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RESEARCH ARTICLE

RECYCLING GRANITE AND MARBLE SLUDGE AS A CONSTRUCTION MATERIAL

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ABSTRACT

Two environmental targets were addressed in this study; decreasing the demand on cement and consequently the amount of generated polluting carbon oxide by the cement industry as well as recycling the granite-marble sludge as a construction material rather than dumping it. This study presented the results of an experimental program that investigated the recycling of the sludge particles as a replacement for cement in mortars. The effect of replacement percentages 10%, 20% and 40% on the basic properties of cement and compressive strength of corresponding cement mortars were determined. Among the studied replacement percentages, the 10% provided optimum acceptable basic cementitious properties such as loss on ignition, soundness, setting times, alkalis and pH value as well as highest mortar compressive strength which fulfilled the ASTM C 150 strength limits at 3, 7 and 28 days. Also, this study showed that the sludge powder is acting as a filler material and that for mixes with sludge, one should control the actual water/cement ratio rather than the ratio of thewater to the sum of cement and sludge powder

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INTRODUCTION

More than 5% of carbon oxide contamination is caused by the cement industry (Worrel, 2001). Consequently, demand on cement must be decreased by introducing a substitute which enables us to produce acceptable cementitous products. Simultaneously, in Saudi Arabia the sludge generated during the saw cutting of marble and granite is usually dumped. Through a questionnaire sent to 20 companies in Jeddah city (Alattar, 2013), approximately 1.5 kg of sludge is generated for every square meter of cut panels with a total of 650 ton per month (250 m^3/month). If we assume that the sludge is dumped at 50 cm thick layers, then we will cover about 500 m^2/month. Several researchers used natural materials as concrete and mortar constituents; the majority focused on the effect of their additive material on the resulting compressive strength. The first group of the researchers replaced cement with natural materials while the second group replaced fine and coarse aggregates. Among the first group of researchers, Morsy *et al.* (2010) observed an increase in compressive strength from 47.1 to 50.5 M Pa when they replaced cement by nano-clay at 8%.

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Ergun (2011) observed an increase in concrete compressive strength when he replaced cement by marble powder ratios up to 7.5%. Uysal (2012) replaced cements with limestone powder, basalt powder and marble powder; he observed an increase in concrete compressive strength from 75 to 78 M Pa at 20% replacement. Rodriguez (2006) reported improved early concrete compressive strength, tensile strength and air permeability as the cement was replaced with incinerated Rice-Husk Ash. On the other hand, Corinaldesi *et al.* (2010) observed a decrease in mortar compressive strength as they replaced cement with marble powder. Belaidi *et al.* (2012) reported a decrease in compressive strength for cases of cement replacement with mixtures of pozzolana and marble powder. Topcu *et al.* (2009) as well as Gesog˘lu *et al.* (2012) replaced cement with marble powder in self-compacting concrete; they observed a decrease in compressive strength with the increase of the replacement ratio.

The second group of researchers replaced fine and coarse aggregates with natural materials of different sizes. Gencela *et al.* (2012) reported a continuous decrease in concrete compressive strength with the increase of replacement percentages (10, 20, 30 and 40%) of waste marble waste. On the other hand, Siddique (2003) replaced sand with class F fly ash. He observed an increase in concrete compressive strength, tensile strength and modulus of elasticity for replacement percentages up to 50%. Similarly, Hebhoub (2011) as well as Binici (2008) replaced sand by sludge. They reported an optimum sand replacement at 50% which provided the highest concrete compressive strength.

The above contradicting trends of increase and decrease of strength is explained in the discussion section based on the arguments of amount of generated calcium silicate hydrate (C-S-H), the reactivity of added materials according to its nature (filler, hydraulic or latent hydraulic), the material specific surface area as well as the effective water/cement (w/c) ratio. Such factors are considered in this study where the recycling of primitively processed sludge as a substitute or addition to cement mortar is investigated.

MATERIALS AND METHODS

The used cement was ordinary Portland cement (Type I) conforming to ASTM C 150 -07 while sand was conforming to ASTM C 33 -07. The sludge was obtained from one of the major companies for cutting marble and granite in Jeddah, Saudi Arabia. Table 1 shows the chemical composition of the cement, sludge powder and the subsequent mixes at different replacement ratios. The specific surface area for the used cement was equal to 3420 cm^2/gm while the sludge particles mainly passed sieve # 30 and were retained on sieve # 200. With this sludge relatively coarse particle size, the specific surface area of different cement-sludge mixes is definitely below the 3420 cm^2/gm.

Table 2 shows the constituents of the different mortar mixes. The original 740 gm of cement was replaced by 10%, 20% and 40%. These replacement percentages correspond to equivalent addition percentages of 11.1, 25.0, and 66.7% respectively.

In order to avoid literature contradicting trends of strength with the increase of replacement percentages, two approaches were adopted. In the first approach (mixes 2 through 4), the ratio of water to the sum of cement and sludge powder, w/cm, (referred to in the literature as the water cementitious ratio) was maintained constant at 0.48 and subsequently the actual w/c ratio increased to 0.53 at 10% replacement, 0.60 at 20% replacement and 0.80 at 40% replacement. In the second approach (mixes 5 through 7), the w/c ratio was maintained constant at 0.48 and subsequently the w/cm ratio decreased to 0.43 at 10% replacement, 0.38 at 20% replacement and 0.29 at 40% replacement as shown in Table 2. Unlike most researchers who focused on the compressive strength, the current study presented the results of an experimental program that investigated the effect of replacing cement with sludge powder (at 10%, 20% and 40%) on the basic properties of cement such as consistency, setting, loss on ignition, insoluble residue, soda equivalent factor, pH value and soundness as well as the corresponding mortar compressive strength at 2, 3, 7 and 28 days.

RESULTS AND DISCUSSION

X-Ray results for the different sludge replacement percentages

Table 1 shows the chemical composition of the cement (0% replacement) and sludge particles (100% replacement) as well as the expected chemical composition of different mixes at 10, 20 and 40% replacement percent ages.

Table 1. Chemical composition by XRF and corresponding lime saturation factor

Replacement (%)	% SiO_2	% Al_2O_3	% Fe_2O_3	% CaO	% MgO	% SO_3	% K_2O	% Na_2O	% MnO	% TiO_2	Lime Saturation Factor (%)
0	20.25	5.41	3.46	63.2	1.37	2.78	0.6	0.13	NA	NA	0.94
10	24.57	5.94	4.95	57.56	1.27	2.53	0.95	0.36	NA	NA	0.70
20	28.5	6.42	6.57	51.35	1.15	2.24	1.28	0.6	NA	NA	0.54
40	35.9	7.52	10.46	39.71	0.93	1.66	1.94	0.95	NA	NA	0.33
100	61.31	10.91	16.47	3.34	0.12	NA	4.01	2.97	0.15	0.19	NA

Table 2. Mix constituents

Mix №	Cement (gm)	Sludge (gm)	Sludge Replacement (%)	Equivalent Sludge Addition (%)	Water (cc)	Sand (gm)	w/c	w/cm
1	740	0	0	0	359	2,035	0.48	0.48
2	666	74	10	11.1	359	2,035	0.53	0.48
3	592	148	20	25.0	359	2,035	0.60	0.48
4	444	296	40	66.7	359	2,035	0.80	0.48
5	666	74	10	11.1	323	2,035	0.48	0.43
6	592	148	20	25.0	287	2,035	0.48	0.38
7	444	296	40	66.7	215	2,035	0.48	0.29

The sludge consisted mainly of SiO_2, Fe_2O_3 and $Al_2O_3$and was free of SO_3.The percentages of MgO and the CaO were less than those found in the cement. Consequently, any replacement percentage would reduce the resulting SO_3, MgO, and CaO in the mix. Also, Table 1 shows the lime saturation factor which ranged from 0.33 to 0.94.

Most of the sludge chemical constituents were not expected to possess hydraulic activity since they were subjected to H_2O during the long slurry phase resulting from the cutting process. As for latent hydraulic activity, current results of compressive tests did not support its existence as will be discussed later. Hence, the sludge particles acted as a filler material that blocked voids and decreased shrinkage of cement-sludge mixes as compared to cement pastes.

Effect of replacement percentage on loss on ignition, soda equivalence, insoluble residue, pH value, consistency, setting time and soundness

Table 3 shows that the loss on ignition percentage decreased from 2.4 to 1.04 % as the percentage of sludge replacement increased from 0% to 40%. The upper limit for loss on ignition set by ASTM C 150-94 is 3%. The relatively lower loss on ignition values is attributed to lower MgO and CaO percentages in the cement-sludge mixes (see Table 1) and hence, lower ability for these compounds to carbonate upon exposure to the environment.

The soda equivalent factor given by % Na_2O + 0.658 * % K_2O was calculated for different cement-sludge mixes as given in Table 3. As expected, the soda equivalent factor increased with the increase of replacement percentage. The corresponding high soda equivalent value limits the use of cement-sludge mixes to aggregates of limited active silica which is the case for mortars where natural sand is mainly used. Table 3 shows that the insoluble residue percentage increased with the increase of sludge percentage. This observation is attributed to the fact that the sludge particles were acting as inactive filler precipitates.

Also, the table shows the relative pH values given by the ratio of the pH value for the mix/pH value for cement. The relative pH value was about 1.0 which infers that acids used in cleaning the slurry pipelines on site (Alattar 2013) did not affect the sludge neutral nature; the acid was washed out by the large amount of cooling water used during the cutting process. Table 3 also shows the w/cm ratio necessary to achieve standard consistent pastes and setting time for different sludge replacement percentages. The table shows that w/cm ratio increased with the increase of sludge added percentage. This increase in the amount of the water needed to achieve the consistent paste is attributed to the fact that sludge particles surround themselves with an adhered film of water necessary for lubrication and hence less water was left for the cement hydration process.

The relative setting time is the setting time at each replacement percentage normalized by setting time at 0% replacement. Table 3 shows that the initial setting time was not affected by the addition of sludge particles as compared to the final setting time which decreased with the increase of added sludge particles. Such setting behavior is due to the fact that the initial setting is controlled by the hydration process which comprises the dissolution of anhydrous compounds into constituents, formation of hydrates in the solution and precipitation of hydrates from the supersaturated solution. Hence, the initial setting will be initiated irrespective of the presence of the sludge particles. On the other hand, the final setting time was positively decreased since it is controlled by paste hardening which is affected by the presence of the sludge particles. Le Chatelier soundness test was also conducted where all mixes passed the test. This is attributed to the fact that the addition of a filler material effectively decreased the inherent tendency of volumetric changes of cement as well as the decrease of the amount of free lime and magnesia (see Table 1) which are responsible for unsoundness.

Table 3. Loss on ignition, soda equivalent factor, insoluble residue, relative pH value, consistency and relative setting time

Replacement %	Loss on ignition %	Soda equivalent factor %	Insoluble residue %	Relative pH value#	w/cm ratio for consistency test	Relative setting time *	
						Initial	Final
0	2.40	0.55	1.2	1.00000	0.28	1.000	1.000
10	1.91	0.98	6.0	1.00085	0.31	1.050	0.939
20	1.61	1.44	13.0	0.99915	0.32	1.025	0.809
40	1.04	2.22	30.0	0.99658	0.34	1.000	0.744

pH value at each replacement percentage normalized by pH value at 0% replacement

*Setting time at each replacement percentage normalized by setting time at 0% replacement.

Table 4. Averaged cube compressive strength and coefficient of variation

Approach	Age (days)	Compressive Strength (MPa)				Coefficient of Variation			
		0%	10%	20%	40%	0%	10%	20%	40%
w/cm ratio was constant	2	18.03	16.41	14.35	9.30	0.067	0.088	0.082	0.072
(0.48) while w/c ratio	3	20.23	18.67	19.89	13.06	0.054	0.045	0.055	0.054
varied (0.48, 0.53, 0.60	7	28.88	28.13	22.33	18.06	0.041	0.043	0.048	0.045
and 0.80)	28	31.27	28.94	24.53	18.52	0.031	0.043	0.044	0.044
w/c ratio was constant	2	18.03	15.49	14.71	5.17	0.088	0.050	0.073	0.079
(0.48) while w/cm ratio	3	20.23	22.04	18.74	4.15	0.054	0.048	0.072	0.059
varied (0.48, 0.43, 0.38	7	28.88	27.04	29.24	4.71	0.053	0.036	0.047	0.050
and 0.29)	28	31.27	32.15	28.57	17.09	0.041	0.036	0.042	0.064

Effect of sludge percentage on the mortar compressive strength

The discrepancy in the compressive strength reported by the cited researchers is mainly attributed to the amount of generated calcium silicate hydrate (C-S-H) which in turn is dependent on the finess of the added material and its nature being filler, hydraulic or latent hydraulic. On replacing cement by a filler (inactive) powder material, the subsequent C-S-H decreases by the increase of the replacement ratio. In such cases, the cement content and water/cement (w/c) ratio have to be controlled rather than water/cementitious (w/cm) ratio. On the other hand, if the additive material is hydraulic or latent hydraulic, the amount of generated C-S-H and the subsequent compressive strength may increase depending on the reactivity of added material. The cited researchers reported materials with specific surface area that ranged from 3500 to 480000 cm^2/gm, and hence, its chemical reactivity and consequently the amount of generated C-S-H as well as the subsequent properties were affected in different ways.

Table 4 presents the averaged cube compressive strength as well as the coefficient of variation for the two adopted approaches. In general, the 10% replacement provided the highest 28 days compressive strength among replacement percentages. Also, the coefficient of variation decreased with age. Figure 1 presents the results for the first approach where the w/cm ratio was maintained at 0.48 while actual w/c ratio increased to 0.53, 0.6 and 0.8 for 10%, 20% and 40% replacements respectively as shown in Table 2. The figure shows that among the replacement percentages, the 10% provided the highest 28-days strength and also provided strengths at 3, 7 and 28 days that complied with the specified ASTM C 150-07 lower strength limits (12, 19 and 28 MPa for 3, 7 and 28 days respectively). Generally, beyond 10% replacement, the compressive strength decreased at most ages with the increase of sludge percentage. This decreasing trend is attributed to the increase of the actual w/c ratio from 0.53 at

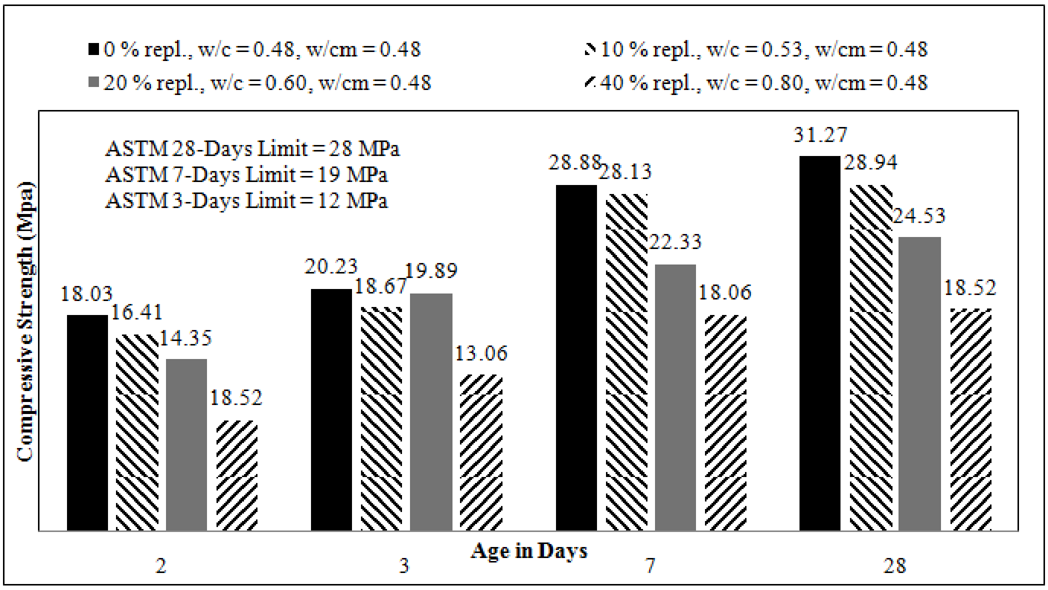

Fig.1. Compressive strength for different cement/sludge mixes of constant w/cm ratio 0.48 and varying w/c

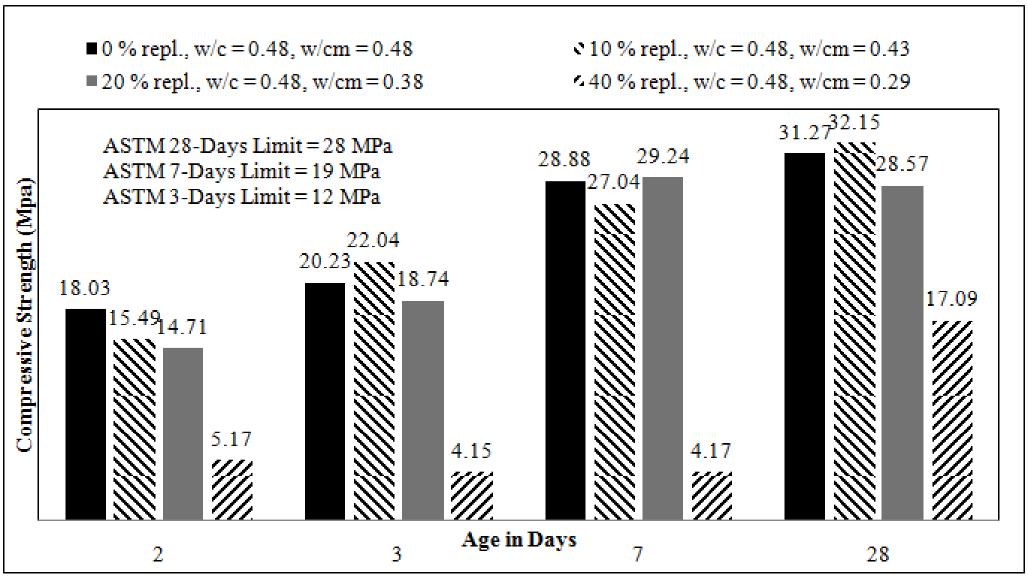

Fig. 2. Compressive strength for different cement/sludge mixes of constant w/c ratio 0.48 and varying w/cm

10% to 0.60 at 20% replacement and finally to 0.80 at 40% replacement. Hence, the classical decrease of compressive strength with the increase of w/c ratio is observed. However, this decrease in strength occurred though the w/cm ratio was maintained constant at 0.48. Figure 1 shows that for every replacement percentage, a progressive increase in strength by time was observed. However, the acquired strength at 7 days was slightly less than that of the 28 days. The difference in strength between 7 and 28 days was less than the corresponding difference of acceptable limits required by ASTM C 150 - 07. Such slight increase in strength from 7 to 28 days infers that the sludge did not add to 28 days strength due its filler nature.

Figure 2 presents the results for the second approach where the w/c ratio was maintained at 0.48 while the w/cm ratio decreased to 0.43, 0.38 and 0.29 for 10%, 20% and 40% replacements respectively. Again, the figure shows that among the replacement percentages, the 10% provided the highest 28-days strength and also provided strengths at 3, 7 and 28 days that complied with the specified ASTM C 150 – 07 lower strength limits. Beyond 10% replacement, the compressive strength decreased at most ages with the increase of sludge percentage. The decreasing trend of strength has been observed though the w/cm was decreasing. One would have expected the conventional increase in strength with the decrease in w/cm ratio if the added sludge possessed a hydraulic or latent hydraulic nature that compensates for the decrease in cement content. Hence, the decrease in strength with the increase of sludge replacement percentage and corresponding decrease in the w/cm ratio suggests that the sludge addition reduces the compressive strength due to its filler nature. Hence, the classical increase of strength with the decrease in w/cm ratio has to be wisely considered when using filler additives.

Apart from the above w/c or w/cm arguments, the decreasing trend of strength beyond 10% replacement of figures 1 and 2 is generally attributed to the lower generated C-S-H. The lower generated C-S-H is attributed to the decreasing amount of cementitious material with the addition of the filler sludge as well as to the reduction in the specific surface area for of cement-sludge mixes. Simultaneously, the added filler sludge particles are relatively fine when compared to the used sand particles, hence, the specific surface area of sand and sludge mixes increase with the increase of the sludge in the mortar mix. Such increase in specific surface area of the filler materials (sand and sludge) causes the reduction in compressive strength. For 10% replacement, one can observe from figures 1 and 2 that the acquired compressive strengths were slightly alternating around the strengths of the corresponding 0% replacement; sometimes slightly higher and sometimes slightly lower. Hence, after considering the coefficients of variation one can conclude that the 10% replacement ratio did not affect the compressive strength to an intolerable extent.

Conclusions

- The sludge produced over the cutting process of granite and marble can be used as a filler additive in mortar mixes. This filler additive will allow us to reduce the cement content in the mortar mixes by 10% while maintaining basic cementitious properties such as loss on ignition, soundness, alkalis content and the compressive strength at ASTM required levels. Hence, the demand on cement and the corresponding pollution resulting from the cement industry as well as the sludge dumping problems will be alleviated.
- The used sludge particles with primitive grinding and sieving to pass sieve # 30 and to be retained on sieve # 200 did not improve the mortar compressive strength. This decrease in strength with the increase of sludge replacement percentage and corresponding decrease in the w/cm ratio suggests that the used primitively processed sludge possessed a filler nature.
- The 10% replacement provided faster final setting time without affecting the initial setting time.
- The 10% replacement ratio did not affect the compressive strength to an intolerable extent when compared to cement mortar free of sludge (0% replacement). Also, among actual replacement ratios it provided highest mortar 28-days compressive strength which fulfilled the ASTM strength requirements at 3, 7 and 28 days.
- In cases where the cement is replaced by a filler material, the cement content and the w/c ratio must be our main monitored variables. Simultaneously, the classical increase of strength with the decrease in w/cm ratio has to be wisely considered when using filler additives.

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