

Article

Synergistic Sintering of Multi-Source Petrochemical Wastes for High-Strength Ceramsite: Process Optimization and Environmental Safety

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Abstract

The sustainable management of typical petrochemical hazardous wastes, such as oil sludge (OS), spent fluid catalytic cracking catalysts (SFCCs), and petrochemical-contaminated soil (PCS), poses a significant challenge. This study developed a synergistic sintering strategy that utilizes the complementary properties of these materials, with OS serving as an organic source, SFCCs and PCS providing an aluminosilicate framework, and waste glass powder (GP) acting as a fluxing agent to produce an environmentally friendly, high-strength ceramsite (OSPG-Opt). Single-factor experiments were first conducted to investigate the effects of OS content, sintering temperature, and duration. Subsequently, the Box–Behnken design was employed to optimize the process for maximizing aggregate strength. The optimal conditions were determined to be 30.5% OS content, a sintering temperature of 1142 °C, and a sintering time of 32 min. Under these conditions, the resulting ceramsite demonstrated a compressive strength of 23.12 MPa, along with a bulk density of 1012.50 kg/m³ and low water absorption of 1.61%, meeting the requirements of the Chinese standard T/CSTM 00548-2022 for structural materials. Microstructural analysis identified the presence of quartz, anorthite solid solution, hematite, and albite. The remarkable mechanical strength is attributed to an interlocking structure of anorthite solid solution within a glassy matrix, which also contributes to effective heavy metal immobilization, ensuring the excellent environmental performance of the final product.

Keywords: oily sludge; hazardous waste; high-strength ceramsite; heavy metals; anorthite solid solution



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1. Introduction

Petroleum refining and catalytic processing generate various solid wastes, among which oily sludge (OS), petrochemical-contaminated soil (PCS), and spent fluid catalytic cracking catalysts (SFCCs) represent three typical hazardous waste categories [1–3]. OS originates from crude oil tank bottoms, oil–water separators, on-site wastewater treatment ponds, and other sources. It is a complex mixture of pollutants rich in various organic contaminants [4]. SFCCs are generated due to the hydrothermal deactivation of catalysts and metal deposition, containing high levels of heavy metals such as nickel and vanadium [2]. It is listed in the National Hazardous Waste Catalogue (HW50, China). PCS originates from petroleum hydrocarbon contamination, affecting extensive areas (approximately 23.6% of

Chinese oilfields) [3]. Aged hydrocarbons are adsorbed by soil clay minerals, exhibiting low bioavailability and posing significant remediation challenges [5]. Both PCS and OS are listed in the National Hazardous Waste Catalogue (HW08, China).

Currently, the hazardous wastes mentioned above are primarily processed through single-material separation methods. Technologies such as OS incineration and solvent extraction often generate secondary residues. SFCCs pose environmental risks in resource utilization due to heavy metal migration, and PCS pyrolysis–desorption treatment is predominantly used for low-value backfilling, failing to achieve upcycling of its rich mineral components such as clay and feldspar [6–8]. This traditional disposal-centric model not only incurs high disposal costs but also leads to inefficient resource conversion and secondary pollution risks, becoming a bottleneck for the green development of industry [9,10].

The conversion of hazardous solid waste into construction materials represents a promising strategy for large-scale resource recovery. Among various valorization routes, the sintering-based production of ceramsite has attracted considerable interest owing to the exceptional stability and versatility of the resulting product [11,12]. The high-temperature sintering process not only ensures complete detoxification of the waste but also transforms it into market-ready construction materials with well-defined functional value [13,14]. In particular, high-strength ceramsite can serve as a high-quality aggregate in concrete applications, contributing directly to the enhanced mechanical performance and durability of the final product [15]. While previous studies have explored the use of various solid wastes, such as sewage sludge, fly ash, and coal gangue, in ceramsite production, existing approaches often rely on feedstocks sourced from multiple sectors, often incurring substantial transportation costs [11,16]. Moreover, for hazardous waste systems with highly complementary physicochemical characteristics, such as OS, SFCCs, and PCS, systematic strategies for synergistic utilization remain underdeveloped. The absence of such integrated methodologies hinders the resolution of inherent challenges in individual waste processing, including difficulties in microstructure control, poor shaping behavior, and risks of heavy metal mobility [17–19].

To address these challenges, this study introduces a synergistic sintering strategy utilizing multiple hazardous waste streams based on their functional complementarity [20]. Through systematic formulation design, three types of hazardous waste were successfully converted into high-performance ceramsite. A synergistic system was established using OS, SFCCs, and PCS as the core raw materials, where OS acts as a pore-forming and endogenous heat source via pyrolysis, SFCCs provide the structural framework, and PCS enhances plasticity while supplementing silica sources. Waste glass powder (GP) was incorporated as an efficient fluxing agent to reduce the eutectic temperature and facilitate liquid phase formation, thereby overcoming the inherent limitations of individual solid wastes [21,22]. By targeting high-strength ceramsite, process optimization led to significant improvements in mechanical properties, rendering the product suitable for construction applications with demanding strength requirements. Furthermore, this study elucidates the mechanisms of structural evolution and heavy metal immobilization during multicomponent sintering, offering theoretical assurance for the environmental safety and long-term stability of the product. This study is expected to provide practical solutions for the integrated management of petrochemical waste.

2. Materials and Methods

2.1. Raw Material Characterization

The OS, PCS, and SFCCs used in this study were sourced from a petrochemical plant in Wuhan, China, with waste glass powder (GP) employed as an auxiliary material. All raw materials were oven-dried at 105 °C for 24 h, subsequently pulverized, and sieved through

a 200-mesh sieve (particle size ≤ 0.075 mm). The resulting powders were stored in sealed containers for subsequent use. Their chemical compositions were determined by X-ray fluorescence (XRF, Zetium, Malvern Panalytical, Almelo, The Netherlands) spectroscopy, as summarized in Table 1.

Table 1. Chemical compositions of raw materials (wt.%).

Chemical Composition (wt.%)	OS	PCS	SFCC	GP
SiO ₂	24.31	56.91	37.61	71.57
Al ₂ O ₃	21.81	23.20	54.73	1.66
Fe ₂ O ₃	15.01	3.47	0.47	0.12
Na ₂ O	1.26	1.84	0.30	14.96
K ₂ O	0.69	4.49	0.16	0.21
CaO	15.75	2.26	0.16	7.46
P ₂ O ₅	2.04	0.13	0.56	0.02
MgO	1.88	0.71	0.94	3.59
SO ₃	13.48	/	0.22	0.21

The mineral phases of the raw materials were identified using X-ray diffraction (XRD; Malvern PANalytical Empyrean, Almelo, The Netherlands), and the patterns are presented in Figure 1. The XRD analysis reveals that OS is primarily composed of quartz (SiO₂), along with calcite (CaCO₃) and olivine (Mg, Fe)₂SiO₄. SFCCs predominantly consist of an amorphous phase, evidenced by a broad diffuse hump, with distinctive crystalline diffraction peaks identified as corundum (Al₂O₃) and quartz (SiO₂). These crystalline phases are expected to constitute the primary micro-skeleton of the ceramsite. PCS exhibits a more complex mineralogical composition, containing typical subsurface minerals such as quartz, kaolinite, muscovite, albite, and K-feldspar. In contrast, the XRD pattern of GP displays no sharp diffraction peaks, confirming its amorphous nature.

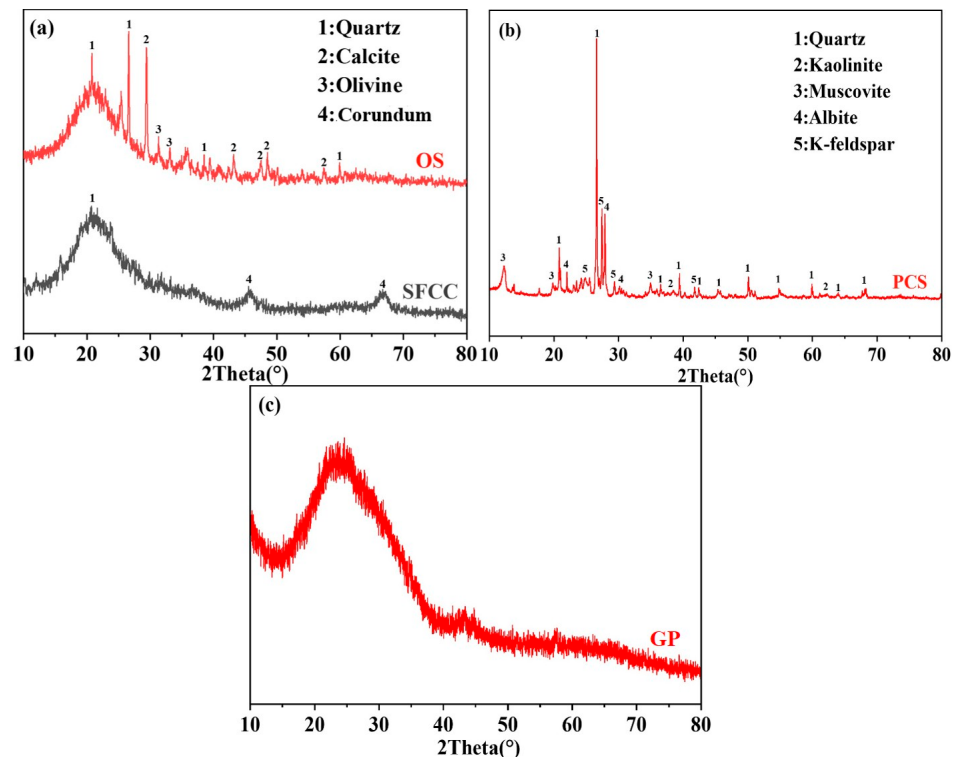


Figure 1. Mineral compositions of raw materials: (a) OS, SFCC, (b) PCS, and (c) GP.

2.2. Preparation of the Ceramsite Samples

According to the Riley phase diagram, when the chemical composition of the ceramsite raw materials meets a SiO₂ content of 40–79 wt.%, an Al₂O₃ content of 10–25 wt.%, and a

fluxing oxide RO (CaO, MgO, Fe₂O₃, Na₂O, and K₂O) content of 13–26 wt.%, the prepared ceramsite exhibits excellent performance [23]. Guided by this principle, five distinct formulations were devised, with their specific proportions and chemical compositions detailed in Table 2.

Table 2. Ratio of raw materials and chemical composition in ceramsite production.

Sample	OS (%)	PCS (%)	SFCC (%)	GP (%)	H ₂ O (%)	SiO ₂ (wt.%)	Al ₂ O ₃ (wt.%)
A1	20	50	10	20	30	51.39	21.77
A2	30	40	10	20	27	48.13	21.63
A3	40	30	10	20	24	44.87	21.49
A4	50	20	10	20	21	41.61	21.35
A5	60	10	10	20	18	38.35	21.21

The ceramsite preparation procedure, illustrated in Figure 2, consisted of four main stages. (1) Mixing: Predetermined amounts of OS and PCS were combined with fixed proportions of SFCCs (10%) and GP (20%). The water was added at a proportion relative to the total dry mass of the mixture and optimized based on the OS content. Raw materials were first dry-mixed for 5 min to achieve a uniform blend in appearance. The optimized amount of water was then added, followed by wet mixing for an additional 5 min until a homogeneous moist mixture was obtained. (2) Pelleting: The mixture was manually shaped into spherical pellets, each weighing approximately 1.5 g, to ensure uniform size after sintering. (3) Pre-sintering: The pellets were first dried at 105 °C for 1 h, followed by thermal treatment in a muffle furnace at 550 °C for 30 min with a heating rate of 10 °C/min. (4) Sintering: The pre-sintered pellets were heated to target temperatures (ranging from 1100 to 1180 °C) at 5 °C/min, held for specified durations (10–50 min), and finally cooled to room temperature under a programmed regime. The sintering temperature was confined to this specific range because temperatures below 1100 °C resulted in severe powder shedding, while exceeding 1180 °C caused over-firing and structural collapse. The entire sintering process was conducted in an air atmosphere.

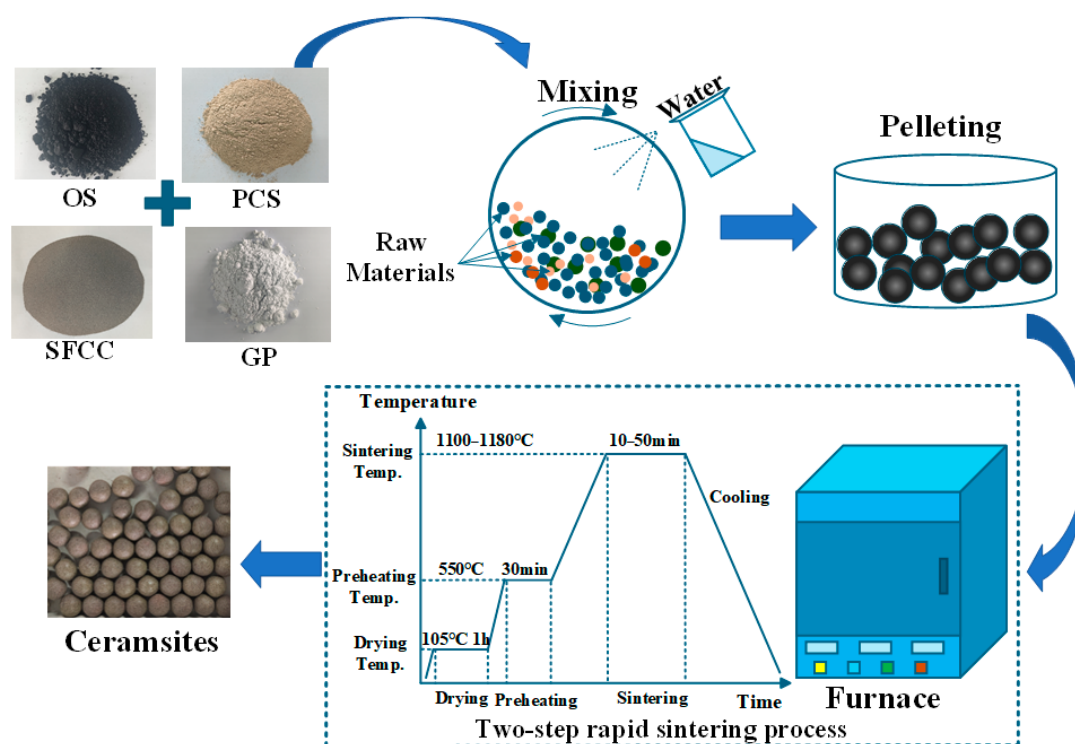


Figure 2. Flowchart for the preparation of high-strength ceramsite.

2.3. Experiment Design

2.3.1. Single-Factor Experiment

Single-factor experiments were first conducted to determine the baseline ranges for subsequent response surface optimization. Initially, the OS content was varied (denoted as levels A1 to A5) while maintaining a constant sintering temperature and time. Subsequently, the optimal OS content identified from this step was fixed, and the sintering temperature was varied (1100–1180 °C) at a fixed sintering duration. Finally, with both OS content and sintering temperature fixed at their optimal levels, the sintering time was tested across different intervals (10–50 min). The optimal condition, along with two adjacent conditions, was selected to define the range for process optimization. The ceramsite produced under these single-factor conditions is referred to as OSPG.

2.3.2. Response Surface Optimization

Response Surface Methodology (RSM) is a statistical technique used to model and analyze relationships between multiple independent variables and their responses. A Box–Behnken Design (BBD) with three factors and three levels was implemented using Design-Expert 13.0.5 software to optimize the compressive strength of ceramsite. The investigated factors included OS content (X_1), sintering temperature (X_2), and sintering time (X_3). Based on single-factor experimental results, each factor was tested at three coded levels (−1, 0, +1), as detailed in Table 3. By using variance analysis and regression analysis to calculate the experimental data, the quadratic equation is shown as in Equation (1):

$$Y = \beta_0 + \sum_{i=1}^k \beta_i X_i + \sum_{i=1}^k \beta_{ii} X_i^2 + \sum_{j>1}^k \sum_{i>1}^k \beta_{ij} X_i X_j + \varepsilon \quad (1)$$

Table 3. Box-Behnken design encoding, design variables, and levels.

Encoding	Design Variables	Levels		
		−1	0	1
A	OS concentration (%)	20	30	40
B	Sintering temperature (°C)	1120	1140	1160
C	Sintering time (min)	20	30	40

Y represents the response variable, k is the number of design variables ($k = 3$), X_i and X_j ($i = 1, 2, 3, j = 1, 2, 3$) are the coded independent variables, β_0 is the constant coefficient, β_i , β_{ii} , and β_{ij} represent the coefficients for linear, quadratic, and interaction terms, respectively, and ε is the random error term.

The significance of the model ($p < 0.05$) and the goodness-of-fit were evaluated through analysis of variance (ANOVA) and the coefficient of determination (R^2), respectively.

2.4. Performance Characterization of OSPG

2.4.1. Basic Characterization Testing

The apparent density and 1 h water absorption of OSPG products were determined following the Chinese standard T/CSTM 00548-2022 for solid waste-derived high-strength lightweight aggregates [24,25]. The compressive strength of individual OSPG particles was measured according to the method developed by Yashima et al. [26], using a KQ-3 particle press. The strength was calculated using Equation (2):

$$S = \frac{2.8 \times P_C}{\pi X^2} \quad (2)$$

where S is the compressive strength of a single OSPG (MPa), P_c is the crushing load (N), and X is the particle diameter (mm). To ensure the reliability of the measurement data, the same batch of ceramsite was divided into three parallel sets for each performance test.

2.4.2. Composition Characterization of OSPG

The mineral phases of the raw materials and OSPG products were identified using X-ray diffraction (XRD; Malvern PANalytical Empyrean, Almelo, The Netherlands) with Cu K α radiation. Data were collected in the 2θ range of 5–80° with a step size of 0.02° and a counting time of 0.5 s per step. Phase identification was performed using HighScore Plus 3.0.5 software. The thermal behavior of raw materials was investigated using simultaneous thermogravimetric and differential scanning calorimetry (TG-DSC, STA449F3, NETZSCH, Selb, Germany) in air, from room temperature to 1200 °C at a heating rate of 10 °C/min. Chemical functional groups were characterized using Fourier-transform infrared spectroscopy (FTIR, Nicolet 6700, Thermo Fisher, Waltham, MA, USA) in the wavenumber range of 400–4000 cm^{−1}.

The leaching toxicity of heavy metals in the Raw Pellets-Opt and OSPG products at different sintering temperatures was evaluated using the Toxicity Characteristic Leaching Procedure (TCLP). Specifically, the raw material mixture and crushed OSPG-Opt particles were sieved to ≤ 9.5 mm and extracted with acetic acid solution (pH 2.88 ± 0.05) at a liquid-to-solid ratio of 20:1 (v/w) on a horizontal shaker for 18 ± 2 h, then filtered through a 0.45 μm membrane. The concentrations of heavy metals in the resulting leachate were determined using an Inductively Coupled Plasma Atomic Emission Spectrometer (ICP, Teledyne Leeman Labs, Prodigy7, Hudson, NH, USA).

2.4.3. Morphology Characterization of OSPG

The internal microstructure of the OSPG particles and the fracture surface morphology of the final products were examined by scanning electron microscopy (SEM; QUANTA FEG 450, FEI Co., Ltd., Hillsboro, OR, USA). Elemental composition and distribution at selected micro-regions were analyzed using an energy dispersive spectrometer (EDS; IE300X, Oxford Instruments, Oxfordshire, UK) attached to the SEM.

3. Results

3.1. Thermal Analysis of Raw Materials

Figure 3 presents the TG–DSC curves for OS, PCS, and SFCCs. For OS, the major mass loss below 500 °C is attributed to the evaporation of free and bound water, followed by the exothermic combustion of residual petroleum hydrocarbons, as evidenced by a distinct exothermic peak at 543.8 °C [27]. The subsequent mass loss (Stage III) primarily stems from the decomposition of calcite, corresponding to the endothermic peak at 719.0 °C. A slight mass loss in Stage V, accompanied by an endothermic peak at 1034.2 °C, is likely due to the decomposition of residual anhydrite [28]. For PCS, mass loss occurs due to dehydration (Stage I) and dehydroxylation of clay minerals at 463.9 °C (Stage II) [29,30]. The endothermic peak at 574.9 °C corresponds to the α – β quartz phase transition, while the prominent endotherm at 1130.3 °C indicates the melting of aluminosilicate components [31]. Regarding the SFCC, it exhibits a total mass loss of only 2.51% during moisture removal (Stage I), followed by a minor loss associated with the pyrolysis of carbonaceous deposits (Stage II) [32]. The negligible mass loss of 0.29% in Stage III confirms its high thermal stability. The two exothermic peaks observed at 984 °C and 1040 °C are attributed to the crystallization of primary mullite and the formation of cristobalite, respectively [33]. Based on the decomposition of anhydrite in OS at approximately 1034.2 °C, the formation of cristobalite in SFCCs at around 1040.7 °C, as well as the prominent endothermic melting

peak of aluminosilicate components in PCS at about 1130.3 °C, which marks the onset of liquid-phase sintering, the sintering temperature range was set as 1100–1180 °C to ensure the complete progression of key reactions in each component and to utilize an effective window for viscous flow sintering.

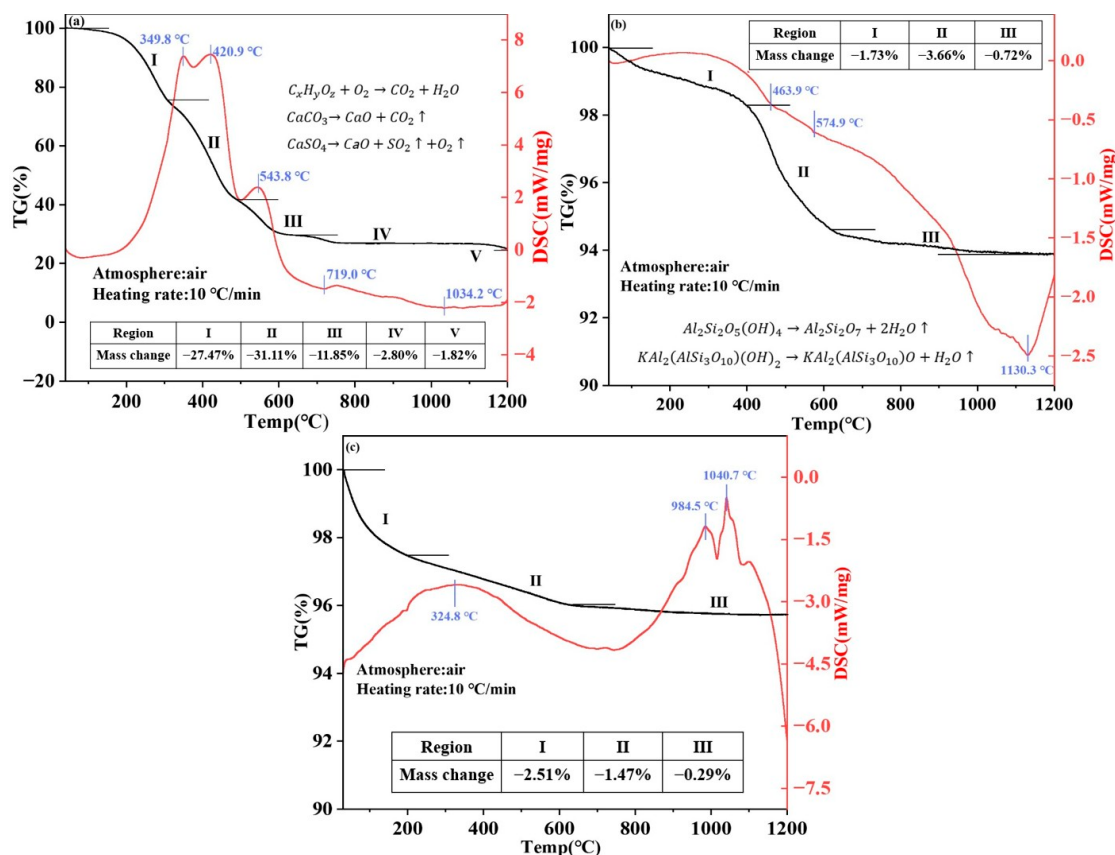


Figure 3. TG–DSC curves of raw materials. (a) OS, (b) PCS, (c) SFCC.

3.2. Single-Factor Analysis

During the high-temperature sintering of OSPG, increasing the sintering temperature and extending the holding time promote interparticle bonding and reduce porosity, leading to progressive microstructural densification. This evolution enhances both the mechanical strength and apparent density, resulting in a strong correlation between these two properties [4].

As shown in Figure 4a, the 1 h water absorption gradually decreased with increasing OS concentration and stabilized at higher levels. The initial reduction is attributed to the combustion of organic matter, which creates micropores, while simultaneous melt formation fills pores and reduces interconnected channels, collectively decreasing water absorption. Beyond a certain threshold, excessive organic volatilization increases porosity; however, this effect is counterbalanced by the diminishing skeletal components (SiO_2 and Al_2O_3) and the progressive formation of continuous glassy networks, which stabilize water absorption and indicate a transition from open to semi-closed pore structures. Both the bulk density and compressive strength peaked at an OS concentration of 30% before declining. The initial enhancement (20–30% OS) is attributed to the reduced proportion of skeletal oxides and elevated fluxing oxides, promoting extensive melting that forms surface glaze-like layers and internal liquid phases, thereby increasing density and strength. Further addition of OS (>30%) introduces excessive organic content. Its combustion creates open pores that disrupt the matrix continuity, generating stress concentration points and

reducing compressive strength. Additionally, gases trapped by viscous liquid phases form closed pores, collectively decreasing the apparent density.

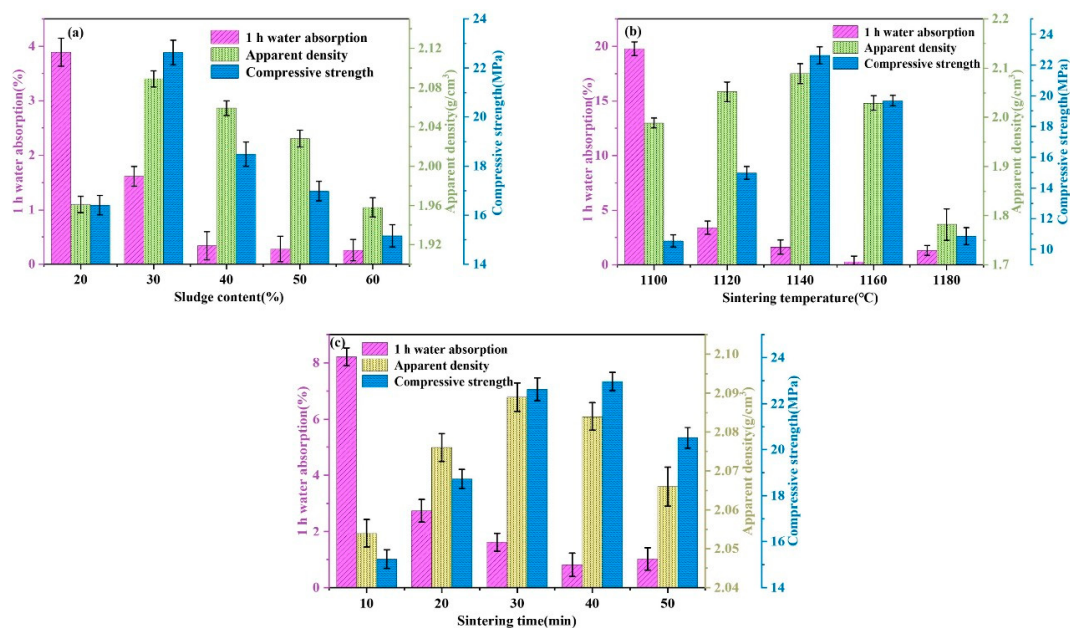


Figure 4. Effect of different OS concentrations and sintering conditions on the basic properties of OSPG. (a) OS concentration, (b) sintering temperature, and (c) sintering time.

As illustrated in Figure 4b, sintering temperature significantly influences the basic properties of OSPG. Increasing temperature progressively reduces 1 h water absorption, with a pronounced decline observed between 1100 °C and 1120 °C. Conversely, the apparent density and compressive strength initially increase before decreasing, peaking at 1140 °C. This behavior arises from enhanced liquid phase formation at elevated temperatures, promoting complete vitrification that develops a glazed surface layer while inducing internal microstructural densification [34]. When the temperature exceeded 1140 °C, an excessive liquid phase induced compressive over-firing, causing the interconnection of previously isolated pores and degradation of densification, manifesting as reduced strength and density. At 1180 °C, extreme material melting resulted in compressive deformation accompanied by the substantial deterioration of mechanical properties. Figure 4c demonstrates the effect of holding time. Water absorption continuously decreased from 8% to 1.5% with prolonged sintering, while the apparent density and compressive strength initially increased and then declined, peaking between 30 and 40 min. This pattern stems from more complete solid-phase reactions among the raw materials facilitated by longer sintering duration. The resultant increase in liquid phase volume formed pore-sealing glassy networks upon cooling, thereby reducing absorption while elevating density and strength. Beyond 40 min, extended sintering promoted pore interconnection through excessive liquid phase formation.

In summary, the compressive strength of the ceramsite reached its respective maximum within the experimental ranges at an OS content of 30%, a sintering temperature of 1140 °C, and a sintering time of 40 min. Consequently, the observed strength peak directly served as the criterion for defining the 0-level of both OS content and sintering temperature. Considering that excessively prolonged sintering durations also result in heightened energy consumption, a comprehensive evaluation identified an oil sludge content of 30%, a sintering temperature of 1140 °C, and a sintering time of 30 min as the central point (0 level) for the response surface design. Regarding the subsequent naming of OSPG, except for the

marked conditions, all other conditions are the zero-level conditions for response surface analysis (OS content 30%, sintering temperature 1140 °C, and sintering time 30 min).

3.3. Response Surface Analysis on Strength

3.3.1. Model Construction and Significance Analysis

The experimental design for the BBD model was based on the results of single-factor experiments, as shown in Table 4. The experimental data were fitted using a quadratic regression model in Design-Expert 13.0.5.

Table 4. Experimental scheme and the corresponding results.

Test Number	OS Concentration (%)	Sintering Temperature (°C)	Sintering Time (min)	Compressive Strength (MPa)
	A	B	C	
C1	20	1120	30	10.16
C2	30	1140	30	22.57
C3	30	1160	40	15.83
C4	30	1140	30	23.22
C5	40	1160	30	13.25
C6	20	1140	20	12.60
C7	30	1140	30	22.75
C8	40	1140	40	15.85
C9	30	1160	20	13.89
C10	20	1160	30	13.09
C11	30	1140	30	22.42
C12	30	1140	30	22.32
C13	40	1120	30	12.88
C14	40	1140	20	14.22
C15	30	1120	40	14.79
C16	30	1120	20	9.93
C17	20	1140	40	15.70

As summarized in Table 5, the model exhibits a high F-value of 168.82 and a p -value < 0.0001 , indicating that it is highly statistically significant. The lack-of-fit p -value of 0.1738 is not significant relative to the pure error, suggesting a 17.38% probability that such a value could occur due to noise. The coefficient of determination ($R^2 = 0.9954$) closely matches the adjusted R^2 ($R^2_{Adj} = 0.9895$) and the predicted R^2 ($R^2_{Pred} = 0.9481$), which indicates that the regression model adequately explains the process. Furthermore, a low coefficient of variation (C.V. % = 2.91 < 10) and a high adequate precision ratio (34.7859 > 4) substantiate the model's reliability and precision.

Table 5. Variance analysis of the compressive strength regression model.

Source	F-Value	p -Value	
Model	168.82	< 0.0001	significant
A	12.18	0.0101	
B	38.79	0.0004	
C	74.86	< 0.0001	
AB	7.38	0.0299	
AC	2.43	0.1627	
BC	9.60	0.0173	
A ²	412.67	< 0.0001	
B ²	604.84	< 0.0001	
C ²	219.18	< 0.0001	
Lack of Fit	2.79	0.1738	not significant
$R^2 = 0.9954$	$R^2_{Adj} = 0.9895$	$R^2_{Pred} = 0.9481$	

To validate the accuracy of the regression model, all model terms were assessed at a significance level of $\alpha = 0.05$. Terms with p -values less than 0.05 were considered significant and retained in the final model, whereas those with p -values greater than 0.1 were deemed non-significant and excluded. According to this criterion, the linear terms (A , B , C), interaction terms (AB , BC), and quadratic terms (A^2 , B^2 , C^2) are significant, while the AC interaction term is not. After removing the non-significant terms, the refined regression model for compressive strength is given by Equation (3):

$$Y = 22.66 + 0.58 \times A + 1.04 \times B + 1.44 \times C - 0.64 \times A \times B - 0.37 \times B \times C - 4.66 \times A^2 - 5.66 \times B^2 - 3.4 \times C^2 \quad (3)$$

3.3.2. Analysis of Response Surface Interactions

To intuitively analyze the interactive effects of oil sludge (OS) content, sintering temperature, and sintering time on the compressive strength of ceramsite, response surface analysis was performed based on the fitted regression model. This analysis generated three-dimensional response surface plots and two-dimensional contour plots under extreme conditions, as shown in the accompanying figure.

Figure 5 illustrates the interactive effects of OS concentration and sintering temperature on compressive strength. As shown in Figure 5a, elliptical contour lines reveal a synergistic effect between OS content and sintering temperature. This synergy arises because OS content alters the oxide composition of the system, while temperature governs reaction kinetics. The balanced interaction between these two factors within the optimal window promotes the formation of a densified, high-strength microstructure. The central location of the extremum point suggests this region represents a potential optimal parameter combination. Figure 5b demonstrates that under a fixed holding time, compressive strength initially increases, then decreases as the OS concentration varies from 20% to 40%. This trend arises because moderate OS addition ($\leq 30\%$) reduces skeletal components ($\text{SiO}_2/\text{Al}_2\text{O}_3$) while increasing fluxing oxides (CaO , MgO , Fe_2O_3 , Na_2O , and K_2O), promoting intergranular melting where feldspathic materials penetrate crystalline interfaces, thereby enhancing strength. Beyond 30% OS, excessive organic volatilization generates gas pockets trapped by viscous melts, coupled with diminished aluminosilicate framework formation, collectively reducing strength. When varying the sintering temperature between 1120–1160 °C at a fixed OS concentration, the response surface exhibits more pronounced changes than OS variation at a fixed temperature, although there is a similar rise–fall pattern. Specifically, insufficient densification at lower temperatures yields porous structures with compromised strength. Progressive temperature elevation increases liquid phase formation, developing surface vitreous layers and internal densification that augment strength. However, exceeding 1140 °C induces over-sintering, where interconnected pores disrupt microstructural integrity, ultimately diminishing strength [35].

Figure 6 illustrates the interactive effects of sintering temperature and holding time on the compressive strength of OSPG at a fixed OS concentration of 30%. As shown in Figure 6a, the elliptical contour lines with dense and uniform distribution indicate statistically significant interactions between these two parameters within specific ranges. Under constant OS content, compressive strength initially increased with rising sintering temperature, reaching a maximum of 22.75 MPa at 1140 °C, beyond which it gradually declined. Similarly, at fixed sintering temperatures, strength exhibited a comparable rise–fall pattern with prolonged holding time, peaking at 30 min. This enhancement is attributed to extended solid-state reactions that generate an additional liquid phase, which forms pore-filling glassy phases upon cooling that facilitate microstructural densification and improve mechanical strength.

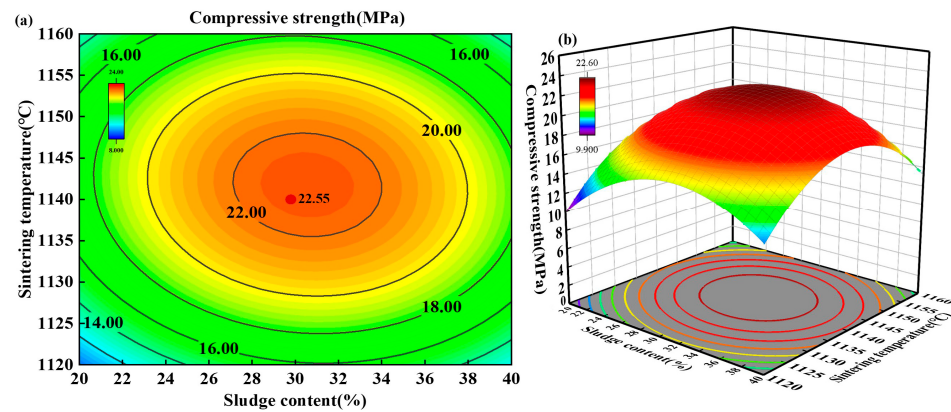


Figure 5. (a) The contours and (b) response surfaces of the interaction of A (OS concentration) and B (sintering temperature) on compressive strength.

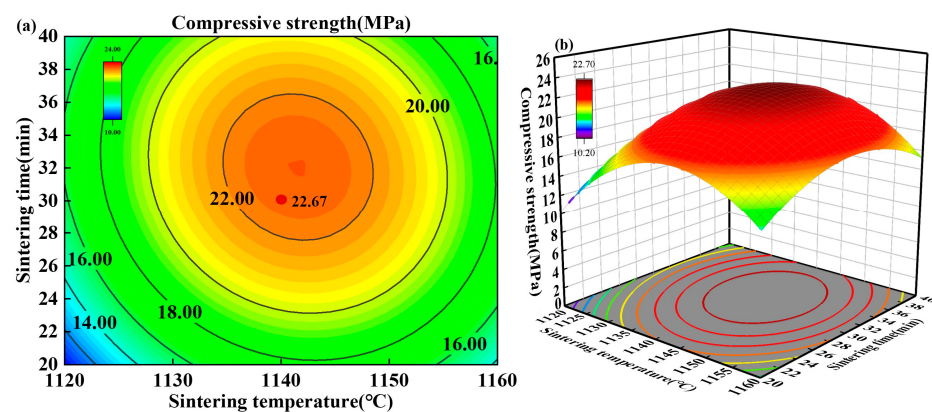


Figure 6. (a) The contours and (b) response surfaces of the interaction of B (sintering temperature) and C (sintering time) on compressive strength.

3.3.3. Model Validation

In the response surface methodology experimental design, the normal probability plot of residuals is used to verify model assumptions and detect potential outliers. As shown in Figure 7, the observed points align closely with the reference line, indicating that the residuals from the compressive strength regression analysis exhibit a random distribution with no significant outliers. This confirms that the developed mathematical model accurately represents the experimental system. The symmetric distribution of the residuals around the diagonal further supports the robustness of the parameter estimates and reflects a strong agreement between the model predictions and the experimental data.

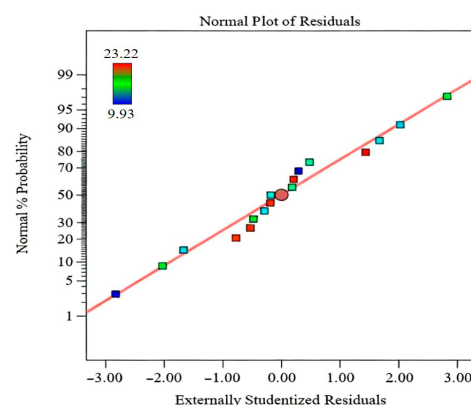


Figure 7. Compressive strength residual plot.

Furthermore, model validity and reliability were evaluated by comparing predicted values with experimental measurements. As shown in Figure 8, the relationship between actual and predicted compressive strength values demonstrates high consistency, evidenced by data points clustering evenly about the diagonal line with minimal deviation. This close alignment confirms that the BBD-derived optimal conditions are experimentally verifiable and statistically robust.

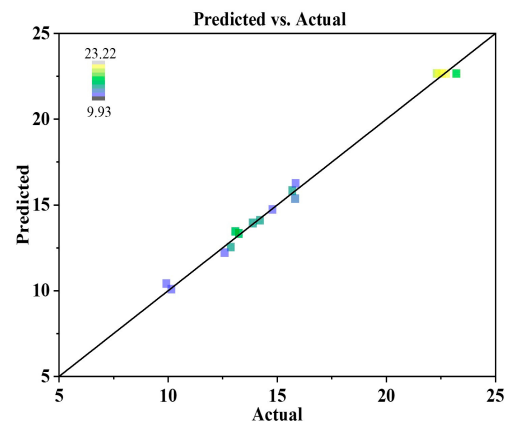


Figure 8. Graph of the relationship between the actual compressive strength values and the predicted values.

3.3.4. Experimental Optimization and Verification Characterization

The numerical optimization module of the Design-Expert software was employed to determine the sintering parameters that maximize particle strength within the experimental range. As summarized in Table 6, the model-predicted optimum conditions were 30.49% OS content, a sintering temperature of 1141.51 °C, and a sintering time of 32.01 min, yielding a predicted maximum particle strength of 22.85 MPa. To facilitate practical experimentation, these values were slightly adjusted to 30.5% OS content, 1142 °C, and 32 min. Three independent validation experiments conducted under these adjusted conditions gave an average measured particle strength of 23.12 MPa. The relative deviation between the experimental and predicted values was only 1.18%, well below the 5% threshold, confirming the high predictive accuracy and practical reliability of the developed model.

Table 6. Optimal solution under comprehensive conditions as generated by the software, and the actual value.

Factor	A	B	C	Compressive Strength (MPa)
Predicted	30.5%	1142 °C	32 min	22.85
Actual				23.12
Relative deviation (%)	-	-	-	1.18

Furthermore, the fundamental performance of the OSPG (OSPG-Opt) prepared under optimal conditions was assessed, with the findings presented in Table 7. The data demonstrate that OSPG-Opt fully complies with the Chinese standard T/CSTM 00548-2022 for high-strength lightweight aggregates produced from solid waste, meeting all specified requirements for structural and building materials [25].

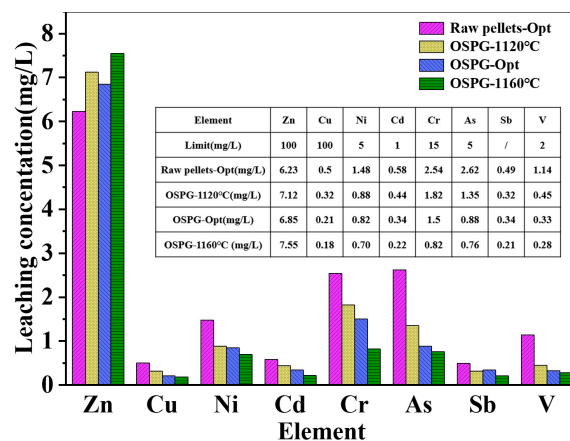
Table 7. Basic performance and indicators of OSPG-Opt.

Indexes	OSPG-Opt	Limit Value
Cylinder compressive strength (MPa)	15.58	≥ 10.5
Bulk density (kg/m^3)	1012.50	≤ 1200
Apparent density (kg/m^3)	2089.02	/
Water absorption (%)	1.61	≤ 10
Softening coefficient	0.83	≥ 0.8

Light aggregate with high strength prepared from solid waste, T/CSTM 00548-2022, PR China.

3.4. Leaching Toxicity Analysis

Heavy metal leaching toxicity is a standard method to evaluate the environmental safety of ceramsite, assessing the potential release of heavy metals that could pose risks to ecosystems and human health. The target heavy metals Zn, Cu, Ni, Cd, Cr, As, Sb, and V) were selected based on their prevalence in the raw materials and regulatory significance (GB 5085.3-2007) [36]. Figure 9 compares the leaching concentrations of key heavy metals between unsintered raw pellets-Opt and samples sintered at different temperatures. With increasing sintering temperatures, the leaching concentrations of all detected heavy metals except Zn decreased significantly. This reduction is attributed to the formation of liquid phases and the crystallization of stable mineral phases during sintering, which encapsulate heavy metal ions within the glassy phase and incorporate them into crystal structures, thereby enhancing their chemical stability. Additionally, a dense, glaze-like surface layer formed on the ceramsite, further inhibiting heavy metal leaching. In contrast, the slight increase in Zn leaching is due to its relatively low boiling point, causing partial volatilization during sintering and subsequent recondensation on pore surfaces, which increases its accessibility to leaching agents [37].

**Figure 9.** Heavy metal leaching concentrations of the ceramsite at different sintering temperatures.

Therefore, the production of ceramsite from solid waste offers significant environmental benefits. This approach not only facilitates the recycling of solid waste, reducing the risk of heavy metal contamination from stockpiling, but also decreases the consumption of natural aggregates. The addition of GP further helps lower the sintering temperature, contributing to energy savings. Although long-term stability under practical conditions requires further investigation, most heavy metals are effectively encapsulated within stable mineral phases. Moreover, the dense glazed surface of ceramsite exhibits low reactivity with water and air [38]. As ceramsite is primarily used in construction and often embedded in cement matrices, its exposure to the environment is further minimized.

3.5. XRD Analysis

Figure 10a,b present the XRD patterns of OSPG prepared with different OS contents and sintering temperatures. The primary crystalline phases identified in all OSPG samples are quartz (SiO_2), anorthite solid solution ($\text{Na}_{1.92}\text{Ca}_{2.08}\text{Si}_{10.00}\text{Al}_{6.00}\text{O}_{32.00}$), hematite (Fe_2O_3), and albite ($\text{NaAlSi}_3\text{O}_8$). While varying the OS content and sintering temperature induces no phase transformation, it significantly affects the diffraction peak intensities, indicating crystallographic evolution. As shown in Figure 10a, increasing the OS content leads to a progressive attenuation of the quartz (SiO_2) diffraction peaks, coupled with a concurrent enhancement of plagioclase solid-solution reflections. This phase evolution is driven by the increasing Ca/Si ratio (from 0.11 to 0.19 as OS content rises from 20% to 40%), which promotes the albite-mediated conversion of quartz into the anorthite solid solution, thereby suppressing discrete albite crystallization [39]. The resulting interlocking structure between the anorthite solid solution and the glass phase significantly contributes to the mechanical strength and stability of OSPG. Concurrently, the elevated OS content introduces more Fe_2O_3 into the raw material mixture. Under the oxidative sintering atmosphere, iron remains stable as hematite, leading to a corresponding increase in its diffraction peak intensity.

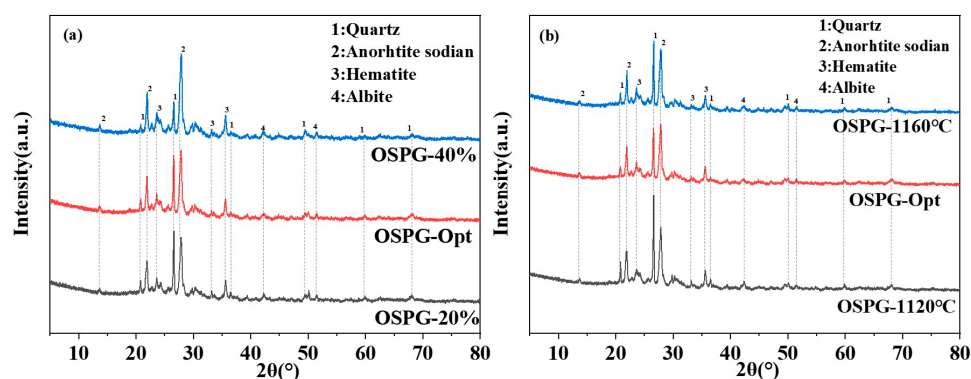


Figure 10. XRD spectra of OSPG under different OS concentrations and sintering conditions: (a) OS concentration, (b) sintering temperature.

The XRD patterns of OSPG sintered at different temperatures are displayed in Figure 10b. No phase changes are observed across the temperature range studied. However, as the temperature increases from 1120 °C to the optimum (OSPG-Opt), the quartz peak intensity decreases notably, while the anorthite solid solution diffraction peaks intensify. No further significant changes occur beyond the optimal temperature, indicating that a stable anorthite solid solution forms under the optimized sintering conditions. Hematite (Fe_2O_3), once formed in an oxidizing atmosphere, exhibits high-temperature stability. Its crystal structure remains intact despite variations in sintering parameters; thus, the corresponding XRD peak intensity depends solely on its original content.

3.6. FTIR Characterization Analysis

Figure 11 presents the FTIR spectra of OSPG ceramics prepared under different sintering conditions, together with the spectrum of the unsintered raw pellet with the optimal formulation (raw pellet-Opt). In the raw pellet-Opt spectrum, the peaks at 3697 cm^{-1} and 3620 cm^{-1} are attributed to the O-H stretching vibrations of crystalline water [40,41]. Peaks at 1460 cm^{-1} and 1324 cm^{-1} correspond to the asymmetric stretching vibration of CO_3^{2-} and the stretching vibration of SO_4^{2-} , respectively [42]. The prominent peak at 1034 cm^{-1} primarily originates from the Si-O stretching vibration in amorphous SiO_2 . After high-temperature sintering, the intensity of this peak decreases, indicating the consump-

tion of amorphous SiO_2 through reactions with other metal oxides to form new silicate phases, which modifies the Si-O bonding environment [43]. When the OS content increases from 20% to 40%, the Si-O peak intensity further diminishes, which is consistent with the reduced proportion of SiO_2 in the initial mixture. Additional features include a peak at 536 cm^{-1} assigned to Al-O stretching, a weak peak at 776 cm^{-1} related to Si-O-Si stretching of polymerized glass networks, and peaks at 457 cm^{-1} and 568 cm^{-1} associated with Fe-O vibrations [44–46]. While the positions of these functional groups remain largely unchanged in OSPG, their relative intensities vary. A broad peak emerges at 3436 cm^{-1} due to O-H stretching vibrations [47]. Collectively, the FTIR analysis confirms that high-temperature sintering facilitates the formation of an aluminosilicate network structure from Si, Al, Ca, and Fe constituents, which is responsible for the enhanced mechanical strength and chemical stability of the resulting ceramsite [48].

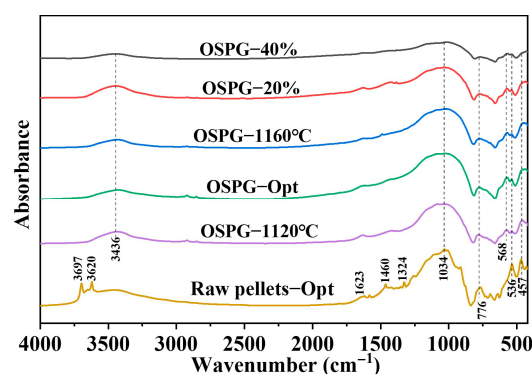


Figure 11. FTIR spectra of raw pellets-Opt and OSPG under different sintering conditions.

3.7. Microstructure Analysis

Scanning electron microscopy (SEM) images of OSPG ceramsites sintered at different temperatures reveal a clear microstructural evolution (Figure 12a–f). Increasing the sintering temperature promoted particle fusion and viscous flow, leading to smoother internal surfaces and the development of multiscale porosity. A key microstructural feature is the formation of abundant lamellar anorthite solid solution crystals (5–20 μm in length), which create a continuous three-dimensional network and form an interlocking structure with the glassy matrix. This interlocked architecture, combined with optimized pore structures, significantly enhances the mechanical strength of the material. As shown in Figure 12a,b, at a sintering temperature of $1120\text{ }^{\circ}\text{C}$, insufficient solid-state diffusion, and limited interparticle bonding prevail in the OSPG matrix, reflecting incomplete sintering at this temperature. Under these suboptimal thermal conditions, only sparse acicular anorthite solid solutions precipitate from the liquid phase, and their growth is restricted within the glass matrix, resulting in truncated crystal dimensions. This underdeveloped microstructure corresponds to diminished compressive strength in the resultant ceramsite. When the sintering temperature is increased to the optimum level (Figure 12c,d), enhanced melting generates a substantial viscous glass phase. This facilitates the growth of well-developed anorthite solid solution crystals while simultaneously trapping gaseous by-products formed during sintering. The subsequent release of these gases, when internal pressure exceeds the surface tension of the surrounding liquid phase, results in the formation of multiscale pores. The remaining non-crystallized glass phase solidifies upon cooling, yielding a densified microstructure characterized by semi-enclosed pores and a consolidated surface texture, with minimal large macropores [49]. The presence of such pores is beneficial for heavy metal immobilization. However, at $1160\text{ }^{\circ}\text{C}$ (Figure 12e,f), near-complete component melting generates an excessive liquid phase that reduces melt viscosity, enabling small pores to coalesce into detrimental macropores that compromise

structural integrity [50]. Simultaneously, overgrown lamellar anorthite solid solutions undergo agglomeration, creating crack-prone interfaces under stress, while expanded melt erosion further degrades mechanical performance.

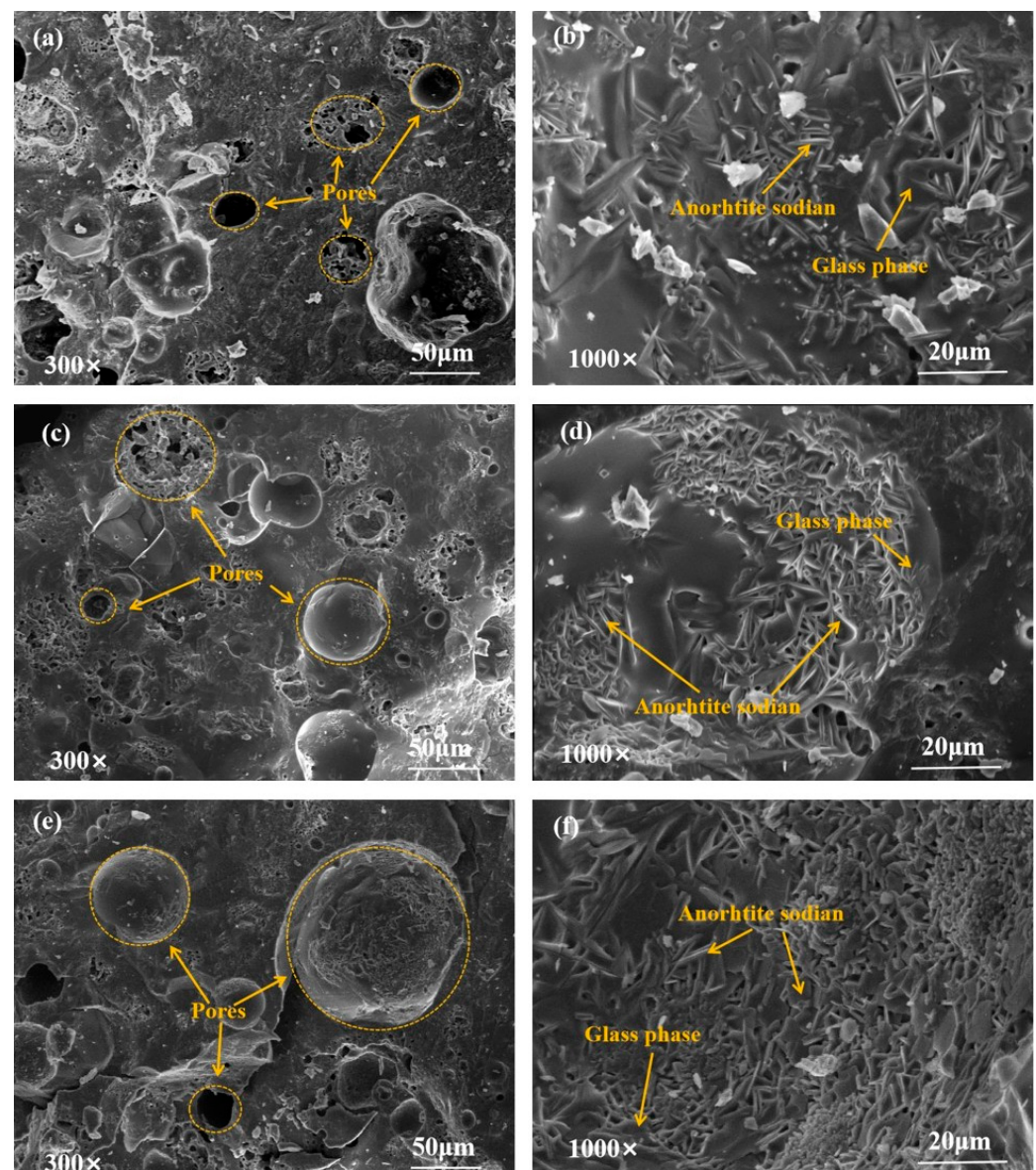


Figure 12. SEM images of OSPG at different sintering temperatures: (a,b) OSPG-1120 °C, (c,d) OSPG-Opt, (e,f) OSPG-1160 °C.

The SEM–EDS results of the OSPG-Opt sample are shown in Figure 13. The elemental composition analysis shows that the region is predominantly composed of O, Si, and Al, confirming the development of an aluminosilicate-based liquid phase during sintering. The atomic ratios of Na, Ca, Si, Al, and O at Point a closely match the theoretical composition of the anorthite solid solution ($\text{Na}_{1.92}\text{Ca}_{2.08}\text{Si}_{10.00}\text{Al}_{6.00}\text{O}_{32.00}$) within the boundaries of experimental error, identifying the layered crystals as this phase. The detected Fe signal at this location suggests the coexistence of hematite within the anorthite matrix, which aligns with the XRD findings. In contrast, Points b and c exhibit the typical composition of a glassy phase, containing O, Si, and Al as the primary elements, along with Na, K, Ca, Mg, and Fe in solid solution. The presence of Mg signifies that MgO from the decomposition of olivine dissolved into the amorphous glass phase, where it functioned as a fluxing agent to enhance the liquid-phase sintering process.

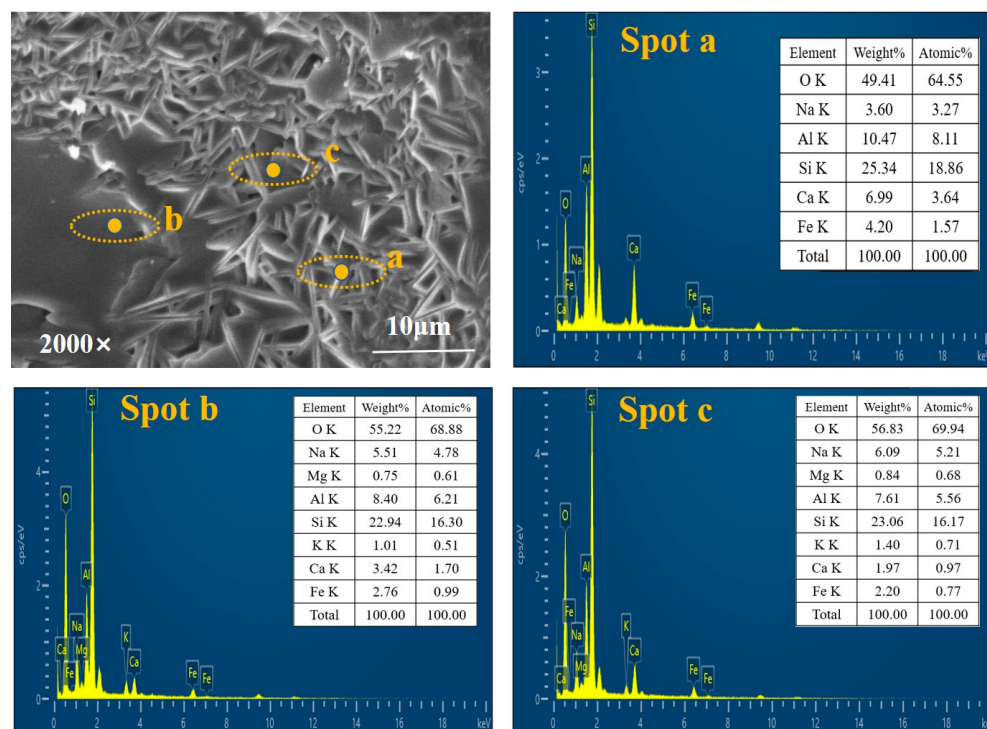


Figure 13. SEM–EDS analysis of OSPG–Opt.

3.8. Mechanism of OSPG Formation

Figure 14 schematically illustrates the mechanisms underlying strength development and heavy metal immobilization in OSPG. Initially, raw materials are thoroughly mixed and granulated. During early-stage sintering, the decomposition of small-molecule water and organic pollutants generates interparticle pores (Equation (4)). As temperature increases, crystalline water evolution and thermal decomposition of certain minerals release additional gases, forming predominantly closed pores while the solid phase remains incompletely melted (Equations (5) and (6)). In the final stage, carbonates and sulfates decompose, releasing CO_2 and SO_2 (Equations (7) and (8)), while magnesium–iron olivine undergoes thermal decomposition as the solid phase begins melting (Equations (9) and (10)). SiO_2 and Al_2O_3 react with flux agents (e.g., CaO and Na_2O), forming a liquid phase of specific viscosity. Exceeding the optimal sintering temperature further reduces system viscosity, interconnecting closed pores into open pores that compromise mechanical strength and may cause structural collapse. Organic matter combustion provides additional thermal energy, while flux agents lower the melting temperature, reducing energy consumption [51].

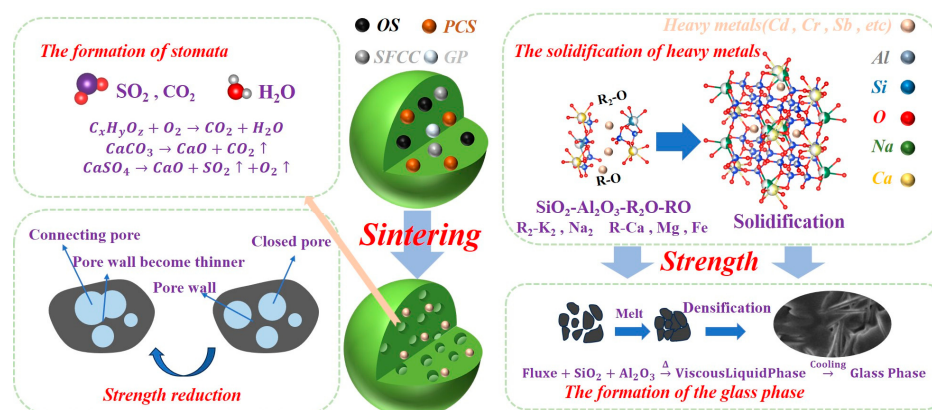
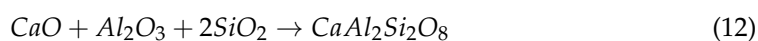
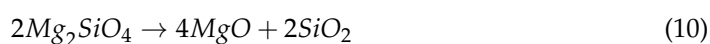
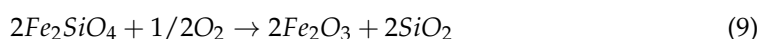
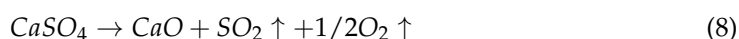
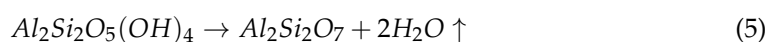


Figure 14. Formation of OSPG strength and heavy metal immobilization process.

The high mechanical strength of OSPG originates from crystalline phases—including anorthite solid solution, albite, and hematite—formed during high-temperature sintering, coupled with the interlocking structure between the anorthite solid solution and the glassy matrix [52]. Notably, the anorthite solid solution, formed through the atomic-scale integration of anorthite and albite components, constitutes the material's microstructural framework (Equations (11) and (12)). Concurrently, magnesium derived from olivine dissolution acts as a flux in the glass phase, enhancing liquid-phase sintering without forming separate magnesium-bearing crystalline phases (Equation (10)). The sintering process significantly reduces heavy metal leachability through two mechanisms: incorporation of heavy metal ions into the crystal lattice of the anorthite solid solution, and their encapsulation within the dense glassy phase. A glaze-like surface layer provides an additional leaching barrier. Therefore, OSPG-Opt, fabricated through the co-sintering of multi-source petrochemical hazardous wastes, meets requirements for environmentally friendly construction materials and demonstrates strong practical application potential.



4. Conclusions

This study successfully fabricated high-strength eco-friendly ceramsite (OSPG-Opt) via the synergistic sintering of petrochemical hazardous wastes—oil sludge (OS), spent fluid catalytic cracking catalyst (SFCC), and petrochemical-contaminated soil (PCS)—assisted by waste glass powder (GP) as a flux. The main conclusions are as follows:

1. Process optimization based on response surface analysis established that interactions among OS content, sintering temperature, and time critically influence particle strength. Under optimal conditions (30.5% OS, 1142 °C, 32 min), the ceramsite achieved a compressive strength of 23.12 MPa, conforming to the Chinese standard T/CSTM 00548-2022 for high-strength lightweight aggregates from solid waste.
2. The ceramsite exhibits excellent environmental compatibility, with heavy metal leaching concentrations below GB 5085.3-2007 limits. Effective immobilization results from the structural incorporation of metals into the anorthite lattice and encapsulation within the dense glass phase, reinforced by a sealing surface glaze.
3. Microstructural characterization identifies quartz, anorthite solid solution, hematite, and albite as key crystalline phases. Mechanical strength originates from an interlocking architecture between anorthite crystals and the glass matrix, promoted by MgO fluxing from decomposed olivine.

This work presents a sustainable and integrated approach to valorizing multi-source petrochemical wastes, demonstrating their feasibility in producing structurally and environmentally viable construction materials.

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