

# Light Weight Plasters Containing Vermiculite and FGD Gypsum Wastes for Sustainable and Energy Efficient Building Construction Material

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**Research Article** 

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## Additional Declarations:

Tables 1-6 is available in the Supplementary Files section.

## **Light Weight Plasters Containing Vermiculite and FGD** 1 **Gypsum Wastes for Sustainable and Energy Efficient** 2 **Building Construction Material** 3

4 Abstract: Development of lightweight plasters for mortar rendering utilizing Flue gas desulfurization (FGD) gypsum 5 have been reported here. Lightweight plasters prepared using FGD gypsum and exfoliated vermiculite were characterized 6 and studied in detail for interior wall applications. Different gypsum vermiculite plasters (GVP) with variable amounts of 7 vermiculite were characterized by X-ray diffraction (XRD), scanning electron microscopy (SEM) and thermal 8 gravimetric analysis (TG-DTG). The physicochemical and mechanical properties of all the samples are determined 9 and considered to be efficient for interior applications. An optimum mix composition was selected based on its 10 compressive strength, water absorption and porosity. Water absorption and porosity studies restrict the usage of GVP 11 only to interior wall purposes. The acoustic performance of the materials revealed good sound absorption ( $\alpha = 0.65$ ). 12 Plasters exhibit satisfactory durability under severe conditions of winter and summer weather. GVP shows excellent 13 fire resistance under BS 476-1997 fire resistance classification with thermal conductivities (< 0.161 W/mK) much 14 lower than standard building materials, which makes them fit for energy efficient insulation materials. These studies 15 depict the efficient utilization of thermal power plant waste, FGD gypsum in interior wall insulation for mortar rendering and can be further extended to exterior construction applications by reducing water absorption. 16 Keywords: FGD gypsum, light weight plaster, GVP, acoustic properties, thermal conductivity. 17 18

#### 19 1. Introduction

20 Flue Gas Desulfurization Gypsum (FGDG) is obtained from coal-based electric power plants. Burning of coal 21 produces harmful flue gases like  $CO_2$ ,  $NO_x$ , and  $SO_x$ .  $SO_x$  is removed from the flue gases by the process of 22 desulfurization via different techniques. Scrubbing method is most extensively used for the process of desulfurization. 23 'Wet-type scrubbing process' and 'dry or Semi-Dry scrubbing process' are generally two scrubbing methods. Among 24 them 'Wet-type scrubbing' is the most widely used technique for desulfurization [1,2]. The chemical composition of 25 FGD gypsum is CaSO<sub>4</sub>.2H<sub>2</sub>O, identical to natural gypsum. Chemical reactions involved in the process of conversion 26 of SO<sub>2</sub> gas into FGD gypsum is as follows [3,4,5]-

27	$SO_2 + H_2O \rightarrow H^+ + HSO_3^-$	(1)
28	$H^+ + HSO_3^- + 1/2O_2 \rightarrow 2H^+ + SO_4^{2-}$	(2)
29	$2H^+ + SO_4^{2-} + Ca(OH)_2 \rightarrow CaSO_4.2H_2O$	(3)

30 In this process, slurry of lime or limestone, which is forced oxidized used as a sorbent for sulfur dioxide and 31 results in the generation of FGD gypsum. Due to the huge available amounts of FGD gypsum, it needed to be placed 32 in disposal areas. This process is expensive as well as causes various kinds of pollution problems [6]. The FGD 33 gypsum's composition is comparable to that of natural gypsum in terms of CaO and SO<sub>3</sub>, although it contains less 34 Al<sub>2</sub>O<sub>3</sub> and Fe<sub>2</sub>O<sub>3</sub>. This is because the FGD gypsum lacks the clays and iron minerals found in the natural material [7]. 35 According to a CSE report, India is about to become the largest emitter of SO<sub>2</sub> in the world by replacing China, 36 as its emission has increased by 50% in the last decade. In order to prevent this extreme situation, a significant share

37 (90%) of India's thermal power capacity is likely to opt for wet limestone FGD technology due to its well-proven

- 38 operational and technological track record across the world. It is estimated that after the installation of FGD units,
- 39 India's power plants would produce around 12–17 million tonnes of gypsum by 2022 with a consumption 32,000
- 40 tonnes of limestone per annum by a 500 MWh unit. Under this scenario, the unit would produce 54–60,000 tonnes of
- 41 gypsum per annum. At this rate of generation, FGDG stockpiled and due to its ability to absorb toxic elements like F,
- 42 As, Hg, Cd, Cr and Pb etc. [8] will be pernicious to the environment and possess detrimental effects to land, water and
- 43 air. Therefore, it is necessary to explore the utilization potential of this waste material, as it can be beneficial for both
- 44 the economy and the environment.
- 45 Gypsum is an integral component of cement production and the sector relied on costly imported or poor quality 46 synthetic gypsum. Gypsum is a soluble source of essential plant nutrients like calcium and sulfur and can improve 47 overall plant growth [9]. Gypsum amendments improve the physical properties of some soils (especially heavy clay 48 soils) [10,11]. It helps reduce erosion losses of soils and nutrients, and the concentrations of soluble phosphorus in 49 surface water runoff. FGD gypsum is being used in agriculture in US and China. Since ancient times, gypsum has also 50 been used as a building material due to its unique hydration and dehydration property. It is well known that gypsum 51 building materials are light weight, provide thermal insulation, fire resistance [12] and possess good acoustic properties. 52 Moreover, rapid population growth and urbanization have increased demand for raw materials such as cement and 53 aggregates many folds in the construction industry worldwide. The mission of sustainable development has led to 54 pressure to improve environmental performance in the construction process by reducing consumption of natural 55 resources extracted.
- 56 Researchers around the world utilized FGD gypsum for the production of various kinds of value added building 57 materials such as wallboard [1,13], blocks [14,15], tiles [16,17] etc., after conversion into  $\beta$ -hemihydrate gypsum. 58 Several researchers demonstrated the procedure for the production of structural board from FGD gypsum. A process 59 for the production of a multilayer thermal insulating board containing different power plant coal residues such as Fly 60 ash, FGD gypsum etc., has been patented. FGD gypsum has also been used in mixtures applied as fire proof coatings 61 for steel structures [18]. Apart from its use in wallboard products, research also has been carried out on the 62 development of FGD gypsum markets mainly in two areas: (a) as an admixture in cement and (b) agriculture uses, 63 such as a soil amendment. Thus, utilization of waste gypsum (FGD) offers not only the solution of disposal problems, 64 but also help to save cement, reduce carbon foot prints by reducing green house gases emission, conserve natural 65 resources for meeting increased demand of aggregates and save energy and environment. Additives are typically 66 incorporated into the gypsum matrix to improve the material's insulating qualities and increase its fire resistance. The 67 most common additions used for fire protection include vermiculite, mica, alumina, perlite, and ceramic hollow 68 spheres [19,20]. Vermiculite, a mineral that resembles mica and has sparkling flakes, belongs to the phyllosilicate 69 family. When heated between 650 and 950°C, it expands up to 30 times its original volume [21]. Vermiculite's physical 70 characteristics may vary depending on how its chemical composition changes [22]. Due to the apparent volume 71 expansion following high-temperature calcining, vermiculite is a type of multi-layered silicate rock with a very high 72 specific surface area. Vermiculite undergoes numerous cycles of interlayer water release during the calcining process 73 before becoming expanded verniculite (EV), which has a highly porous structure [23]. EV is suitable for a wide range

74 of applications across a variety of fields due to its low thermal conductivity, low bulk density, relatively high 75 refractoriness, chemical inertness durability, and environmental safety. Increased workability, reduced unit weight, improved sound and thermal insulation, and improved fire resistance are all benefits of employing EV in the matrix 76 77 [24]. These facts justify the use of vermiculite as a construction material and simultaneously as a heat isolation and 78 sound absorption material [25,26] Gypsum plasters' 28-day compressive strength was reported to be reduced by 79 19.46% and 32.22%, respectively, with the addition of 10% and 20% EV (size 0.145 mm). On the other hand, the 28-80 day bending strength was improved by 16.25% and 35%, respectively, with the addition of 10% and 20% EV [27]. It 81 has been also reported that the addition of EV at values ranging from 5% to 25%, by weight, decreased the mechanical 82 strength of fibrous gypsum plasters. As the EV content increases, this reduction also increases [28]. The tiles have 83 been manufactured from cement and exfoliated vermiculite and the EV incorporation was 5-50%. According to the 84 findings, the amount of exfoliated vermiculite decreased with increasing compressive strength and flexural strength 85 [29]. Raw, inert materials of various densities have been widely used and known for producing lightweight cements. 86 Lightweight aggregates like expanded perlite and vermiculite, among others, are typically combined with gypsum and 87 cement to lessen the density of the finished product [30]. In order to create a novel lightweight plaster material with 88 improved thermal qualities for industrial uses in accordance with building rules, the compatibility of polyamide 89 powder waste with plaster has been investigated [31].

90 In the present work, we proposed to develop light weight plaster with FGD gypsum using vermiculite for 91 rendering work in the construction industry. Several mixes of  $\beta$ -hemihydrate gypsum and exfoliated vermiculite were 92 envisaged in different proportions to develop light weight gypsum vermiculite plasters (GVP). Raw FGD gypsum was 93 calcined to obtain  $\beta$ -hemihydrate form. The gypsum vermiculite plasters with vermiculite content of 3, 4, 5, 8, 10 & 94 12% were casted into cubes and studied for their mechanical properties such as compressive and flexural strength. 95 The cubes were characterized by X-ray diffraction (XRD), scanning electron microscopy (SEM) and thermal 96 gravimetric analysis (TG-DTG). GVP samples were investigated for their water absorption, porosity, thermal 97 conductivity, fire resistance, acoustic properties and durability under severe environments.

### 98 2. Materials & Methods

#### 99 2.1 Raw materials

100 Different mix compositions of FGD gypsum (FGDG) and vermiculite were used for the development of light 101 weight plaster. FGDG was procured from NTPC (National Thermal Power Corporation) operated coal-based power 102 plant, VSTPS (Vindhyachal Super Thermal Power Station) located in Singrauli, Madhya Pradesh. Vermiculite crystals 103 of grade, 1-2 mm (after grit removal) were used. Diethylenetriaminepentaacetic acid (DTPA) was used as a chemical 104 retarder to delay fast setting of binders. The FGDG sample is greyish in appearance and vermiculite particles reflect 105 light to dark brown particles. The collected samples of FGDG were sun dried, ball milled for an hour, and sieved 106 through a 90 µm sieve prior to its various physical and chemical analyses as per IS: 1288-1982 [32]. The physical 107 properties of the materials are summarized in Table 1. These results suggest that FGDG conforms to the requirements 108 of IS: 12679-1989 [33]. A pycnometer was used to measure the specific gravity of the samples.

109 2.2 Characterization

Scanning electron microscopy (SEM; Carl Zeiss-Ultra plus55), powder X-ray diffraction (XRD; Bruker-D8
 Advance, Cu-Kα) and thermogravimetry/ differential thermal analysis (TGA/DTA; EXSTAR, SII 6300) were used
 for characterization of the samples.

113 Microscopic images of FGDG show euhedral prismatic rod-like particles ranging from 1 µm to 200 µm in length 114 without any sign of agglomeration. SEM images of exfoliated vermiculite depict flaky lamellar structures 115 corresponding to multi-layered trioctahedral structure of vermiculite made of Mg-Fe-Al-Si. This lamellar structure 116 contains air in-between layers that explain the exfoliation of the vermiculite [34]. XRD data of the materials were 117 recorded on a RIGAKU 2200 diffractometer at 30 kV and 10 mA for Cu-K $\alpha$  ( $\lambda = 1.54050$  Å), with a step size of 0.02° 118 in 20. The XRD pattern of FGDG revealed that the main crystalline phases present are gypsum and CaO, 119 corresponding to more than 90% of gypsum content determined by chemical analysis IS:1288-1999. XRD pattern of 120 vermiculite shows major peaks of calcium magnesium aluminum silicate (#) at 27.59°, 29.92°, 35.10°, 35.46° and 121 45.81°; calcium aluminum silicate (\*) peaks observed at 20 of 9.44° and apart from it, a peak of quartz (Q) is also 122 observed at 26.64°.

TGA/DTA has been realized to determine the phase stability and different species present in the materials. The weight loss as a function of temperature was determined using TGA and details of the experiment are given somewhere else [35]. TGA curve of FGDG sample shows two signatures of weight loss at ~ 140 °C and ~ 660 °C that signifies endothermic loss of 3/2 moles of H<sub>2</sub>O from calcium sulfate dihydrate to form calcium hemihydrate (Eqn. 4) and weight loss due to decomposition of carbonate content (Eqn. 5), respectively.

- 128  $CaSO_4.2H_2O \xrightarrow{\Delta} CaSO_4.1/2H_2O + 3/2H_2O$  (4)
- 129

$$CaCO_2 \xrightarrow{\Delta} CaO + CO_2$$
 (5)

TGA curve of vermiculites accounts for 5% weight loss on heating upto 1000 °C. Early weight loss ~ 100 °C
 accounted for the loss of surface absorbed water molecules. However, the majority of weight loss occurs between 500
 to 900 °C, which could be a result of dehydration of chemically bound water molecules [36].

133 2.3 Mechanical Properties

The mechanical properties of all the mixes were determined as per IS: 2542-1978 (Part I) [37]. Setting times of
 pastes were determined using normal Vicat's apparatus according to IS: 2547-1976 (Part II) [38].

#### 136 2.4 Water Absorption and Porosity

To determine water absorption and porosity of hemihydrate plaster, 25mm x 25mm x 25mm cubes were casted and treated as mentioned above [30,39]. The initial weight of the cubes was measured and then immersed in water for a particular period of 2, 8 and 24 hours, and their weight was measured again after each period to calculate the percentage of water absorption using the following expression-[40]

141 
$$Water absorption (\%) = \frac{W_2 - W_1}{W_1} \times 100$$
(6)

- 142 where,  $W_1$  is the initial weight of the specimen and  $W_2$  is the weight of the specimen after immersion in water.
- 143 The porosity of the specimens was determined from the following expression-

144 
$$Porosity = \frac{W.d_S}{d_W}$$
(7)

145 where, W is the weight loss,  $d_S$  is the dry density of the gypsum binder and  $d_W$  is the density of water used.

#### 146 2.5 Thermal Conductivity Measurement

147 The guarded hot plate method was used to determine the thermal conductivities of GVP samples. It follows a 148 steady-state method that requires the specimen insulating material to be in equilibrium with its surroundings for

- accurate measurements. The thermal conductivities of the GVP mixes were measured as per IS: 3346-1980 [41].
- 150 Finally, the thermal conductivity values of the GVP mixes were determined using the equation given below-

151 
$$\kappa = \frac{V \times I \times d}{[2 \times A(T_H - T_C)]}$$
(8)

where,  $\kappa$ , d, V, I,  $T_H$  and  $T_C$  are the thermal conductivity (in W/m.K), thickness of specimen (in mm), voltage (in V), current (in A), temperature of hot-plate and cold-plate (in *K*), respectively.

#### 154 2.6 Acoustic Properties

The acoustic performance of samples was measured on a Holmarc (ITA219) impedance tube system consisting of an impedance tube, two microphone locations, and a digital frequency analysis. The acoustic transfer matrix is calculated from the pressure and particle velocity, or the acoustic impedance of the traveling waves on either side of the specimen. Transmission loss (*TLN*) and sound absorption coefficient ( $\alpha_n$ ) are extracted from this transfer matrix using the following equations-

$$TLN = 10 \log_{10} \frac{W_i}{W_t}$$
(9)

 $\alpha_n = 1 - |\Gamma|^2 \tag{10}$ 

162 where,  $W_i$  and  $W_t$  are the power of incident and transmitted sound.  $\alpha_n$ , *TLN* and  $\Gamma$  are the sound absorption coefficient, 163 transmission loss and pressure reflection coefficient, respectively.

164

## 165 3. Results & Discussion

#### 166 **3.1 Mix proportions**

167 The pre-treated FGD gypsum was used to generate calcium sulfate hemihydrate ( $\beta$ -hemihydrate), which is later 168 utilized to develop hemihydrate plaster and gypsum vermiculite plaster (GVP) as per standard methods.  $\beta$ -hemihydrate 169 was obtained by firing the FGDG at 150 - 155 °C for 3 - 4 hours with intermittent spatulation every 45 minutes to 170 obtain a uniform product. The XRD data of β-hemihydrate obtained after calcination is shown in Fig. 1a. It depicts 171 that after calcination, the major content of dihydrate gypsum gets converted to hemihydrate with major crystalline 172 peaks observed at 2θ values 14.74°, 25.66°, 29.71°, 31.87°, 42.23°, 49.31° and 54.11° are indexed for β-hemihydrate 173 plaster. XRD analysis reveals that  $\beta$ -hemihydrate content is about 85-88 % that matches well with similar findings 174 observed during chemical analysis. Unreacted gypsum and anhydrite form accounts for 4 - 6% and 5 - 8%, 175 respectively, with some traces of calcium oxide in the  $\beta$ -hemihydrate sample.

176TG-DTG curve of β-hemihydrate plaster is shown in Fig. 1b, which exhibits a small (1.2%) mass loss at 86 °C177owing to removal of free water and an endotherm ~ 125 °C that accounts for 5.0% mass loss. This clearly indicates178that more than 90% of the FGD gypsum gets converted to hemihydrate form after calcination. The CaSO<sub>4</sub>.2H<sub>2</sub>O179content was determined by the chemical analysis as per IS:2547-Part 2: 1976. However, this 5.0% loss is due to the180inversion of remaining fully or partially dihydrated gypsum to hemihydrate plaster.

181 The gypsum vermiculite plaster (GVP) was developed from  $\beta$ -hemihydrate and vermiculite in different 182 compositions along with a suitable retarder (DTPA) to increase the setting time as per BIS 2542:1978. For mix 183 compositions, blending was carried out in a ribbon powder mixture for a desired interval of time to form a 184 homogeneous mixture. Several mix compositions of gypsum vermiculite plaster with varying percentages of 185 vermiculite (1.0 – 12.0%) were prepared. The composition of these trial mixtures is given in Table 2.

**3.2 Physical and Chemical Properties of GVP** 

187 The 25 mm cubes of all GVP mixes were characterized for their chemical and physical properties as per IS:
188 2542-1978 [37]. The physical characteristics of the samples are given in Table 3 and 4.

#### 189 3.3 Compressive and Flexural Strength

190 Compressive strength results of all the samples determined at 7 and 28 days of hydration are presented in Fig. 2. 191 It is observed that the compressive strength of all the samples increases with hydration period due to the completion 192 of the hydration reaction. However, on replacement of FGD gypsum with vermiculite, compressive strength reduces 193 gradually as we increase the percentage of vermiculite. GVP3 exhibits compressive strength of 12.6 MPa followed by 194 GVP4 > GVP5 > GVP 8 > GVP10 > GVP12 in order after 28 days. This is known to be attributed to lower gypsum 195 content and porous nature of vermiculite which results in poor compressive strength. Although the mixes with GVP0, 196 GVP1, and GVP2 showed the highest compressive strength, they failed to meet the minimum density requirement of 197 at least 750kg/m3 according to BIS: 2547-Part 2: 1976 for plaster. Flexural strength results of the samples show a 198 similar trend as observed for compressive test results. Flexural strengths of the GVP mixes are shown as a bar diagram

**199** in Fig. 3.

#### 200 3.4 SEM & XRD Analysis

SEM images of 28 days hydrated GVP3 plaster were captured and shown in Fig. 5. A perusal of SEM micrographs shows intermingled vermiculite and gypsum particles that indicate an excellent interlocking of gypsum plaster crystals with vermiculite granules. Due to this agglomeration, there observed a high bonding of the plaster, which ultimately results in higher mechanical strength of GVP3 plaster.

XRD of hydrated gypsum vermiculite plaster is shown in Fig. 5a, which depicts major peaks of calcium sulfate
 dihydrate (G) at 20 of 11.69°, 20.78°, 23.45°, 29.17°, 33.42° etc. This shows that hemihydrate plaster gets converted
 into dihydrate gypsum after hydration reactions, responsible for binding properties. Peaks of Calcium Magnesium
 Aluminum Silicate (#) are observed at 29.01° and 31.23° along with peaks of Calcium Aluminum Silicate (\*) peaks at
 9.44° present in vermiculite.

TG-DTG curves of GVP-3 plaster are shown in Fig. 5b. There observed two prominent weight loss signatures from the analysis of DTG curve. Major endothermic peak observed at 110 - 140 °C accounting for ~ 18% weight loss corresponds to FGD gypsum due to dehydration of 3/2 moles of water. The second endotherm observed between 500 - 900 °C corresponds to weight loss attributable to vermiculite due to dehydration of chemically bound water molecules.

The distribution of vermiculite particle in 28 days hydrated GVP3 and GVP 12 at different scale and magnifications are shown in Fig. 4. A perusal of SEM micrographs shows euhedral prismatic, tabular and twined shaped gypsum particle along with flaky lamellar structures of vermiculites. It was also noticed that vermiculite 218 particles are intermingled with gypsum particles that indicate an excellent interlocking of gypsum crystals with 219 vermiculite granules. In GVP3, a high bonding of the plaster with vermiculite is observed due to presence of higher 220 amount of gypsum, which ultimately results in higher mechanical strength of GVP3 plaster.

## 221 **3.5** Determination of Water Absorption and Porosity of GVP

222 The calculated values of water absorption and porosity of the gypsum vermiculite plasters at 2, 8 and 24 h are 223 shown in Table 5. The results revealed that as the percentage of vermiculite increased from 1 to 12% in the plaster, 224 water absorption and porosity increased while the compressive strength and dry bulk density (powder) decreased. The 225 above observations are in good agreement with Martias et al. and Gencel et al. [43,44]. GVP3 mix shows the highest 226 strength and dry bulk density of 12.60 MPa and 790-810 kg/m<sup>3</sup>, respectively, after 28 days of curing. Water absorption 227 of 32.5 - 48.6% is observed for GVP mixes after 24 h of immersion, with GVP3 showing the lowest water absorption 228 in comparison to other mixes. GVP12 shows an almost two-fold increase in water absorption in contrast to GVP3. 229 Like water absorption, the porosity of GVP mixes shows an upward trend with a higher amount of vermiculite in the 230 plasters. This gradual rise in the water absorption and porosity of plasters with addition of vermiculite is attributed to 231 the layered porous structure that entraps water molecules in the cavities [45]. The higher water absorption and porosity 232 limit the applicability of GVP only to interior building proposes. Therefore, we recommend the GVP3 mix for further 233 studies and the feasibility of commercial production.

## 234 3.6 Durability Studies of Gypsum Vermiculite Plaster

Different effects of environmental conditions on the plaster were investigated by durability studies. Winter and summer like weather conditions created in the laboratory and performance of the GVP samples was studied by way of compressive strength. For this hydrated sample of the plaster was exposed to extreme cold, heat & rain/humidity (rain) and compressive strength was determined.

239 3.6.1 Effect of Cold on the Performance of Gypsum Vermiculite Plaster

240 The similar weather conditions as generally observed during winters in Roorkee (29°51' N; 77°53' E) were 241 achieved in the laboratory with the help of an environmental chamber. Atmospheric temperature in the day and night 242 in the January-March varies from 15 - 27 °C and 3 - 12 °C, respectively, with the low humidity of 70 - 75%. In order 243 to replicate the above conditions, the environmental chamber was maintained at a temperature of 18 - 20 °C during 244 the day and 3-5 °C in the night at a humidity of 70-75%, and the mechanical strength of the GVP3 was determined 245 after 7 days and 28 days of treatment. The compressive strength of GVP3 at 7 and 28 days is represented in Table 6. 246 Interpretation of results revealed a decrease in compressive strength (about 10 - 11%) of the plaster compared to the 247 strength attained by the plaster treated at 40 °C for the same period. A progressive increase in the strength development 248 of the plaster was noticed with an increase in the curing period without any variation in the weight of the plaster cubes

249 during the exposure test.

## 250 3.6.2 Effect of Heat Exposure at High Temperatures

In order to contemplate the effect of exposure of hydrated samples of GVP3 to high temperatures, 2.5 cm size were placed in two different ovens maintained at 50 °C and 60 °C under 90% humidity. The compressive strength values of the cubes were determined after 7 and 28 days of treatment in the respective ovens and results are shown in

Table 7. The perusal of results demonstrates a small increase in compressive strength of binders with an increase in

curing temperature. Around 5% and 7% gain in compressive strength was observed at 50 °C and 60 °C, respectively,

in contrast to the strength attained in hardening the plaster at 40 °C for the same period of time. The water retentivity

257 of the cubes decreases due to the evaporation of the water at higher temperatures, hence it affects the strength of the

258 cubes.

#### 259 3.7 Sound Absorption

The normal incidence sound absorption coefficient *vs.* frequency of the GVP3 plaster is shown in Fig. 6a. In the 100 – 1500 Hz frequency range, sound absorption initially increases to a maximum of 0.65 at 400 Hz and then decreases to 0.06 around 1300 Hz. In the higher frequency range 1500 - 4000 Hz, it shows a peak maxima (0.48) at 2500 Hz. However, the transmission loss *vs.* frequency plot (Fig. 6b) exhibits a zig-zag graph within the range of 30 – 45 dB, depicting the highest loss upto ~ 45 dB at 1600 Hz. These results suggest good insulation performance of gypsum vermiculite plaster, GVP3. It may be due to the porous structure of plaster containing a large number of interpenetrating interlinked pores that facilitates sound absorption and leads to noise reduction [42].

#### 267 3.8 Efflorescence Studies

268 Efflorescence generally referred to the white powder generated at the surface of porous building materials [46]. 269 It occurs due to the reaction of CO<sub>2</sub> in the atmosphere with hydroxide ions (OH<sup>-</sup>) that evaporate to the surface from 270 the core of the binder (Eqn. 8) and form carbonate salts with alkali metal ions [47]. The level of efflorescence in the 271 lightweight gypsum vermiculite plasters was measured by mixing 0.1% iron oxide pigment in the plaster mixes. The 272 pigment imparts red color to the plasters in order to clearly distinguish white patches of efflorescence developed on 273 the surface. Circular discs of diameter 10 cm and thickness 0.5 cm were casted and cured at 40 °C for a period of 28 274 days. The specimen discs were placed in a tray filled with water up to a height of 2.5 cm height and the water was 275 allowed to rise in the specimens, followed by drying at an ambient temperature. The test cycles were repeated for a 276 period of 3, 7 and 28 days and examined for any efflorescence produced in the same period. No efflorescence was 277 observed up to a period of 7 days. However, on the 28th day, slight efflorescence was observed on the edges of the 278 specimens.

## 279 3.9 Fire Evaluation and Thermal Conductivity Studies

280 Effect of fire on the performance of gypsum vermiculite plaster (GVP3) specimen was studied as per BS 476-281 1997 (Part 7) [48]. This test evaluates the durability of the material in case of any accident of fire. Lateral spread of 282 flame along the surface of a specimen of a product orientated in the vertical position under opposed flow conditions 283 is measured, and materials are classified based on the rate and extent of the spread of flame. Specimens of size 900 284 mm x 270 mm x 07 mm were developed and the extent of flame spread after 1.5 min and after 10 min was determined. 285 The fire test results reveal that the GVP3 plaster is resistant to any susceptible fire accident as no spread of flames was 286 observed without any gas emission during the test. Based on the above findings, gypsum vermiculite plasters are 287 classified as Class I materials. Moreover, no loss of mechanical stability or any deformation was observed in the 288 specimen panel throughout the experiment.

Thermal conductivity gives an idea about the suitability of building materials for wall insulation applications and is one of the most important parameter for insulating materials to be energy efficient. Thermal conductivities measured for gypsum vermiculite plasters vary from 0.140 - 0.161 W/mK. GVP12 mix demonstrates the lowest conductivity of 292 0.140 W/mK and it increases as we decrease the amount of vermiculite in the mix, with GVP3 showing thermal 293 conductivity of 0.161 W/mK. The thermal conductivity of gypsum vermiculite plasters exhibits good insulating 294 properties with reference to some commercially available building materials such as brick tile, burnt clay brick, cement 295 plaster and mortar [48]. The downfall trend of thermal conductivity in lightweight plasters is chiefly attributed to the 296 lamellar structure of vermiculite that entraps a large volume of air, which reduces the thermal conductivity [49]. Larger 297 the content of vermiculite in the mix, higher will be the porosity of the mix, which facilitates a larger volume of air 298 that serves as an excellent insulator and leads to lower thermal conductivity values. Therefore, lightweight GVP are 299 suitable materials for internal wall insulation with their substantial compressive strengths.

300

**301 4. COST ESTIMATION** 

4.1 Cost of hemihydrate plaster using FGD gypsum	
Cost of raw FGD gypsum per 1.33 tonne	=₹1330/- (\$16.41)
Cost of drying, grinding and calcination of	= ₹2000/- (\$24.68)
FGD gypsum per tonne	
1.33 tonne of FGD gypsum gives	= 1.0 Tonne of POP
Cost of retarder @₹400/kg	= ₹200 (0.50 kg/tonne) (\$2.
Packaging per tonne @ 25bags of 40 kg	= ₹300/- (\$3.70)
Cost of hemihydrate plaster per tonne	Total = ₹3930/- (\$48.49)
	=₹5000/- (\$61.69)
DSR rates (CPWD) of POP per tonne	- (3000/- (401.09)
4.2 Cost of Gypsum Vermiculite Plaster (one tonne)	- (3000/- (301.09)
	= ₹3424/- (97 %) (\$42.25)
4.2 <u>Cost of Gypsum Vermiculite Plaster (one tonne)</u>	
<ul> <li>4.2 Cost of Gypsum Vermiculite Plaster (one tonne)</li> <li>Cost of hemihydrate plaster @ ₹3530/-</li> </ul>	= ₹3424/- (97 %) (\$42.25)
<ul> <li>4.2 Cost of Gypsum Vermiculite Plaster (one tonne)</li> <li>Cost of hemihydrate plaster @ ₹3530/-</li> <li>Cost of Vermiculite per tonne @ 10000</li> <li>Blending &amp; Packaging per tonne @ 25 bags</li> </ul>	= ₹3424/- (97 %) (\$42.25) = ₹300/- (3 %) (\$3.70)

The cost of the developed composite binder has been determined as per DSR as shown above. The rates gypsum
Vermiculite Plaster are less (₹4124/Tonne) as compared to the DSR rate (₹6000/Tonne). It is proved that the Gypsum
Vermiculite Plaster is also economically feasible construction material.

325

326 5. LIMITATION AND FUTURE SCOPE

327 Limitation: The water absorption of FGD gypsum is a little higher. Therefore, it cannot be used for external surfaces

328 and that is why it is recommended for internal applications.

329 Future Scope:

- 330 1. More and more utilization of by-product FGD gypsum, a waste generated from thermal power plants during the
- **331** desulphurization process, by developing a vermiculite-based composite binder.

332 2. Further, the aim of the study is to develop a lightweight low density interior textured plaster using vermiculite in333 which paint is not required and no curing is needed.

#### **6.** Conclusion

335 The gypsum vermiculite plaster developed using waste gypsum shows good applicability for internal purpose. Several 336 mixtures were tried with different replacements of gypsum with vermiculite (3, 4, 5, 8, 10 & 12%). These plasters 337 show improved coverage and adhesion with dry weight less than half that of traditional sanded plaster. XRD analysis 338 confirms > 90% purity of FGD gypsum, with gypsum being the major phase in the pattern. SEM depicts the 339 agglomeration of vermiculite particles with FGD gypsum particles for strength generation. The strength of the GVP 340 was measured to be 5.5 - 12.6 MPa after 28 days of hydration, with GVP3 being the toughest. Gypsum vermiculite 341 plasters show high water absorption (32.5 - 48.6%) at 24 h of immersion) and porosity due to the layered porous 342 structure of vermiculite present. GVP3 mix was selected as optimum composition as it exhibits the highest mechanical 343 strength and lowest water absorption. The performance of GVP3 under extreme cold and hold weather resulted in 344 good durability for the light weight plaster. These plasters provide reverberation (echo) control by absorbing sound 345 radiations with highest sound absorption coefficient observed of 0.65. There were no signs of efflorescence observed 346 in the plasters after 7 days. These materials were classified as Class I materials as no lateral spread of flame observed 347 nor any signs of deformation or gas emission. Thermal conductivities 0.140 - 0.161 W/mK were measured for GVP 348 that indicate towards excellent insulation behaviour of plasters. An economical and efficient way of waste mitigation 349 was devised to develop light weight plaster materials for interior applications exhibiting thermal as well as sound 350 insulation. This work can further be extended to reduce water absorption for exterior applications of these plasters. 351 **Supplementary Information** 

- 352 The online version contains supplementary material available at https://doi.org/xxxx.
- 353 Acknowledgements
- We are very thankful to the Director, CBRI Roorkee, for granting permission to publish this work.
- 355 Data Availability
- 356 The authors confirm that the data supporting the findings of this study are available within the article and its
- 357 supplementary materials.

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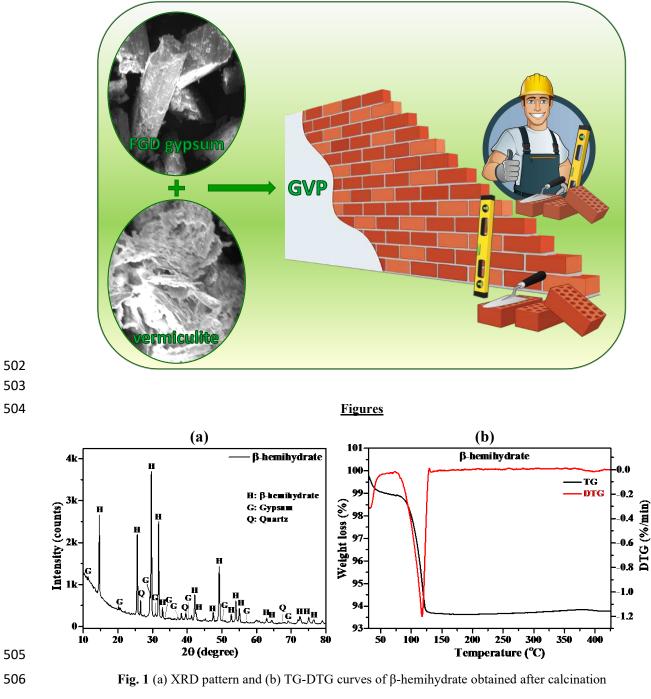
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## Graphical Abstract



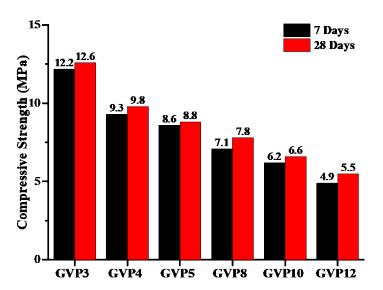
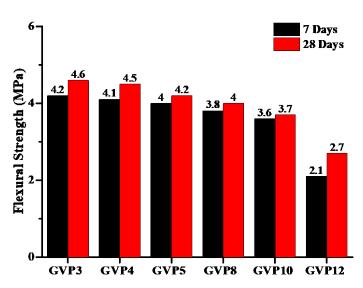






Fig. 2 Compressive strength results of GVP mixes at different hydration periods.





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Fig. 3 Flexural strength results of GVP mixes at 7 days and 28 days of hydration.

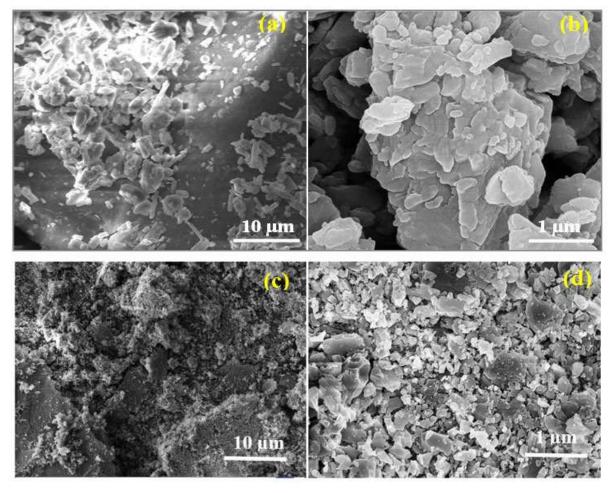
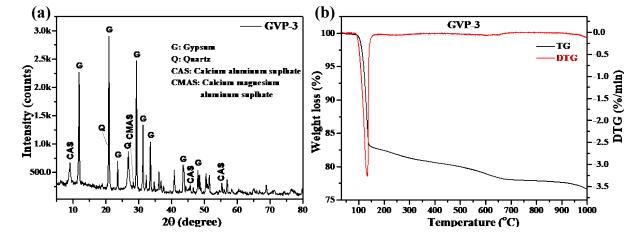
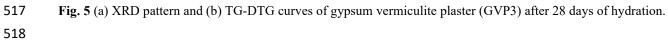




Fig. 4 SEM images of hydrated gypsum vermiculite plaster after 28 days of hydration GVP3 (a,b) and GVP12 (c,d)
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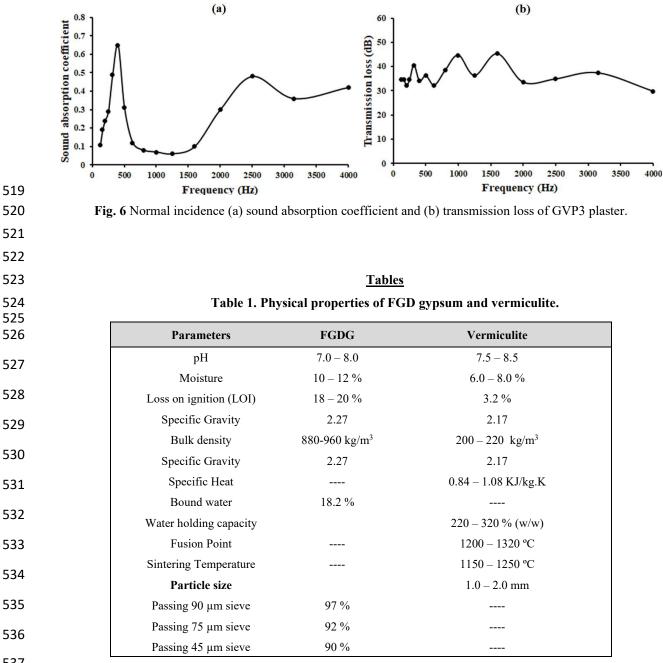


Table 2. Mix compositions for development of Gypsum Vermiculite Plaster (GVP)

Mix Designations	β-hemihydrate plaster (%)	Vermiculite (%)	Retarder (DTPA) (%)
GVP3	97.0	3.0	0.05
GVP4	96.0	4.0	0.05
GVP5	95.0	5.0	0.05
GVP8	92.0	8.0	0.05
GVP10	90.0	10.0	0.05
GVP12	88.0	12.0	0.05

540		

 Table 3. Physical properties of gypsum vermiculite plaster (GVP)

Parameters	Values					
1 al alletel s	GVP3	GVP4	GVP5	GVP8	GVP10	GVP12
pH	7.40	7.38	7.35	7.30	7.28	7.26
Loss on ignition (LOI)	8.80 %	8.90 %	9.0 %	8.70 %	8.60 %	8.50 %
Dry Bulk Density (powder)	$\begin{array}{c} 790-810\\ kg/m^3 \end{array}$	$\begin{array}{c} 770-790\\ kg/m^3 \end{array}$	$\begin{array}{c} 760-780\\ kg/m^3 \end{array}$	$\begin{array}{c} 750-760\\ kg/m^3 \end{array}$	$\begin{array}{c} 690-710\\ kg/m^3 \end{array}$	$\begin{array}{c} 640-660\\ kg/m^3 \end{array}$
Set Bulk density of cube (dried at 42°C)	$\begin{array}{c} 1370-1390\\ kg/m^3 \end{array}$	$\begin{array}{c} 1260-1280\\ kg/m^3 \end{array}$	$\begin{array}{c} 1210-1230\\ kg/m^3 \end{array}$	$\frac{1100-1120}{kg/m^3}$	$\frac{1010-1030}{kg/m^3}$	$\frac{1000-1010}{kg/m^3}$
Specific gravity	2.17	2.12	2.07	1.96	1.86	1.76
Fineness: Retention on 45 µm sieve	8.0-12.0 %	8.0-12.0 %	8.0-12.0 %	10.0 - 15.0 %	10.0 - 15.0 %	10.0 - 15.0 %
Setting time	20 – 25 min	20 – 25 min	20 – 25 min	20 – 25 min	25 – 30 min	30 – 35 min

## Table 4. Water absorption and porosity of gypsum vermiculite plaster (GVP)

Material	Wa	ater absorption (	%)		Porosity (%)	
	2h	8h	24h	2h	8h	24h
GVP3	32.10	32.20	32.50	43.35	43.50	43.85
GVP4	34.60	34.60	35.00	43.20	43.20	43.70
GVP5	36.20	36.50	36.60	43.60	43.95	44.10
GVP8	38.70	38.85	39.20	40.30	40.40	41.20
GVP10	43.80	44.00	44.10	40.90	41.85	41.95
GVP12	47.15	48.20	48.60	42.40	43.40	44.05

## Table 5. Compressive strength of GVP3 under exposed atmosphere during winter

Curing period	Compressive strength (MPa)
7 days	11.00
28 days	11.25

## Table 6. Compressive strength of GVP3 under exposed atmosphere during summer

Curing period	Compressive strength (MPa)			
Curing period	50 °C	60 °C		
7 days	12.75	13.00		
28 days	13.20	13.50		