EVALUATION OF MARBLE SLURRY WASTE FOR PREPARATION OF COMMERCIAL GRADE CEMENT

Wisal Shah*, Muhammad Nafees**, Muhammad Iqbal***

ABSTRACT

Marble processing is generating a large amount of slurry waste which poses serious impact on the environment. This study was conducted with the objective to demonstrate the practicability of using marble slurry wastes in the manufacturing of cement on commercial scale. Waste marble slurry was obtained from marble processing units and compares it with the calcium carbonate used in the cement industry by analyzing it for physical and chemical characteristics. Cement was made in the laboratory according to standard formulation. Commercial brand available in the market was used as control for product comparison. Physical and chemical properties of both cements and mechanical properties of their concrete specimens were tested according to American Society for Testing Materials (ASTM) standards. The test results showed that the cement prepared from marble slurry waste have similar properties like commercial brand. It was concluded that marble slurry can be used as a substitute of calcium carbonate raw-material in the manufacturing of commercial cement.

Keywords: *Marble Slurry waste, Cement, Concrete Mixture, Compressive Strength, Alternative Resource.*

INTRODUCTION

Marble is a semi ornamental stone category use for ages in the palaces, monuments and other construction purposes. Marble quarrying and processing produces a large amount of waste in the form of slurry (mixture of water and marble dust) which causing environmental problems[1]. The marble stone processing industry consumes huge amount of fresh water as a results of which large quantity of semi liquid stone slurry is generated. During the cutting and polishing of marble stone, water is showered on gang saws and blades for cooling purpose. As estimated by Pakistan stone development company, the average wastage/loss of marble mineral is 85% during quarrying processing. This means processing of 1 ton irregular shaped marble block results in 850 kg of waste[2]. The wastage is further supplemented when these irregular shaped marble blocks are used for further processing finished goods. During cutting and processing of 1m^3 marble stone into 2cm thick slabs, 25% marble stone is wasted. As a result 250 litter/m^3 marble slurry waste is produced. This proportion increases when the thickness of cutting slabs decreases[3]. Marble slurry waste mainly consists of $CaCO_3$, CaO, MgO, SiO_2, Al_2O_3, K_2O_3 and Na_2O. This marble slurry waste is disposed off in open areas and water bodies which causes serious ecological problems. Besides this, open dumping of marble slurry waste find its way into agricultural field lands and reduce soil fertility and plant growth[4,5]. When the slurry dries, its fine particles causes air pollution which has got adverse impacts on human health[6]. However, this fine particles can be used as by-products[7,8]. With increasing demands of construction materials day by day, the utilization of this waste for construction purposes is one of the sensible and logical means for recycling and reduction of waste marble slurry rather dumping[9,10]. Studies conducted on the utilization of waste marble slurry in concrete mixture shows that the concrete having more evenly distributed limestone contents have high strength characteristics[11]. Marble Stone Slurry wastes predominantly, consist of by 99% calcium carbonate. Various researchers worked on waste marble slurry waste in many applications like; utilization of marble dust in the development of high strength cementitious binders materials[12] usability of marble slurry waste in self compacting concrete[13], making concrete blocks, manufacturing of light weight bricks and floor tiles[14,15] and its suitability for construction purposes[16]. It is also used in Asphalt mixture as a filler material[17], utilized in ceramics industry[18] and also used in the process of desulphurization[19].

The literature survey shows that marble slurry waste has got many applications and can be used as a raw material. In Pakistan, Mineral Testing Laboratory, Hayatabad prepared low-grade cement[20] which has got limited use but was not evaluated for commercial scale. Therefore, the aim of this research study was to investigate the feasibility of using waste marble slurry in the

* Department of Enviornmental Sciences, University of Haripur
** Department of Enviornmental Sciences, University of Peshawar
*** Department of Statistics, University of Peshawar

manufacturing of commercial cement instead of raw calcium carbonate and evaluate it for physical, chemical and mechanical properties.

METHODOLOGY

Characterization of Raw Materials

Marble stone slurry was obtained from nearby marble processing units and dried in sunlight. Common raw $CaCO_3$, used in the manufacturing of commercial grade cement was taken from cement industry. Both the raw materials analyzed for physico-chemical characterization. Physical characterization includes average particle size, porosity and density were done according to ASTM C373-88 & ASTM C136-01 designation[21,22]. For this purpose sample was dried in oven at 110 ºC. Chemical analysis of waste marble slurry generating from marble processing units and raw calcium carbonate was done by using X-ray Fluorescence spectrometer (Panalytical Axios Max WDXRF) according to ASTM C25-11 designation[23].

Preparation of Portland cement

Test specimens were prepared in the laboratory by using marble slurry waste as a source of raw calcium carbonate, used in cement manufacturing process. In the laboratory 78% of marble slurry was mixed with 12% silica sand, 5% iron oxide, and 5% aluminum-silicates clay minerals Figure (1a). All of these were mixed together and saturated with water to form slurry. This slurry was heated in a batch rotary kiln, inside high alumina brick with 2 feet internal diameter, 5 feet length having capacity of 50 kg at 1400 ºC at a rotational speed of 10rpm and then cooled by using centrifugal exhaust fan. After that solid prismatic stone like clinker materials were formed Figure (1b). These solid aggregates were crushed into fine powder. After this 5% gypsum by weight was added for settling of cement.

Evaluation of Portland cement

The cement used for evaluation purpose in the experiment was commercial grade Portland cement available in market say as CEM I and cement prepared in laboratory from marble slurry powder called as CEM II. For evaluation of both types of cement physical, chemical and mechanical properties of their concrete mixture were determined.

Physical analysis

Initial and final settling times for both specimens were determined invoice with ASTM-C 266 by using Gilmore needles methods along with volume expansion by Le-Chattlier method[24]. The density was also determined by using pycnometer according to ASTM C373-88 designation. To determine the fineness of hydraulic cement two methods are generally used i.e. (i) sieve methods for particles size determination and (ii) Blaine air permeability method. In current study sieving method was used (Standard EN 196-6)[25].

Chemical analysis

Chemical analysis of commercial cement (CEM I) and cement prepared in the laboratory (CEM II) were carried out for SiO_2, Al_2O_3, Fe_2O_3, CaO, MgO, K_2O_3

(a)

(b)

Figure 1: (a & b): Showing Raw material used in the manufacturing of Portland cement and Clinker formed after heating.

and Na_2O by using Panalytical Axios Max WD XRF spectrometer XRF Whereas free lime was determined by using X-Rays Diffraction (Bruker-Germany D8 Discover XRD) . The loss on ignition (LOI) was determined by heating both cements samples at 950-1000ºC in a furnace and subtracted the loss in weight.

MECHANICAL PROPERTIES

Preparation of Concrete Specimens

Making concrete mixture of cement prepared in laboratory and commercial cement available in the market with sand, gravel and cement (S:G:C) ratio of 10:10:80, 20:20:60, 30:30:40, 40: 40:20 respectively. For this purpose crushed stones and dry clean natural river sand were used in concrete mortar (Figure 2a & b).

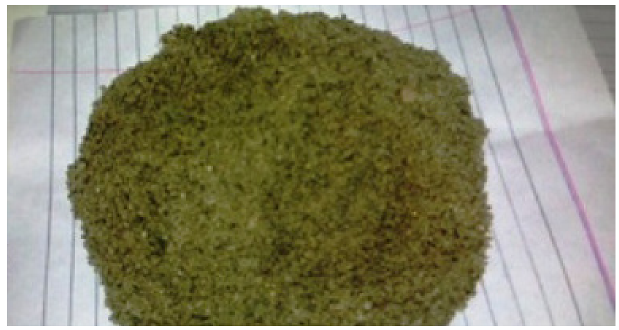
Cylinderical Concrete block Specimens of 6x3 inch (150x30 mm) were casted and de-molded after 48 hours and were tested at 7 to 28 days of curing age. For this purpose overall 16 cylindrical blocks were prepared, 4 cylindrical block of the each specimen/ratio were prepare and each cylinder block was tested for water absorption capacity and compressive strength at the interval of 7, 14, 21, and 28 days (Figure 3a & b).

Water absorption capacity

Water absorption capacity test was done for the determination for pore structure of concrete specimens to check the durability of both types of cements. With the help of water absorption capacity test of the concrete gives us the reachable volume of total pores capacity of the hardened concrete. Water absorption capacity also investigate the apparent porosity increase in porosity means decrease in the quality of concrete materials or more porosity weaker concrete[26]. The test of Water Absorption capacity (Wa) was carried out according to ASTM C373-88 (2006) designation by immersing the tested specimen in water for 48 hours after 7, 14, 21, and 28 days curing time. After curing the specimens were dried in oven at 110ºC to evaporate occluded water. The value of (Wa) was calculated by the following formula (Equation 1).

$$Wa = \frac{W2-W1}{Wa} \times 100 \qquad (1)$$

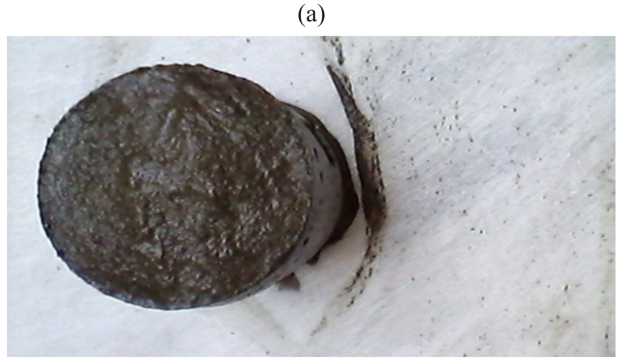
(a)



(b)

Figure 2: (a & b). Showing Gravel (a) and sand (b) used in the preparation of concrete mixture.

(a)



(b)

Figure 3: a & b: showing preparation of cylindrical mould specimens made of concrete mixtures for compressive strength.

Wa = water absorption capacity
W1= weight before immersion
W2= weight after immersion

Compressive strength test

This is the most important test to check the quality of hardened concrete. The concrete should be tested for its compressive test prior to use it in construction works. To avoid subsequent cracking and shrinkage compressive Strength tests were not performed on the paste of neat cement. Compressive Strength of concrete specimens made of different cement – sand-gravel mortar in specific proportion. Removing irregularities and capping of cylindrical specimens were done by using grinding technique and sulfur lock mortar. For compressive strength of testing specimen IPC Global Servo Hydraulic Universal Compressive Testing Machine (UTM 100) was used. The common breakage pattern observe in the testing specimen were conical fracture pattern. All of the above procedure was invoice of ASTM 31-12[27] and ASTM C39[28].

RESULTS AND DISCUSSION

The density value of dried marble slurry waste was about 3.8g/cm^3 which were a little higher than that of raw calcium carbonate materials 2.76g/cm^3 that used in the manufacturing of commercial cement. This high density of marble slurry was due to the presence of iron abrasive particles from gang saws during sawing the rocks. Chemical analysis of Marble slurry and raw calcium carbonate aggregate shows (CaO) calcium oxide as the major component (>50%) with loss of ignition around (>40%), and small amounts of SiO_2 (<2%), MgO(<1%), and Fe_2O_3(<1%), as indicated in the Table 1.

In the physical properties the cement paste setting time are categorized into initial and final setting time. initial setting time is the approximate time at which the fresh cement paste begin to stiffen, while final setting time indicate the approximate time at which the cement paste has hardened and can support load. Generally average initial settling time for a cement used for construction purpose are between 60-240 minutes, while for final settling the range is 180-360 minutes and the volume expansion of commercial cement should not be more the 0.8% of the volume of tested specimen[29]. The results of both types of cement pastes were satisfactory and the results of CEM -II initial and final settling time were show less difference as compare to CEM- I results. The soundness or volume expansion is an important test of cement physical quality assessment. The volume expansion of the cement dominate over the attractive crystallization forces as a results of which no hardening takes place or the hardening effect of cement decreases[30,31]. The results of volume expansion for both types of cements

Table 1: Physico-Chemical propereties of Marble slurry powder and raw calcium carbonate used in cement manufacturing process.

Physical Characteristics	Marble Slurry	Raw $CaCO_3$
Average density (g/cm^3)	3.8	2.76
Average particle size (µm)	5-25	>100
Porosity (%)	1.96	1.3
Chemical Characteristics (%)		
SiO2	1.43	1.52
Al2O3	0.14	0.17
Fe2O3	0.05	0.10
CaO	54.30	52.10
MgO	0.50	0.80
K2O3	0.12	0.10
Na2O	0.10	0.12
LOI	42.20	44.10

Table 2: Physical Characteristics of commercial grade Portland cement (CEM I) and cement prepared from marble slurry waste (CEM II)

S.No	Properties	CEM- I	CEM -II	Pakistan Standards (PS-232)
1	Initial setting (min)	220	240	45
2	Final setting (min)	300	320	60
3	Volume expansion (mm)	1	1	10
4	Density (g/cm3)	2.9	3.0	--
5	Sieve analysis (passing %)			
	Particle size 100µm	100	100	72 µm ‘or’ 225m^2/kg ‘or’ 2250 cm^2/g
	Particle size 75µm	94	85	
	Particle size 50µm	88	80	
	Particle size 25µm	65	58	
	Particle size 15µm	60	55	
	Particle size < 5µm	20	25	

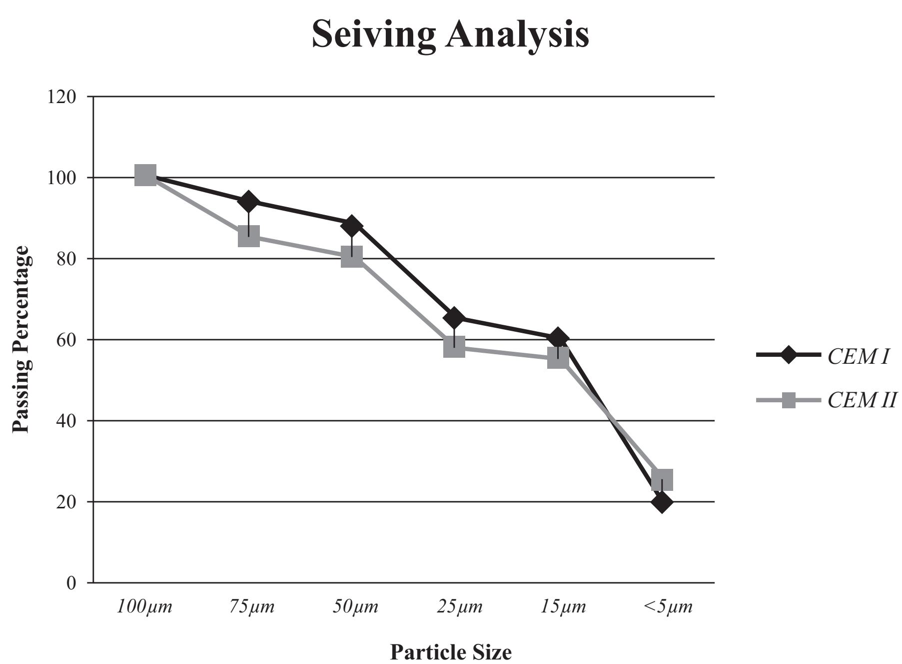


Figure 4: showing average particle size of CEM I and CEM II

were 1mm which is less than the standards maximum allowable value of 0.8%. Density and average particle size results for both types of cement were marginally different (Table 2 & Figure 4).

The results of chemical parameters were shown less significant difference from each other except slight change in silica contents, iron oxide and loss on ignition (Table 3). According to ASTM and ACI cement standards Magnesium oxide should be present in Commercial cement up to 6%, loss on ignition 3% and alkalis (Na_2O, K_2O) 0.6%.

Table 3: Chemical characteristics of ordinary commercial Portland cement (CEM I) and cement (CEMII) prepared from marble slurry (WEIGHT %)

Parameters	CEM-I			CEM-II			
	Min	Max	Average	Min	Max	Average	Standards
SiO_2	23.2	23.9	23.6±0.2	20.0	20.3	20.2±0.1	23.5-25.5*
Al_2O_3	5.6	7.0	6.3±0.5	5.4	7.2	6.3±0.6	8.0-9.0*
Fe_2O_3	0.3	3.5	1.9±1.1	2.1	5.5	3.8±1.1	3.0-4.0*
CaO	58.1	68.2	63.2±3.4	56.5	69.8	63.2±4.5	--
MgO	1.5	2.1	1.8±0.2	1.7	1.8	1.8±0.0	4.0-6.0**
K_2O	0.5	1.1	0.8±0.2	0.9	1.0	1.0±0.0	0.75*
Na_2O	0.2	0.2	0.2±0.0	0.1	0.5	0.3±0.1	0.75*
SO_3	0.5	1.5	1.0±0.3	0.6	1.6	1.1±0.3	3.25**
Free Lime	0.8	1.0	0.91±0.5	1.0	1.2	1.1±0.3	1.5*
LoI	4.5	5.5	5.0±0.3	5.4	6.0	5.7±0.2	3-4**

** Pakistan Standards PS:232 – 1983 (R)

* British Standards BS:12-1996

Table 4: Curing age and Composition wise water absorption capacity of Concrete cylindrical block made of CEM I & CEM II concrete cement mixtures

Specimen	Sand (%)	Gravel (%)	Cement (%)	Curing period (days)	Water Absorption Capacity (%)	
					CEM I	CEM II
1	10	10	80	7	05	06
				14	4.5	05
				21	04	4.5
				28	03	04
2	20	20	60	7	5.5	06
				14	05	05
				21	05	5.5
				28	4.5	5.5
3	30	30	40	7	6.0	7.0
				14	5.5	6.5
				21	4.0	4.0
				28	3.8	4.0
4	40	40	20	7	7.0	7.0
				14	6.8	7.0
				21	6.5	6.5
				28	6.0	6.5

Table 5: Curing age and Composition wise Compressive strength of Concrete cylindrical block made of CEM I & CEM II concrete cement mixtures

Specimens	Sand (%)	Gravel (%)	Cement (%)	Curing period (days)	Compressive strength (psi)	
					CEM I	CEM II
1	10	10	80	7	1800	1600
				14	3200	2800
				21	4200	3600
				28	4700	4500
2	20	20	60	7	1600	1400
				14	2600	2500
				21	3800	3500
					4200	4000
3	30	30	40	7	1400	1300
				14	2400	2300
				21	3400	3200
				28	3900	3900
4	40	40	20	7	1100	900
				14	1800	1300
				21	2300	2100
				28	2900	2600

Note: According to Pakistan PS:232 – 1983 (R) standards for concrete quality, at 28th day the compressive test should not less than 2800 PSI or 197.0 kg/cm^2

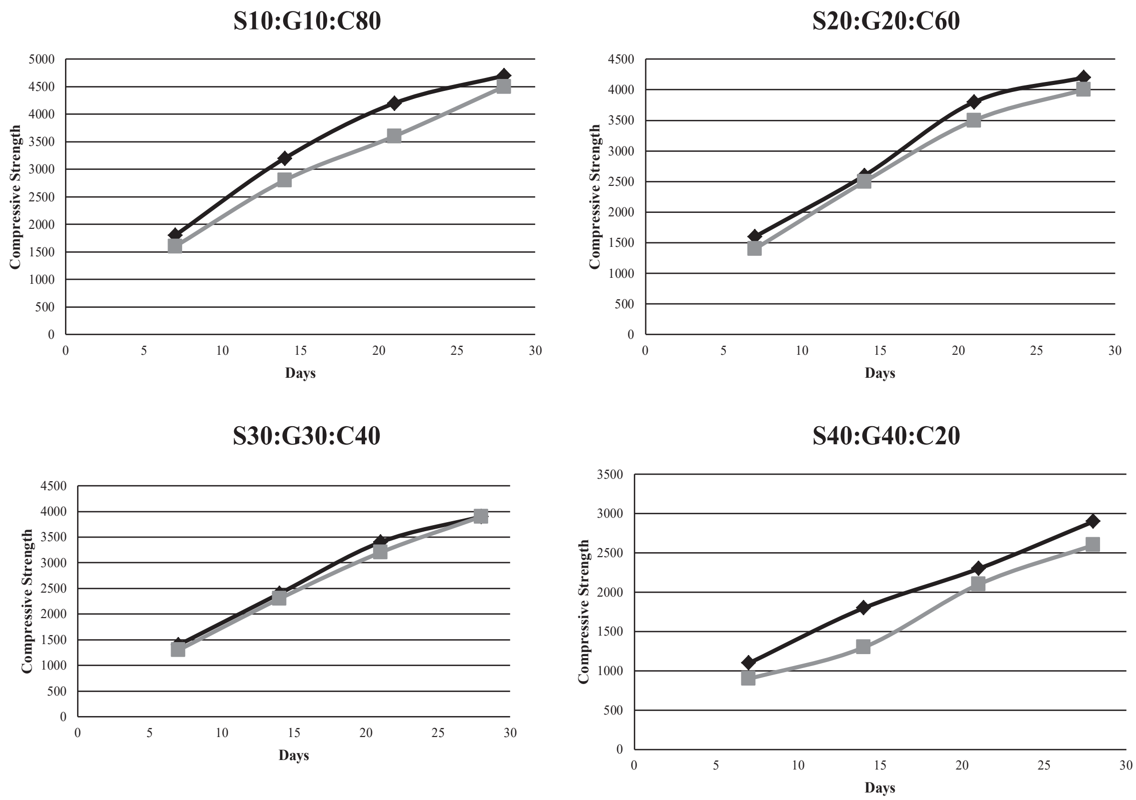
Comparison of Water absorption capacity

Water absorption capacity of concrete depends upon the types of aggregates used in the concrete mixture. If coarse aggregates will be used the water absorption capacity increases due to formation of high pores spaces as compare to fine aggregates concrete mixture. The water absorption capacity of 30 minutes emersion concrete should be <3% for low, 3-4% for average and >4% for high absorption concrete[32]. (Table 4)

Comparison of Compressive Strength Test:

The cylindrical specimens were made and tested for 7, 14, 21 and 28 days of curing age strength (Table 5). Compressive strength requirement for concrete are 2500 psi for residential purposes and 4000 psi to higher in commercial structures[33]. Compressive test results of specimens 1,2 and 3 shows excellent strength as compared to Pakistan PS:232 – 1983 (R) standards for concrete quality which is 2800 psi (Table 5).

Compressive strength was also graphically presented to shows the trend of compressive strength for different concrete specimens made by both types of cements (Figure 5). All the four types of tested specimens show good compressive strength results in different curing ages. At the SGC ratio 30:30:40, same compressive strength was recorded on the 28th day. It is suggested that the specimen number 1 and 2 would be use for high commercial structures construction while specimen 3 and 4 are suitable for residential purposes.



CEM I ◆ CEM II ■
S=Sand, G=Gravel, C=Cement

Figure 5: showing graphically the Curing age and Composition wise Compressive strength of all four tested specimen for both types of cement concrete mixtures.

CONCLUSION

Calcium carbonate waste marble slurry dust is similar in characteristics of the raw calcium carbonate use in cement manufacturing process. In Pakistan marble slurry waste is produce in large quantity and disposed off into the environment which deteriorates the physical chemical and biological segment of environment. The utilization of marble slurry waste as an alternative resource of calcium carbonate in industrial manufacturing process of cement has been the objective of this study. By recovery, reusing and recycling of this waste material in the manufacturing of cement have a great contribution to the environment and economy by minimizing pollution load coming from marble processing industry and by utilizing waste into valuable product. The cement prepare from Marble slurry dust having almost similar standards when it was compare to commercial cement during evaluation because the mean compressive strength test result shows less difference with each other. Compressive strength testing of specimen in the study was used for the acceptance of cement, quality control, evaluation or for the estimating the concrete strength in a structure for the purpose of scheduling construction operation. Beside from this a policy should be made under which cement industry compelling to utilize the waste of marble industry into its production process.

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