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Reuse of phosphogypsum as a sustainable retarder in PCB40 cement production

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KEYWORDS

Phosphogypsum
Natural gypsum
Cement
PCB40
Environment

ABSTRACT

Vietnam faces a significant challenge due to its limited natural gypsum resources, which have been imported to meet the demand of cement production. Besides, the country is grappling with the environmental issue of thousands of tons of phosphogypsum waste discharged annually from Diammonium Phosphate (DAP) fertilizer production. This industrial byproduct poses substantial environmental concerns, highlighting an urgent need for sustainable waste management solutions. This study addresses these critical issues by reusing phosphogypsum as a sustainable alternative to natural gypsum in the manufacturing of PCB 40 cement. This work investigates the effect of varying phosphogypsum content, ranging from 1.5% to 5%, on cement properties, including standard water content, soundness, setting time, and compressive strength. To establish a comparative analysis, the experimental results were compared against a reference cement that incorporates 4% natural gypsum. The findings demonstrate that phosphogypsum can effectively substitute natural gypsum without compromising the quality or performance of the cement. Furthermore, the study confirms that cement produced with phosphogypsum consistently meets all established Vietnamese national standards for the examined properties. In an innovative step, the study also leveraged a 4th-degree polynomial regression model to predict the optimal phosphogypsum content. The model identified an optimal dosage of 3.5% phosphogypsum for maximizing compressive strength. The optimized 3.5% phosphogypsum formulation achieved a compressive strength of 58.3 MPa, which is 3% higher than that of the natural gypsum control. This prediction illustrated remarkable alignment with the experimental data, underscoring the potential of predictive modeling in cement production. This study paves a promising way in the reuse of phosphogypsum in construction materials, aiming at reducing impact on environment and sustainable development.

1. Introduction

Cement, a fundamental binding material, plays an important role in the construction industry. Its production is a highly energy-intensive process, primarily involving the thermal decomposition of limestone and clay at extremely high temperatures, typically around 1450 ˚C, to yield an intermediate product known as clinker [1]. This clinker then undergoes a grinding process, combined with gypsum and various essential additives to produce Portland cement blended [2]. These additives include retarders, hydraulic additives, and fillers, each contributing to the desired properties of the final cement product.

Among these crucial additives, gypsum is paramount for controlling the harden rate of cement. Traditionally, natural gypsum ($CaSO_4.2H_2O$) has been the preferred and almost indispensable additive, typically incorporated at a content percentage by weight ranging from 3 % to 5 % [3]. However, Vietnam faces a significant challenge in this issue due to the lack of natural gypsum mines. Consequently, Vietnam is depending on importing natural gypsum from neighboring countries such as Laos, Thailand, and China. The dependency on imports gives rise to several economic and logistical hurdles. It directly escalates the production cost of cement within Vietnam, making it less competitive on the global market. Furthermore, it undermines the proactive control over raw material sourcing, which can lead to supply chain vulnerabilities and impact production stability.

In contrast to this reliance on imported natural gypsum, Vietnam's industrial landscape presents a unique opportunity for sustainable resource management. Particularly, the Diammonium Phosphate (DAP) Dinh Vu fertilizer factory in Hai Phong has discharged substantial quantities of phosphogypsum as a solid waste. Annually, hundreds of thousands of tons of this phosphogypsum, primarily composed of gypsum ($CaSO_4.2H_2O$), are discharged [4]. The improper management of this massive volume of industrial waste, such as untreated phosphogypsum leads to severe environmental pollution and public health risks, including soil and water contamination, and occupies vast tracts of land, thereby diminishing available space for other essential uses [5], [6], [7]. Moreover, its accumulation can present various potential health hazards to surrounding communities. Therefore, finding a suitable approach to reuse phosphogypsum is an urgent task aiming to protect the environment from the harmful impact of this solid waste. Due to the main composition of phosphogypsum is calcium sulfate dihydrate ($CaSO_4.2H_2O$), it has been widely used as a substitute material to natural gypsum in the construction materials, such as cement [8], [9], [10], [11], brick [12], [13], [14], [15], concrete materials [16], [17], [18] and agriculture industries [19], [20], [21].

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To solve the dual challenge in Vietnam, this study directly addresses these issues by investigating the viability and effectiveness of utilizing this phosphogypsum as a comprehensive substitute for natural gypsum in the production of PCB 40 cement. The primary objective is to evaluate its performance as a setting retarder, a crucial additive that controls the hydration process and setting time of cement [22]. The study aims to demonstrate that phosphogypsum can not only effectively replace natural gypsum but can do so without compromising the fundamental quality, mechanical properties, or long-term performance of the resulting cement. Key parameters such as standard water content, setting time, soundness, compressive strength will be thoroughly examined to ensure that the cement produced with phosphogypsum meets all Vietnamese national standards and specifications for PCB 40 cement. Beyond the technical aspect, this study also promotes resource efficiency, significantly reducing industrial waste, and fostering truly sustainable industrial activities within Vietnam. By transforming a discarded industrial byproduct into a valuable retarder in cement production, this study promotes a circular economy approach. This innovative repurposing not only mitigates the environmental burden associated with phosphogypsum disposal but also lessens the reliance on natural resources. Ultimately, the successful implementation of these findings has the potential to unlock a future where Vietnam's construction industry is characterized by both robust economic resilience and unparalleled environmental stewardship, setting a precedent for sustainable development in the region.

2. Materials and Methods

2.1. Clinker

The clinker used in this study was taken from the Van Hoa clinker plant (Figure 1), a prominent producer known for its consistent quality and adherence to Vietnamese national standards, TCVN 7024-2013. Detailed analysis and characterization of the Van Hoa clinker were conducted to ascertain its chemical composition and physical properties. The comprehensive results of these analyses are presented in Table 1. This table includes key parameters such as the percentages of major oxides (CaO, SiO_2, Al_2O_3, Fe_2O_3), and mineralogical composition (alite (C_3S), belite (C_2S), aluminate (C_3A), ferrite (C_4AF) phases) which directly influence the clinker's hydraulic properties and strength development in cement.

Figure 1. Van Hoa Clinker.

2.2. Natural gypsum

The natural gypsum used in this study was taken from Laos, Figure 2. Its chemical characteristics are detailed in Table 2.

Table 1. The specifications of Van Hoa clinker.

Specification Parameter	Unit	Specification TCVN 7024:2013	Test Result
CaO	%	58 ÷ 67	64.64
SiO_2	%	18 ÷ 26	20.82
Al_2O_3	%	3 ÷ 8	4.67
Fe_2O_3	%	2 ÷ 5	3.51
MgO	%	≤ 5.0	4.76
Free CaO	%	≤ 1.5	0.90
$Na_2O_{eq} = Na_2O + 0.658.K_2O$	%	≤ 1.0	0.48
C_3S	%	-	61.97
C_2S	%	-	12.94
C_3A	%	-	6.44
C_4AF	%	-	10.68
Insoluble residue	%	≤ 0.75	0.10
Loss on ignition	%	≤ 1.0	0.35
Compressive strength - 3 days ± 45 minutes - 28 days ± 8 hours	MPa	 ≥ 25 ≥ 50	 31.3 54.4

Figure 2. Natural gypsum.

Table 2. The specifications of natural gypsum.

Specification Parameter	Unit	Test result
SO_3	%	43.26
Insoluble residue	%	1.42
Structural water	%	19.31

2.3. Phosphogypsum

Figure 3. Phosphogypsum.

The phosphogypsum used in this study is taken directly from the Dinh Vu Gypsum Joint Stock Company, Figure 3. This particular type of gypsum has undergone rigorous quality assessment and has been officially certified by the Institute of Construction Materials, a reputable body operating under the Ministry of Construction. The certification confirms that the phosphogypsum meets the Vietnamese national standards and regulations, specifically adhering to the requirements outlined in QCVN 16:2023/BXD. This compliance is a crucial indicator of its suitability for construction applications. The characteristics of this gypsum are presented in Table 3.

Table 3. The specifications of phosphogypsum.

Specification Parameter	Test results (%)
Humidity	8.0
SO_3	39.8
P_2O_5	0.65
Insoluble residue	10.5

2.4. Limestone

Limestone was taken from Quang Binh province as shown in Figure 4. A comprehensive analysis of the limestone's chemical composition was conducted to ensure its suitability as a filler material within the framework of this study. The detailed data presented in Table 4 outlines the precise percentages of key oxides and other components.

Figure 4. Limestone.

Table 4. The specifications of Limestone.

Specification Parameter	Unit	Test result
SO_3	%	0.14
SiO_2	%	3.52
Al_2O_3	%	0.23
Fe_2O_3	%	0.82
CaO	%	51.66
MgO	%	1.51
Loss on Ignition	%	40.38
Insoluble residue	%	2.77

2.5. Slag

Figure 5. Hoa Phat Slag.

The slag used in this study was taken from the Hoa Phat Steel Plant, as depicted in Figure 5. This material, a byproduct of steel manufacturing processes, is commonly recognized for its pozzolanic properties and potential for use in construction materials. The specific quality and chemical composition of the slag utilized in this study are presented in Table 5.

Table 5. The specifications of Hoa Phat slag.

Specification Parameter	Unit	Test result
SO_3	%	0.15
SiO_2	%	33.62
Al_2O_3	%	11.95
Fe_2O_3	%	2.24
CaO	%	46.32
MgO	%	7.05
Loss on ignition	%	0.94
Insoluble residue	%	0.20

2.6. Mix Proportion

To investigate the effect of phosphogypsum on the characteristics of cement. The phosphogypsum content was added to the cement product in a range from 1.5 % to 5 % by weight. These specific mix proportions are shown in Table 6. For comparative analysis, a conventional cement mixture containing 4 % natural gypsum was utilized as a reference. This particular proportion of natural gypsum is standard and commonly employed in cement plants throughout Vietnam, making it an appropriate benchmark for evaluating the performance of the phosphogypsum blends. The careful selection of these mix proportions allowed for a thorough examination of how different levels of phosphogypsum content influence the properties of cement, providing valuable insights into its potential as a sustainable alternative.

Table 6. Mix proportions (weight percent) of cement samples with varying phosphogypsum content.

Sample	Clinker (%)	Gypsum (%)	Limestone (%)	Slag (%)	Total (%)
M0	85.0	4.0*	6.0	5.0	100
M1	85.0	1.5	8.5	5.0	100
M2	85.0	2.0	8.0	5.0	100
M3	85.0	2.5	7.5	5.0	100
M4	85.0	3.0	7.0	5.0	100
M5	85.0	3.5	6.5	5.0	100
M6	85.0	4.0	6.0	5.0	100
M7	85.0	4.5	5.5	5.0	100
M8	85.0	5.0	5.0	5.0	100
*: natural gypsum as reference					

2.7. Soundness

In this study, the soundness of the various cement samples was carried out using the Le Chatelier's apparatus, as depicted in Figure 6. This method is a widely accepted and standardized procedure for assessing the expansion of cement pastes. The test was performed in adherence to the specifications outlined in the Vietnamese national standards, TCVN 6017:2015. This particular standard details the precise methodology, including the preparation of the cement paste, the curing conditions, and the measurement of the expansion.

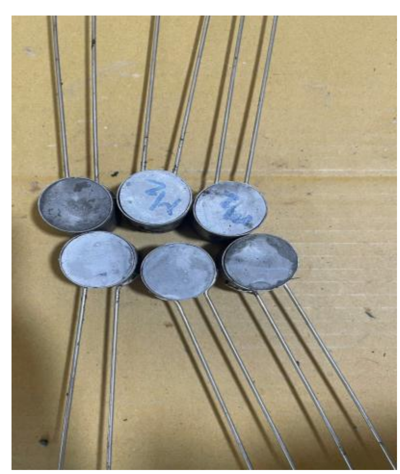

Figure 6. Le Chatelier's apparatus is used to determine the soundness of cement samples.

2.8. Standard water content

The standard water content was determined using the Vicat apparatus, as depicted in Figure 7. The testing methodology rigorously adhered to the guidelines stipulated by the Vietnamese national standards, TCVN 6017:2015.

Figure 7. Vicat apparatus is used to determine the standard water content of cement samples.

2.9. Setting Time

For the cement samples under investigation, both the initial and final setting times were determined using vicat apparatus (Figure 8 and Figure 9) in strict adherence to the Vietnamese national standard, TCVN 6017:2015. This standard, which specifies the methods for determining the setting time of hydraulic cement, provides a robust framework for consistent and reliable measurements.

Figure 8. Vicat apparatus is used to determine the initial setting time.

Figure 9. Vicat apparatus is used to determine the final setting time.

2.10. Compressive strength

To thoroughly assess the impact of varying phosphogypsum content on the mechanical properties of cement samples, the compressive strength was evaluated after 3, 7 and 28 curing days. The preparation of the cement mortar mixture adhered strictly to established standards to ensure consistency and comparability of results. A precise ratio of cement mass: sand: water = 1:3:0.5 was maintained for all mixtures, a proportion carefully selected to reflect typical construction applications while allowing for distinct observation of gypsum's influence. The mixing procedure itself was carried out in accordance with Vietnamese national standard, TCVN 6016:2011. Following uniform mixing, the fresh cement mortar was cast into standardized molds, each with dimensions of 40 mm × 40 mm × 160 mm (as depicted in Figure 10). These specific dimensions are internationally recognized for prism-shaped mortar specimens used in compressive strength testing, allowing for direct comparison with a wide range of research data. The cast samples were then subjected to an initial curing period of 24 hours within their molds. After this initial curing period, the samples were carefully demolded and immediately immersed in a water tank. Subsequently, for each sample, a set of three specimens was tested at each of the predetermined intervals: 3, 7, and 28 days. The compressive strength measurements were performed using a 300kN compression machine manufactured by Control, Italy (illustrated in Figure 11).

Figure 10. Mortar samples in mold with dimensions of 40 mm × 40 mm × 160 mm.

Figure 11. The compressive strength testing machine manufactured by Controls.

3. Results and Discussion

3.1. Standard water content

The standard water content, a critical parameter in determining the workability and consistency of cement paste, exhibited remarkable stability across all tested samples. As illustrated in Figure 12, the measured values regularly fell within a narrow range of 25.4 % to 26.2 %, which is within the range of the Vietnamese national standard, TCVN 6017-2015. This consistent range was observed even with varying amounts of phosphogypsum, specifically from 1.5 % to 5 %. Notably, the cement paste containing 4 % natural gypsum of the common PCB 40 type also aligned perfectly within this established range, further underscoring the consistent behavior of the mixtures. These findings strongly suggest that the incorporation of phosphogypsum, within the tested range, has a negligible impact on the standard water content of the cement paste. This is a significant observation as it indicates that phosphogypsum can be effectively utilized as a supplementary cementitious material without compromising the fundamental rheological properties of the fresh cement mixture. The ability to maintain a consistent standard water content is crucial for various applications, ensuring predictable mixing and workability characteristics of the cement paste.

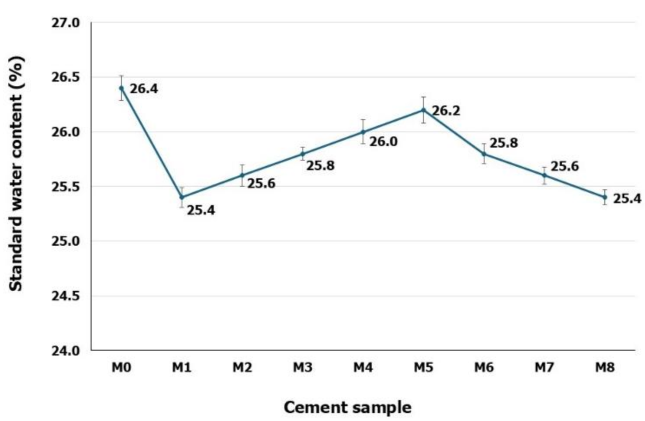

Figure 12. Standard water content of different cement samples.

3.2. Soundness

To evaluate the effect of phosphogypsum on the ability to resist volume changes after setting and hardening of cement, a critical aspect of material performance, we conducted an examination of the soundness test for all prepared cement samples. The results of these soundness tests demonstrate remarkable volumetric stability across all studied samples and reference, as shown in Figure 13. Specifically, the measured soundness values were either 0.2 mm or 0.3 mm, which is significantly less than the limit of the Vietnamese national standard, TCVN 6060-2020 (≤ 10 mm). These exceptionally low values are a strong indicator of good volume stability within the hardened cement paste. This indicates that the incorporation of phosphogypsum, within the studied concentrations, does not introduce any risk of excessive expansion or detrimental internal stresses that could lead to cracking or degradation over time. Such stability is crucial for the long-term durability and structural integrity of concrete applications where these cements would be utilized. The findings thus underscore the suitability of phosphogypsum as a viable component for cement production, particularly in mitigating the potential for undesirable volumetric changes.

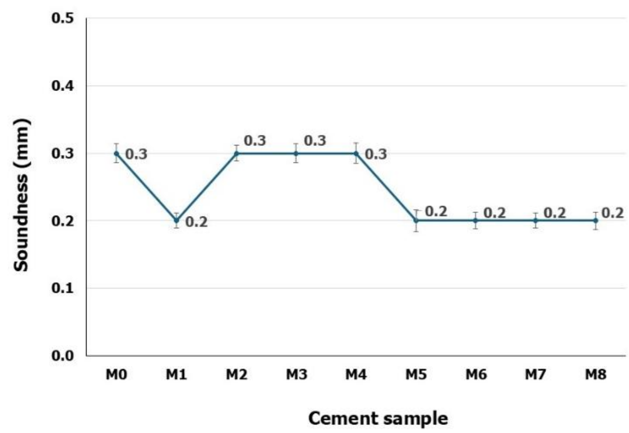

Figure 13. Soundness of different cement samples.

3.3. Setting Time

The influence of phosphogypsum content on the setting times of cement samples was systematically investigated and the results are presented in Figure 14. A consistent trend observed across all samples was a general increase in both initial and final setting times as the percentage of phosphogypsum increased. In particular, the initial setting times increase from 150 minutes to 190 minutes and final setting times increase from 185 minutes to 239 minutes when the content of phosphogypsum was added from 1.5-5 %, respectively. Compared to the same content of natural gypsum (4 %), the initial and final setting times are slightly higher by 10 minutes. This observation is crucial as it indicates the active role of phosphogypsum in modulating the hydration kinetics of cement. Despite this increase, all tested cement samples rigorously adhered to the requirements of the Vietnamese national standard, TCVN 6268:2020. This standard mandates that the initial setting time must not be less than 45 minutes, ensuring adequate workability, while the final setting time must not exceed 375 minutes, allowing for timely hardening and strength development. The fact that all samples remained well within these defined limits underscores the suitability and safety of incorporating phosphogypsum within the studied concentration ranges. The observed prolongation of setting times strongly suggests that phosphogypsum effectively functions as a retarder in cement hydration. This retarding effect is a desirable property in various construction applications, as it provides a longer window for transportation, placement, and finishing of concrete mixes, particularly in large-scale projects or in hot weather conditions. The consistent compliance with national standards further validates the efficacy and reliability of phosphogypsum as a component in cement formulations, indicating its potential to optimize cement performance without compromising structural integrity or practical applicability.

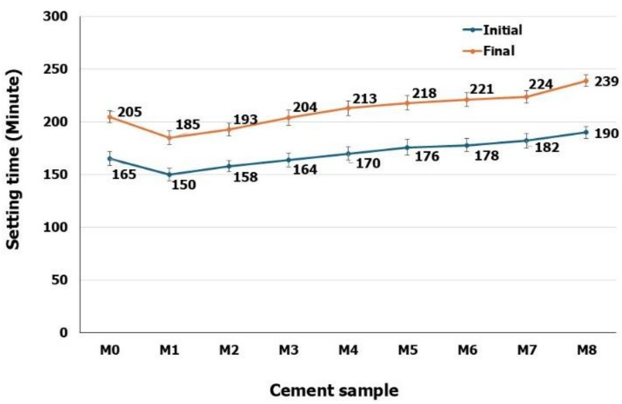

Figure 14. Initial and final setting time of different cement samples.

3.4. Compressive Strength

The investigation into the compressive strength of cement samples with varying contents of phosphogypsum revealed distinct performance trends, as presented in Figure 15. Analysis of early compressive strength development indicated that cement samples with the studied phosphogypsum content exhibited 3-day compressive strength ranging from 28.5 MPa to 33.7 MPa. This is a significant increasing early compressive strength compared to requirement of the Vietnamese national standard, TCVN 6062-2020, in which the 3-day compressive strength of 18 MPa. Phosphogypsum demonstrated a significant advantage, extending to the 7-day compressive strength, with values ranging from 38.9 MPa to 44.5 MPa. This early compressive strength of phosphogypsum used cement was 5 MPa higher than that of the natural gypsum at 4 %. These findings suggest that phosphogypsum accelerates the hydration process, leading to improved early-age strength gain.

For 28-day compressive strength, cement samples showed an increase from 52.8 MPa to approximately 58 MPa as phosphogypsum content rose from 1.5 % to 3.0 %. This compressive strength largely remained around 58 MPa within the 3 %-4 % phosphogypsum range. Notably, the cement sample with 4 % phosphogypsum slightly outperformed natural gypsum used cement. However, compressive strength decreased when phosphogypsum content exceeded 4.0 % to 5.0 %.

Based on these results, the optimal phosphogypsum content for cement, designated M4 with 3.0 % phosphogypsum, served effectively as a retarder and significantly outperformed the reference sample M0, which used 4.0 % natural gypsum. Specifically, sample M4 achieved a 28-day compressive strength of 58.4 MPa, representing an approximate 2.1 % increase compared to M0's 28-day compressive strength of 57.2 MPa. This substantial improvement underscores the potential of phosphogypsum as a more effective and possibly more sustainable alternative to natural gypsum in cement manufacturing. Furthermore, a direct comparison at equal content revealed compelling results. Sample M6, containing 4.0 % phosphogypsum, achieved a 28-day compressive strength of 58.2 MPa. This value is 1.8 % higher than the reference sample M0, which also contained 4.0 % natural gypsum. This finding is particularly significant as it demonstrates that even at equivalent replacement levels, phosphogypsum can contribute to a slightly enhanced long-age compressive strength. The consistent superior performance of phosphogypsum samples, especially M4 (3.0 % phosphogypsum), highlights its potential to optimize cement performance, particularly in applications where early and moderate long-age compressive strength development is crucial.

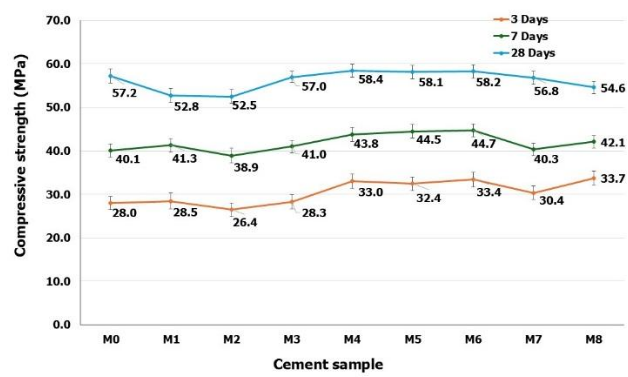

Figure 15. Compressive strength of different cement samples at 3, 7 and 28 days.

3.5. Machine learning model

To precisely determine the optimal phosphogypsum content for producing PCB 40 cement that meets to Vietnamese national standards, a sophisticated machine learning model was developed and applied. This model was trained using comprehensive experimental data, specifically focusing on the compressive strength at 28 days across various contents of phosphogypsum. The chosen model for this predictive analysis was the 4th degree polynomial regression, implemented in Python using the open-source sckit-learn library. This particular model was selected due to its ability to capture complex, non-linear relationships within the obtained experimental data, providing a more accurate representation of how varying gypsum content influences the final compressive strength. The results generated by this model are represented in Figure 16. A 4th degree polynomial regression model achieved a high coefficient of determination (R^2 = 0.9572), indicating it explains 95.7% of the variance in the data. The Root Mean Squared Error (RMSE) was 0.4410 MPa, which is a low value relative to the strength measurements, confirming a high-precision fit. As illustrated in Figure 16, an optimal 28-day compressive strength of 58.95 MPa is predicted by the model. This compressive strength is achieved at a phosphogypsum content of 3.51 %. A crucial aspect of this modeling effort was the validation of its predictions against actual experimental outcomes. The predicted optimal 28-day compressive strength of 58.95 MPa showed excellent agreement with the experimentally obtained compressive strength of 58.1 MPa. This close correlation is further highlighted by the minimal difference between the experimental data and the prediction, which stands at a mere 1.5 %. This high level of agreement underscores the reliability of the machine learning model in accurately predicting the optimal phosphogypsum content for PCB 40 cement production, ensuring compliance with the Vietnamese national standards while maximizing material performance.

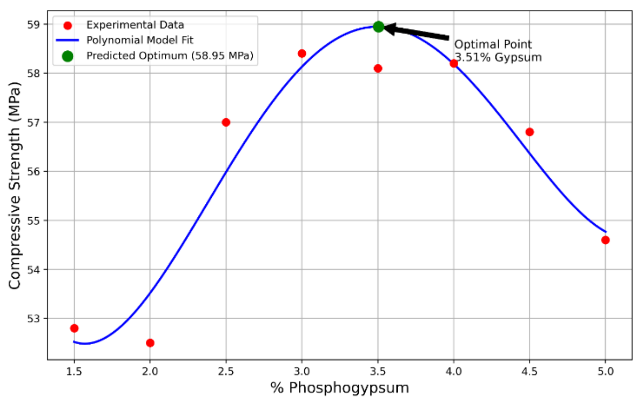

Figure 16. A 4th-degree polynomial regression model showing the predicted relationship between phosphogypsum content and 28-day compressive strength.

4. Conclusions

A comprehensive investigation into the use of phosphogypsum as a substitute for natural gypsum in cement production, specifically for PCB 40 grade, has yielded positive results. In the range of phosphogypsum contents from 1.5 % to 5 % by weight in this study, cement samples have been tested for the standard water content, soundness, and setting time. The resulting cement samples consistently meet all Vietnamese national standard requirements. Beyond merely meeting baseline requirements, the early-age and long-age compressive strength of the studied cement samples has exhibited significant improvement. Comparative analysis reveals an impressive increase of nearly 2 % in 28-day compressive strength compared with the reference cement sample that utilizes 4 % natural gypsum. The experimental results have shown optimal content for phosphogypsum in the range 3.0 % to 4.0 %. By applying a sophisticated machine learning model with a 4th degree, the optimal phosphogypsum content at 3.5 % is predicted and in good agreement with experimental observation. This convergence of experimental data and predictive modeling provides a high degree of confidence in the recommended content, underscoring the reliability and accuracy of the study outcomes. The implications of these findings are multifaceted and profound. The phosphogypsum at this optimized level not only contributes to the creation of a stronger, more resilient cement product but also contributes to crucial environmental and economic objectives. By promoting the effective reuse of industrial byproducts, this approach significantly reduces the dependence on natural gypsum, thereby conserving natural resources and mitigating the environmental impact associated with extraction and processing. This sustainable approach aligns with the goals of circular economy principles and responsible resource management, offering a pathway toward more environmentally conscious and efficient cement manufacturing.

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