH alkali solution. Limited WMP replacement was
 134 experimented as excessive usage may lead to deterioration in properties of GPM, as learned
 135 from previous studies. The mechanical performance was judged based on water absorption,
 136 ultrasonic pulse velocity, compressive strength, and flexural strength tests. In addition,
 137 microstructural modifications were assessed by scanning electron microscopy, energy
 138 dispersive spectroscopy, X-ray diffraction and Fourier transform infrared spectroscopy.

139 2. Material and methodologies

140 2.1. Materials for geopolymer mortar

141 2.1.1. Fly ash and marble powder

142 The primary binder in this study is class-F fly ash which meets ASTM C 618 (2014) criteria .
 143 It was collected from a coal-fired power plant in Karnataka. The chemical composition of FA
 144 is summarised in **Table 1**. Fly ash particles have a spherical shape and they act as fillers
 145 resulting in a compact and denser morphology (Sinsiri et al. 2010). The concentration of silica
 146 in FA is 59.62%. Marble powder was acquired from processing facility situated near Panipat,
 147 India. As marble contains a high amount of calcium i.e., 55.96%, compounds which can
 148 provide great potential to generate additional gels in the mix such as calcium silicate hydrate
 149 (CSH) and calcium aluminate silicate hydrate (CASH) (Saloni et al. 2021b). These products
 150 are responsible for strength gain in the produced material.

151 **Table 1. Chemical Composition of Fly ash & WMP**

Chemical Composition	Fly Ash (%)	WMP (%)
Silica (SiO ₂)	59.62	0.65
Alumina (Al ₂ O ₃)	25.79	0.23
Iron Oxide (Fe ₂ O ₃)	5.53	0.23
Total Sulfur (SO ₃)	0.45	0.12
Calcium Oxide (CaO)	6.35	55.96
Potassium Oxide (K ₂ O)	1.23	0.16
Sodium Oxide (Na ₂ O)	0.31	0.14
Loss on Ignition (LOI)	0.72	42.51

152
 153 XRD analysis also confirms the presence of amorphous silica in fly ash as the presence of
 154 quartz, mullite, mellite, and calcite can be seen in **Fig. 1 (a)** while the SEM image in **Fig. 1 (b)**
 155 shows the spherical shape of FA particles as reported in the literature. Marble powder had a

156 specific density of 2.76 which is much higher than 1.93 of fly ash particles as shown in **Table**
 157 **2**. With a comparable mean particle size of 17 μm and Blaine fineness of 3728 cm^2/g , WMP
 158 can potentially replace FA in the mixes.

159 **Table 2. Physical properties of fly ash and WMP**

Properties	Fly ash	WMP
Specific density	1.93	2.76
Blaine fineness (cm^2/g)	3,918	3,728
Mean particle size (μm)	21	17

160

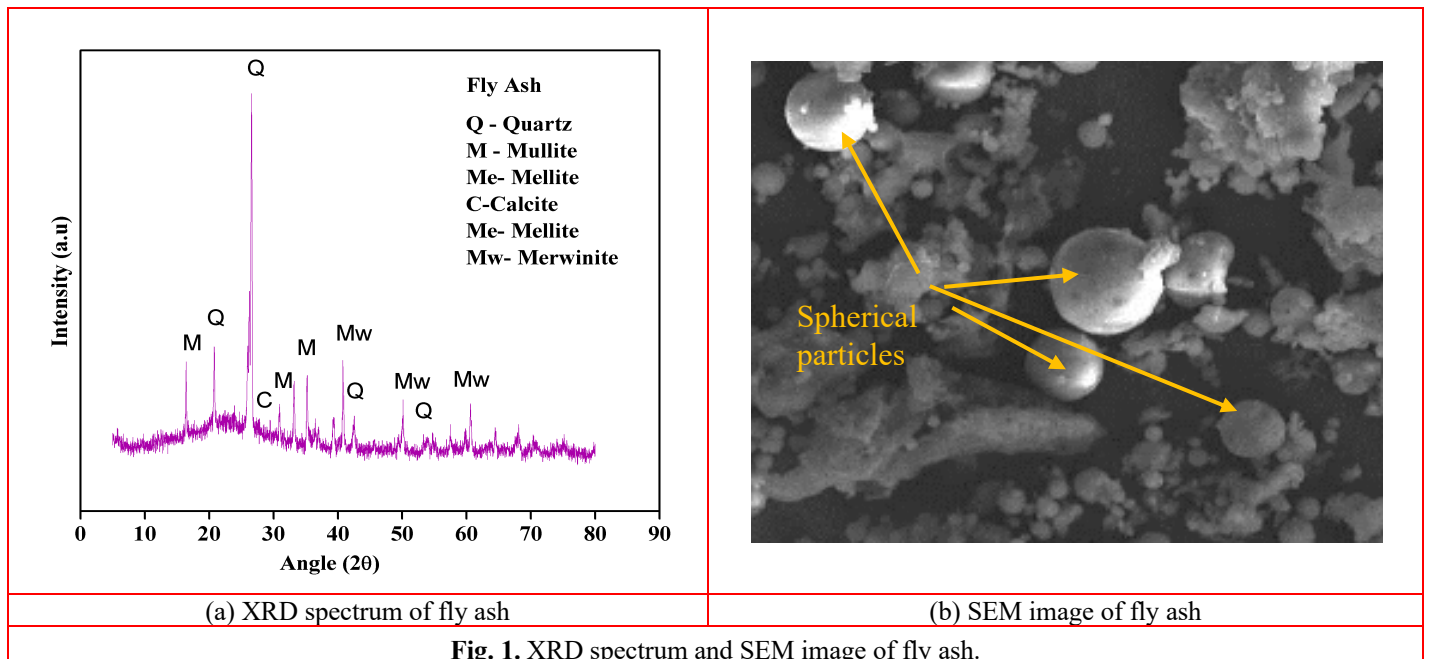


Fig. 1. XRD spectrum and SEM image of fly ash.

161

162 2.1.2. Alkaline activator

163 By mixing 99% pure NaOH pellets with tap water, a solution of NaOH was prepared with 8M
 164 concentration. For producing an alkaline activator liquid (AAL), NaOH solution was mixed
 165 with sodium silicate (Na_2SiO_3) and was kept for 5 minutes. The ratio between Na_2SiO_3 and
 166 NaOH was 2.5 to achieve desirable outcomes as suggested in previous studies (Lloyd and
 167 Rangan 2010; Anuradha et al. 2012; Ferdous et al. 2013; Junaid et al. 2015). Preparation of
 168 this mixture was done 24 hours before the final mixing to reduce the excessive heat released
 169 when NaOH is combined with Na_2SiO_3 .

170 2.1.3. Superplasticiser

171 Increased workability and flowability of the resultant mixture can be obtained by adopting a
 172 superplasticiser. In this study, a reducing agent polycarboxylate ether was used as a
 173 superplasticiser with water as suggested in a previous study (Ushaa et al. 2015).

174 **2.1.4. Fine aggregates**

175 **Table 3** shows the chemical composition of Yamuna river sand procured from a local supplier,
 176 and utilized as fine aggregates. Natural fines were predominantly composed of silica (80.11%)
 177 and alumina (11.65%). All the physical properties of fine aggregates comply with requirements
 178 of ASTM C 33-13 (2013) and are shown in **Table 4**. For instance, specific gravity and water
 179 absorption of natural sand, (2.63 and 0.71%, respectively), fulfilled the ASTM C127 (2009)
 180 conditions. Sieve analysis of fine aggregates resulted in a fineness modulus of 2.82 and the
 181 particle size distribution (PSD) curve is shown in **Fig. 2**.

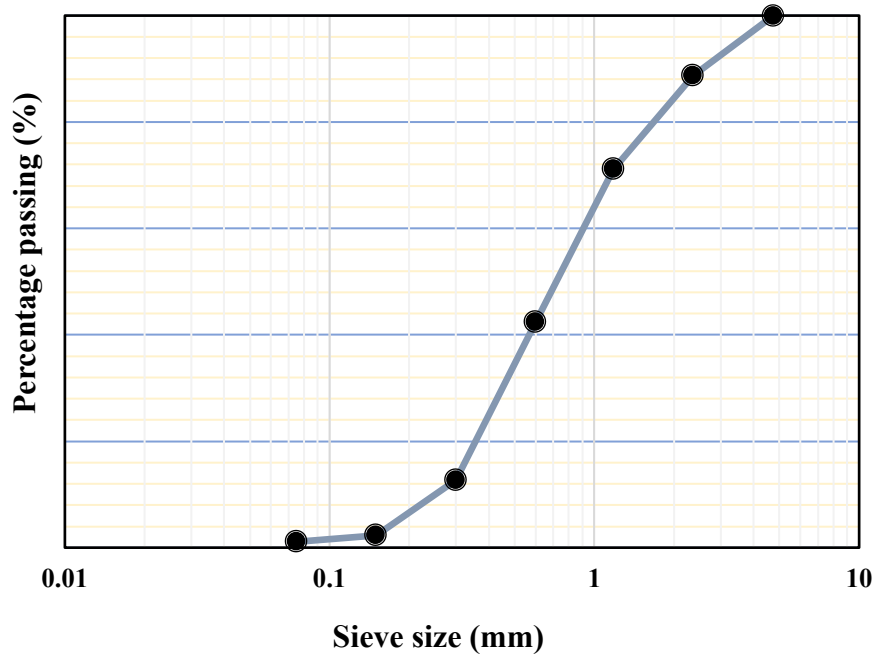
182 **Table 3. Chemical composition of fine aggregates**

Chemical Composition	(%)
Silica (SiO ₂)	80.11
Alumina (Al ₂ O ₃)	11.65
Iron Oxide (Fe ₂ O ₃)	2.25
Calcium Oxide (CaO)	2.57
Magnesium Oxide (MgO)	0.44
Potassium Oxide (K ₂ O)	0.77
Sodium Oxide (Na ₂ O)	0.81
Loss on Ignition (LOI)	1.4

183

184 **Table 4. Physical characteristics of fine aggregates**

Physical Property	Fine Aggregates (%)	Limits [ASTM C33 (2013)]
Specific gravity (ASTMC127, 2009)	2.63	>2.5
Water absorption (%) (ASTMC127, 2009)	0.71	<1
Impact value (BS812-112, 2015)	16.3	<25
Crushing value (BS812-110, 1990)	19.8	<25
Abrasion test (ASTMC535, 2009)	26.7	<50
Bulk density (kg/m ³) (ASTMC29/C29M, 1997)	1580	1200-1760
Voids content (%) (ASTMC29/C29M, 1997)	36.38	33-42



185
186 **Fig. 2.** PSD curve of fine aggregates used in the GPM mix.

187 **2.2.Mix Proportion**

188 Fly ash was used as the primary binder in all the GPM mixes while WMP was used as a
189 supplementary binder. A total of five mixes were prepared as summarised in **Table 5**. The first
190 mix 100F0M serves as a reference mix, in which “100F” indicates that the mix contains 100%
191 FA and “0M” shows the percentage of WMP replacement (0% for reference mix). Other mixes
192 had FA replaced by WMP with different percentages up to 20%.

193

Table 5. Mix Proportion

Mix ID	Fly Ash (gm)	Marble Powder (gm)	Sand (gm)	NaOH (gm)	Na ₂ SiO ₃ (gm)	Extra Water (gm)	Plasticizer (gm)
100F0M	100	—	150	13	32	20	2
95F05M	95	5	150	13	32	20	2
90F10M	90	10	150	13	32	20	2
85F15M	85	15	150	13	32	20	2
80F20M	80	20	150	13	32	20	2

194 **2.3.Mixing, casting and curing**

195 All the materials were dry mixed using a pan mixer for around 5 minutes. Afterwards, the
196 activator solution was added to the dry mixture. The mixes were then cured by heating in an
197 oven for 24 hrs at about 60°C. Heat curing was adopted to obtain the maximum compressive
198 strength as possible. To avoid evaporation of the samples, polyvinyl sheets were used to seal

199 all the samples. Next, all the samples were then stored in a laboratory until testing. The
200 laboratory temperature was between 25° - 27°C.

201 **2.4. Testing of specimens**

202 **2.4.1. Ultra-sonic pulse velocity test**

203 ASTM C597 (2016) guidelines were adopted to conduct ultra-sonic pulse velocity (UPV) tests.
204 A grinder was used to polish the faces of the cylindrical specimens. For the reception and
205 transfer of ultrasonic waves, piezo-transducers were used. The first transmitter was connected
206 to one end of the sample while the receptor transducer was attached to the other end. To
207 eliminate air, lubricant was used. A standard plastic bar of cylindrical shape having fixed values
208 of the wave velocity was used for equipment calibration before each experiment, which helps
209 in attaining proper readings.

210 A digital device triggered an actuator (JSR DPR 300). Picoscope V6.4.64.0 was used for
211 processing the datagrams. Time (t) taken by the pulse to pass through the sample was monitored
212 by a detecting sensor on a digital metre. This time is known as the ‘time of flight’. When both
213 of the transducers were placed at the centre of the specimen, the acoustic pulse travelled 200
214 mm. The equation for computing UPV value is given below:

$$215 \qquad \qquad \qquad \text{UPV} = l/t \qquad \qquad \qquad (1)$$

216 in which, the unit of UPV is in kilometres per second,

217 l denotes the length of the specimen

218 t is the time taken by the pulse to travel along the whole length of the specimen.

219 **2.4.2. Mechanical properties**

220 ASTM C348 (2002) based three-point loading system was adopted for the determination of the
221 flexural strength. The size for the beam specimens was 160 × 40 × 40 mm. The formula used
222 for the determination of flexural strength is given as follows:

$$223 \qquad \qquad \qquad S_f = 0.0028 P$$

224 where S_f denotes the flexural strength in MPa and

225 P denotes the maximum load in N.

226 For the determination of the compressive strength, ASTM C109-based guidelines were taken
227 into consideration. Three identical cylindrical specimens were used for each test.

228 **2.4.3. Water absorption tests**

229 For determining the porosity of the specimens, the water absorption tests were performed
230 according to ASTM C642-13 (2013). The dimensions of the cylindrical specimens were 100 ×
231 50 mm. Three identical cylindrical specimens were used for each test.

232 **2.4.4. Microstructural characterisation and spectroscopy analysis**

233 More advanced testing was conducted at 90 days to gain more in-depth understanding on their
234 microstructure. The sample was kept inside an enclosed chamber for an electron beam to strike
235 it. An electron microscope was used in this examination. A contrast detector was used for the
236 backscattered electrons which provides a contrast between various chemical constituents and a
237 clear SEM image is produced.

238 EDS analysis was also performed for wavelength differentiation. An energy dispersive detector
239 was used for this purpose which analyses X-Ray radiations. Afterwards, XRD analysis was
240 conducted which monitored the scattering angles and intensity of the X- Rays emitted by the
241 sample (Kim et al., 2012). For this purpose, the sample was first bombarded with X- Rays. A
242 curve was then plotted between the angle of scattering and the values of intensities obtained.

243 FTIR analysis was carried out according to ASTM E1252 (2013). The purpose of this test is to
244 detect the organic compounds inside the mixture produced. In this analysis, compounds
245 containing carbon and hydrogen can be distinguished. The specimen was powdered and kept
246 in a cup inside a diffuse reflectance device. The output was obtained in the form of an infrared
247 spectrum.

248 **3. Results and discussions**

249 **3.1. Water absorption**

250 The water absorption test was performed to evaluate the outcome of geopolymerization on the
251 pore structure of fly ash and WMP-based geopolymer matrix. Accordingly, the effect of marble
252 replacement on the water absorption of geopolymer mortar was also examined. The tests were
253 carried out after a curing period of 7, 14 and 28 days, and the results are presented in **Fig. 3**. In
254 general, the water absorption of all the mixes did not change significantly, varying within the
255 range of 1.98% to 2.36% for all the investigated ages.

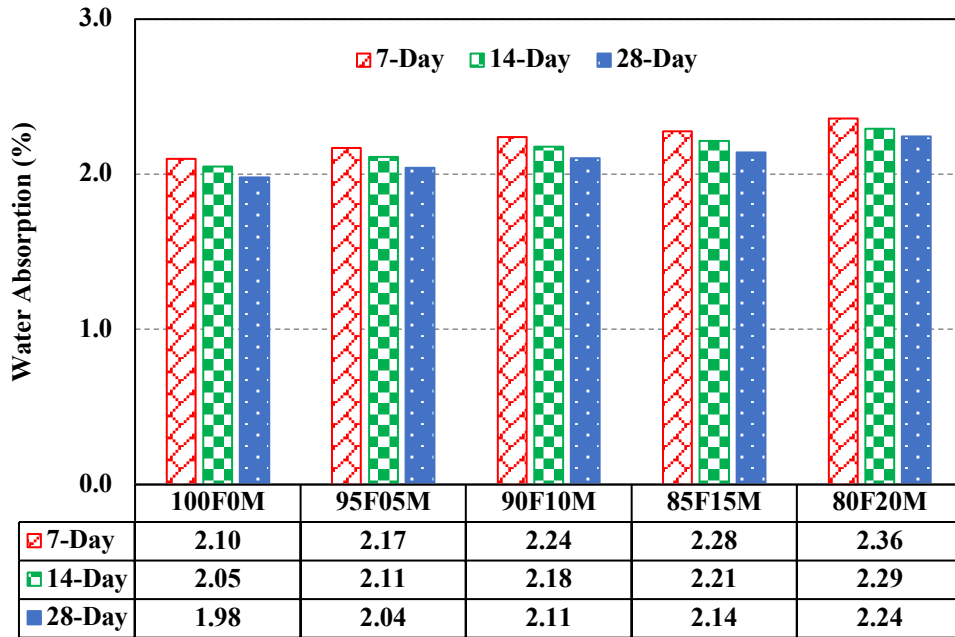
256 The least water absorption was observed by the control mix without marble powder inclusion
257 (100F0M) for all ages. From the results, it was observed that the water absorption of WMP-

258 based geopolymer mortar for all curing ages only slightly increased as compared to the control
259 mortar. Meanwhile, other mixes also showed a similar range with a bit higher value of the
260 water absorption. The percentage of water absorption at 28 days of all the geopolymer mortar
261 mixes ranged from 1.98% to 2.24%. Since the average particle size of WMP is smaller than
262 FA, it creates a closed packing density by filling the micro-pores in the matrix. However, non-
263 reacted particles of marble waste, which are presented later, did not participate in the
264 polymerisation. Therefore, these remained non-reacted particles did not improve the denseness
265 of the microstructure in the matrix.

266 When comparing the 7-day water absorption of mixes 95F05M, 90F10M, 85F015 and
267 80F020M with 100F0M, an increment of 3.33%, 6.66%, 8.57% and 12.38% respectively was
268 noticed. This may be due to the immaturity of the specimen since waste marble creates a
269 hindrance due to its non-reactive particles in the polymerisation process. This trend was similar
270 for all the investigated curing periods. Meanwhile, the specimens cured at 28 days showed
271 marginally lower water absorption percentages. This can be attributed to the additional
272 polymerisation of fly ash with time and the development of geopolymer gel around unreacted
273 waste marble powder particles. Despite all the facts, the water absorption percentage of mixes
274 95F05M, 90F10M and 85F15M was lower than 3% at all ages, and it justifies the presence of
275 WMP in the geopolymer mortar did not considerably increase its water absorption.

276 In general, the addition of marble powder had two opposing effects in geopolymer formation.
277 First it decreases available aluminosilicate precursor content, which decreases strength.
278 Secondly, marble particles worked as extra fine un-reactive aggregates and provide a suitable
279 nucleus for the formation of a network of interlinked polymer chains. Marble particles failed
280 to form interfacial transition zone (ITZ) with GPM matrix, but due to WMP pore-filling effect,
281 the GPM pore structure did not degrade considerably as evidenced by only a marginal increase
282 in the water absorption.

283 Yamanel et al. (2019) revealed that inert marble dust as cement mass replacement (5, 10, 15
284 and 20%) did not contribute to hydration of mortar mixture. Furthermore, marble dust increased
285 the porosity and hence the water absorption capacity from 6% in the reference mix to 8% in
286 the mix with the highest marble content. Another study (Komnitsas et al. 2021) showed that
287 the water absorption increased by 25.66% when 30% WMP was used as a binder substitute in
288 metakaolin-based GPM. These results from the previous studies showed the similar influence
289 of WMP on the water absorption.



290

291

Fig. 3. Percentage water absorption of geopolymer mortar mixes at various ages.

292

3.2. Ultrasonic pulse velocity test

293

The ultrasonic pulse velocity test (UPV) was performed to measure the stress wave velocity in the specimens and thus examine the internal structure of geopolymer matrix. Assessment of cracks and their bonding ability with WMP can be assessed by UPV. Mean UPV values for each GP mix at distinct ages are shown in **Fig. 4**. Evaluating the speed of ultrasonic pulses travelling through geopolymer matrix made with/without marble powder provides a good indication for denseness of the microstructure. It was observed that UPV of geopolymer mortar made up solely of fly ash as source material was higher than other mixes with WMP inclusion. Meanwhile, an increase in UPV values was observed in all the mixes with respect to curing ages, which ensures the progressive formation of internal structure with ages. At 7 days, the UPV of mixes 95F05M, 90F10M, 85F15M and 80F20M reduced by of 12%, 18.2%, 24.34% and 32.38% as compared to that of 100F0M, respectively. A similar trend was observed for 14-day cured specimens.

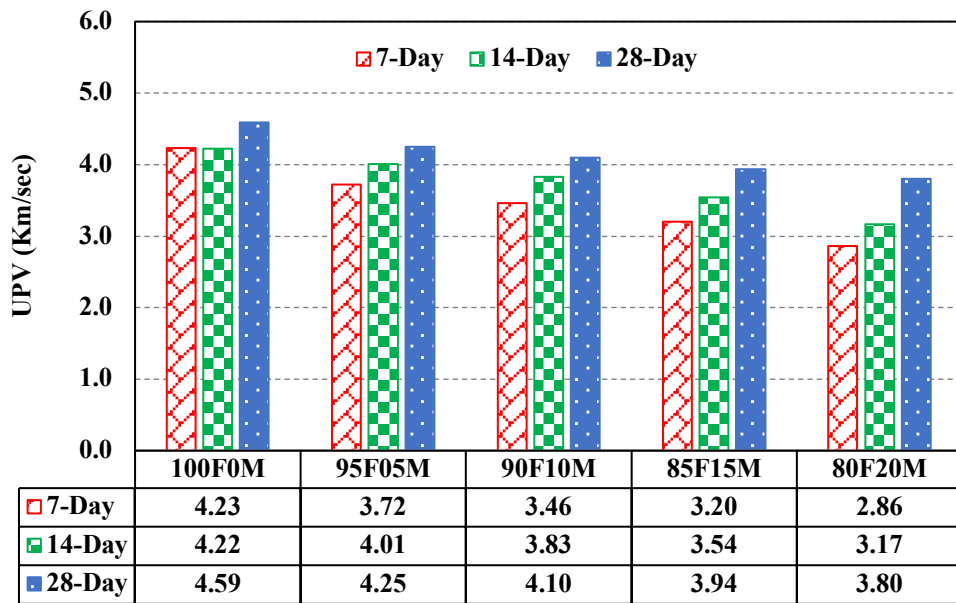
305

From the test results, it was observed that the 28-day UPV of mixes 100F0M, 95F05M and 90F10M was respectively 4.59, 4.24 and 4.10 km/s and these values are categorised as excellent according to BS 1881–203 (1986) guidelines (**Table 6**). The 28-day UPV performance for mixes 85F15M and 80F20M was observed as medium quality. The UPV of mixes 95F05M and 90F10M reduced respectively by 7.4% and 10.6% as compared to that of mix 100F0M, but fall in excellent category. The relatively poor performance of mixes

310

311 85F015M and 80F20M may be due to cavities created around excess un-reactive marble
 312 particles. In addition, the decrease of UPV can be attributed to the reduction of the compressive
 313 strength of the mixes with WMP replacement. Marble powder is a substitution to binder content
 314 and its replacement leads to a reduction in the compressive strength (f_c), which is proportion to
 315 the modulus of elasticity (E_c). As a result, a reduction of the compressive strength leads to
 316 decrease in the elastic modulus but with a slower rate. The UPV test measures the velocity of
 317 stress wave in concrete, which can be calculated as $V = \sqrt{\frac{E_c}{\rho}}$, where ρ is the density of
 318 concrete. When the density of concrete remains almost unchanged or is expected to have a
 319 minor change, the UPV is proportional to the elastic modulus. Therefore, a decrease in the
 320 compressive strength of mortar led to a decrease in its UPV.

321 Similar finding was also reported by Seghir et al. (2020) who attributed the decline in UPV
 322 (3.16% with 15% marble replacement) to the increased porosity and reduced hydrate products
 323 in cement-based mortar, when incorporating WMP as a binder substitute.



324
 325 **Fig. 4.** Ultrasonic pulse velocity of geopolymer mortar mixes at various ages.

326
 327

Table 5. Quality Interpolation from UPV

Age	Average Ultrasonic Pulse Velocity (km/Sec)				
	100F0M	95F05M	90F10M	85F15M	80F20M
28-day	4.591	4.248	4.103	3.941	3.799
Quality	Excellent	Excellent	Excellent	Medium	Medium

328 3.3.Compressive strength

329 The compressive strength of all the mixes at 7, 14 and 28 days is presented in **Fig. 5**. Each
330 result of the compressive strength is the average of three identical specimens tested at different
331 ages. All the comparisons are made with reference to the control mix, 100F0M. Irrespective of
332 age, the compressive strength of the reference mix, 100F0M, was greater than all the other
333 mixes. The 7-day compressive strength of mixes 95F5M, 90F10M, 85F15M and 80F20M
334 reduced by 3.68%, 6.90%, 10.86% and 18.97 % as compared to that of mix 100F0M,
335 respectively. The corresponding compressive strengths at 14 and 28 days also revealed the
336 same trend. Mixes 95F5M and 90F10M exhibited the 28 days compressive strength comparable
337 to that of the control mix (24.95 MPa). Although mix 80F20M showed a remarkable decrease
338 in the 28-day strength, by 4.24 MPa as compared to the control mix, but percentage wise the
339 28 days strength reduction (16.9%) is less than the results of 7 days testing (18.97%). This
340 observation indicates that marble powder delayed strength development at early stages of
341 geopolymer formation. The compressive strength reduced as the replacement level of marble
342 powder increased suggested that marble particles served primarily as fillers and they did not
343 effectively participate in geopolymer reaction, which is further confirmed by XRD and FTIR
344 analyses. Furthermore, marble powder used as a replacement for fly ash (5-15%) resulted in
345 about 4-17% decrease in the compressive strength at 28 days.

346 Temuujin et al. (2010) explained that the compressive strength of geopolymer mortar remains
347 essentially same for varying sand aggregate content because the strength of mortar primarily
348 depends on strength of hardened geopolymer gel. Therefore, a decrease in the compressive
349 strength of GPM due to marble powder addition is a result of less geopolymer gel formation
350 and poor interfacial bonding between inert marble particles and geopolymer gel. Also marble
351 particles themselves possess weaker mechanical properties than geopolymer gel and natural
352 aggregates. The compressive strength of geopolymer mortar primarily depends on volume and
353 strength of geopolymer gel and aggregates, and the bond between aggregates and geopolymer
354 gel.

355 The fly ash content in geopolymer decreases with an increase in marble powder content which
356 results in increased AAL to fly ash ratio and increases the probability of dissolution of fly ash
357 for complete geopolymerisation. However, a previous study indicated that each fly ash patch
358 has a different optimum Al/FA ratio to achieve complete activation depending on fly ash
359 fineness and amorphous content (Hadi et al. 2018). Based on preliminary studies done in this

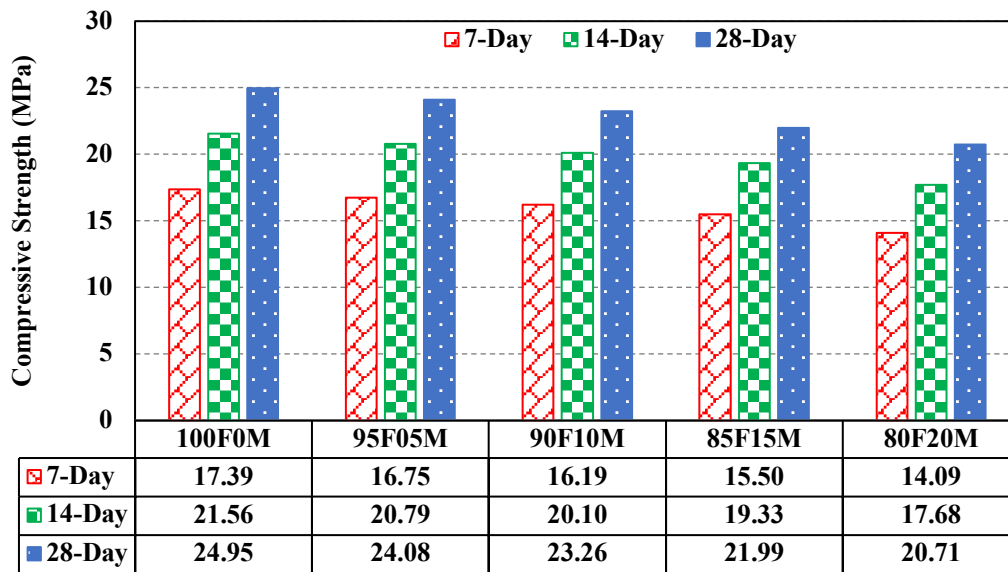
360 study the AAL content of 0.45 was sufficient for dissolution of fly ash and any further increase
 361 would not lead to the formation of additional geopolymeric gel and increase in GPM strength
 362 (Hardjito et al. 2004).

363 The compressive strength pattern obtained in this study is in agreement with an existing study
 364 by Komnitas et al. (2021) who used WMP as binder replacement in metakaolin-based
 365 geopolymer mortar. The decrease in the compressive strength of specimens with an increase in
 366 waste MP to metakaolin mass ratios of 0.3, 0.7 and 1.5 was found to be 20.41%, 36.1% and
 367 60%, respectively; and was attributed to poor alkali activation potential of marble powder.

368 At 28 days, the compressive strength of mixes 95F05M, 90F10M, 85F15M and 80F20M
 369 reduced by 3.48%, 6.77%, 11.86% and 16.99% as compared to that of mix 100F0M,
 370 respectively. From these results, an empirical equation can be derived to estimate the
 371 compressive strength of the mix at 28 days as follows:

372
$$f_c = 25 - 16.98 (WMP/FA) \quad R^2 = 0.977$$

373



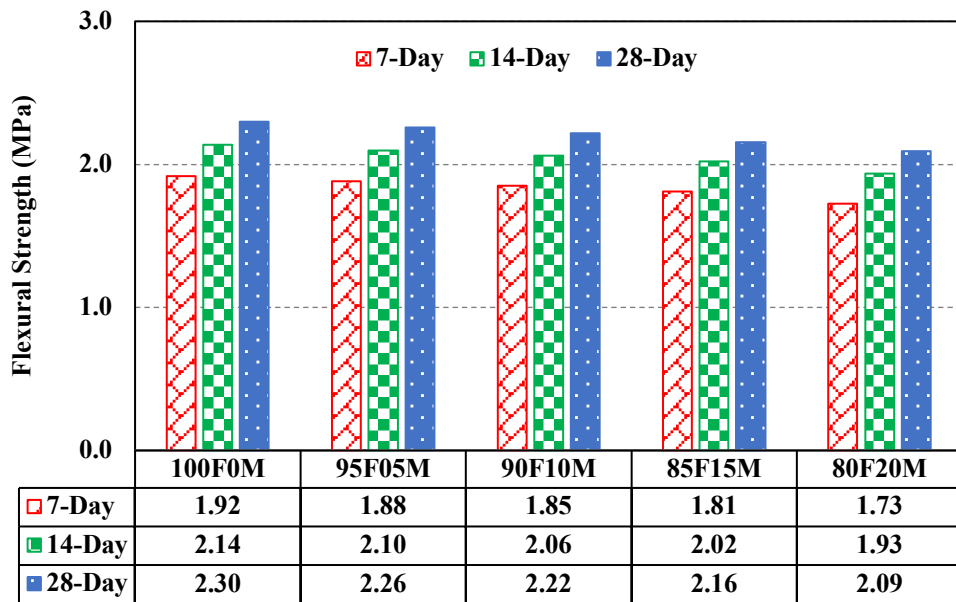
374

375 **Fig. 5. Compressive strength of geopolymer mortar mixes at various ages.**

376 **3.4.Flexural Strength**

377 **Fig. 6** depicts the flexural strength of the beam specimens for all the mixes at 7, 14 and 28
 378 days. The results reveal that the flexural strength of mix 100F0M was also the highest among
 379 all the other mixes. Therefore, it could be inferred that fly ash plays a vital role in geopolymer

380 mortar while marble powder was not effective in gaining strength. Irrespective of the curing
 381 period, the flexural strength of mix 95F05M was found almost similar to the control mix. The
 382 minimum flexural strength was observed in mix 80F20M, e.g., its flexural strength at 7, 14 and
 383 28 days was 1.92, 2.14 and 2.30 MPa, respectively. At 28 days, the flexural strength of mixes
 384 95F05M, 90F10M, 85F15M and 80F20M decreased by 1.73%, 3.47%, 6.08% and 9.13%
 385 regarding mix 100F0M, respectively. This in the flexural strength may be due to the poor
 386 bonding of marble particles with GPM matrix. The reduction in flexural strength results was
 387 due to higher porosity of the interfacial transition zone (ITZ) with marble particles. Although,
 388 the flexural strength of a mortar is directly related to its performance under compressive load,
 389 strength reduction percentage in flexure was less as compared to its compressive strength.

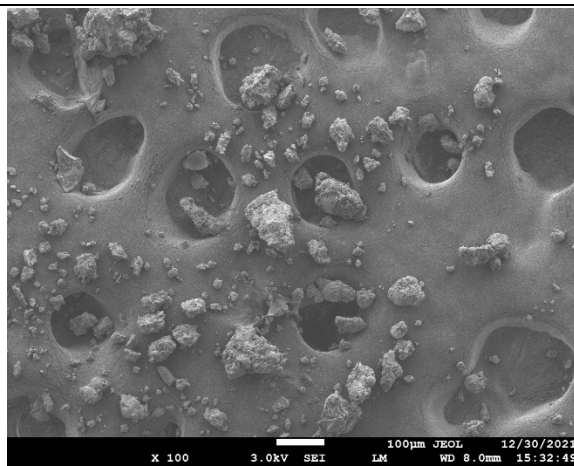


390
 391 **Fig. 6. Flexural strength of geopolymer mortar mixes at various ages.**

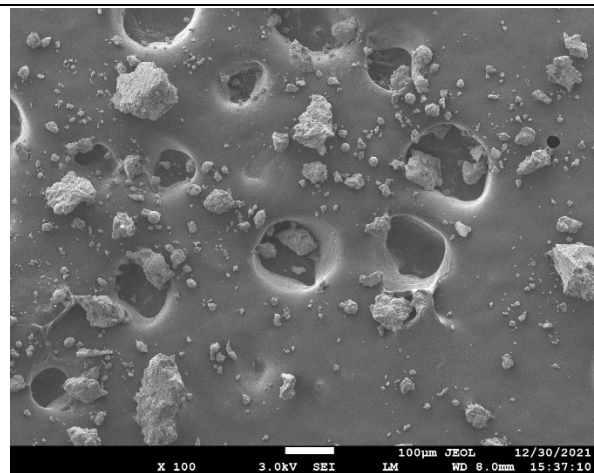
392 3.5.SEM and EDS analyses

393 Scanning electron microscopy (SEM) examination was conducted after 28 days and the
 394 resulting images are shown in **Fig. 7**. SEM images were used to investigate the GPM
 395 microstructure in detail and the bonding characteristics between geopolymer matrix and marble
 396 powder. At the microscopic level the matrix appears to be homogenous which suggests that
 397 unreactive micro-sized marble particles get evenly distributed in the GPM matrix. A close
 398 inspection of the interfacial region suggests negligible dissolution of the WMP particles which
 399 result in almost non-existent bond between marble particles and geopolymer binder. So, it can
 400 be concluded that the marble powder was mostly unreactive in geopolymer as no sufficient

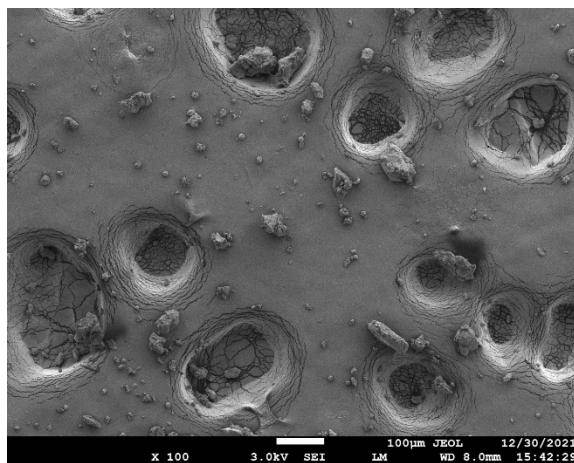
401 bond was formed with the matrix. Due to weak adhesion with geopolymer gel, the marble
402 particles act merely as void fillers but did not affect the mortar matrix homogeneity
403 significantly. The SEM image of 100F0M shows that the control GPM mix, without marble
404 powder, had more compact and homogenous structure when compared to the SEM image of
405 the other mixes with marble powder replacement. The SEM images of mixes 95F05M,
406 90F10M, 85F15M and 80F20M show a higher degree of pores, unreacted particles and frequent
407 micro cracks, which justifies their high water absorption and low strength performance. Fine
408 marble particles which affect the geopolymer gel formation and the weaker inter-transition
409 zone is found in SEM images of mixes 85F015M and 80F20M.



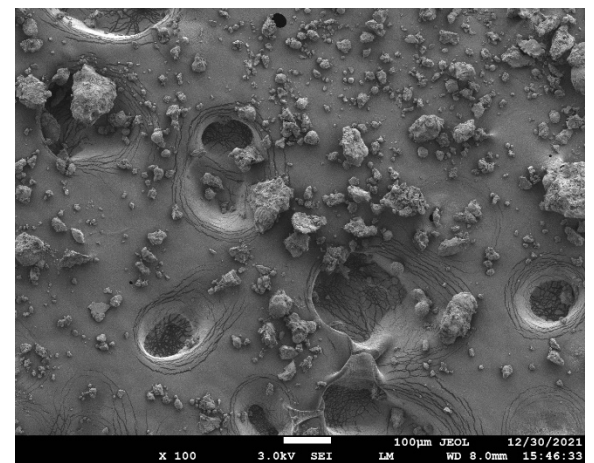
(a) Compact and homogenous microstructure of 100F0M



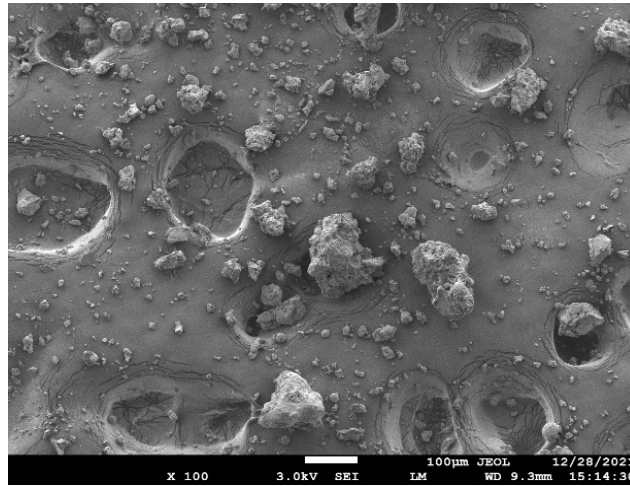
(b) Even distribution of marble particles in the 95F05M matrix



(c) Porous structure observed in 90F10M



(d) Early signs of micro-cracking in 85F15M

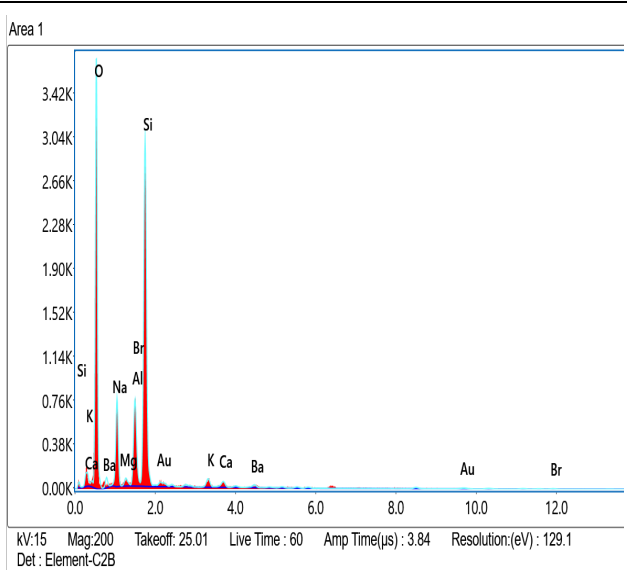
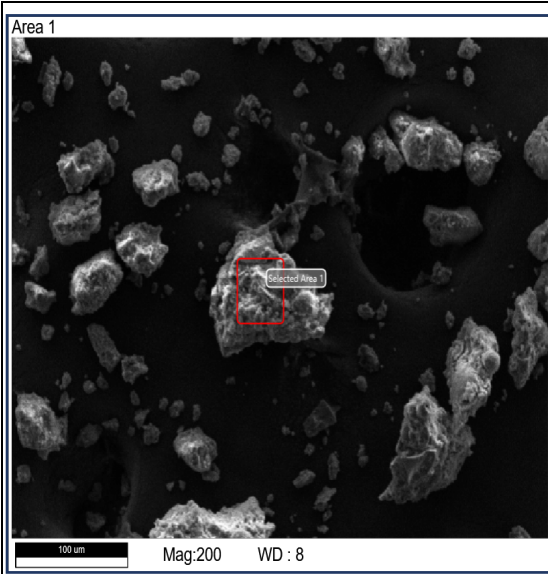


(e) Unreacted marble particles in 80F20M

410

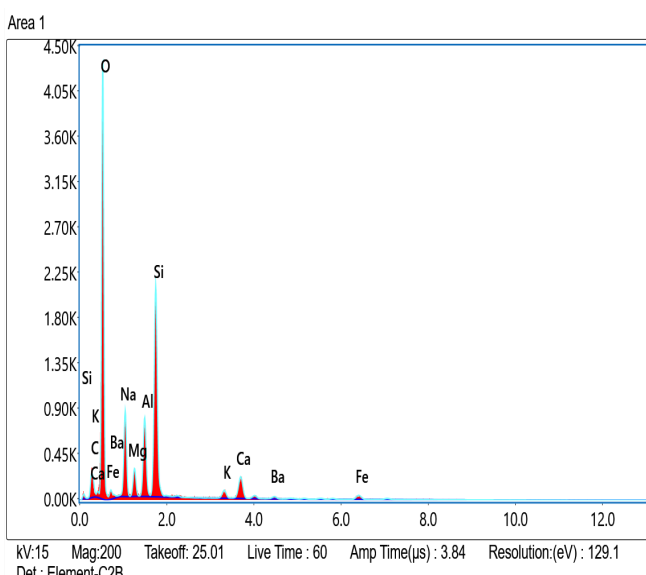
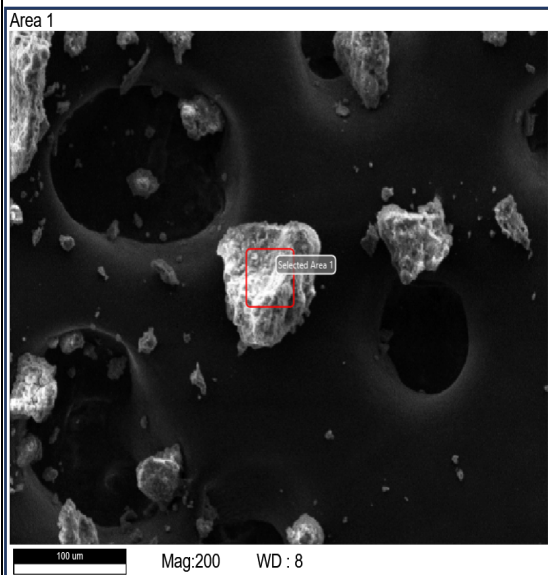
Fig. 7. SEM images of all the mixes.

411 **Fig. 8** presents the microstructural investigations using EDS tests, which show that Na, Al and
412 Si make up the majority of the glassy matrix in all the GPM mixes and are the essential
413 constituents of a geopolymer gel. According to the elemental atomic ratios of GPM calculated
414 in **Table 8**, the geopolymeric matrix's Si/Al ratio slightly reduces with WMP replacement
415 percentage, which has a detrimental effect on the development of geopolymer gel (Duxson et
416 al. 2007; Wang et al. 2020). It is because when incorporating WMP, the fly ash content
417 decreases and AAL/FA ratio increases, which leads to a decrease in dissolution of unreacted
418 fly ash particles and release of less aluminosilicates in the geopolymer gel. The Ca/Si ratio
419 shows a marginal decrease with the WMP content, which proves that the incorporated marble
420 particles were almost unreactive in surrounding alkaline media and did not produce significant
421 calcium ions in the geopolymer mix. Furthermore, the carbon compounds percentage increases
422 from 8.91% to 16.07% (**Table 8**) due to carbonation of excessive alkaline solution, as AAL/FA
423 ratio increases.



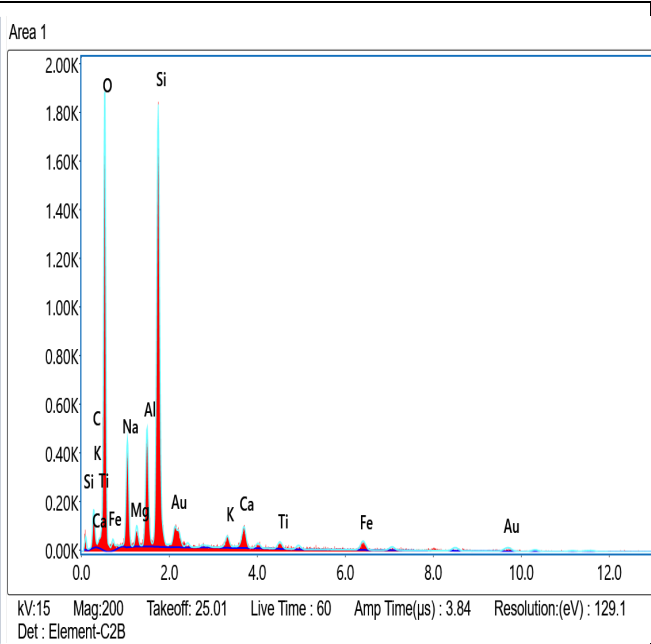
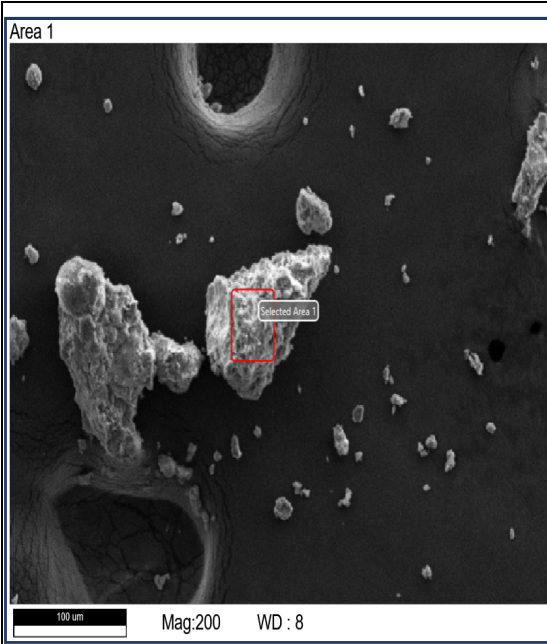
(a) 100F0M at 28 days

EDS analysis at 28 days (100F0M)



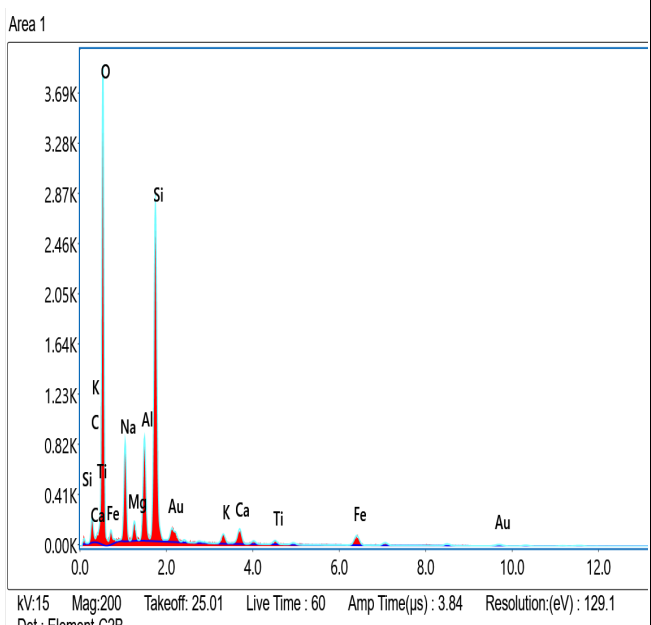
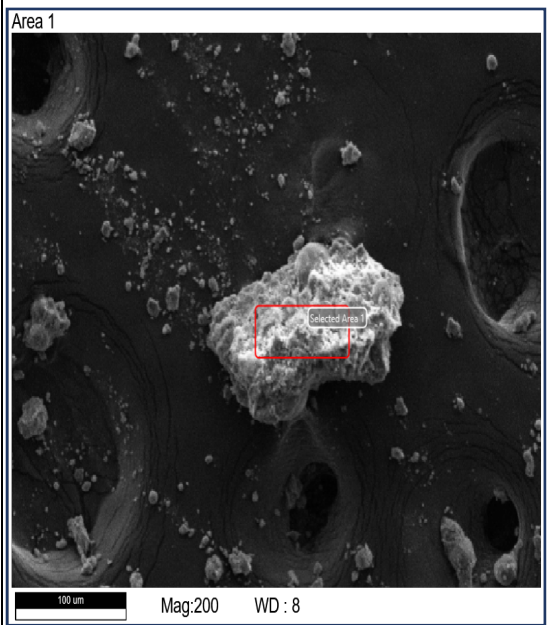
(b) 95F5M at 28 days

EDS analysis at 28 days (95F5M)



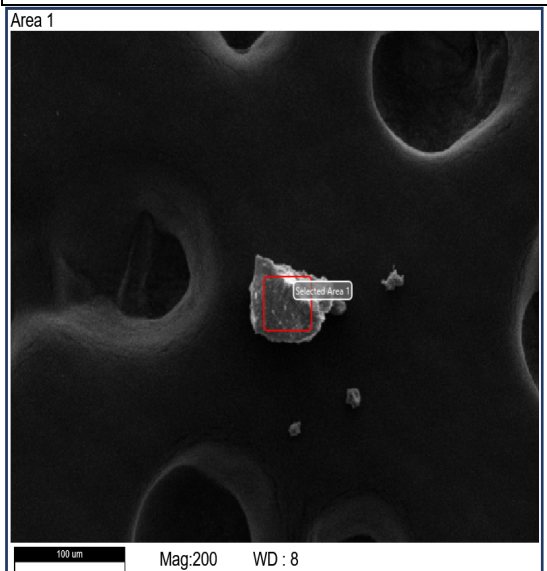
(c) 90F10M at 28 days

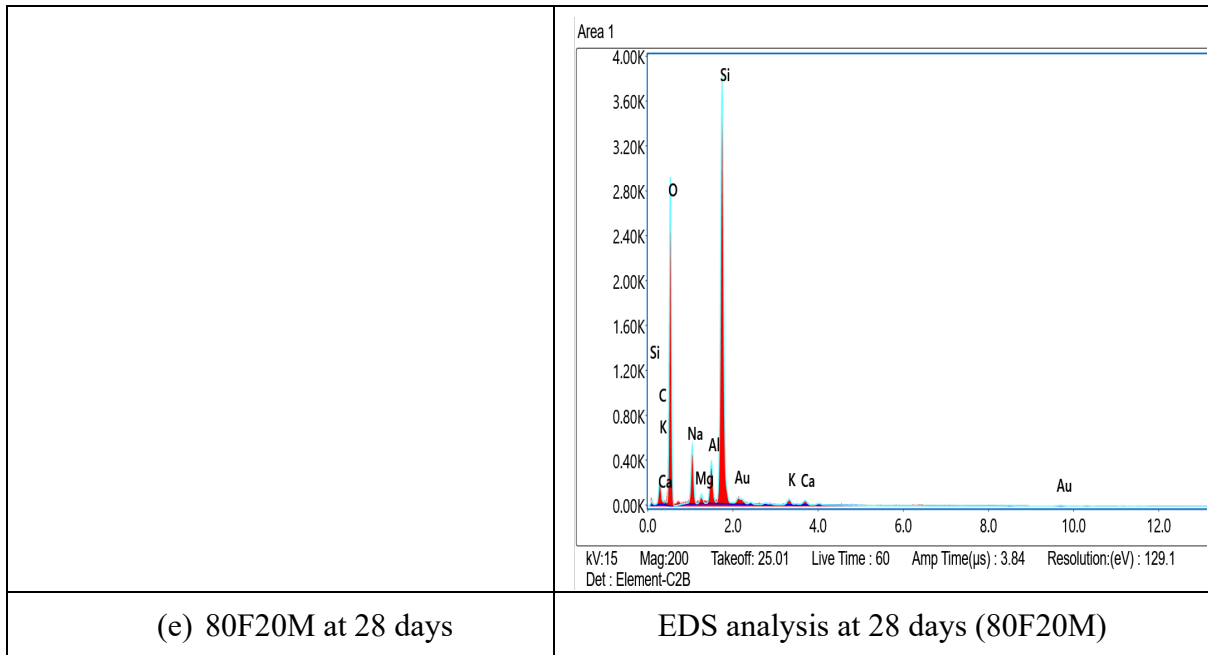
EDS analysis at 28 days (90F10M)



(d) 85F15M at 28 days

EDS analysis at 28 days (85F15M)





424

Fig. 8. EDS analysis results (Note: Needles at Ca and Si)

425

Table 6. Atomic ratios of elements.

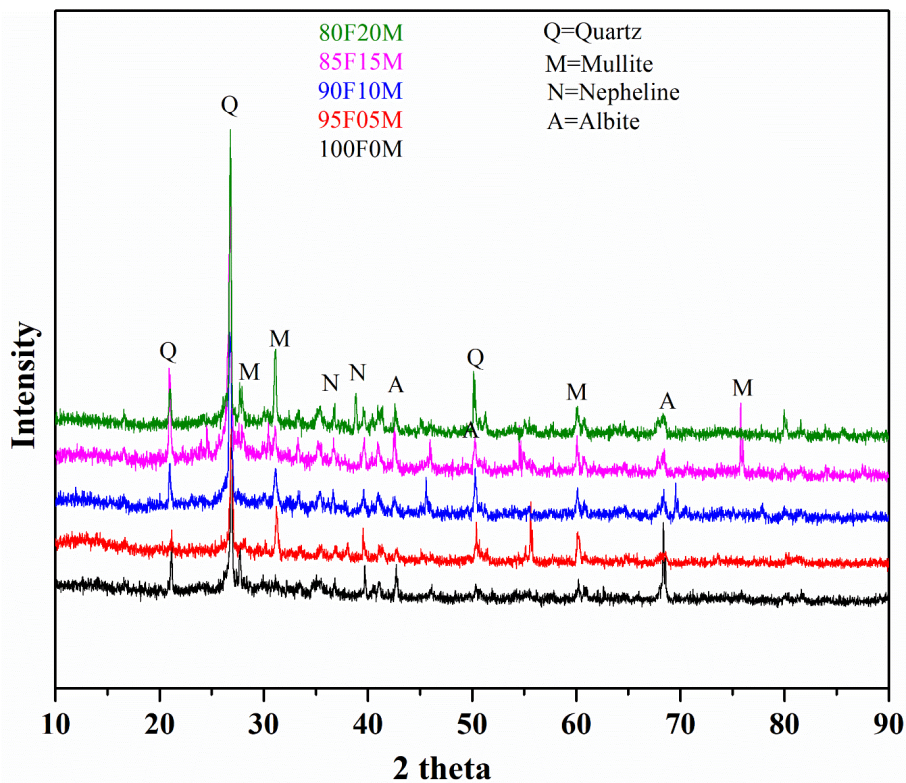
	100F0M	95F05M	90F10M	85F15M	80F20M
Element	Atomic (%)	Atomic (%)	Atomic (%)	Atomic (%)	Atomic (%)
C K	8.91	10.21	12.98	14.53	16.07
O K	56.07	55.8	53.8	52.26	52.21
Na K	9.56	8.93	7.91	7.84	6.88
Mg K	1.51	1.94	1.34	1.18	0.83
Br L	1.69	0.77	1.15	0.29	0.78
Al K	4.23	4.32	4.39	4.68	4.81
Si K	12.45	12.42	12.43	13.01	13.36
Au M	0.1	0.1	0.37	0.57	0.14
K K	0.76	0.6	0.67	0.65	0.48
Ca K	3.59	3.52	3.39	3.21	3.14
Ba L	0.32	0.23	0.16	0.31	0.16
Ti K	0.42	0.37	0.52	0.63	0.43
Fe K	0.39	0.79	0.89	0.84	0.71
Ca/Si	0.288	0.283	0.273	0.247	0.235
Si/Al	2.943	2.875	2.831	2.780	2.778

426

427 **3.6.X-Ray Diffraction (XRD)**

428 The existence of distinct phases in GPM specimens after 28 days was determined by XRD
 429 analysis as shown in **Fig. 9**. The crystalline content of a sample produces sharp diffraction
 430 peaks when bombarded with X-rays. The control GPM mix shows the presence of crystalline

431 minerals which were intrinsic phases of the aluminosilicate raw material used, as crystalline
 432 components are incapable of dissolution in the polymerisation reaction. Crystalline phases such
 433 as mullite, nepheline and albite are common to both fly ash and its geopolymer mortar. These
 434 crystalline feldspar minerals are composed of aluminate compounds. The absence of mullite
 435 and quartz peak near 32° and 51° , respectively, in control GPM was due to higher dissolution
 436 of fly ash and consumption of amorphous silica in the alkaline media. From the XRD results,
 437 it is inferred that the presence of quartz was dominant in all the mixes. Quartz mineral peaks
 438 in the control GPM were due to the crystalline silica component of fly ash and natural fine
 439 aggregates. The dispersion peaks in the region of 22° to 36° are the characteristic amorphous
 440 substances in geopolymer. This broad hump in **Fig. 9** is noticeably displaced to the right in
 441 comparison to the XRD patterns of fly ash in **Fig. 1 (a)** (12° to 28°), indicating the production
 442 of new amorphous substances (Na-Al-Si-H) in the geopolymer reaction products.



443

444

Fig. 9. XRD graphs of geopolymer mortar specimens

445 Furthermore, the position and size of dispersion peaks of reaction products of each mix were
 446 similar, implying that the degree of polymerisation and reaction products were relatively the
 447 same for WMP-based GPM. By comparing different phases present in XRD graphs of fly ash-
 448 based geopolymer control mix and its engineered mortar mixes, it is clear that there were no
 449 peaks and humps that indicate the presence of calcium-based compounds in GPM mortars.
 450 Thus, using marble powder as a partial substitute for fly ash did not alter the phase composition

451 qualitatively. However, marble powder did change the phase ratios. It is evident from the XRD
452 results that marble powder primarily remains inert to the alkaline activator solution and lacks
453 potential to take part in the geopolymerisation reaction. Komnistas et al. (2021) also reached
454 to similar conclusion while inspecting the valorization potential of marble powder through
455 alkali activation.

456 **3.7. Fourier Transform Infrared Spectroscopy (FTIR)**

457 FTIR analysis report can be seen in **Fig. 10**. During the geopolymerisation reaction of the
458 control mix, 100F0M, the Si–O–Si/Si–O–Al bending band can be seen at 440 cm^{-1} , while the
459 band at 542 cm^{-1} appears due to AlO_4^- vibrations. The band at 1019 cm^{-1} is due to asymmetric
460 stretching of Si–O and Al–O bonds resulting from dissolution of precursor fly ash. The
461 relatively weak band at 1385 cm^{-1} represents stretching vibration of CO_3^{2-} ion. This peak
462 becomes more noticeable when WMP is introduced in the GPM mix due to the presence of
463 CaCO_3 in marble. This observation further establishes that the calcium content of marble
464 remains bound, which renders it impotent to form new bonds with geopolymer gel. In all the
465 geopolymeric mixes, bands in the regions of 1640 and 3440 cm^{-1} which were attributed to
466 bending vibrations (H–O–H) and stretching vibration (–OH), respectively and represent the
467 bound water present in the polymerisation products (Hou et al. 2009). Bound water molecules
468 were largely adsorbed on the geopolymer gel surface and some were trapped in the GPM
469 cavities.

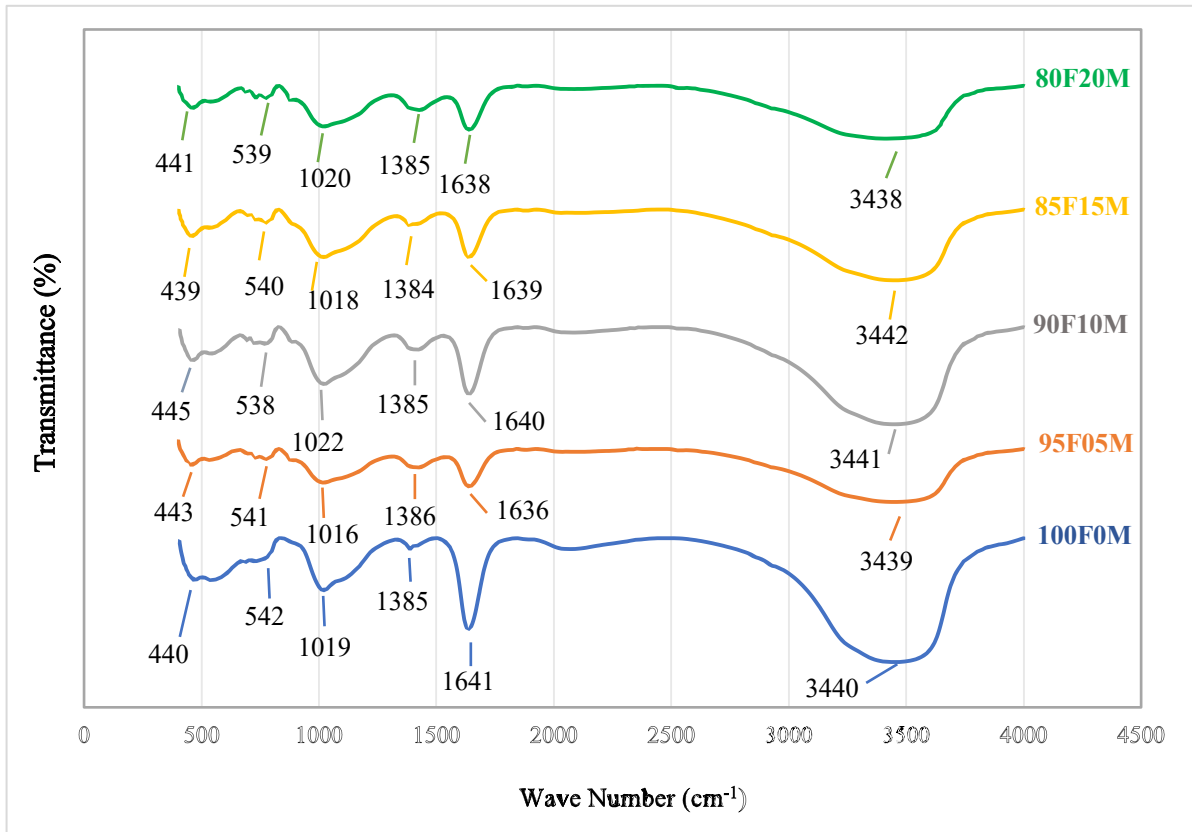


Fig. 10. FTIR spectra of each geopolymer mortar mix.

470
471

472 All the above bands are indicative of the formation of the aluminosilicate network in a
 473 geopolymer gel. It is quite complex to determine the extent of geopolymerization based on
 474 location and intensity of these bands, but their presence is solely due to the presence of
 475 amorphous phase of the alumino-silicate raw material.

476 It can be observed that GPM mixes with marble powder addition did not display new peaks,
 477 which indicates that marble powder has not formed additional chemical bonds with geopolymer
 478 matrix and acts as a completely inert ingredient.

479 **4. Conclusions**

480 This study examined the feasibility of WMP as binder supplement in fly ash-based GPM based
481 on mechanical performance and microstructural investigations. Following main conclusions
482 can be made:

- 483 1. With an increase in WMP replacement, the water absorption increased as high specific
484 surface area of WMP and unreacted marble particles introduced additional void volume in
485 the matrix. However, all mixes showed a satisfactory WA percentage of less than 3%.
- 486 2. The decrease in UPV values with WMP is attributed to reduction in geopolymer gel volume
487 proportion that deteriorated the overall matrix denseness and strength. However, after 28
488 days all the specimens exhibited UPV values which were classified well above the medium
489 quality mortar.
- 490 3. Replacement of FA with WMP consistently reduced the compressive and flexural strength
491 of GPM, due to WMP inability to contribute to geopolymer gel formation.
- 492 4. Further, 28 days compressive strength of all the mortar mixes prepared in this study, was
493 above 20 MPa which could be used to make masonry mortar bricks and non-traffic
494 pavement blocks, as per strength requirements of IS 2250 (1981) and IS 15658 (2006).
- 495 5. SEM images show unreacted WMP particles occupying interstitial spaces. Further, EDS
496 analysis shows a marginal decrease in Ca/Si ratio with increasing WMP content, which
497 revealed that WMP remained unreactive in surrounding alkaline media and did not produce
498 additional calcium compounds. The percentage of carbon compounds increased with
499 WMP/FA ratio, which could be attributed to carbonation of unused alkaline solution.
- 500 6. FTIR and XRD examinations confirmed that WMP has no noticeable role in
501 geopolymerisation process and acted as a mere filler material, since no new compounds
502 and chemical bonds were revealed during these microstructural studies.

503 Based on mechanical test results and microstructural analyses conducted in this study, it is
504 suggested that WMP integrated GPM at low content (<20%) can find sustainable applications
505 in various construction activities without significant reduction in mechanical strength.

506 **Compliance with Ethical Standards and Competing interests**

507 ☒ The authors declare that they have no known competing financial interests or personal
508 relationships that could have appeared to influence the work reported in this paper.

509

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