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The Characteristics of Cement Mortars Utilizes the Untreated Phosphogypsum Wastes Generated From Fertilizer Plant, Aqaba- Jordan

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Abstract

Huge amounts of phosphogypsum (PG) are produced as by-products of phosphoric acid manufacture process in Jordan. Millions of tones of PG wastes are stockpiled over open areas. Major negative environmental impacts are highly expected due to dissolution of various hazardous chemicals the stockpiled contains. This will cause a serious threat to the soil, surface and groundwater bodies. X-Ray Fluorescence revealed that the main oxides were SO_3 and CaO with 39% and 31.9%, respectively. What reflected its mineralogy were gypsum, as main phase, and calcite, quartz and apatite, as minor. Two PG samples were used, first dried to 60°C (PG1) and then heated up to120°C (PG2). Both samples were used in sand-clinker and clinker–sand-pozzolanic tuff mortars to investigate compressive strength, setting time, consistency and other physical properties. The results showed that the addition of 2-4% of PG1 gave an average of 52 N/mm^2 and 46.4 N/mm^2 at 28 days for the two mixing ratios, respectively. Meanwhile, PG2 showed lower compressive strength values. Moreover, lower consistency was achieved with PG1 more than with PG2 samples, due to the fact that PG2 contains more anhydrite. Setting time is higher and shows an increasing trend in PG1, which contradicts PG2 samples. The result approves the possibility for the utilization of raw untreated PG wastes instead of expensive pure gypsum in cement industry, because it showed good setting time, minimizes the W/C ratio, and increases the compressive strength of the mortars. Besides, it will decrease the production cost and convert these wastes into useful industrial material.

Keywords: Phosphogypsum, Clinker, Compressive strength, Setting time, LE-CHATE, Pozzolana

1. Introduction

Natural materials are usually targeted as a replacement of ordinary cement past in order to attain a high compressive strength, longer workability, lower water consumption, better soundness, etc. El-Hasan and Al-Hamaideh (2012) used Tripoli as a partial replacement of OPC, and they reported that increasing the replacement ratio of Tripoli did not affect the setting time or soundness; however, it increased the volume of the required water to produce the past. Smadi *et al.* (1999) studied the behavior of the compressive strength of cement with different replacement ratios of purified PG and calcined temperature up to 900 °C, finding an increasing trend in compressive strength which improved the initial and final setting time. Bhadauria & Thakare (2006) utilized the phosphogypsum wastes in cement additive as past or mortar in concrete. They carried these applications after purifying the phosphogypsum. They found it suitable for concrete in terms of workability and 28 days compressive strength. PG is also known worldwide for its applications as a binder or cement. Therefore, it was also studied by many researchers (e.g. Gutt 1978; Ouyang *et al.* 1978; Ghafoori, 1986; Akın & Yesim, 2004; Degirmenci, 2008; Lysandrou & Pashalidis, 2008; and Yang *et al.*, 2009).

According to the European Fertilizer Manufacturing Association (EFMA), there are two phosphoric acid production processes; the first, at temperature 70 -80 °C that yields 26-32% P_2O_5, and gives acid and dehydrated phase ($CaSO_4.2H_2O$); the second, at temperature 90 - 110 °C, which yields 40-52% P_2O_5, and gives acid and Hemihydrate phase ($CaSO_4.1/2H_2O$) (EFMA 2000). The Phosphogypsum is found to be radioactive due to the presence of naturally occurring uranium and radium in the phosphate ore (Dippel, 2004). The PG is known for having some chemical impurities like its source rock (i.e., phosphates). Thus, PG is classified as a hazardous waste and is currently being disposed and stockpiled on sandy soil (Berish, 1990; Azouazi *et al*., 2001; Dueñas *et al*., 2007). The PG is supposed to be treated for these impurities; therefore, PG without treatment is referred to here as raw or impure PG.

There are various storage processes of PG wastes, such as ocean disposal (Wissa, 2001), dry stacking on land, which is the common practice (EPA 1999), and wet stacking on basins (Anon, 1998), as well as the use of PG as mine backfill disposal as suggested by (Dippel, 2004).

At the industrial complex at Aqaba, south of Jordan, a daily average of 900-1310 tons of phosphoric acid are produced (http://www.jpmc.com.jo/?q=node/166). For every ton of phosphoric acid produced, about 5 tons of PG are generated, depending on the quality of the phosphate ore (http://ardaman.com/waste.php3). Therefore, about 5000 tons of (PG) are produced each day. Al-Hwaiti *et al.* (2005) reported that heavy elements concentrations in the PG stacks are stable with age, which indicated that trace elements were not leached from the stacks in any significant amount. Whereas, Abed (2011) suggested that Uranium is concentrated in the phosphoric acid

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then into Di-Ammonium Phosphate (DAP) in the fertilizer industry and not in the phosphogypsum.

The possible use of PG as a major constituent in construction materials is vital in order to solve the serious negative environmental impacts caused by the dispersion of the harmful chemicals contained in PG into surrounding environment. Several studies were carried out on purifying the PG by washing it with water to minimize the impurities content before utilizing it in various industrial aspects (Smadi *et al*., 1999).

To enlighten the environmental concerns of the huge PG stockpiled at the industrial complex at Aqaba, this study aims to examine the possible utilization of these untreated PG wastes replacing the expensive pure gypsum in cement industry, presenting the best scenario and mixing ratios to attain the best cement mortar properties.

Cement – Phosphogypsum mixes

In case of cement phosphogypsum mixes, the materials used were untreated phosphogypsum, clinker, tap water, and pozzolana.

Phosphogypsum

Phosphogypsum, used in this study, was obtained from the industrial complex at Aqaba, south of Jordan. Its chemical composition and mineralogical constituents are illustrated in Table (1) and Fig. (1).

Clinker

The Clinker used here is obtained from Fuhais Cement Factory located at Amman city; its code is (OPC type 1). The Clinker was procured from local market and in one lot to maintain uniformity throughout the investigation.

Pozzolana

Tuff as pozzolanic material was obtained from Tal-Hassan which is located about 120 km northeast of Amman.

Water

Ordinary tap water was used for mixing and curing operation.

2. Analytical procedures and equipments

Ordinary Portland Cement type-1 (OPC), from Lafarge Cement Factory at Al-Fuhais area, was mixed with sand to prepare reference standard mortar following ASTM C109. Two samples of PG were heated separately up to 60°C (PG1) and 120°C (PG2) using automatically controlled electrical oven. Grounded clinker (OPC Type-1) was used to produce different mortars of clinker-sand-phosphogypsum and tested following ASTM C109.

The 5*5*5 cm cubes were cured at laboratory temperature for 2, 7 and 28 days. Compressive strength of samples was tested using Digital Compression Machine (Type: ADR- Auto manufactured by ELE); a computerized compression machine was used to determine the compressive strength of multi size concrete cubes. The initial and final setting time for clinker-phosphogypsum mortar was tested using the Vicat needle according to ASTM C191.

The chemical composition of PG was investigated through X-Ray Fluorescence analysis. It was done using the machine (XRF-Pioneer F4), manufactured by Broker at the labs of Natural Resources Authority (NRA), Amman. The machine comes with an attached 72-position sample changer. Pellets were made by fusing 0.8 g of sample powder and 7.2 g of $L_2B_4O_7$ in Au/Pt crucible using a flexor machine (Leco 2000) for 3 – 4 minutes at 1200 ºC. The melt was poured in a mold and left to cool to form a glass disc; trace elements were analyzed. The machine was calibrated with international standards, particularly the Geological Survey of Japan (GSJ) geochemical standards (i.e., Japanese slate JSl-1 and JSl-2). The analytical error was within 5%.

Moreover, mineral constituents were determined using X-Ray Diffraction analysis. It was executed using the machine (XRD-Philips Expert MPD) at the labs of Natural Resources Authority (NRA), Amman. The samples were scanned between 2º and 65º 2θ, using Ni-filtered Co K α radiation, 40 kV/40mA, divergent and scattering slits of 0.02ºmm, a receiving slit of 0.15mm, with stepping of 0.01º and scanning speed of 3º/min.

3. Results and Discussion

3.1. Phosphogypsum chemistry and mineralogy

The chemical composition of the Jordanian PG is shown in Table (1). CaO and SO_3 formed about 71.1 wt %, Loss On Ignition (LOI) (24.4 wt %) with some SiO_2 and P_2O_5. It is well known that PG contains a lot of impurities inherited from its parent phosphatic rocks, such as U, Cr and Ni (Jiries *et al*., 2004; El-Hasan, 2006; Abed *et al*., 2008, Batarseh & El-Hasan, 2009; and Abed, 2011). Therefore, it is considered as an environmental waste (Dippel, 2004; and Bhadauria & Thakare, 2006). It is clear from Table (1) that the main mineral will be gypsum, which is suitable for being used as a blinder or cement past.

Table 1: The chemical composition of the Jordanian Phosphogypsum (wt%).

Oxides	Concentrations (wt%)
SiO_2	4.11
Al_2O_3	0.087
Fe_2O_3	0.068
TiO_2	0.016
Na_2O	0.21
K_2O	0.48
MgO	0.002
CaO	31.9
MnO	0.001
P_2O_5	0.95
SO_3	39.2
Cl	0.001
LOI	24.4

The chemical composition confirms the mineral constituents of PG which was revealed from the X-Ray diffraction, the main phase is gypsum, with minor calcite quartz, apatite and montmorrillonite (Fig. 1). This is in agreement with the findings of Al-Hwaiti *et al.* (2005). As have been reported by Taylor (1990), between 100 -150ºC the gypsum is converted into the metastable phase Hemihydrates ($CaSO_4.1/2H_2O$), and above 170ºC it changed into the Anhydrite ($CaSO_4$). Therefore, in our experiment, cold PG (i.e., 60ºC) represents gypsum and heated PG (i.e., 120ºC) represents the hemihydrates phase, sometimes called bassanite. Because this phase is metastable, it reacts quickly with normal tap water and returns to its complete hydrated form (gypsum), with an exothermic reaction.

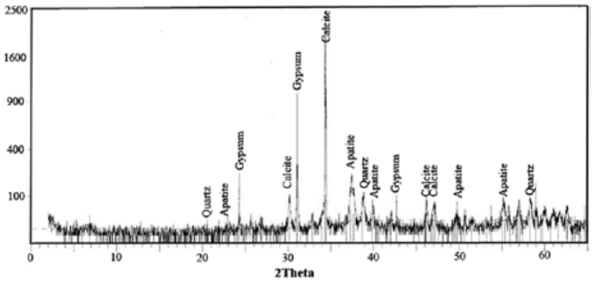


Figure 1: X-Ray diffraction chart for the Jordanian Phosphogypsum used in the mixtures.

3.2. Cement mortar without Pozzolana

3.2.1. Blaine and pH

The Blaine and pH results of produced mortars of both (PG1) and (PG2) resemble those of OPC, Tables (2, 3, 4 and 5). The Blaine was high enough to provide a larger surface area relative to grain size, which caused an acceptable setting time and compressive strength. Moreover, pH of cement water paste was similar to OPC, which facilitates the alkali-pozzolanic reaction, where, clinker compounds react with water through hydration reaction to produce calcium silicate hydrate (C-S-H) and calcium alumina silicate hydrate (C-A-S-H). The time and intensity of these reactions depend on the pH.

3.2.2. Setting time

The setting time for cold raw PG shows a higher initial and final setting time at PG% (4%). Meanwhile, at lower ratios (2%), the heated (PG2) has a higher initial and final setting time. However, at 3%, they show similar initial and final setting time (Fig. 2). Thus 3% may illustrate the optimum mixing ratio of both PG1 & PG2. This can show that at a higher mixing ratio of PG1, more workability can be attained. On the contrary, a lower mixing ratio of PG2 can give better workability behavior of cement mortar.

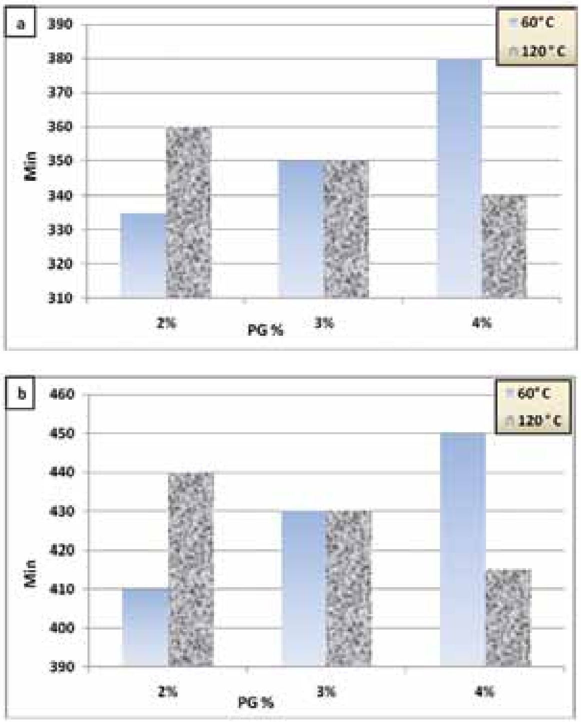


Figure 2: Initial (a) and Final (b) Setting Time (Minutes) difference between PG1 & PG2 for the three PG mixing ration.

3.2.3. Consistency

Water/cement ratio was found increase with increasing the PG replacement percentages for both PG1 & PG2 in the produced mortars, Fig. (3). PG2 has higher percentages than PG1. This might be attributed to the fact that PG2 converted into anhydrite, which needs more water than gypsum, thus PG1 mixing ratio shows lower water/cement (W/C) %, which is a good advantage of cement mortar. The increase in W/C is very small (21.1 - 22%) for PG1 mixed mortars Fig. (3), this may be due to the fact that the PG replacement percentages are small. However, the PG1 shows larger W/C% (21-22.5%), and the highest was in PG1 mixing ratio of (4%) due to anhydrite higher water demand. Bhadauria & Thakare (2006) found that at PG replacement percentage of 5%, the consistency was very near to standard OPC. Furthermore, any further addition causes W/C % to increase rapidly. Both PG1 and PG2 mixing ratios have consistency, lower than OPC standard value (Tables 2 and 4).

Table 2: Generalized table showing complete measurements for unheated raw PG (PG1) mixtures (up to 60°C) without Pozzolana.

Sample No.	**Samples components**			**Blaine**	**pH**	**Compressive Strength (N/mm2)**			**Consist.**	**Setting time (Minutes)**		**LE-CHAT**	
	K	PG1	Pozz	cm^2/g		2 days	7 days	28 days	(W/C)%	Initial	Final	(mm)	
2%	98	2	-	3377	13	21.7	41.8	53.3	21.2	335	410	1	Lab
3%	97	3	-	3356	13	24.3	42	52.3	21.8	350	430	1	Lab
4%	96	4	--	4404	13	22.3	41.9	53	22	380	450	1	Lab
OPC	-	-	-		13	27.2	41.7	53	25	165	215	1	Lab

Table 3: Generalized table showing complete measurements for unheated raw PG mixtures (PG1) (up to 60°C) with Pozzolana; $SO_3 = 2.8\%$.

Samples No.	**Samples components**			**Blaine**	**pH**	**Compressive Strength (N/mm^2)**			**Consist.**	**Setting Time (Min)**		**LE- CHAT.**	
	K	PG1	Pozz	cm^2/g		2 days	7 days	28 days	(W/C)%	Initial	Final	(mm)	
1	96.3	3.7	-	3412	13	22.6	41.7	52.1	22.6	375	455	1	Lab
2	86.3	3.7	10	4200	12	20.1	39	47.7	23.2	365	445	1	Lab
3	96.3	3.7	-	3412	13	22.6	41.7	52.1	22.6	160	210	1	Sun
4	86.3	3.7	10	4200	12	20.1	39	47.7	23.2	145	195	1	Sun

Table 4: Generalized table showing complete measurements for heated raw PG mixtures (PG2) (heated up to 120°C) without Pozzolana.

Sample No.	**Samples**			**Blaine**	**pH**	**Compressive Strength (N/mm²)**			**Consist.**	**Setting Time (Min)**		**LE-CHAT**	
	K	PG2	.Pozz	cm²/g		days 2	days 7	days 28	(W/C)%	Initial	Final	(mm)	
2%	98	2	-	3410	13	19.9	39.3	49.1	21.6	360	440	1	Lab
3%	97	3	-	3385	13	20.5	39	46.6	22.4	350	430	1	Lab
4%	96	4	-	3369	13	19.7	37	43.9	22.6	340	415	1	Lab
OPC	-	-	-	-	13	27.2	41.7	53	25	165	215	1	Lab

Table 5: Generalized table showing complete measurements for heated raw PG mixtures (PG2) (heated up to 120°C) with Pozzolana; SO3 = 2.8%.

Sample No.	**Samples**			**Blaine**	**pH**	**Compressive Strength (N/mm²)**			**Consist.**	**Setting Time (Min)**		**LE-CHAT.**	
	K	PG2	.Pozz	cm²/g		days 2	days 7	days 28	(W/C)%	Initial	Final	(mm)	
1	95.5	4.5	-	3387	13	21.2	40.5	49.1	22.4	370	450	1	Lab
2	86.3	3.7	10	4218	13	17.8	36.7	44.3	23.4	355	435	1	Lab
3	95.5	4.5	-	3387	13	21.2	40.5	49.1	22.4	155	205	1	Sun
4	86.3	3.7	10	4218	13	17.8	36.7	44.3	22.4	135	185	1	Sun

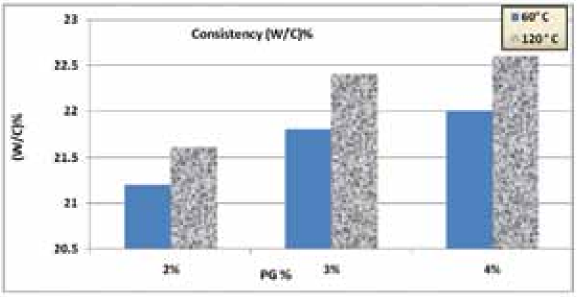
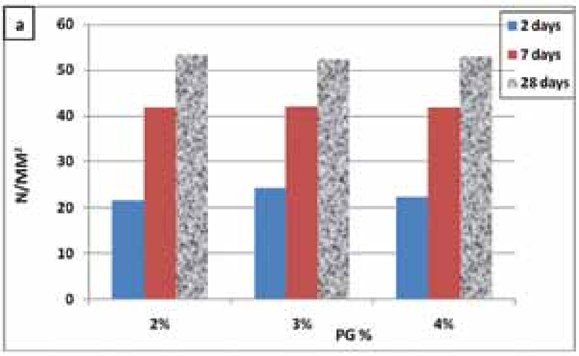


Figure 3: Consistency (W/C)% differences between PG1 & PG2, for the three PG mixing ratios.

3.2.4. Compressive Strength

Obviously, PG1 showed a higher compressive strength than PG2, Fig. (4). Also, PG1 showed an increasing compressive strength starting from 2 days up to 28 days, Fig. (4a). They showed very near compressive strength values to the OPC; the only difference was at 2 days curing time, as shown in Table (2). On the contrary, PG2 showed a decreasing trend in compressive strength, the lowest compressive strength was in the 4% mixing ratio, Fig. (4b). They showed different compressive strength values compared to those of OPC; all mixing ratios have lower values than those of OPC at all curing times, Table (4). This might be due to the increased demand of mixing water and hence higher W/C %.



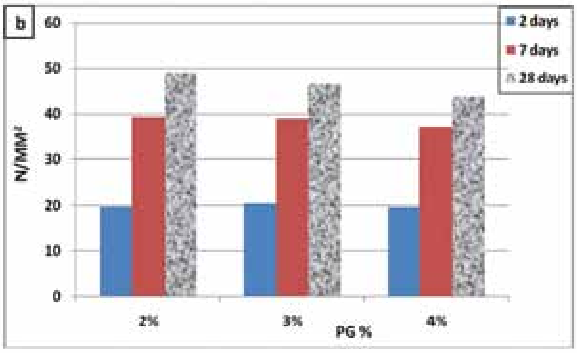


Figure 4: Plotting showing the Compressive Strength (N/mm2) differences between used PG (a) cold at 60°C (PG1) and (b) heated at 120°C (PG2), for the three PG mixing ratios.

3.2.5. Soundness (LE-CHAT)

The LE-CHAT values are shown in Tables (2, 3, 4 and 5); it maintained its value at 1 mm, which means that no volume changes were noticed, be it an expansion or a shrinkage. This is similar to LE-CHAT value of the OPC, which is an additional advantage of the suitability of using raw PG in cement mortar. This shows that the produced mortars show a perfect soundness with either PG1 or PG2 mixes. This is in agreement with the results reached by Bhadauria & Thakare (2006).

4. Cement mortars with Pozzolana

4.1. Setting time

In the second experiment that includes the addition of Pozzolana (10%) to the PG-clinker past, the addition of Pozzolana (Mixture No. 2) slightly lowered the initial and final setting time than the mixture without Pozzolana (Mixture No. 1). The effect of temperature was clear, when the same mixture was done under Sun (Mixtures 3 & 4), where the setting time dropped sharply, compared with those under room temperature (Mixtures 1 & 2), as shown in Fig. (5). Again the PG1 mixtures showed higher initial and final setting time in all mixtures. A very slight decrease in initial and final setting time for PG2 was noticed; this might be due to the dehydration of PG2 to anhydrite.

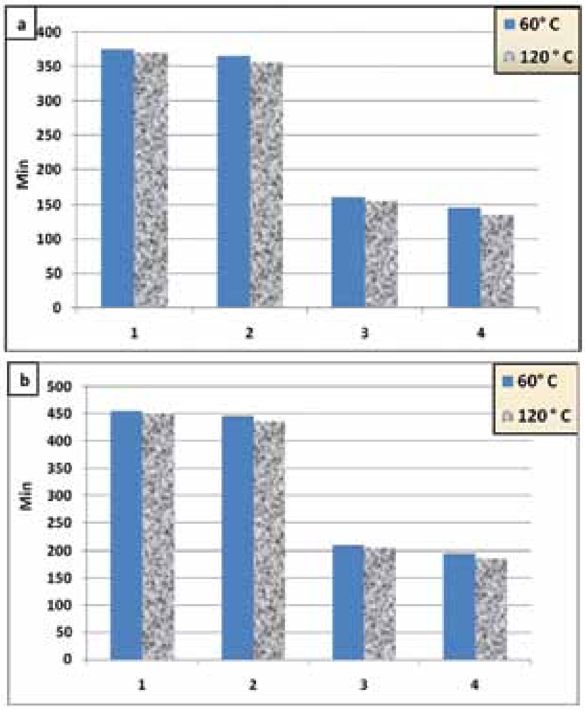


Figure 5: Plotting showing Initial (a) and Final (b) Setting Time (Minutes) difference between PG1 and PG2 for samples (1): Clinker 96.3 + PG 3.7 under room temperature (2): Clinker 86.3 + PG 3.7 + Pozzolana 10 under room temperature (3): Clinker 96.3 + PG 3.7 under Sun (4) Clinker 86.3 + PG 3.7 + Pozzolana 10 under Sun.

4.2. Consistency

The water consumption was also higher in the mixture that used Pozzolana (Mixture No. 2), and the PG2 has the higher (W/C%), as shown in Fig. (6). This behavior changed under the Sun temperature (mixture No. 4). Meanwhile, the mixture with Pozzolana showed the opposite behavior where PG1 (mixtures 1&3) had a higher (W/C%), whether at room temperature or under the sun. In general, the addition of Pozzolana (10%) is too large that decreases the clinker to (86.3%). Pozzolana addition to the mixture slightly increased the W/C% in PG2 that mixed at room temperature (No. 2). However, under the sun, pozzolanic mixture sharply dropped in W/C% as in mixture (No. 4), which might be due to the utilization of heated (PG2), as shown in Fig. (6).

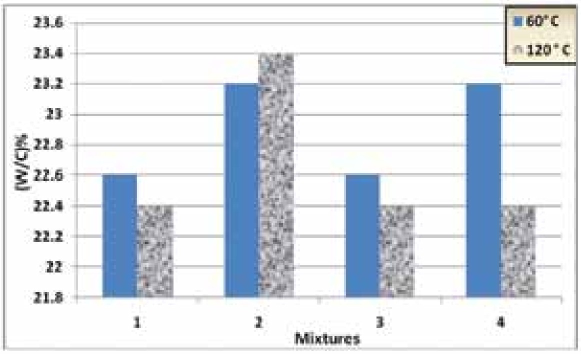


Figure 6: Consistency (W/C)% differences between PG1 & PG2, for mixtures (1): Clinker 96.3 + PG 3.7 under room temperature (2): Clinker 86.3 + PG 3.7 + Pozzolana 10 under room temperature (3): Clinker 96.3 + PG 3.7 under Sun (4) Clinker 86.3 + PG 3.7 + Pozzolana 10 under Sun.

4.3. Compressive Strength

Clearly, the addition of Pozzolana (10%) decreased the compressive strength of 2, 7 and 28 days for PG1 & PG2, Fig. (7). The same trend was noticed for mixtures solidifying either under room temperature or under the sun, as shown in Fig. (7). This might be due to the addition of more Pozzolanic materials (i.e., 10%), which sharply decreased the clinker share in the mortar, causing a decrease in the compressive strength.

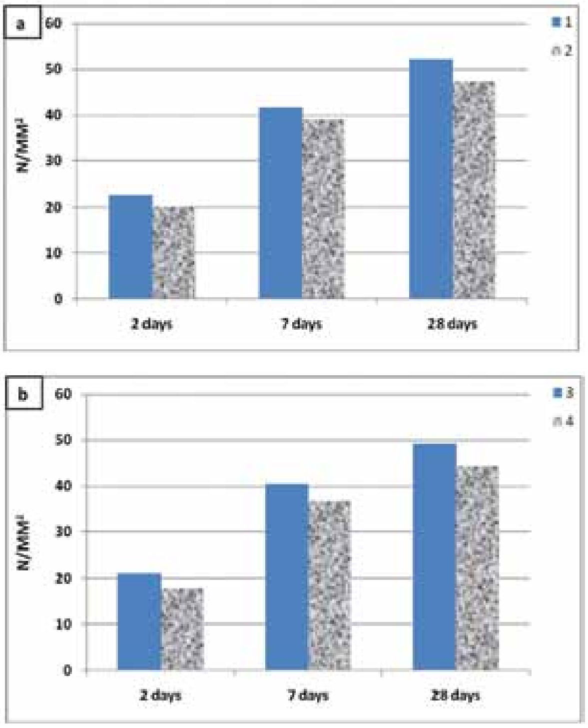


Figure 7: Plotting showing the Compressive Strength (N/mm2) differences between PG1 & PG2, for mixtures (1): Clinker 96.3 + PG 3.7 under room temperature (2): Clinker 86.3 + PG 3.7 + Pozzolana 10 under room temperature (3): Clinker 96.3 + PG 3.7 under Sun (4) Clinker 86.3 + PG 3.7 + Pozzolana 10 under Sun.

5. Conclusions

Colder phosphogypsum (PG1) can easily and successfully replace gypsum in the production of ordinary cement. The setting time (i.e., workability), consistency and compressive strength showed an acceptable value. On the contrary, heated phosphogypsum (PG2) showed an imperfect setting time, consistency and compressive strength. The addition of 10% pozzolana showed a negative impact because of its large mixing ratio. In general, pozzolanic material is added with certain percentages as mineral admixtures to cement in order to increase the compressive strength of the concrete. In our case. Pozzolana was added on the expense of clinker, which resulted in decreasing the compressive strength of the tested samples. The cement mortars using PG had similar Blaine and pH values to OPC. Moreover, they showed perfect soundness values.

Experimental results recommend the use of raw PG without treatment (heating) in cement production. This will eliminate a serious environmental source of pollution; besides, it will decrease the cost of cement production. It is also recommended that further investigations be executed to explore the possibility of extracting gypsum from PG that can be used directly in cement industry.

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