The Use of Phosphogypsum for Soil Bricks Manufacturing as an Alternative for Its Sustainable Destination

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Abstract

This research studies soil-compacted bricks using Phosphogypsum (PG) in their dosage. Aiming to assess the physical characteristics of these bricks, laboratory tests were performed. To obtain the bricks, two dosages were used: 4 and 7% of Phosphogypsum (PG) concentration. Bricks with no Phosphogypsum (PG) in their mixture were also assessed as a benchmark. The brick's physical characteristics were obtained for bricks dried by the sun and in an oven at 900^{o}C for 96 hours. The results of the laboratory tests were analyzed through statistical analysis to explore the differences between the means for each studied condition (dosage and drying method). We found no statistically significant difference between the mean strength of bricks with 4% of Phosphogypsum (PG) in their dosage and bricks with no PG. Beyond 4% of Phosphogypsum (PG) dosage, the brick's strength presented a reduction. For all dosages, bricks dried in the oven showed higher strength than bricks dried by the sun. However, according to the Brazilian Technical Standards, all studied bricks presented enough strength to be used in regular constructions. We conclude that Phosphogypsum (PG) for brick manufacturing can be an alternative way for its disposal, which can help mitigate the civil construction environmental impacts.

1. Introduction

Civil construction has become a prominent consumer of natural resources, among all economic activities, due to its constant growth, mainly associated with urban areas. Many researchers have attempted to develop viable solutions to reduce the environmental impact of civil construction. Among these solutions, the use of (industrial) solid wastes for replacing natural raw materials for manufacturing construction materials can be pointed out. However, industrial solid waste needs particular care in its management and disposal since it is often associated with potentially polluting substances [18], [29] To reduce the generation of industrial solid wastes, it is essential to maintain proper management of the manufacturing processes, including destination and final disposal of those which sustainable principles should drive [22].

Among industrial solid waste, phosphogypsum (PG) is the subproduct of the primary raw material used by the fertilizer industry: phosphoric acid [11]. According to [12] and [2], PG can be classified as a Naturally Occurring Radioactive Material (NORM), which means that its reuse may pose risks to humans and the environment from the radiological protection point of view. PG (which is mainly composed of calcium sulfate dihydrate) consists of a waste that is generated by the production of phosphoric acid by wet processing of the phosphoric rock, formed mainly by apatite mineral ($Ca_5(PO_4)_3OH$)) with sulphuric acid. The PG is taken to a suspension with water and pumped to storage rafts, where it decants and dries out. The chemical equation of phosphoric acid production is as follows [26] [2]:

$$Ca_5F(PO_4)_3+5H_2SO_4 + 10H_2O \rightarrow 3H_3PO_4 + 5CaSO_4.2H_2O + HF \quad (1)$$

Based on this equation, nearly 5 tons of PG ($CaSO_4.2H_2O$) are produced from 1 ton of phosphoric acid (H_3PO_4) [14][17] [30] [23]. PG consists of a powdery material with little or no plasticity, with particle density between 22.7 and 24.0 kN m^{-3} and bulk density varying among 9.0 and 17 kN m^{-3} [28]. From the morphological point of view, PG is characterized by a crystal structure, mostly in rhombic and hexagonal forms. PG also contains impurities as H_3PO_4, $Ca(H_2PO_4)$ H_2O, $CaHPO_42H_2O$ and $Ca_3(PO_4)_2$, residual acids, fluorides (NaF, NaF_2SiF_6, Na_3AlF_6, Na_3FeF_6, and CaF_2), sulfate ions, trace metals, and organic matter as aliphatic compounds of carbonic acids, amines, and ketones, adhered to the surface of gypsum crystals [23].

Due to its low economic value for companies, PG usually ends up being landfilled or discharged into the environment without any prior treatment, thus resulting in environmental contamination and pollution of soil and water, including seawater [3]. An estimated 100–280 million tons of PG are generated worldwide per year. Only 15% of these are recycled as soil stabilization amendments, fertilizers, and building materials, mainly due to their strong acidity ($pH < 3$) and high moisture content [3] [2] [30][28] [23]. Approximately 3 billion tons of PG are stored in deposits of different sizes in over 50 countries [12]. Due to the possible acid and heavy metals infiltration, storage of PG may cause pollution of soils and waters [1]. The storage and management of PG are considered the main challenges facing the phosphoric acid production industries worldwide. They require the mobilization of significant resources and occupy large land areas [3].

In Brazil, PG is a severe environmental liability. The amount of PG generated as waste is about 4 to 6 times higher than the amount of phosphoric acid produced, making the storage and disposal of this waste product a challenge, especially for fertilizer industries. According to [12], Brazilian production of PG reaches 12 million tons per year. The industrial complex of the city of Uberaba/Brazil, the biggest producer of phosphoric acid in Brazil, generates more than 3 million tons of PG per year: the PG waste is stored in a 1 million m^2 area, in piles that are 30 m high [13]. Another fertilizer industry in the city of Catalão produced in 2008 an amount of 600.000 tons of PG that is disposed of in landfills. [13] comments that paper and cement industries and agriculture have reused only 10% of the PG produced by this industry.

The interest in PG as a source of secondary raw materials has increased over the past decade [15]. Initially, PG was considered mainly a component for construction, cement, road-building, and agricultural industries [17] [[29] [30]. However, over the past 10–20 years, the focus has shifted, given the increase of anthropogenic pressure on the environment and the resulting shortage of natural sources of raw materials. PG, which has many valuable elements, is considered a source of calcium, phosphorus, rare-earth elements, trace elements, and a mineral resource in technological, environmental protection processes. Research increasingly focuses on finding reliable and efficient ways to manage and reuse PG, especially civil construction [3] [17]. An assessment of its radiological impact is required, mainly due to the radionuclides content and radon exhalation [12]. [13] points out that the use of PG for civil construction purposes can be a valuable way to reduce the environmental impacts caused by this economic sector. Like [18], many authors point out that PG can be used for alternative construction materials manufacturing, including bricks, tiles, and mortar.

Several studies have explored the use of PG in the base and sub-base of roads and embankments and as a final layer of earthworks to improve soil properties, minimize the possible environmental impacts caused by the disposal, and insert a new material on the market. However, the primary use of PG remains in cement manufacturing, where it substitutes natural gypsum (about 5%) [1] [16]. Researchers have studied the possibility of using PG in construction materials such as raw blocks and fired bricks, and promising results have been found [1] [30]. PG can be reused with fly ash and Portland Cement in the building industry [29]. Such results suggest that PG can become an alternative raw material for the civil construction sector, reducing the impact of landfills close to the chemical industries.

For such applications, the natural radioactivity of PG, mainly from ^{226}Ra, remains a challenge [19]. Other radioactive elements derived from phosphate rocks may be present, such as Pb^{210}, Po^{210}, U^{238}, and U^{234} [20]. PG that exceeds 370 Bq kg^{-1} (10pCig^{-1}) of radioactivity has been banned from all uses by the USA Environmental Protection Agency (EPA) since 1992 [20]. The European Atomic Commission (EURATOM) prescribed a limit of 500 Bq kg^{-1} (13.5 pCig^{-1}). Despite such characteristics, however, [20] points out that PG cannot be classified as toxic waste since PG elements are not corrosive and the average total concentration of elements classified as toxic (e.g., Ba, As, Cr, Cd, Hg, Pb, Se, and Ag) by the USA Environmental Protection Agency is lower than the EPA allowable limits for toxic, hazardous waste.

This study characterizes the physical parameters of fired and non-fired bricks produced with PG in their dosage. It also studies the influence of PG dosage on these physical characteristics. The main objective is to evaluate the potential use of PG as an alternative construction material, thus reducing the environmental impacts caused by this solid waste and civil construction activities.

2. Methodology

2.1 Laboratory tests for determination of physical properties of soils and bricks

A representative soil sample was collected at 1.5 m depth from the Experimental Field for Soil Mechanics in the University Nove de Julho, São Paulo, Brazil. The laboratory tests were carried out at the Soil Mechanics Laboratory and the Civil Construction Materials Laboratory at Universidade Nove de Julho, in São Paulo, Brazil.

Testing soil samples with different PG proportions were prepared: soil + 0% PG, soil + 4% PG, and soil + 7% PG. To characterize the collected soil and the prepared testing samples, granulometric analysis tests and determination of consistency limits were conducted according to Brazilian Association of Technical Standards (ABNT NBR) ABNT NBR 7181:2018, ABNT NBR 6459:2017, and ABNT NBR 7180:2016, respectively. After the tests, the testing samples were classified by the Unified Soil Classification System (ASTM D 2487–06).

The PG material was donated by the Energy and Nuclear Research Institute (Ipen) in plastic bags sealed in dihydrate form. The PG producer's name will be omitted in this paper; however, it is located in the Minas Gerais/Brazil. Before its use, the PG underwent a drying process in an oven at a temperature of 100°C for 24 hours. Because of this drying, the PG was transformed from dihydrate to hemihydrate. The samples were prepared according to ABNT NBR 6457:2016.

After classification, compaction tests were carried out with normal proctor energy for each of the mixtures (soil + 0% PG, soil + 4% PG, and soil + 7% PG) according to ABNT NBR 7182:2016 in order to determine the maximum dry bulk density and the optimum humidity of the mixtures under study.

After that, solid bricks were compacted in each of the studied dosages. Sixty bricks were compacted, 30 were dried in the open air, and 30 were burned in an oven at 900°C for 96 hours. The bricks were subjected to compressive strength as recommended by ABNT NBR 13279:2005.

To analyze the results, three groups were considered: a control group (soil + 0% PG) and two experimental groups (soil + 4% PG and soil + 7% PG).

Descriptive statistical methods were used for data analysis, verification of data adherence to the normal distribution (Kolmogorov-Smirnov and Shapiro-Wilk tests), homogeneity analysis tests of variances (Levene test), ANOVA, and post hoc Tukey's HSD test. For data not adhering to the normal distribution, the Kruskal-Wallis non-parametric test was used, and the Mann-Whitney test was performed as a post-hoc test.

2.2 Determination of activity concentration of radionuclides and effective annual dose of radiation due to external exposure

All building materials contain varying amounts of natural radionuclides. In the case of PG, this material contains a concentration of natural radionuclides, which, depending on its magnitude, can cause an increase in the radiation dose received by users of a dwelling is used as a building material [27].

The assessment of the effective annual dose inside a residence was established by the methodology proposed by [24] [25], based on the concept of a standard room [27]. The absorbed dose is calculated using the following equation:

$$DR = (qRA.\, CRA + qTH.\, CTH + qk.\, CK).\, mi$$

2

Where DR = rate of dose absorbed in the air (nGy.h-1); qRA, qTH, qK = conversion factors for the concentration of ^{226}Ra, ^{232}Th, and ^{40}K, respectively; CRA, CTH, CK = activity concentration of ^{226}Ra, ^{232}Th, and ^{40}K, respectively; mi = percentage fraction of mass of "i" type building material in a standard room.

The following equation can calculate the effective annual external dose:

$$E = DR.T.f.FCD.0.000001$$

3

where E = effective annual dose due to external exposure (mSv a^{-1}); DR = rate of dose absorbed in air ($nGy.h^{-1}$); T = 8760 hours / year; f = occupancy factor of the residence; DCF = conversion factor from dose absorbed in air to effective dose (Sv Gy^{-1}).

3. Results And Discussions

3.1 Physical characterization of the materials

The properties of the soil samples are shown in Table 1. According to the Unified Soil Classification System, the soil samples can be classified as a CH type.

Table 1
The properties of the test soil.

Average properties	**Soil Sample**
Classification (USCS)	CH
% passing through sieve #200	> 50%
Liquid Limit (LL)	52%
Plasticity Index (PI)	29%
Optimum water content	27.6%
Maximum dry specific weight	14.9 kN m^{-3}
Specific weight	27.0 kN m^{-3}
Average properties	**Soil + 4% PG**
Liquid Limit (LL)	50%
Plasticity Index (PI)	17%
Optimum water content	27.0%
Maximum dry specific weight	14.8 kN m^{-3}
Average properties	**Soil + 7% PG**
Liquid Limit (LL)	47%
Plasticity Index (PI)	15%
Optimum water content	25.0%
Maximum dry specific weight	14.8 kN m^{-3}

Table 2 shows the activity concentration of ^{226}Ra, ^{232}Th, and ^{40}K obtained are shown next in the PG material, as measured by the researchers.

Table 2
From samples, activity concentrations of ^{226}Ra, ^{232}Th, and ^{40}K (Bq kg^{-1}) were taken.

Sample	**^{226}Ra**	**^{232}Th**	**^{40}K**
Phosphogypsum A	1277 ± 39	445 ± 14	< 26
Clayey Soil (CH)	64 ± 2	120 ± 6	155 ± 23

3.2 Ultimate compressive strength obtained for the fired bricks

The ultimate compressive strength values are shown in Table 3.

Table 3
Results of ultimate compressive strength for each PG dosage.

Dosage	N	Mean (MPa)	Std. Deviation	Std. Error	95% Confidence Interval for Mean		Minimum (MPa)	Maximum (MPa)
					Lower Bound	Upper Bound		
0%	10	0.73	0.11	0.05	0.58	0.88	0.60	0.87
4%	10	0.68	0.12	0.05	0.52	0.83	0.53	0.82
7%	10	0.39	0.05	0.02	0.32	0.46	0.30	0.46
Total	30	0.60	0.18	0.04	0.50	0.70	0.30	0.87

Table 3 shows that the ultimate compressive strength is reduced when the proportion of PG increases. To verify if this behavior is statistically significant, an ANOVA analysis was performed after the Kolmogorov-Smirnov and Shapiro Wilk tests did not reject normal distribution ($p > 0.05$), and Levene's test did not reject the hypothesis of equal variances ($p > 0.05$).

The ANOVA showed a significant difference among the studied groups ($p < 0.005$). Figure 1 shows the differences among the groups. The groups 0%PG and 4%PG showed no statistical difference ($p > 0.05$). Thus, it can be stated that PG concentrations until 4% caused no influence on the bricks' compressive strength. Meanwhile, when these groups are compared to the 7% PG group, a significant difference in the compressive strength can be found, indicating a lower compressive strength for this group.

3.3 Ultimate compressive strength obtained for the non-fired bricks

The results for the ultimate compressive strength of non-fired bricks are shown in Table 4.

Table 4
Results of ultimate compressive strength for each dosage in non-fired bricks

Dosage	N	Mean (MPa)	Std. Deviation	Std. Error	95% Confidence Interval for Mean		Minimum (MPa)	Maximum (MPa)
					Lower Bound	Upper Bound		
0%	10	3.64	0.73	0.32	2.72	4.55	2.6	4.3
4%	10	2.92	0.22	0.10	2.63	3.20	2.6	3.2
7%	10	2.46	0.27	0.12	2.12	2.79	2.1	2.8
Total	30	3.00	0.66	0.17	2.63	3.37	2.1	4.3

The ultimate compressive strength was lower at a high percentage of PG. Kolmogorov-Smirnov and Shapiro Wilk tests were run and showed $p > 0.05$. However, according to Levene's test, the variances are not homogeneous. Thus, the Kruskal-Wallis test was performed. Figure 2 shows the box-plot chart for ultimate compressive strength for each dosage.

The results point to a significant difference among the studied groups ($p < 0.05$), indicating that the PG percentage in each group influence the non-fired bricks' ultimate compressive strength. The Mann-Whitney posthoc test was run, and three conditions were taken into account: 0%PG versus 4%PG; 0% PG versus 7%PG and 4% PG versus 7% PG. The test returned the following results: $s = 0.15$ ($p > 0.05$), $s = 0.01$ ($p < 0.05$) and $s = 0.013$ ($p < 0.05$) respectively. These point to no difference between 0%PG and 4%PG and statistically significant differences between these groups and 7%PG. Thus, it can be stated that the compressive strength does not present any notable influence until 4% of PG concentration; however, between 4 and 7% of PG concentration, the compressive strength decreases.

3.3 Ultimate compressive strength obtained for the fired bricks

The average compressive strength obtained for fired bricks is shown in Table 5.

Table 5
Results of ultimate compressive strength for each dosage in fired bricks.

Dosage	N	Mean (MPa)	Std. Deviation	Std. Error	95% Confidence Interval for Mean		Minimum (MPa)	Maximum (MPa)
					Lower Bound	Upper Bound		
0%	10	12.92	1.64	0.73	10.87	14.96	11.5	15.4
4%	10	13.72	2.05	0.92	11.16	16.27	12.3	17.3
7%	10	4.86	0.88	0.39	3.76	5.95	3.9	6.1
Total	30	10.5	4.40	1.13	8.06	12.93	3.9	13.3

The compressive strength decreases for a high PG percentage. The Kolmogorov-Smirnov and Shapiro Wilk tests did not reject normality ($p > 0.05$), but Levene's test showed non-homogeneous variances. Thus, the Kruskal-Wallis test was performed. Figure 3 shows the box-plot chat for ultimate compressive strength for each dosage.

According to the Kruskal-Wallis test, the PG percentages in each group influenced the compressive strength of the fired bricks. The trend of strength reduction when PG percentage gets higher was verified again. The Mann-Whitney posthoc test was performed for three comparisons: 0%PG versus 4%PG; 0% PG versus 7%PG and 4% PG versus 7% PG. The results of the test were: s = 0.10 ($p > 0.05$), s = 0.01 ($p < 0.05$) and s = 0.012 ($p < 0.05$) respectively. These point to no difference between 0%PG and 4%PG and statistically significant differences between these groups and 7%PG. Thus, it can be observed that the compressive strength percentage does not present any notable influence until 4% of PG; however, between 4 and 7% of PG concentration, the compressive strength decreases.

3.4 Comparison between ultimate compressive strength obtained for fired and non-fired bricks.

After determining the compressive strength values for non-fired and fired bricks, they were compared among themselves. Thus, a test for paired samples was carried out to determine if the fired process used for the bricks influenced the compressive strength of the studied bricks. The performed test results are shown in Table 6.

Table 6
Test of ultimate compressive strength for paired samples of fired and non-fired bricks.

Pair	Description	Mean	Sd.	Std. error mean	95% Confidence Interval of the Difference		t	df	s
					lower	upper			
1 (0%PG)	CNFx CF	-9.28	1.51	0.67	-11.16	-7.39	-13.6	4	**0.000**
2 (4%PG)		-10.8	1.87	0.83	-13.12	-8.47	-12.8	4	**0.000**
3 (7%PG)		-2.40	0.83	0.37	-3.43	-1.36	-6.40	4	**0.003**

CNF - Ultimate compressive strength for non-fired bricks; CF - Compressive strength for fired bricks.

Table 6 shows that the difference between the two conditions' means was large enough not to be random. The table provides the difference in the means between the scores: Pair 1 = -9.28; Pair 2 = -10.80; Pair 3 = -2.40. The table also shows the standard deviation of the differences and the standard error between the specimens' scores in each condition. If the values of t are negative, the average of the compressive strengths without oven is less than the average of the compressive strengths with the oven. Therefore, it is concluded that after the specimens pass through the oven, they present greater resistance to compression. The 95% confidence intervals do not contain zero (both limits are negative), indicating that the value of the average difference is unlikely to be zero. Therefore, one can be confident that the data does not represent random samples from the same population. To calculate the effect size r and to convert a t-value to an r-value, the following equation was used:

$$r = \sqrt{\frac{t^2}{t^2 + gl}}$$

4

The effect size values r for all three pairs are > 0.95, indicating a substantial effect (above 0.5). Therefore, in addition to being statistically significant, this effect is also large.

3.5 Effective dose of external exposure

The determination of the activity concentration of ^{226}Ra, ^{232}Th, and ^{40}K (Bq kg^{-1}) and the values of DR and E were carried out for the material studied with the highest dosage of PG, that is, 7% concentration. The values obtained are presented in the following Tables.

Table 7
The samples were concentrated at ^{226}Ra, ^{232}Th, and ^{40}K (Bq kg^{-1}).

Sample	Concentrations (Bq kg^{-1})		
	^{226}Ra	^{232}Th	^{40}K
Soil + PG (7%)	173 ± 6	146 ± 7	159 ± 23

Table 8
Average value of DR (nGy.h^{-1}) and E (mSv a^{-1}) obtained for brick with 7% PG.

Sample	DR (nGy.h^{-1})	E (mSv a^{-1})
Soil + PG (7%)	21.06	0.10

The values of E per year recommended by CNEN - Brazilian National Nuclear Energy Commission [27], by the European Commission, and by UNSCEAR - United Nations Scientific Committee on the Effects of Atomic Radiation are, respectively, E = 1.0 mSv; E = 0.3 mSv and E = 0.48 [27]. The material studied in this research had an E value lower than those recommended by these institutions.

4. Conclusions

From the results obtained in the tests carried out in this research, it can be concluded that:

For the flexural, compressive tests, the strength showed a downward trend as the concentration of PG in the mixtures increased; however, when analyzing these data statistically, it is possible to notice that the specimens with soil mixtures with 4% PG presented mean values similar to those obtained for the specimens without the addition of PG.

Concerning the compressive strength tests, the specimens were analyzed without using the oven and using the oven. It is possible to identify the same tendency presented in the flexural compressive tests from the obtained data. When analyzing the specimens statistically with mixtures of soil with 4% PG, it is noticed that they do not present significant differences when compared with the specimens without PG. The conclusions for the specimens that went to the oven are the same for the specimens without the oven.

Although mixtures of soil with 4% PG are statistically equivalent to mixtures without the addition of PG, mixtures of soil with 7% PG, which showed worse properties in the tests, can also be used. The average of the compressive strengths (with oven) meets the requirements of the ABNT NBR 15270-1 / 2017 standard for the manufacture of ceramic blocks and bricks for masonry. According to the standard, the mixture with a concentration of 4% PG can be used for solid bricks for the sealing of class 40. This mixture can also be used for solid structural bricks of classes 60, 80, 100, and 120. In the case of mixtures with a concentration of 7% PG, it can be used for solid bricks for the sealing of class 40.

The study results indicate that the possibility of reducing PG waste by incorporating it in the manufacture of bricks may be viable, representing a sustainable solution to reduce the number of raw materials extracted for such use.

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Figures

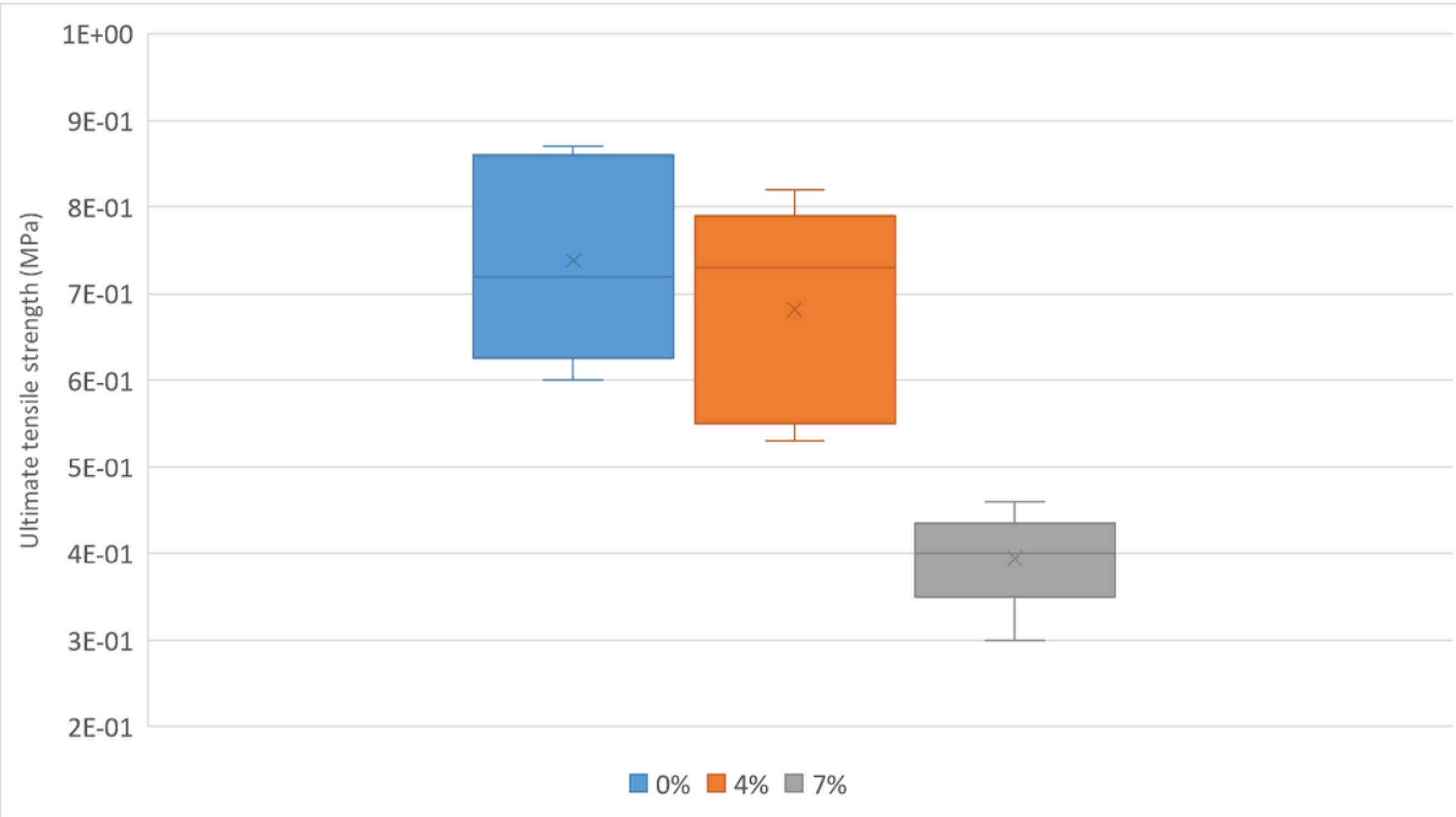

Figure 1

Box-plot chart for ultimate compressive strength for each dosage.

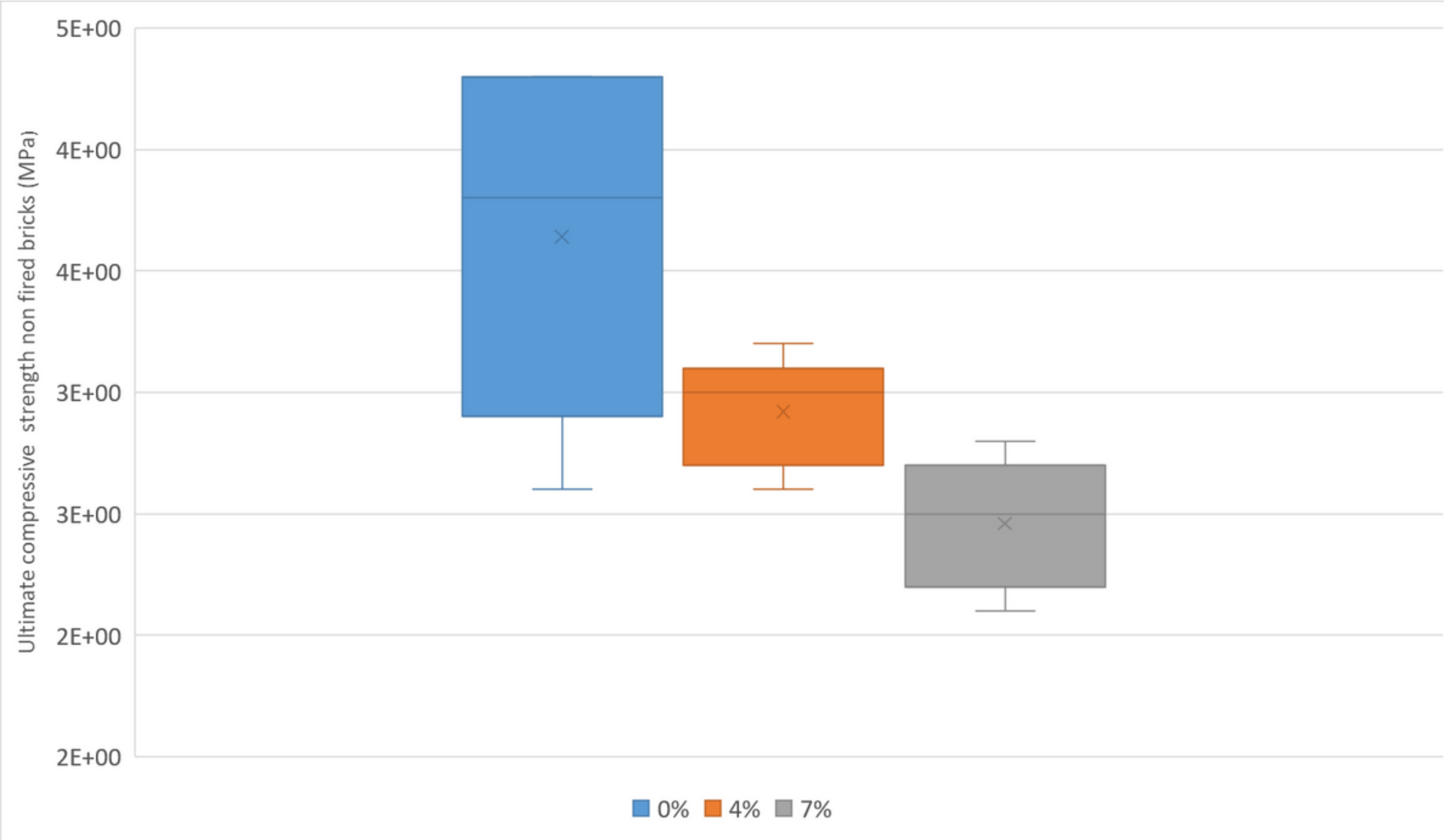

Figure 2

Box-plot chart for ultimate compressive strength for each dosage.

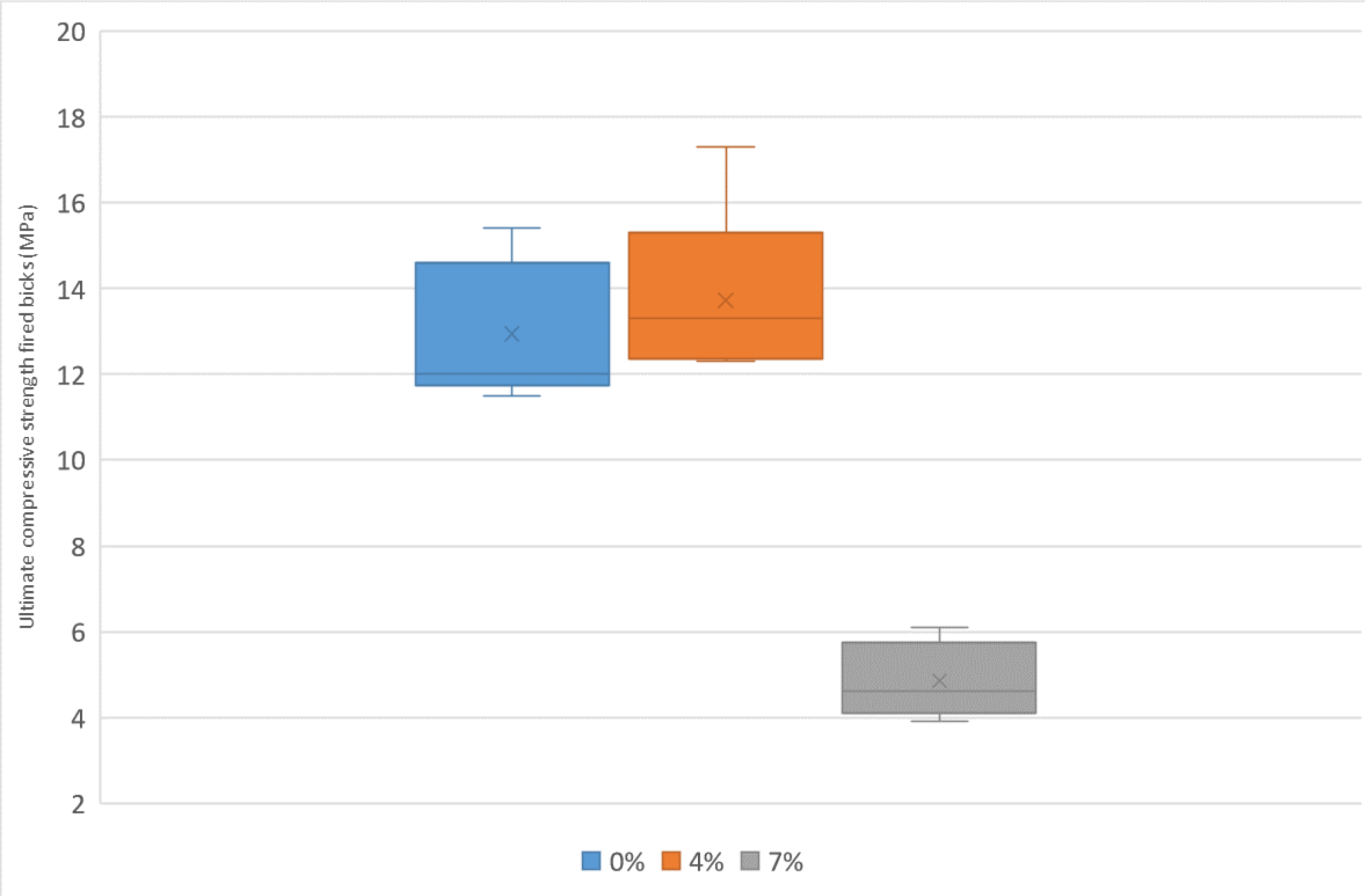

Figure 3

Box-plot chart for fire bricks’ ultimate compressive strength for each dosage.

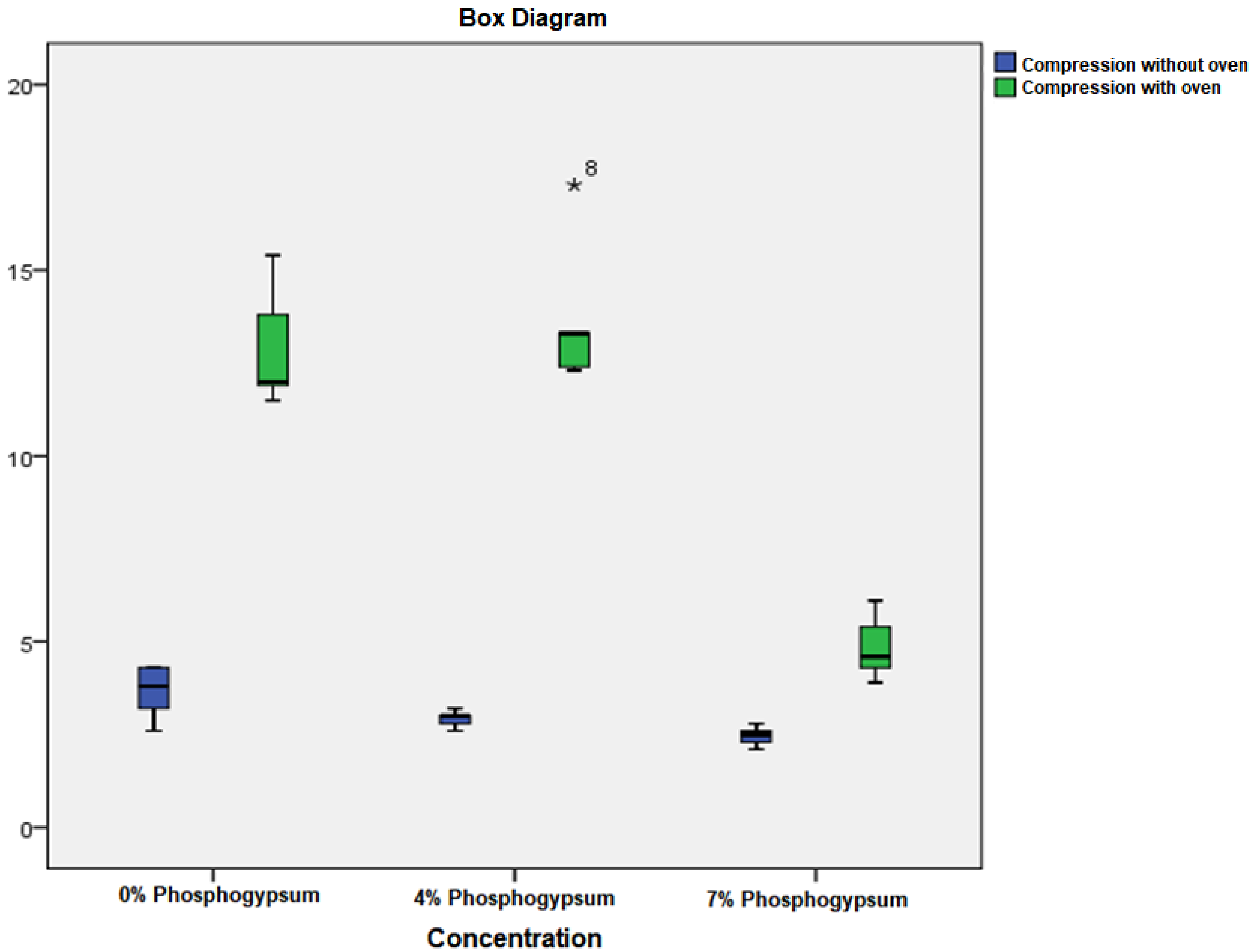

Figure 4

Comparison between compressive strength (MPa) of non-fired and fired bricks.