Thermal and Mechanical Properties Enhancement of Cement Mortar using Phosphogypsum Waste: Experimental and Modeling Study

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ABSTRACT

This research presents an in-depth investigation into the application of phosphogypsum (PG), a by-product of phosphate fertilizer plants and chemical industries, as a replacement material for cement in mortar, with a focus on enhancing its thermal and mechanical properties. The influence of PG as a partial replacement for cement on the compressive strength of mortar after 3, 7, and 28 days is investigated. Utilizing the Box-Behnken design within Response Surface Methodology, this study analyzed factors, such as sulfuric acid concentration, washing time, calcination temperature, and PG to cement ratio. Results indicate that optimal PG levels enhance mortar strength, particularly at 28 days, through sustained ettringite formation and microstructure optimization. Sulfuric acid concentration and calcination temperature were identified as the most significant elements influencing compressive strength, with the latter improving PG quality and reactivity. A PG to cement ratio up to 10% was found beneficial, while washing time had a negligible effect. The research highlights a critical synergy between the sulfuric acid concentration applied during the purification of PG and the calcination temperature. A significant improvement of 21% in compressive strength was achieved, underscoring the combined effect of chemical and thermal treatment on PG's efficacy in mortar. The increased sulfuric acid concentration is presumed to purify the PG by removing impurities, thus improving its reactivity. Concurrently, calcination alters the PG's crystalline structure and diminishes its organic composition. This interdependent optimization is instrumental in enhancing the structural integrity of PG-modified mortar. The potential for raw PG to be used as an insulating material is more pronounced at higher replacement rates (10%), while sulphuric acid treated PG (SCPG) and heat treated PG (HTPG) seem to be unable to provide a clear insulative advantage.

Keywords-waste management; phosphogypsum; thermo-mechanical improvement; cement mortar; RSM modeling

I. INTRODUCTION

The use of solid waste as a partial replacement in cement mortar has emerged as an interesting subject, not only regarding the construction industry, but also for environmental reasons [1-3]. The global concrete market size was valued almost half a trillion dollars in 2020 and should grow about 5% each year until 2028 based on forecasts, strongly indicating a huge possibility for utilizing solid waste as a partial replacement in cement mortar [4]. Among all solid wastes, fly ash is one of those that have received wide attention [5]. It has been established through several investigations that fly ash can improve workability and durability along with decreasing permeability and CO_2 emission potential of cement mortar [6-8]. Silica fume is a by-product of silicon alloy manufacture that improves strength and durability within cement mortar since it contains highly reactive low size pozzolanic particles [9-11]. Industrial slags, such as blast furnace slag and steel slag, have also been investigated [12]. These materials can enhance the mechanical properties and durability of cement mortar, especially when finely ground [13]. Recycled Concrete Aggregate (RCA), obtained from demolished concrete structures, has been used as a replacement for natural aggregate in cement mortar [14, 15]. It is estimated that the construction industry globally generates around 1.3 billion tons of Construction and Demolition Waste (CDW) per year [16]. Other types of CDW, such as crushed bricks and tiles, have also been used as partial replacements in cement mortar, contributing to waste reduction and recycling in the construction industry, and providing satisfactory performance [17]. The use of solid waste in cement mortar can lesd to the development of sustainable and environmentally friendly construction materials, but the properties and performance of the resulting mortars depend on the type and proportion of the waste used, as well as the specific mix design and curing conditions. Further research is needed to optimize these factors and to assess the long-term performance and environmental impact of these mortars.

The exploration of phosphogypsum (PG), a by-product of the phosphorous fertilizer industry, as a potential substitute for a portion of cement in concrete has garnered interest in recent years [18-20]. Each year, the production of PG by the fertilizer industry surpasses 100 million tons globally [21]. Its physical and chemical characteristics are similar to those of natural gypsum, which makes it a promising option for industrial uses. Early research indicated that substituting 5% of cement with PG could enhance the setting time and increase the compressive strength of cement by nearly 10%, thereby establishing a basis for future studies [22]. Further building on this premise, a more detailed study investigated the effects of substituting 10% - 40% of cement with PG, and found that a 20% replacement could enhance the compressive strength of concrete by as much as 15% [23]. PG may serve as a viable substitute for cement mortar, potentially improving the structural properties of concrete. However, the radioactivity of PG, due to radionuclides, such as Ra-226 and Th-232, has been a significant concern, limiting its broader usage. Authors in [24] sought to establish the safe levels of PG in cement, concluding that radioactivity could be kept within safe limits — typically less than 370 Bq/kg for Ra-226 and less than 200 Bq/kg for Th-232 — with a replacement level of up to 10%. This conclusion paved the way for the safer application of PG in concrete.

As cement production contributes to roughly 7% of global CO_2 emissions, any decrease in the usage of cement could have a substantial environmental benefit [25, 26]. Authors in [27] found that substituting cement with PG could lead to a reduction in these emissions by as much as 10%, suggesting a more eco-friendly approach to concrete production. Presently, ongoing research aims to further enhance the application of PG in mortar. In conclusion, existing studies indicate that PG holds promise as a viable substitute for a portion of cement in mortar, contributing to both improved structural characteristics and significant reductions in CO_2 emissions. However, the radioactivity associated with PG needs to be thoroughly understood, and additional research is needed to refine its application, ensuring safety and efficiency. Regardless of the noteworthy number of research studies on the PG use to ameliorate mechanical properties, few studies have focused on experimental condition optimization of the partial replacement of cement mortar employing PG. In this context, Response Surface Methodology (RSM) design modeling stands out as a valuable analytical technique for investigating the complex effects of various PG processing methods, namely chemical treatment, washing, and calcination and how they interact to improve compressive strength. By conducting a detailed investigation of the individual and combined effects of chemical, washing, and calcination treatments, this study will pinpoint the most effective conditions for significantly boosting compressive strength.

II. EXPERIMENTAL PART

The methodology encompasses the collection and preparation of PG samples and the formulation of various mortar mixtures. The mortar's compressive strength is measured, with the data collected serving as a basis for analysis and comparison against traditional mortar mixtures. RSM is used to refine the processing of PG for cement mortar enhancement. It investigates three PG variants: raw, untreated (RPG), water-washed and acid-treated (CPG), and heat-treated (HTPG). Ordinary Portland Cement (OPC) and well graded sand are used to ensure uniformity in the mortar mixtures with varying ratios of PG to cement.

A. Materials and Methods

PG is the main substance of the current investigation. Table I exhibits the chemical composition of natural gypsum and PG.

TABLE I. CHEMICAL COMPOSITION OF NATURAL GYPSUM AND PHOSPHOGYPSUM.

	Gypsum	**PG**
P_2O_5 (%)	0.08	1.2
Fe_2O_3(%)	0.29	0.13
MgO (%)	8.23	0.26
Al_2O_3(%)	0.89	0.21
SO_3(%)	31.26	43.8
CaO (%)	34.2	30.7
K_2O (%)	0.15	0.08
Na_2O (%)	0.09	0.03
SiO_2(%)	5.1	3.5
F (%)	0.09	1.9
TOC (%)	0	0.64
Cl (ppm)	269	399

The study employs PG in three forms. RPG, is used in the condition it is found within industrial disposal areas. In contrast, the cleaned form CPG is subjected to an intensive purification process including water washing (WCPG) and sulphuric acid treatment (SCPG). In the third form, PG undertakes heat treatment (HTPG) that eliminates contaminants and moisture.

The protocol, presented in Figure 1, is designed to explore a series of treatment methods aimed at purifying and enhancing the quality of PG. It includes procedures for acid treatment using sulfuric acid (SCPG) at different concentrations, water washing (WCPG) to remove soluble impurities, and calcination (HTPG) at varying temperatures to alter the physical and chemical properties of PG.

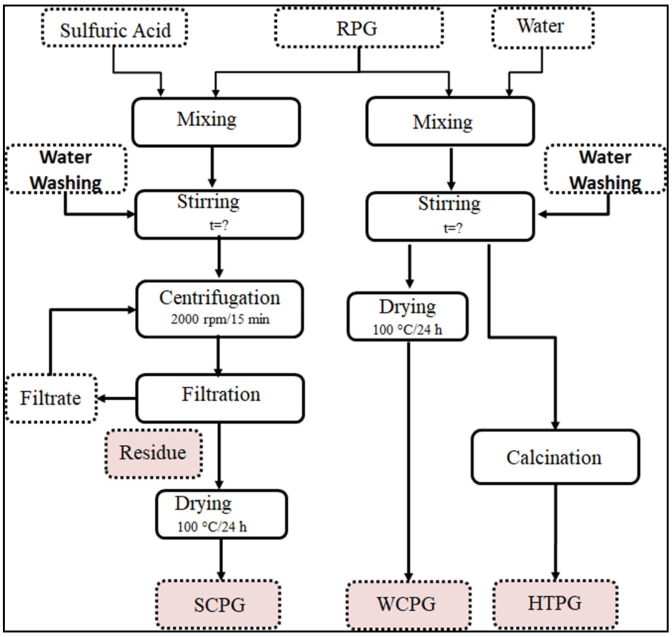

Fig. 1. Protocole of PG treatments.

- Acid Treatment with H_2SO_4: The treatment of raw PG with sulfuric acid is performed at varying concentrations to remove impurities. PG is, soaked, stirred , filtered, rinsed until neutral, and dried at 100°C.
- Water Washing: The Basic Water Washing (BWW) method consists of agitating 100 grams of raw PG in 1 lt of distilled water for 1, 2, and 3 hr to dissolve soluble impurities. Post-washing, the PG is filtered, repeatedly rinsed until the pH is neutral, and then dried at 100°C to remove all water content.
- Calcination: Calcination treatments are applied to PG post-treatment to further modify its properties. The Low-Temperature Calcination (LTC) is conducted at 100°C for 1 hr, focusing on dehydrating the PG without altering its chemical structure significantly. Medium-Temperature Calcination (MTC) is performed at 400°C for 2 hr. The temperature is higher to boost dehydration and possible phase changes. Finally, High-Temperature Calcination (HTC) at 700°C for 3 hr is the most intensive calcination method, designed to complete the thermal treatment and potentially enhance the purity and reactivity of the PG.

Type I OPC is the chosen binding agent due to its widespread use and dependable properties in the field of construction, with a specific gravity of 3.15 to ensure standardized mix ratios. The sand selected for this study is well graded, with a fineness modulus of 2.92, and having removed any dust, clay, or organic debris, thereby assuring its neutral impact on the mortar's performance characteristics. Six mortar mixtures with varying PG-to-cement ratios were prepared according to the proportions detailed in Table II. The preparation process was carefully controlled. Each batch was mixed with precise amounts of cement, sand, PG, and water until homogenous consistency was reached.

TABLE II. MIXTURE PROPORTIONS OF PG - CEMENT MORTAR (FOR 9 CUBES)

Mixture	Cement (g)	Sand (g)	Water (mL)	PG (g)	PG/Cement (% wt.)
M1	740	2035	359	0	0
M2	725.2	2035	359	14.8	2
M3	703	2035	359	37	5
M4	684.5	2035	359	55.5	7.5
M5	666	2035	359	74	10
M6	647.5	2035	359	92.5	12.5

B. *Response Surface Methodology*

RSM is a powerful statistical approach used to model and analyze the effects of multiple variables on a response variable. The Box-Behnken design, a subset of RSM, is particularly efficient for experiments involving three levels of each factor, minimizing the number of experimental runs required while still providing a comprehensive understanding of the system. In our specific application of a Box-Behnken design, we consider 4 factors:

- [H_2SO_4] (M): This continuous factor represents the concentration of sulfuric acid, with 3 levels: 0, 2, and 4 M.
- Washing Time (hr): Another continuous factor, washing time, also with 3 levels: 1, 2, and 3 hr.
- Calcination Temperature (°C): The third continuous factor is the temperature at which calcination occurs, with 3 levels set at 100, 400, and 700°C.
- PG/Cement Weight Percentage: The categorical factor in this design is the ratio of PG to cement, measured in weight percentage. It has 5 levels: 2, 5, 7.5, 10, and 12.5%.

This particular design allowed us to investigate the individual effects of each factor and the interactions between them on the response variable: the Compressive Strength (C.S) of a cement mortar. By using 3 levels of the continuous factors, the design will enable the fitting of a full quadratic model, which includes linear, quadratic, and two-factor interaction effects. The addition of 5 levels for the categorical factor, although not standard for a Box-Behnken design, provides additional support in understanding how the replacement of cement with PG affects the response. The unique combination of continuous and categorical variables within this Box-Behnken design offers a robust framework for optimizing the material properties of cement mortar. In a typical Box-Behnken design for 3-level factors, the number of required experiments (N) is calculated by:

$$N = 2^k (k - 1) + C_0 \qquad (1)$$

where k is the number of factors and C_0 is the number of center points. Since we have a categorical factor with 5 levels, separate Box-Behnken design for each level of the categorical factor was conducted. For a single level of the categorical factor, the total number of runs is: 12 + 3 = 15. Since the categorical factor has 5 levels, the total number of experiments required is: 15 * 5 = 75.

C. *Thermal Conductivity*

To determine the thermal conductivity of cement mortar, a cubic sample with 5 cm thickness was prepared and fully cured. This sample was positioned within a heat flow meter setup, inserted between a heated plate and a cooled plate, while thermocouples monitored the temperatures on each side. The cold plate was set to 20°C, and the hot plate to 30°C, generating a steady-state temperature gradient of 10°C across the mortar. Upon reaching equilibrium, the heat flow meter registered a heat transfer rate of 0.6 W through the sample. Thermal conductivity was then calculated using the steady-state heat flow, the sample's cross-sectional area, and the established temperature difference, resulting in a thermal conductivity value indicative of the mortar's ability to conduct heat. This value is crucial for evaluating the material's effectiveness as an insulator in building structures.

III. RESULTS AND DISCUSSION

A. *Age Influence on Compressive Strength*

When PG is used as a partial replacement for cement in mortar, its impact on the C.S varies with different ages (3, 7, and 28 days) as presented in Figure 2.

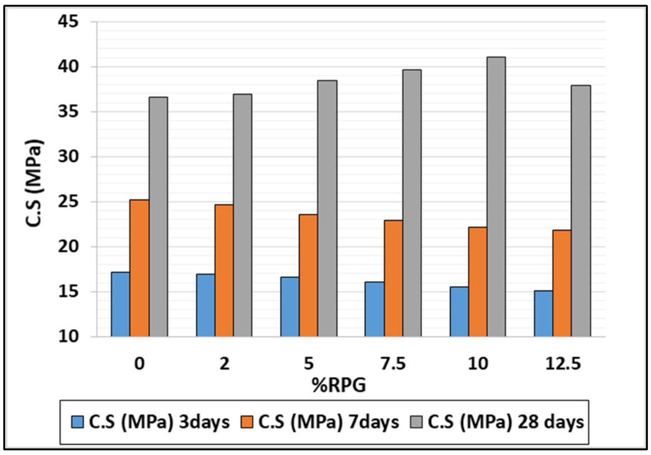

Fig. 2. Compressive strenght of all mixutres after 3, 7, and 28 days.

During the first 3 days, the hydration process of mortar is particularly sensitive to PG. Low PG % can be beneficial, aiding in ettringite formation and thus, early strength, but higher PG levels might introduce detrimental substances, such as phosphates and organics that hinder this process. As the time progresses to 7 days, the ongoing hydration generally results in strength increase due to the presence of more calcium silicate hydrate (C-S-H) and ettringite. For higher PG levels the negative effects of contaminants appear. After 28 days, the mortar's hydration is largely complete, and the full impact of PG is evident: optimal PG levels can lead to strength enhancement via continued ettringite formation and microstructure optimization, whereas excessive PG (12.5%) can undermine strength through mechanisms, like Delayed Ettringite Formation (DEF), phosphate complexation, and the creation of non-beneficial secondary products.

B. *Results of the Experimental Design*

The Box-Behnken design, a component of RSM, investigates the effects of four factors across various levels. This influence of sulfuric acid concentration (0, 2, 4 M), washing time (1, 2, 3 hr), calcination temperature (100, 400, 700 °C), and the PG to cement ratio (2, 5, 7.5, 10, 12.5%) were assessed through a thorough analysis of their impact on compressive strength (C.S). The p-value of each variable is compared against the alpha level of 0.05 to determine statistical significance with respect to the dependent variable. A p-value below 0.05 indicates a significant relationship. The null hypothesis assumes that variables have a zero coefficient, implying no effect on the outcome. This study establishes the alpha at 0.05 to identify significant variables. Regression analysis results are illustrated in Figure 3.

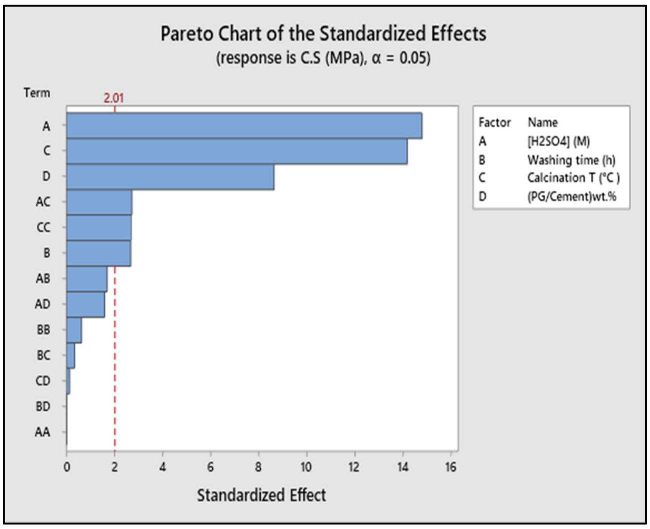

Fig. 3. Pareto chart for C.S response.

The standardized effect Pareto chart of Figure 3 reveals that sulfuric acid concentration, washing time, calcination temperature, and the ratio of PG to cement have essential impacts. Additionally, the interaction between sulfuric acid concentration and calcination temperature has considerable effects on C.S. In addition, the chart indicates that the sulfuric acid concentration and calcination temperature are notably more influential than PG to cement ratio and washing time. All other interactions between factors appear to be insignificant. These interpretations are verified by discrete analysis of the impact of each factor, as illustrated in Figure 4, which shows the individual contribution of each variable to the C.S. A higher concentration of sulfuric acid during the purification process of PG can lead to the removal of impurities, such as soluble salts, heavy metals, and residual phosphates, resulting in a more reactive and pure form of PG. This purified PG, when used as a partial replacement of traditional cement in mortar, has the potential to increase C.S due to its improved bonding and hydration characteristics.

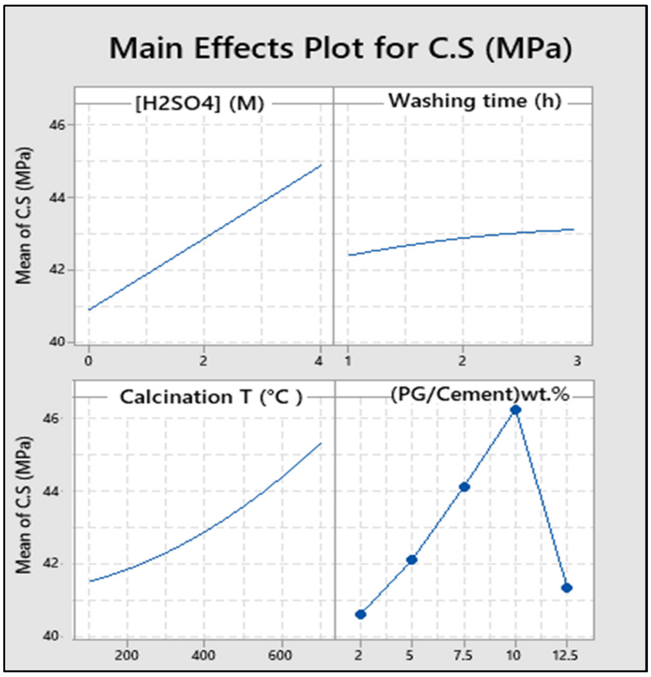

Fig. 4. Main effect plots on C.S.

Calcination temperature also plays a crucial role in determining the quality of PG. Higher calcination temperatures enhance the removal of moisture and organic materials, which in turn can transform the crystal structure of the gypsum. This process creates a calcined product that, when mixed with cement, leads to an increase in the C.S of the mortar. The calcined PG is more chemically active, promoting a stronger bond within the mortar matrix and contributing to the formation of a denser, harder structure. The ratio of PG to cement is another critical factor in achieving optimal compressive strength in mortar applications. An increased PG ratio can improve compressive strength up to 10% by contributing sulfate ions that facilitate the formation of ettringite, a mineral that can positively affect the microstructure and strength of the mortar. Finally, washing time of PG appears to have a marginal effect on the C.S of the mortar, indicating that the main contaminants within PG do not significantly impact the hydration process or are not readily washed away within the tested time frames. This suggests that while washing is a part of the PG purification process, it is less critical than the other factors, like the concentration of sulfuric acid used and the calcination temperature in determining the overall quality and effectiveness of PG as a cement replacement in mortar. These results are confirmed by the influence of washing time on C.S for different levels of acid concentration and calcination temperature as shown in Figures 5 and 6. On the other hand, the interaction between sulfuric acid and calcination temperature, illustrated in Figure 7, is significant according to the Pareto diagram as it shows that C.S is more enhanced when increasing both these operational variables.

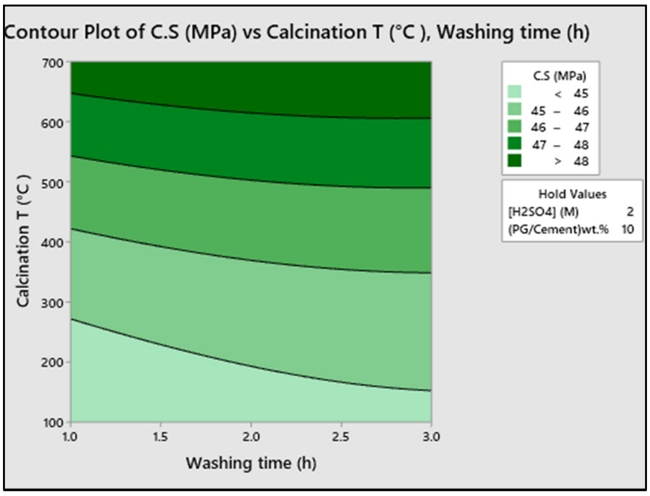

Fig. 5. Washing time influence on C.S for different calcination tempertaures.

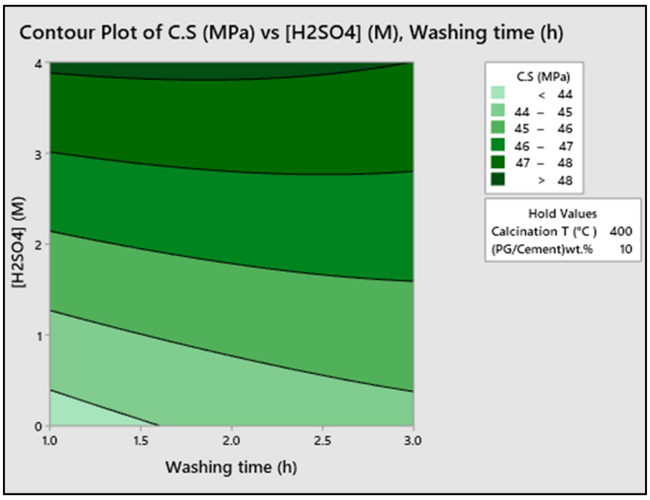

Fig. 6. Washing time influence on C.S for different [H_2SO_4] values.

Finally, the use of PG to cement ratio of 10% and a washing time of 2 hr with extensive chemical and thermal treatment ([H_2SO_4] from 3 to 4 M and calcination temperature of 600 to 700°C), improves the mechanical behavior of mortar samples with TPG. An upgrade in C.S of 21% was achieved in comparison with mortar samples that utilized RPG as a replacement material. In fact, increasing the sulfuric acid concentration causes chemical changes that contribute to the strength of the material, such as creating more cross-linkages or improving the packing density of the material's microstructure. Likewise, increasing the calcination temperature causes physical changes, like the removal of any volatile components, the enhancement of crystallinity, or the sintering of particles, which can also increase the material's strength.

C. Thermal Study Results

A comparative analysis of the thermal conductivity of mortar when different types of PG are used as partial replacement of cement is shown in Figure 8. Three types of PG were considered: RPG, SCPG, and HTPG, with varying replacement percentages, from 0% to 12.5%. The control group's thermal conductivity remains constant at 1.20 W/(m·K) as it represents the standard mortar without any PG replacement, with the consideration that a decrease in thermal conductivity less than or equal to 3% may fall within the experimental error. RPG remains the only PG type which has a potential decrease in thermal conductivity that could be considered significant and not attributable to experimental error, but only at replacement percentages of 7.5% and higher. SCPG and HTPG do not display decreases that would be considered significant. Therefore, the potential for RPG to be used as an insulating material is more pronounced at higher replacement rates, while SCPG and HTPG seem to be unable to provide a clear insulative advantage.

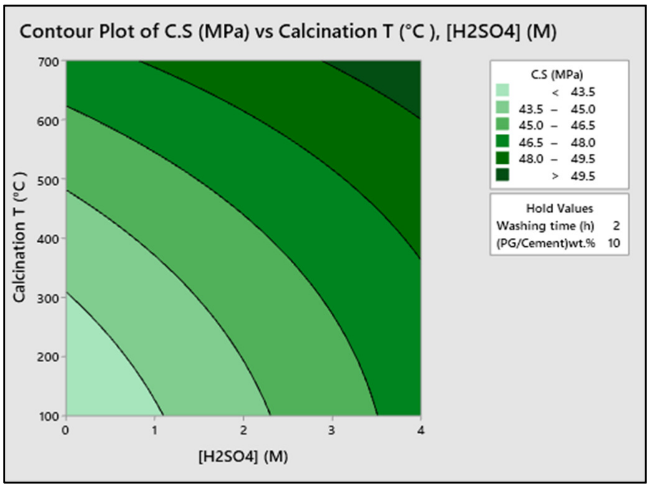

Fig. 7. Calcination tempertaure influence on C.S for different [H_2SO_4] values.

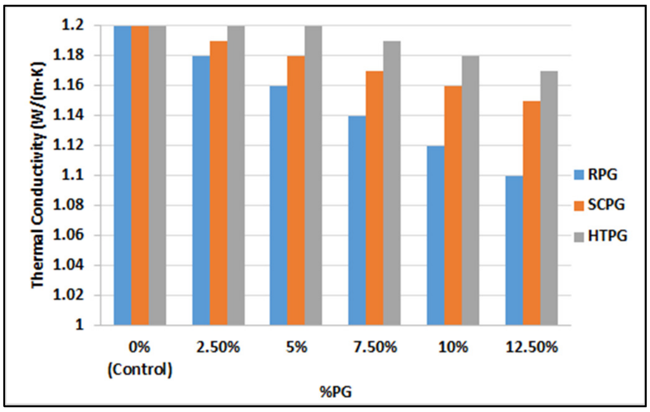

Fig. 8. Comparative analysis of the thermal conductivity of mortar with different types of PG treatments.

IV. CONCLUSION

In conclusion, this study provides valuable insights into the potential use of PG as a partial replacement of cement in mortar. By exploring unprocessed, water-washed, sulphuric acid treated, and heat-treated forms of PG, the research identifies various treatment methods that can optimize the PG quality. The use of Response Surface Methodology, employing the Box-Behnken design, allowed an efficient and structured investigation to be conducted. The latter explored the effects of sulfuric acid concentration, washing time, calcination temperature, and PG to cement ratio on mortar compressive strength.

The results clearly indicate that the treatment of PG with sulfuric acid and the application of appropriate calcination temperatures significantly enhance the compressive strength of the mortar. These treatments effectively remove impurities and improve the microstructure of PG. Furthermore, the study confirms that an optimal ratio of PG to cement exists, at which the benefits of sulfate contributions to mortar strength are maximized without the adverse effects of contaminants.

Washing time, while a part of the purification process, has a less pronounced impact on compressive strength, suggesting that the primary contaminants in PG are either not significantly affecting the hydration process or are not easily removed within the tested time frame. However, it serves as a necessary step in the overall enhancement of the PG quality.

The interaction between sulfuric acid concentration and calcination temperature is particularly noteworthy, demonstrating that a synergistic effect exists when both parameters are increased simultaneously, leading to substantial improvements in compressive strength. This combination of chemical and thermal treatments, particularly at an acid concentration range of 3-4 M and calcination temperatures between 600 and 700 °C, has been shown to significantly improve the mechanical properties of the mortar.

The potential for RPG to be used as an insulating material is more pronounced at higher replacement rates, while SCPG and HTPG seem to be unable to provide a clear insulative advantage.

The study's findings are promising for the construction industry, suggesting that with proper treatment, PG can be a viable partial replacement of cement. This could lead to more sustainable practices by recycling a byproduct of the fertilizer industry and reducing the carbon footprint associated with cement production.

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