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Strength and microstructure of mortar with sand substitutes

Authors:

Assoc.Prof. Jeyaprabha Balasubramanian
Fatima Michael College of Engineering and Techn.
Department of Civil Engineering
Tamil Nadu, India
jeyaprabha@gmail.com

Elangovan Gopal, PhD. CE
University College of Engineering
Department of Civil Engineering
Tamil Nadu, India
gelangoki@gmail.com

Assist.Prof. Prakash Periakaruppan, PhD. Chem
Thiagarajar College
Department of Chemistry
Tamil Nadu, India
kmpprakash@gmail.com

Professional paper

Jeyaprabha Balasubramanian, Elangovan Gopal, Prakash Periakaruppan

Strength and microstructure of mortar with sand substitutes

The strength and microstructure of mortars incorporating manufactured sand (MS) and various levels of granite powder (GP) as substitutes for river sand (RS) are considered in the paper. The optimum RS substitution by GP is 15 % by mass for the maximum compressive and splitting tensile strength. Strength properties of mortars containing MS are better when compared to RS and GP mortars, irrespective of the curing period. The thermal analysis, microstructural analysis, and mineralogical analysis are conducted on MS and GP mortars using TGA, SEM, XRD and FT-IR techniques.

Key words:
manufactured sand, granite powder, mortar, microstructure, strength properties

Stručni rad

Jeyaprabha Balasubramanian, Elangovan Gopal, Prakash Periakaruppan

Čvrstoća i mikrostruktura morta sa zamjenskim materijalima umjesto pijeska

U radu se razmatra čvrstoća i mikrostruktura morta u kojem se kao zamjena za riječni pijesak (RS) koriste drobljeni pijesak (MS) i granitni prah (GP) u raznim udjelima. Za postizanje maksimalne tlačne čvrstoće i vlačne čvrstoće pri cijepanju, optimalni težinski udio GP u RS iznosi 15 %. Svojstva čvrstoće morta koji sadrži MS bolja su od odgovarajućih svojstava mortova RS i GP, bez obzira na vrijeme njege. Za mortove MS i GP provedena je toplinska analiza, mikrostrukturna analiza i mineraloška analiza, a pritom su korišteni postupci TGA, SEM, XRD i FT-IR.

Ključne riječi:
drobljeni pijesak, granitni prah, mort, mikrostruktura, svojstva čvrstoće

Fachbericht

Jeyaprabha Balasubramanian, Elangovan Gopal, Prakash Periakaruppan

Festigkeit und Mikrostruktur von Mörteln mit Materialen als Sandersatz

In dieser Arbeit werden Tragfähigkeit und Mikrostruktur von Mörteln, bei dem Brechsand (MS) und Granitstaub (GP) in verschiedenen Anteilen als Ersatz für Flusssand (RS) angewandt werden, untersucht. Um eine maximale Druck- und Zugfestigkeit bei Spaltversuchen zu erzielen, ergibt sich ein optimaler Gewichtsanteil von 15% GP im RS. Festigkeitseigenschaften von Mörteln mit MS sind besser als entsprechende Eigenschaften von RS und GP Mörteln, unabhängig von der Dauer der Festigung. Für MS und GP Mörtel wurden eine Wärmeanalyse, eine Analyse der Mikrostruktur sowie eine mineralogische Analyse durchgeführt. Dabei wurden die Verfahren TGA, SEM, XRD und FTIR angewandt.

Schlüsselwörter:
Brechsand, Granitstaub, Mörtel, Mikrostruktur, Festigkeitseigenschaften

1. Introduction

River sand (RS) is a commodity that is widely used as construction material all over the world, particularly in the production of cement-sand mortar and concrete. However, RS is not a renewable natural resource. In some areas, RS has been exploited quite excessively, which creates environmental problems. On the other hand, RS is expensive due to exorbitant cost of transport from natural sources, while the exhaustion of RS due to quarrying activities has already caused flooding in many parts of the world. Various governments, non-governmental agencies, and research institutes are striving to identify alternative materials that can be used instead of RS. Hence, extensive research is currently made on RS substitutes, which can chiefly be used in construction industry. According to literature data [1-5], substitute materials for RS comprise manufactured sand (MS), industrial by-products (some forms of slag, bottom ash), recycled aggregates, etc. One of these materials, MS, is receiving considerable attention these days as a replacement for RS [1, 2].

In India, about 6 million tonnes of waste from marble and granite industries are released annually from granite cutting, polishing, processing and grinding [6]. During sawing and polishing of marble and granite rock blocks, a considerable quantity of wastes is produced, mostly composed of SiO_2, Al_2O_3, Fe_2O_3, and CaO [7, 8]. The use of granite sawing powder waste in the production of bricks and tiles is becoming common practice, and this material has been applied successfully in many countries [9 -12].

The marble and granite powder (GP) waste is used as coarse aggregate and fine aggregate to produce various grades of concrete mixes [13-17]. The effect of marble dust on properties of conventional concrete and self compacting concrete has been well documented [18-21]. The effect of using granite as a partial cement replacement on mechanical properties of concrete has been studied by Felixkala [22].

MS particles are generally more angular and have rougher surface texture than RS particles. The shape and texture of MS can lead to development in the strength of concrete due to better interlocking between particles. The microstructure of a material is one of the main links between the process and its final properties. The so called alternatives for RS viz. MS and GP should not escape this rule. Although many investigations have been made on cement substitutes, no work has so far been undertaken to study the microstructure and strength properties of these materials as RS substitutes, which is a constant challenge to researchers, mainly because of the complex and heterogeneous mineralogy. The quality of these alternatives is dependent on the % of crystallinity and other structural properties, which need to be investigated quite thoroughly.

Recognizing the need for an extensive research on RS alternatives, with the focus on strength properties and micro structure, the authors of the study investigate the use of MS and GP as substitute materials for RS in mortar production. Thermal analysis, micro structural analysis, and mineralogical analysis, have been carried out on the cement, MS and GP mortars using TGA, SEM, XRD and FT-IR techniques.

2. Materials

2.1. Cement

The Ordinary Portland Cement (OPC) was used in this study. The main properties are listed in Table 1.

Table 1. Properties of OPC

Br.	Description	Test results	Requirements IS: 12269-2013
A)	**Chemical requirements**		
1.	$CaO-0.70SO_3$ / $(2.8SiO_2+1.2Al_2O_3+0.65Fe_2O_3)$	0.88	0.8 - 1.02
2.	Al_2O_3 / Fe_2O_3	1.21	0.66 min.
3.	Insoluble residue [%]	1.15	4.00 max.
4.	Magnesia [%]	1.01	6.00 max.
5.	Sulphuric anhydride [%]	2.46	3.50 max.
6.	Loss on ignition [%]	2.96	4.00 max.
7.	Chlorides [%]	0.005	0.10 max.
B)	**Physical requirements**		
1.	Fineness [M^2/kg]	310.4	225 min.
2.	Normal consistency [%]	28.5	
3.	Setting time [in min]		
	Initial	180	30 min.
	Final	280	600 max.
4.	Soundness		
	Le-Chat. Expansion [MM]	1.00	10.00 max.
	Auto clave [%]	0.02	0.80 max.
5.	Compressive strength [MPa]		
	3 days ± 1 hour	37.3	27 min.
	7 days ± 2 hours	42.7	37 min.
	28 days ± 4 hours	57.0	53 min.

2.2. River sand

RS passing through 4.75 mm sieve and having a specific gravity of 2.54 was used in this study. The grain size distribution analysis was carried out on fine aggregate according to IS 383 / 1970 [23], as presented in Figure 1. The water absorption of river sand amounts to 0.032 %. The silt content in river sand is 2 %.

2.3. Manufactured sand

MS passing through 4.75 mm sieve and having a specific gravity of 2.57 was used in this study. It has a wide range of particles, as shown in Figure 1. Water absorption of MS is 0.032 %, and it contains 2 % of silt. The rock used in this study to produce the manufactured fine aggregate is petrologically classified as granulite and, according to its mineralogical composition, it consists of alkaline feldspars (15 %), plagioclase (12 %), quartz (42 %), hyperstene (15 %), and hornblende (12 %), with a small quantity of biotite (4 %). The rock structure is categorized as isotropic with no foliations, and its degree of alteration is very limited. It is, however, characterized by the presence of internal fractures that are within the scale of individual mineral grains.

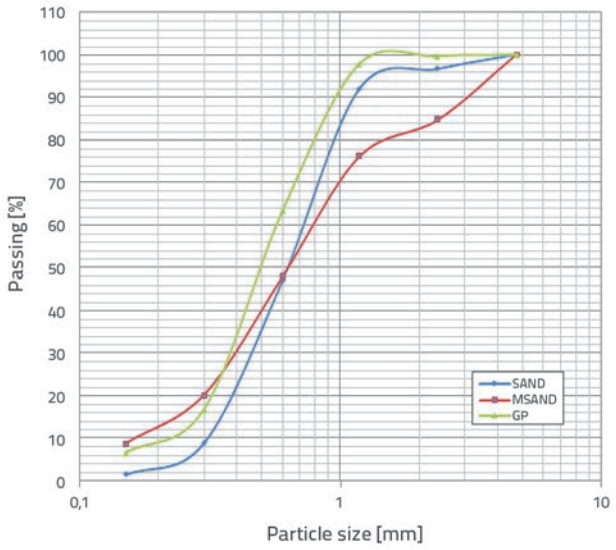

Figure 1. Particle size distribution curve

2.4. Granite powder waste

GP from a granite preparation plant was used throughout the research work. The GP was collected and delivered in wet condition. It was oven-dried prior to use in order to control the mixing water content. Appropriate tests were carried out to confirm the physical and chemical description of the GP. Thus the specific gravity and specific surface area of GP amounted to about 2.19 and 333 m^2/kg, respectively. The grain size distribution analysis was carried out for the GP to validate its physical characterization. It was observed that 63 % of GP particles were less than 600 μm in size, while 17 % of particles were less than 150 μm in size.

Figure 2. shows the XRD pattern of GP. Quartz (SiO_2), sodium feldspar ($NaAlSi_3O_8$), and mica, were present as major minerals, while the potassium feldspar ($KAlSi_3O_8$), biotite and calcite ($CaCO_3$) were present as minor minerals. Despite the high iron content, no peaks for iron, ferrous hydroxide, and ferric oxide were detected. This may be due to their very poor crystallinity [24- 29]. The main constituent, quartz, is present in the (0 1 1) crystal plane (JCPDS: #87-2096).

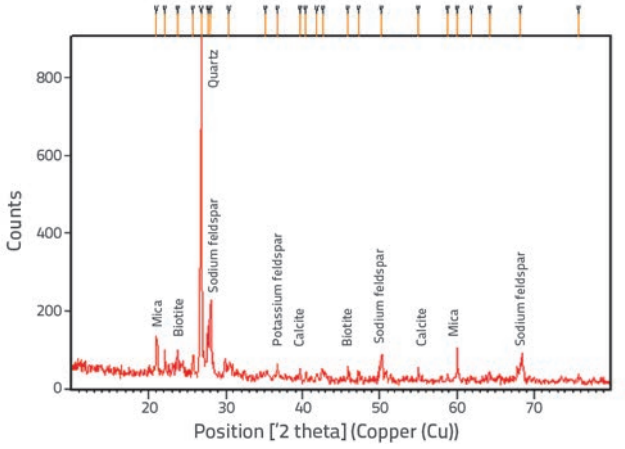

Figure 2. XRD pattern of GP

3. Methods

3.1. Mixture proportions

Mortar mixtures containing 100 % MS and different percentages of GP (5, 10, 15, 20 and 25 %) were cast in the laboratory as RS alternatives. In this experimental study, mortars were produced

Table 2. Mixture proportions of mortar specimens

Specimen	Replacement for sand [%]	Cement	Sand	Water	Granite dust	M Sand	w/c	Flow [%]	Packing density
		Weight in gram							
CM	0	450.0	1350.0	225.0	0.0	0.0	0.5	105.8	0.71
G5M	5	450.0	1282.5	225.0	67.5	0.0	0.5	101.4	0.68
G10M	10	450.0	1215.0	225.0	135.0	0.0	0.5	96.2	0.67
G15M	15	450.0	1147.5	225.0	202.5	0.0	0.5	95.8	0.65
G20M	20	450.0	1080.0	225.0	270.0	0.0	0.5	93.7	0.63
G25M	25	450.0	1012.5	225.0	337.5	0.0	0.5	91.0	0.62
MSM	100	450.0	0.0	225.0	0.0	1350.0	0.5	99.4	0.72

with cement, RS, MS, and GP at various proportions. Seven different mixtures were designated for mortar specimens. The water content of each mixture was kept constant. Mixture proportions of mortar specimens are given in Table 2. The compressive and splitting tensile strength of the mortar was measured on 70.6 mm x 70.6 mm x 70.6 mm cubes and 50 mm x 100 mm cylinders, respectively. All cubes and cylinders were cast in three layers, and each layer was fully compacted using the mortar cube vibrator. After casting, specimens were kept at room temperature for 24 h. Then they were demoulded and transferred to the curing tank where they were stored until their testing dates. The compressive strength and splitting tensile strength were measured at the age of 3, 7, 14, and 28 days using the universal testing machine (UTM) with the capacity of 400 kN. For each mixture, six specimens were tested according to a relevant IS standard.

3.2. Micro structural analysis

The SEM, XRD, FT-IR and TGA examinations were performed to determine the micro structure of the specimens. These tests were performed with specimens after 28 days of curing.

3.2.1. Scanning Electron Microscopy (SEM) analysis

The powdered mortar specimen samples with RS, MS and GP were suspended in a two-part Epotek 301 epoxy to stabilize them for SEM use. Firstly, the epoxy was mixed according to the manufacturer's directions. The powdered samples were then stirred into the mixture by adding small amounts at a time so as to ensure that particles are evenly coated with epoxy. The mixture was then poured into a one inch diameter sample cup, and put under vacuum to remove air bubbles. The samples were then cured at room temperature for a minimum of 24 hours under normal pressure. Next, the samples were removed from sample holders and polished, first with 120 grit sandpaper, and then progressively with higher grit sandpapers until the 1200 grit size was reached. Further polishing continued with the 6 m, 3 m, 1 m and 0.25 m diamond paste. Later on the samples were coated with gold palladium in order to provide a conductive surface for the SEM. The images of mortar samples with RS, MS and GP were taken at various magnifications to identify the shape and texture of the particles.
The size and morphology of the specimens were examined using the SEM model on a JEOL JSM 6390 instrument.

3.2.2. X-Ray Diffraction (XRD) analysis

A small amount of powder sample is put into an aluminium sample holder, and the surface is finished smoothly. The holder is then placed into the X-Ray diffractometer. The samples are scanned by an X-Ray diffractometer using CuK radiation at 40 kV / 20 mA, CPS = 1k, width 2.5, speed 2° / min and scanned with an angle of 2 from 3 - 70°. The analysis is stepped at 0.04 degree increments and continued for a period of 3 seconds. In X-ray diffraction, X-rays are scattered by atoms in a pattern that indicates lattice spacing of elements present in the material analysed. Once the X-ray analysis is completed, the scans are analysed using Jade 7 - X-Ray Diffraction (XRD) software. The JCPDS is used to compare peak intensities at different angles with a database of different minerals and compounds. Compounds with maximum peak intensities matching those of the 2e values are identified and the compounds present in the samples are also determined. The x-ray diffraction (XRD) spectra are recorded and analysed for the specimens with the X-ray diffraction analysis using the XPERT-PRO PW3050/60 diffractometer. The XRD was carried out on cement mortar specimens with the 100 % and 15.0 % manufactured sand and granite dust, respectively, as river sand replacements using 0.5 w/c ratio.

3.2.3. FT-IR analysis

The FT-IR Spectroscopy is a technique based on the interaction between an IR radiation and a sample that can be either solid, liquid, or gaseous. It measures the frequencies at which the sample absorbs radiation, and also the intensities of these absorptions. The frequencies are helpful for the identification of the sample's chemical make-up due to the fact that chemical functional groups are responsible for the absorption of radiation at different frequencies. The concentration of a component can be determined based on the absorption intensity. FT-IR spectra were recorded using a SHIMADZU-FT-IR-8400S spectrophotometer. The spectra for the mortar specimens were recorded by grinding the specimens to powder, mixing the powder with a small amount of KBr powder, and compacting the mixture into a disk.

3.2.4. Thermogravimetric analysis

The thermogravimetric analysis (TGA) was conducted mainly to study the effect of the presence of the manufactured sand and granite dust on the degree of hydration of cement, which is a function of calcium hydroxide content. TGA was performed on samples subjected to temperatures of up to 1832°F (1000°C), with a heating rate of 18°F/min (10°C/min) under helium atmosphere.

4. Results and discussion

4.1. Compressive strength

Development of compressive strength at the age of 3, 7, 14 & 28 days is shown in Figure 3. This figure in fact shows the effect of MS and GP as RS replacements on mortar compressive strength after 3, 7, 14 and 28 days of curing. From this figure, it is evident that all specimens containing MS and GP show an appreciable increase in compressive strength with an increase

in curing periods, compared to cement mortar. The compressive strength of specimens made with GP increases up to G15, and decreases from G20 and G25. The enhancement of the compressive strength of G15 compared to cement mortar for the curing period of 3, 7, 14 and 28 days is 48 %, 57 %, 61 % and 43 %, respectively. The development of compressive strength at 15 % GP waste as RS replacement may be due to the filling effect as a result of using a high-fineness granite dust. On the other hand, the reduction in compressive strength as a result of using 20 % granite dust and more as sand replacement may be due to the reduction in workability. Here the decreased workability is attributed to an increased specific surface area and specific density of the GP waste, which create a demand for cement paste volume resulting in poor compactness. This in turn results in the reduction of compressive strength [30].

The compressive strength of mortars made with MS is found to be greater than that of cement mortars (53 %) and GP mortars (7.14 %). The development of compressive strength of MS mortars compared to cement mortar for the curing period of 3, 7, 14 and 28 days is 48 %, 69 %, 64 % and 53 %, respectively. Surface texture of MS particles has a significant effect on concrete strength, as angular particles have a larger surface area and the rough surfaces enhance the bond between aggregate particles and cement matrix, thus increasing the compressive strength [3].

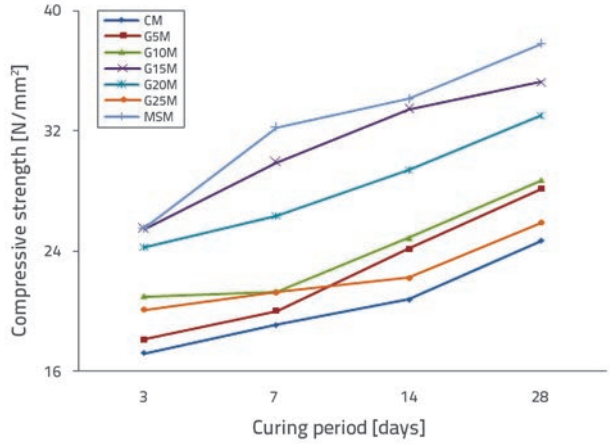

Figure 3 Development of mortar compressive strength

The particle size distribution curve presented in Figure 1 shows that the uniformity coefficient of MS is greater than 6 and that the coefficient of curvature varies between 1 and 3. According to IS: 383/1970 [23], the MS used is well graded and it is under the Zone -II, i.e. fine particles are in proper proportion with fewer voids and better interlocking resulting in higher compressive strength values.

4.2. Splitting tensile strength

The way in which the splitting tensile strength is affected by the use of the MS and GP as RS alternatives, after 28 days of curing, is presented in Figure 4. This figure clearly shows that the use of the MS and GP dust as sand replacements has a positive effect on tensile strength, compared to cement mortar. The maximum tensile strength is achieved with the 15 % GP mortars. MS mortars reveal higher tensile strength values compared to cement and GP mortars. This trend agrees well with the compressive strength test results for mortar modified with the MS and GP dust as RS replacements. The relation between the mortar compressive strength (F_c) and mortar tensile strength (F_t) is given in Figure 5 for the mortar modified with the MS and GP as RS replacements. It can be seen from this figure that the tensile strength of MS and GP mortars amounts to about 12 % and 11-14 % of compressive strength, respectively. Also, it is clear that the values of F_t / F_c are independent from the inclusion of MS and GP as sand alternatives [31].

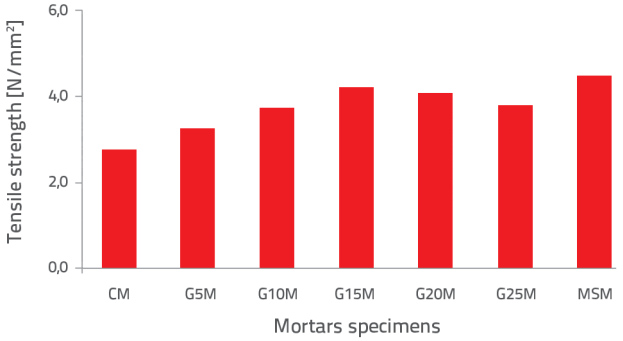

Figure 4. Tensile strength of mortar modified with M sand and GP waste as sand alternatives

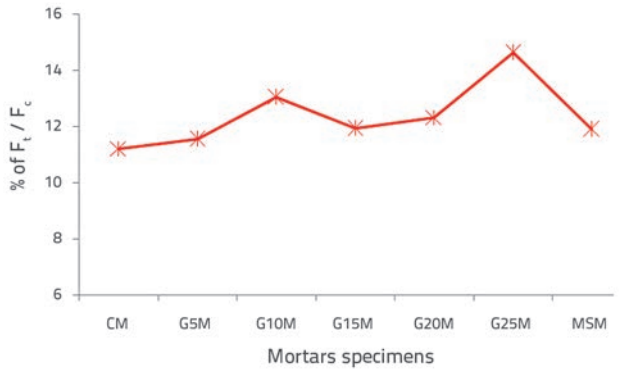

Figure 5. Relation between mortar tensile strength and compressive strength

4.3. Thermogravimetric analysis (TGA)

The TGA is a method for measuring the mass change of a sample as a function of rise in temperature. In cement-based materials, thermal changes result in mass changes, which can be measured by a thermogravimetric analyser [32]. The mass changes imply dehydration of hydration products. This technique is helpful for evaluating changes in the composition of cement-based materials to predict their behaviour upon exposure to fire. Typical plots of TGA curves with respective mass loss for

cement mortars, MS and GP mortars as RS replacements, are shown in Figure 6.

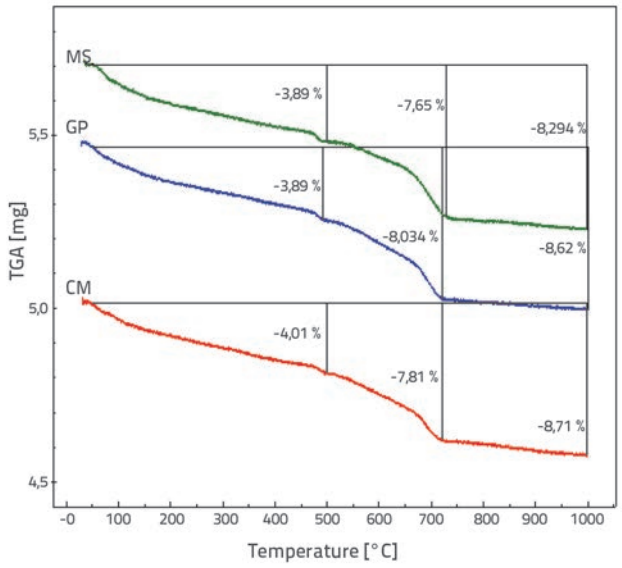

Figure 6. TGA curves of mortars made with CM, GP and MS

TGA curves show that each curve consists of three zones. Zone I covers the range from 100 to 500 ^{0}C and is attributed to dehydration of C-S-H and ettringite. Zone II, ranging from 500 to 700 ^{0}C, is attributed to dehydration of calcium hydroxide. The loss of calcium hydroxide in CM, GP and MS amounts to 3.8 %, 4.14 % and 3.76 %, respectively. An endotherm after 700 ^{0}C indicates the complete decomposition of C-S-H. In all samples (cement mortar, MS mortar or GP mortar samples), the calcium hydroxide peak is at 470-485 ^{0}C, which indicates that there is no change in phase chemistry for different mixtures [31]. However, the variation of calcium hydroxide content between cement mortar, MS mortar and GP modified mortars is insignificant.

4.4. XRD analysis

XRD patterns for the cement mortar, MS mortar and GP mortars, as RS alternatives, are shown in Figure 7. The XRD analysis results are in agreement with thermal analysis, where there is no significant difference in hydration products between all tested mortars. Given the nature of the mortar specimens, with multiple lattice planes at a number of positions, a large amount of sampling "noise" was present due to fine nature of these powder samples. Similar observation was made by De Jong et al. [33]. The spectra were analysed by the search/match function using the JCPDS software. All libraries of the spectra that were available for comparison were considered. Several key components of the powders were identified, as shown in the Figure 7.

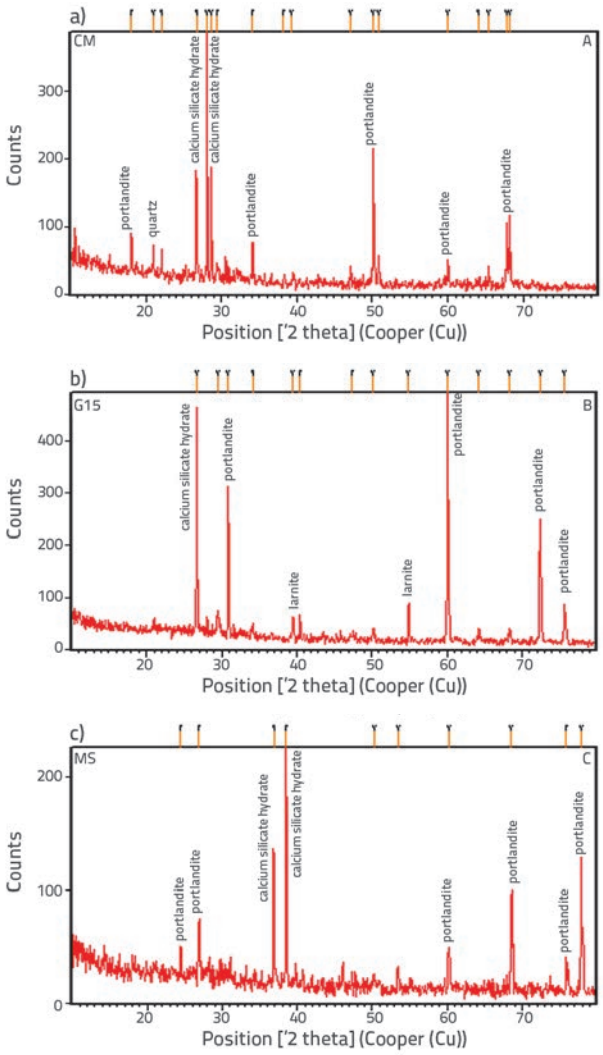

Figure 7. XRD patterns for: a) cement mortar, b) GP mortar, c) MS mortars

4.5. FT-IR analysis

FT-IR spectra (Figure 8) of all samples are almost similar. Major absorption bands in all samples include: 3000 cm^{-1} and 1638 cm^{-1} bands assigned to H-OH stretching and H-O-H bending, respectively (generally known as water bands); 1330 cm^{-1} - 1587 cm^{-1} bands associated with CaO and CaCO3; 1123 cm^{-1} and 926 cm^{-1} bands assigned to Si-O-Si and Si-O-Al bonds, respectively (tricalcium silicate and tricalcium aluminate); and 626 cm^{-1} and 528 cm^{-1} bands assigned to the Si-O bending (dicalcium silicate) [34]. The assigned bands are based on literature data [35]. The FT-IR analysis results are in good agreement with the thermal and XRD analysis, where there is no significant difference in hydration products between all tested mortars. A clearer observation is the high percentage of quartz in mortar modified with GP dust and MS.

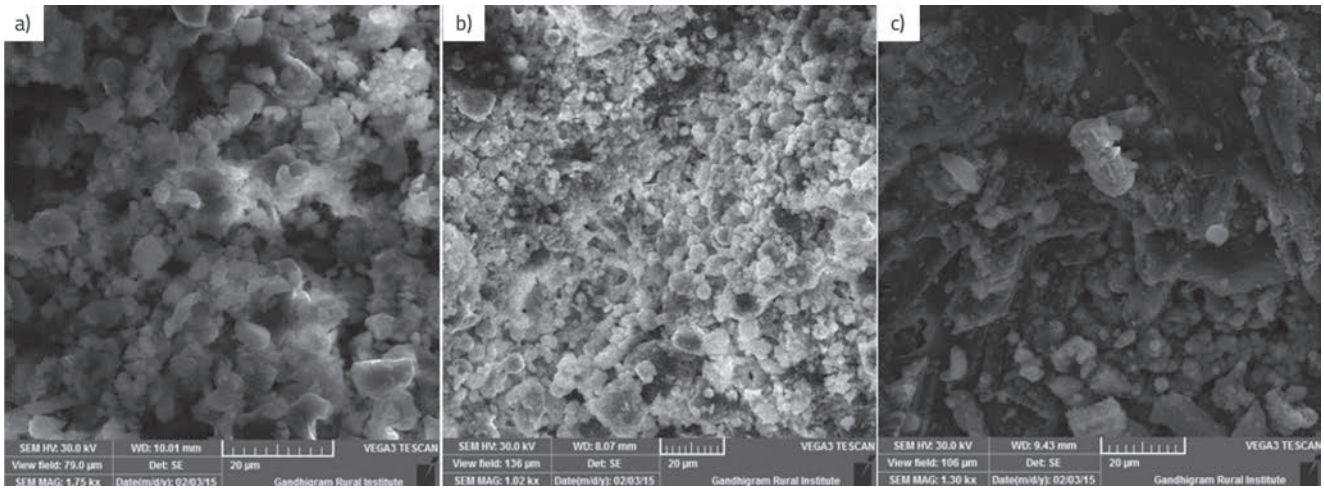

Figure 9. SEM images of a) MS mortar; b) GP mortar, c) CM mortar

4.6. SEM Analysis

The surface texture of MS mortar (Figure 9.a) shows that it has angular particles with a larger rough surface area, which enhances the bond between aggregate particles and cement matrix. It creates better interlocking between the particles and reduces the porosity. Because of this, the strength and durability characteristics are improved [31]. High fineness and spherical shape of GP dust results in good filling effect, clearly visible in Figure 9.b, when compared to control mix (Figure 9.c).

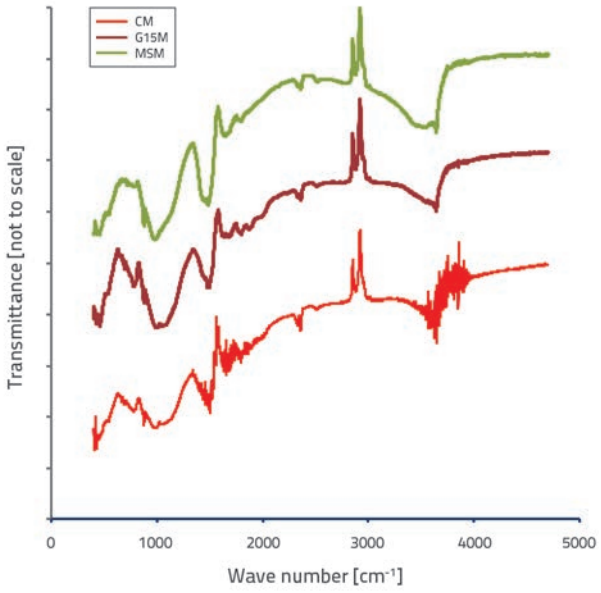

Figure 8. FT-IR spectra of mortar made with cement, GP and MS

5. Conclusion

Based on this experimental study, the following conclusions can be drawn:

- Both the compressive and splitting tensile strength of the mortars containing MS and GP waste as RS alternatives are enhanced compared to cement mortar samples irrespective of the curing period.
- The use of up to 15 % GP waste as RS alternative increases the mortar's compressive and splitting tensile strength. The optimum % of GP waste is 15 %.
- For mortars modified with MS and GP waste, the tensile strength amounts to about 12 % and 11-14 % of mortar compressive strength, respectively.
- The ratio of tensile strength to compressive strength (F_t / F_c) is independent of sand alternatives.
- It is inferred From TGA results that the samples have a stable structural state up to 500 ^{0}C, while the exposure to higher temperatures results in a remarkable decomposition of the hydration products.
- The results of XRD analysis are in agreement with thermal analysis, where there is no significant difference in hydration products between all tested mortars.
- Angular particles with larger rough surface area of MS enhance interlocking between the particles and high fineness and spherical shape of GP dust results in good filling effect, as proven by SEM analysis.
- Due to naturally rough and angular characteristics of MS particles, the MS mortar is superior to GP waste mortars and cement mortars with regard to the compressive and splitting tensile strength.
- There is no change in phase chemistry for mortars modified with MS and GP waste compared to cement mortar where the variation of calcium hydroxide content between cement mortar and modified mortars is insignificant, which is revealed by FT-IR.
- It has been established that the replacement of RS by MS and GP waste (up to 15 %) of any formulation is favourable for mortar making, without adverse effects on the strength criterion.

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