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ARTICLE

Effects of Raw Materials, Sintering Conditions, and Stabilizers on High Volume M3-Alite Synthesis

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ABSTRACT

The study presents the process to synthesize and characterize the M3 polymorph of Tricalcium oxy silicate, also known as alite ($Ca_3O(SiO_4)$), a major component in Portland cement. An optimized solid-state reaction protocol has been established for synthesizing high volume pure M3-alite. The effects of raw materials, sintering conditions, degree of compaction, and stabilizers were studied on the synthesis process of the pure phase. The synthesized M3-alite powder was analyzed using X-ray diffraction (XRD), Brunauer–Emmett–Teller (BET), and particle size analysis. The results indicated that high volume M3-alite polymorph (800 gm in one cycle) can be synthesized with a purity higher than 95% by sintering under optimized raw materials and sintering conditions at 1650 °C for 180 min without compaction techniques. Stabilization of the M3 polymorph also involved the addition of MgO as well as Al_2O_3 as stabilizers. XRD analysis confirmed the formation of the monoclinic structure (M3) of alite. The synthesized powder showed a d_{50} of 13.85 µm, a BET surface area of 777.8 ± 50 m^2/kg, and a density of 3.14 ± 0.05 g/cm^3. This optimization process allows efficient production of a superior quality M3-alite polymorph that is required in low carbon cement research for a better understanding of different attributes after the addition of chemical and mineral admixtures at different environmental conditions.

Keywords: Alite; Doping; Low Carbon Cement; Polymorph; X-ray Diffraction

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1. Introduction

Alite, or Tricalcium oxy silicate ($Ca_3O(SiO_4)$), constitutes 50%–70% of Portland cement clinker which is its main component, due to its rapid rate of hydration leading to an early development of strength[1, 2]. This calcium silicate phase can be found in seven polymorphs which differ based on their crystallographic symmetry and temperature-dependent stability. The order of polymorphic transformation during the heating process starts off with three triclinic (T_1, T_2, T_3), three monoclinic (M_1, M_2, M_3), and one rhombohedral (R) structure, which is common to both pure and doped systems. Industrial clinkers are mostly composed of the monoclinic M_3 polymorph which is known to be stabilized by minor constituents like magnesium oxide (MgO) and sulfur (S) as Calcium ions are substituted in the crystal lattice[3–5]. The stabilisation of some polymorphs depends on chemical dopants and thermal history. The M_3 form seems to be preferentially stabilized by magnesium substituting Ca^{2+} ions and sulfur seems to promote the M_1 form through bonding with aluminum and iron impurities. These substitutions change the position of SiO_4^{4-} tetrahedra and calcium coordination geometries leading to changes in hydration reactivity and mechanical properties of the cement. In cement clinker, M3 polymorph's distorted crystal structure and lattice defect sites enhance its dissolution rate compared to other forms, which contributes directly to the higher early strength developed. The following appears to be the sequence that C_3S goes through when heated[1, 2].

$$T_1 \xleftrightarrow{620\,^{\circ}\mathrm{C}} T_2 \xleftrightarrow{920\,^{\circ}\mathrm{C}} T_3 \xleftrightarrow{980\,^{\circ}\mathrm{C}} M_1 \xleftrightarrow{990\,^{\circ}\mathrm{C}} M_2 \xleftrightarrow{1060\,^{\circ}\mathrm{C}} M_3 \xleftrightarrow{1070\,^{\circ}\mathrm{C}} R \tag{1}$$

where, T = triclinic, M = monoclinic, and R = rhombohedral; subscript numbers denote symmetry variations.

During heating, the ordered polymorphic sequence reflects the increase of symmetry:

Triclinic phases ($T_1 \rightarrow T_2 \rightarrow T_3$): These low-temperature phases with complex orientations of SiO_4^{4-} tetrahedra that retard hydration exhibit lattice distortions in decreasing order.

Monoclinic phases ($M_1 \rightarrow M_2 \rightarrow M_3$): Sequences of intermediate-temperature forms of silicates with simpler arrangements. M_3 possesses structural defects that enhance water retention at reactive sites within the silicate framework.

Rhombohedral phase (R): This high-temperature phase is rarely retained in industrial clinkers due to cooling restrictions but does influence crystallization pathways during sintering.

On cooling, this transition sequence is at least partly reversible. C_3S possesses rhombohedral symmetry above 1070 °C. Furthermore, during the peak temperature excursion of clinkering cycle, the rhombohedral phase incorporates various ions in solid solution such as- Mg, Al, Fe, and so on. Because of the impurity atoms absorbed into the R phase, the transformation sequence may deviate somewhat from that obtained for pure C_3S. Impurities have a stabilizing function, influencing not just the subsequent sequence of minor phase transitions, but also helping to stabilise C_3S. Commercial clinkers are more likely to contain M_3 or M_1, or combinations of the two. **Figure 1** depicts the possibility of C_3S synthesis at different temperatures ranging from 1250 to 2150 °C. When the temperature increases beyond 1250 °C, it is often formed through the reaction of C_2S and CaO.

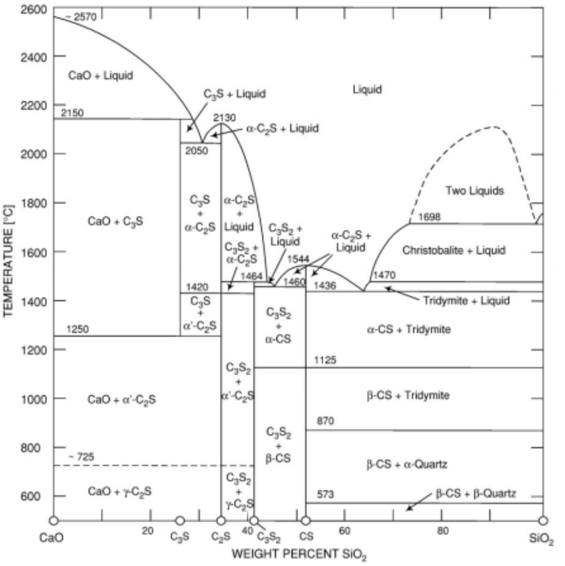

Figure 1. Phase diagram of the binary system CaO–SiO_2[2].

In industrial Portland cement clinkers, the most common form is the M3 monoclinic polymorph[6]. For deeper understanding of cement hydration mechanisms and developing new cementitious materials, it is important to synthesize pure phases of alite under laboratory conditions. However, production of high-purity M3-alite homogeneously with similar characteristics has remained a challenge. Traditional synthesis methods often involve several sintering cycles at very high temperatures for long periods which are both energy intensive and time consuming. The synthesis of C_3S has been reported at temperatures ranging from 1500 °C to 1650 °C[7–27]. Most studies used a heating rate of 5 °C/min but there was no adverse effect when C_3S was heated at 1600 °C for 3 h using the faster rate of 7 °C/min. Some samples needed to be done several times with resistance time ranging from 1 h to 24 h to attain optimal purity of phases. The free CaO content in the synthesized C_3S was generally kept below 0.5%–3%[9, 10, 13, 22–25]. Various silica sources were used, including quartz powder[9, 10, 13, 22–25], Brazilian quartz, gel silica, and amorphous fumed silicas[7, 25]. Some studies incorporated dopants like Al_2O_3, Fe_2O_3, MgO, ZnO, La_2O_3 into the raw mix[8, 9, 11, 18, 19, 23, 24].The raw materials were typically mixed using lab mixers or ball mills for 3–24 h. Some studies included pretreatment steps like calcination at 1000 °C–1100 °C[10, 14, 18] or pressing into pellets/discs before sintering[9, 20–22, 27].

This research paper aims to improve the solid-state reaction route towards the M3 polymorph of alite, a significant constituent in cement using industrial cement, while focusing on property enhancements. To achieve this, the study will focus on several distinct objectives. First, the study attempts to optimize the selection and preparation of the raw materials, so that the best possible ingredients are utilized for the synthesis of M3-Alite. Second, the research intends to find the optimal sintering conditions particular to the sintering of the desired M3 polymorph. The study also examines the effect of several stabilizers on the multiple polymorphs and phase formation to better understand the crystallization mechanisms. Finally, the research concentrates on determining the levels of impurity and crystallinity of the produced M3-alite to ensure high quality products. This work intends to provide a better synthesis protocol that is less time-consuming and results in better product quality to explore the effect of different curing conditions regarding pure alite phase induced with different kinds of chemical and mineral admixtures. Also, improving the synthesis of high quality M3-alite is very important for the cement industry and the construction industry overall.

2. Materials and Methods

The raw materials for synthesis of M3-alite polymorph were Calcium carbonate (>98% pure from M/s. LOBA Chemie Ltd., India) and fumed silica (Aerosil 200 from M/s. Evonik Industries AG, Germany), as shown in **Figure 2**. To reduce the fineness as per Li at al.[7]; silica fume was processed at 70 RPM for 30 min in a ball mill (Make: Retsch; Model: TM300). The mass to charge was maintained in the ratio of 1:2. Both the chemical powders were fine powder having D_{50} size of 3.24 μm and 131.2 nm for Limestone and Fumed silica, respectively because higher fineness causes effective sintering at high temperature[28]. 0.75 wt.% Magnesium oxide (MgO) and 0.35 wt.% corundum (Al_2O_3), were used as stabilizers along with 98.9 wt.% mix of $CaCO_3$ and SiO_2[3, 10, 29]. The raw materials used for synthesizing pure M3-alite phase along with their molar equivalents are given in **Table 1**.


(**a**) (**b**)

Figure 2. Raw materials (**a**) Pure Limestone, and (**b**) Fumed silica.

Table 1. Characteristics and molar equivalents of raw materials.

Raw materials	Purity (%)	Molar Weight (g/mol)	Molar Equivalents
$CaCO_3$	98.26	100.09	2.844
SiO_2	99.81	60.08	0.963
MgO	98.0	40.30	0.064
Al_2O_3	99.85	101.96	0.012

For both the pure raw materials, chemical composition was determined using XRF (Make: M/s. Bruker, S4 Pioneer). It was observed that limestone powder contained Calcium carbonate ($CaCO_3$) as primary component, making up 98.26% of the powder. Fumed silica—Aerosil 200—is made up mostly of silicon dioxide (SiO_2), which makes up 99.81% by weight. The fumed silica has a high surface area, with a value of around 200 m²/g.

Pure calcium carbonate and ground silica fume were employed during the synthesis of M3-Alite phase with optimized stoichiometric ratios between calcium carbonate and silica fume. To ensure the homogeneous mixing of silica fume and $CaCO_3$, pure water was introduced in a ratio of approximately 2:1 to the dry raw materials. After weighing the raw materials as per the mix composition in **Table 1** and **Table 2**; homogeneous mixing was done using a Ball mill (Capacity: 10L, Make: Retsch; Model: TM300) for 08 h. The homogeneous mixture was poured into borosil glass and subsequently maintained in an oven at 150 °C for 8 h. Subsequently, the dry mix was allowed to cool to room temperature. During synthesis, compaction was also done to improve sintering progress and better material grain contact for effective grain diffusion [10, 28]. Therefore, for the compaction process to make pellets for M3-alite synthesis, hard steel (HS) mould having outer and inner diameter as 155 mm and 50 mm along with height of 80 mm was fabricated, as shown in **Figure 3**. The materials were pressed into cylindrical pellets with 50 mm in diameter and 20 mm in height at about 45–50 MPa uniaxial compression pressure before sintering [29]. The hydraulic press (Make: M/s. Projects & Engineering Services/PES, India) was used for this purpose, as shown in **Figure 3**.

(a) **(b)**

Figure 3. Hydraulic press set up without and with HS mould.

The compacted or un-compacted dry mix was stored in platinum (Pt) crucibles during the sintering process. After that, Pt crucibles containing blend, was exposed into a programmable electrical furnace (Make: M/s. Carbolite HTF 1800, Carbolite-Gero Ltd., UK), as shown in **Figure 4**. It is not recommended to use alumina crucibles for synthesizing pure alite phase because addition of aluminum may lead to the formation of tricalcium aluminate (C_3A) or other aluminate phases which are undesired when aiming at pure alite synthesis. The synthesis of pure M3-C_3S phase was conducted at sintering temperature of 1450 °C –1650 °C with Resistance time of 2.25–4 h. The test conditions/protocol which was used for synthesis of pure Alite phase is given in **Figure 5** and **Table 2**.

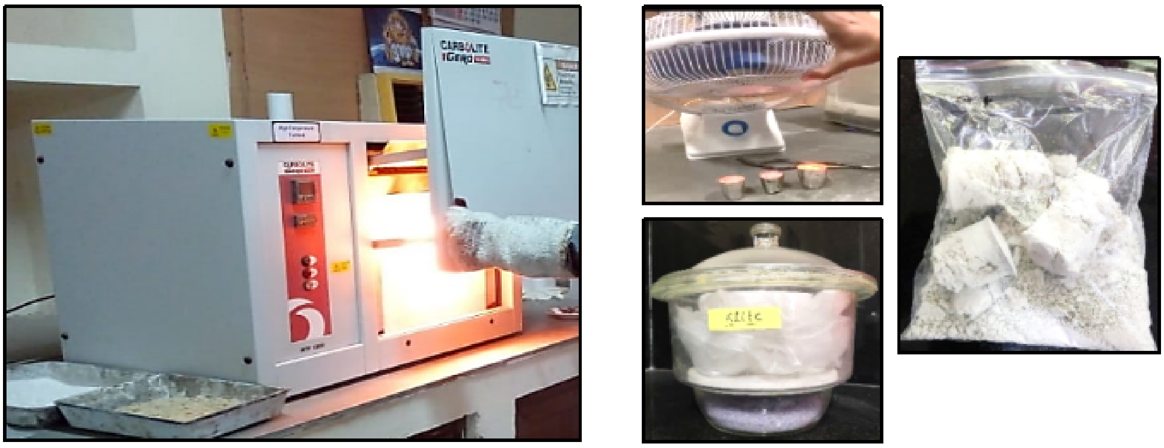

Figure 4. Operation of furnace during synthesis of alite, quenching in air, storage in desiccator of Silica Gel, and Alite sample.

After completion of the sintering process up to about 7–8 h, the Alite sample was quenched using forced (under an electric fan) and/or natural air. After quenching, the alite sample was ground using Planetary ball mill (Make: M/s. Retch GmbH-Germany, model PM 100). The alite sample was then sieved using a 90 µm sieve to produce a fine powder. After grinding, to avoid moisture, the pure phase powder was stored in silica gel desiccator and was used for further experimentation and analysis.

The characteristics of alite powder were analyzed in detail after synthesizing the pure alite phase. The particle size distribution (PSD) of the Alite powder was calculated from Laser diffraction particle size analyzers (Make: M/s. Horiba LA-950V2) which measures the size and distribution of particles. To check the crystalline phases and purity of synthesized alite, X-ray diffraction (XRD) analysis was conducted using a Rigaku DMax-2200 diffractometer with CuKα radiation. Before conducting XRD analysis, the alite powder was ground to a size smaller than 45µm and dried to ensure accurate results. Under these conditions, the XRD measurements were taken at 40 kV/40 mA with data collection for 2θ:10°–70° at a 0.5°/min scan speed. The XRD provided a deeper understanding of the crystal structure and phase composition of the synthesized alite powder. With the aim of further analyzing and interpreting the XRD measurements, the advanced software PANalytical HighScore Plus v5.0 was used. This software provided insights into the composition and purity of alite powder by efficiently identifying and quantifying its crystalline phases.

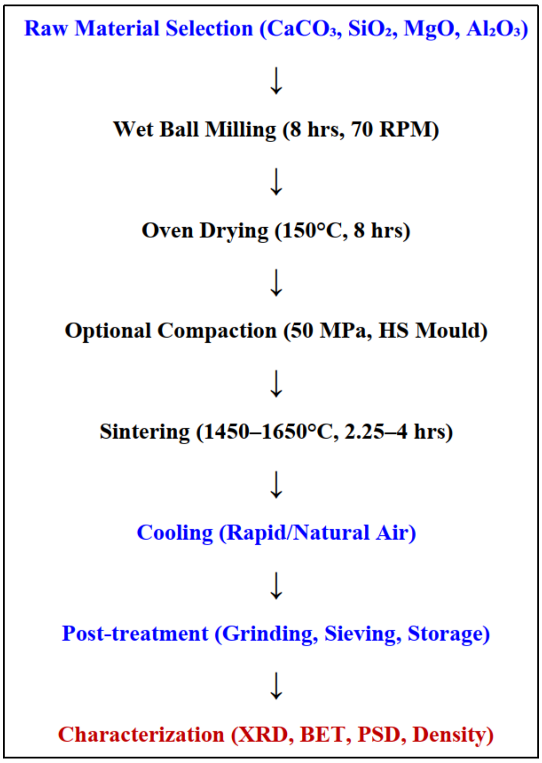

Figure 5. Flowchart summarizing the research methodology.

Table 2. Synthesis protocol for pure Alite synthesis.

Designation	Compaction	Temperature and Resistance Time	Stabilizer	Cooling
A_1	No	1650 °C, 3 h	No	Rapid
A_2	No	1450 °C, 4 h	Yes	Natural
A_3	Yes	1650 °C, 2.25 h	Yes	Rapid
A_4	No	1650 °C, 3 h	Yes	Rapid

3. Results and Discussions

3.1. Raw Material Preparation

The synthesis of high-purity M3-alite polymorph depends on multiple several key factors. One of the most important factors is the application of finely ground available raw materials, whose fineness can be optimized. This combination of materials increases the probability of attaining reactive completeness of the phases to alite. The importance of particle size in this process cannot be overstated, as it has been frequently reported that higher particle sizes increase the concentration of free lime and belite within the concrete[7]. The correlation of the particle size with the efficiency of the reaction is described based on solid-state reaction principles[30]. The increase in particle size presents a decrease in the surface area to volume ratio, which greatly affects the kinetics of these reactions. Solid-state reactions are dominantly dependent on ion diffusion, and the ease of achieving ion diffusion becomes more difficult with increasing particle dimensions. Therefore, with the use of larger particles, the quantity of undesired reactants, mainly an excess of non-reacted free lime (CaO), drastically increases due to incomplete reactions. To counter these issues while achieving desirable outcomes, a wet ball-milling technique was used. This technique applied for 08 h produces a homogeneous mixture with a specified particle size distribution. The pre-sintered mix has particles between 0.5 and 5 µm, with an average particle size (d_{50}) of about 1.75 µm. Such effective distribution of particle sizes ensures efficient reactions and formation of high-quality M3-alite polymorph. Methods like wet ball-milling, which provide precise control of particle sizes, increase the kinetics of reactions as well as their quality and purity. Such methods enable reaction surfaces with efficient ion diffusion to be maximized, greatly lessening the quantities of unreacted materials and unwanted phases. This accuracy of raw materials control is important for attaining M3-alite polymorph of high purity.

3.2. XRD Analysis

The diffractogram included in **Figure 6** elaborates the formation of different phases within various mixtures and elucidates the interplay on composition, cooling rates, and the control of temperature during the sintering process, which was intricate. The investigation showed quite complex interactions between these factors and their impact on the characteristics of Alite synthesis. For Mix A_1, the application of rapid cooling by air, the formation of T3–Alite (92.72%) was observed along with β-C_2S (6.87%) and minor amount of free lime. The lack of stabilizers like MgO and Al_2O_3 affected the mineral composition, leading instead to the triclinic phase of alite[10]. This underscored claim regarding stabilizer's influence on alite's crystalline structure reinforced further careful approach on mix compositions in synthesis of pure cementitious phases. On the other hand, Mix A_2 demonstrated distinct patterns of behavior under slower cooling rates causing the formation of γ-C_2S (40.98%). This example is rather important as it showcases the dependence of phase formation on cooling rates[31]. This information is relevant for cement industry practices where it is critical to keep the C_2S polymorphic form that would allow the cement to possess the best characteristics. The work also emphasizes the most important aspects of temperature control in the sintering stage of the process. It was discovered that, at temperatures lower than 1550 °C, the reactions were incomplete, resulting in a high concentration of free lime[2, 3, 6, 10]. This situation reveals the challenge of accurately controlling the temperature to realize the reaction of the raw materials without leaving undesired free lime in the final blend. On the other hand, it was shown that, if the temperature exceeds 1650 °C, overheating leads to some melting and the formation of non-uniform mixes in the system. This observation highlights the further need for refined control of temperature, as their deviation beyond the set point is likely to alter favorable material properties. Other aspects of the work show that sintering for long periods does improve the purity of the product but does increase the energy needed. This piece of information carries considerable weight in the field of production optimization as it shows that increased sintering times are not likely to improve product value and, in fact, may result in wasteful energy consumption. The cooling rate proved to be very significant in the stabilization of various polymorphs of alite. M3 polymorph was effectively stabilized by rapid cooling, while slow cooling transformed it into triclinic forms. This observation shows the striking need for the control of cooling rates in achieving desired crystal structures and, as a result, product attributes. The XRD results of mixtures A_3 and A_4 supported beyond reasonable doubt the formation of monoclinic M3-alite was the overriding phase which was more than 95% of the composition. This was made by adding 0.75 wt% of MgO and 0.35 wt% of Al_2O_3 which markedly increased the stability of M3 polymorph. Significantly low quantities of β-C_2S (less than 5%), as well as traces of free lime peaks, suggested that there had been almost complete reaction with the raw materials employed in the synthesis. For the samples exhibiting high M3-C_3S content, specifically Mixes A_3 and A_4, the quality of Rietveld refinement was assessed through key statistical parameters. The weighted profile R-factor (R_{wp}) ranged between 10.8% and 12.5%, while the profile R-factor (R_p) was between 8.1% and 9.6%. The goodness-of-fit (χ^2) values were found to be in the range of 1.87 to 2.14, which indicates a robust and acceptable refinement quality for multiphase cementitious systems. These residuals confirm the reliability of the reported phase quantification, particularly in distinguishing the M3-C_3S phase from overlapping peaks in the complex diffraction pattern. Thus, these results highlight the importance of additive compositions in phase formation and the attainment of the desired polymorphs in high purity. The study looked further into the effects of compaction on the purity of the M3-Alite phase. **Table 3** demonstrates that practically the compaction of dry raw materials through palletization prior to sintering did not affect purity significantly. The process of compaction yielded only a 0.80% increase in purity, which is quite modest. This is at odds with the belief that high levels of purity require compacting.

It was concluded that one synthesis cycle was sufficient for achieving optimal purity levels (more than 95%) in a high-volume synthesis of M3 polymorph of alite. This synthesis cycle should consist of a 3-h dwell period at 1650 °C with no compaction, integrated into the heating profile ramping at 8 °C/min. This methodology significantly reduces process steps and energy expenditure while maintaining the high-purity M3-Alite target. The impact of this work is not only of theoretical value to academia but also provides numerous benefits for the cement industry. It appears that by modifying temperature control, cooling rates, additive formulations, manufacturers may enhance product quality while increasing energy efficiency and production rate. The findings from this study pertaining to compaction and extended sintering time also refute industry standards regarding these factors and may invite fresh perspectives for optimizing cost and enhancing efficiency in pure cementitious phase production. It can be concluded that this broad study explores the relations between diverse factors in cement production, including the formation and stability of different existing phases. In scope, this work advances cement chemistry by deepening the understanding of phase composition, temperature, cooling rate, and processing relationships, and adds knowledge on the refinement of industrial processes for cement production.

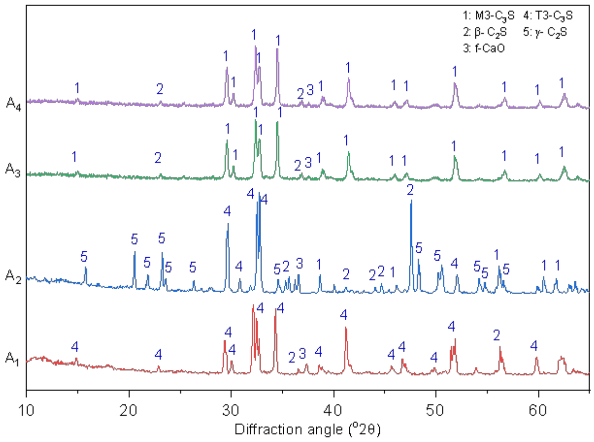

Figure 6. XRD patterns of synthesized alite samples (Mixes A_1–A_4), showing the influence of sintering conditions, compaction, and stabilizers on phase formation. Mix A_1 (no stabilizers, rapid cooling, without compaction) predominantly forms T3-C_3S and β-C_2S; Mix A_2 (stabilized, natural cooling without compaction) results in high γ-C_2S content; Mixes A_3 (with MgO and Al_2O_3 stabilizers, rapid cooling with compaction) and A_4 (with MgO and Al_2O_3 stabilizers, rapid cooling without compaction) yield high-purity M3-C_3S phase with minor β-C_2S and traces of free CaO. Peak assignments correspond to monoclinic M3-C_3S, β-C_2S, T3-C_3S, γ-C_2S, and free lime phases.

Table 3. Summary of the mineral composition.

Mix Designation	M3-C3S, (% by Mass)	β-C2S, (% by Mass)	f-CaO, (% by Mass)	T3-C3S, (% by Mass)	γ-C2S, (% by Mass)
A_1	0	6.87	0.41	92.72	0
A_2	9.01	20.8	2.50	26.71	40.98
A_3	96.30	3.33	0.37	0	0
A_4	95.50	4.02	0.48	0	0

3.3. Physical Properties

The optimized Alite mix, referred to as A_4, showed desirable physical characteristics. Particularly, its density was observed as 3.14 g/cm³ ± 0.05 which indicates that it had a compact material structure. The analysis on particle size distribution was important for understanding the granulometry of the mix. Its d_{10} or lower size cut-off of 10% mass was determined as 6.42 µm. The median particle size d_{50} was 13.85 µm and the particle cut-off value for 90% mass, i.e., d_{90} was measured as 43.56 µm. All these values in combination appear to reflect the wide range of particle sizes in the mix. In addition, the specific surface area of the Alite mix was calculated with the BET method and it resulted in 777.8 ± 50 m²/kg. Nitrogen (N_2) was used as the adsorbate gas during the BET analysis, and the measurement was carried out after vacuum degassing to eliminate moisture that may otherwise occlude accessible pores. This higher surface area indicates that the particles are very fine and highly reactive, that could impact on the material's performance in a wide variety of applications.

3.4. Comparison to Other Methods

The optimized solid-state reaction method offered in this study is an improvement in the field as it offers more advantages over traditional approaches. One of the most striking advantages is the high purity achieved, with M3-alite content greater than 95%. This level of purity is greater than the average range of 80–90% in several previous studies, which shows a remarkable improvement in product value. In addition to this, the processing time is greatly reduced to only 6.5-h sintering cycle as opposed to multiple cycles over 24 h that is common in traditional methods. That time alone increases productivity and decreases energy consumption. Another key benefit is the lower sintering temperature of 1650 ºC which is widely reported to be 1750 ºC. Lowering the temperatures sintering levels saves energy while potentially prolonging the lifespan of high temperature furnace by reducing the risk of unwanted side reactions. The simplicity of the equipment needed is another key benefit because the method is based on regular furnaces and ball mills, eliminating the need for expensive and complex setups. Easier equipment access makes the method more convenient for implementation across different levels of research and industries. This is accompanied by the fact that the process automated in parallel yields greater benefits due to the simplicity of expanding existing procedures. This is highly beneficial for industrial use where mass production is critical. Compared to methods executed in solutions like gel-based Pechini or sol-gel processes, this solid-state methodology is much simpler and less expensive. It is more effective in accomplishing synthesis while minimizing resource expenditure. Lower resource expenditure coupled with the efficiency of this optimized solid-state reaction makes it a strong option for creating high-purity materials in materials science and engineering.

4. Conclusions

In this study, a high-purity M3-alite polymorph was successfully synthesized using an optimized solid-state reaction protocol. The synthesis involved mixing high-purity calcium carbonate (98.26%) and fumed silica (99.81%) with stabilizing agents—0.75 wt.% MgO and 0.35 wt.% Al_2O_3. The mixture was homogenized via wet ball milling for 8 h, oven-dried at 150 °C, and sintered at 1650 °C for 180 minutes without compaction. Rapid cooling was applied to stabilize the M3 polymorph, and the synthesized powders were characterized using XRD, BET, and PSD techniques. The optimized process produced M3-alite with phase purity exceeding 95%, supported by low residual phases (β-C_2S < 4.1%, f-CaO < 0.5%). The synthesized material exhibited a specific surface area of 777.8 ± 50 m²/kg, a median particle size (d_{50}) of 13.85 µm, and a density of 3.14 ± 0.05 g/cm³.

These results confirm the effectiveness of the protocol in achieving high-purity alite with desirable physical properties. The method also reduces energy input by eliminating the need for multi-cycle sintering and compaction, thereby offering a scalable and cost-effective route for producing phase-pure M3-alite relevant to low-carbon cement applications. From the study, it was found that about 800 grams of pure M3-alite, polymorph can be synthesized with a total processing time of 6.5 h while maintaining over 95% purity. This demonstrates that the solid-state reaction method can be efficiently and effectively used to synthesize M3-alite which can then be used in studies on cement hydration to better understand M3-alite's behavior and properties at different curing conditions along with mineral and chemical admixtures.

Thus, the present study primarily focuses on optimizing the synthesis protocol and achieving high phase purity of the M3-alite polymorph. Future investigations will be necessary to assess the structural soundness of the synthesized material, especially for its application in cementitious systems as MgO addition can lead to delayed expansion and compromise volume stability. Soundness refers to the material's ability to maintain volume stability without experiencing delayed expansion, cracking, or disintegration due to the presence of reactive constituents such as unreacted MgO. To evaluate this, standard soundness tests, including the Le Chatelier method and autoclave expansion tests (as per ASTM C151), are planned on pastes incorporating the synthesized M3-alite [32, 33]. These tests will be complemented by microstructural observations using Scanning Electron Microscopy (SEM) to detect internal flaws or hydration-induced damage. Additionally, Thermogravimetric Analysis (TGA) and X-ray Diffraction (XRD) will be employed to monitor the hydration behavior of MgO and quantify any unreacted content, which may be linked to potential volumetric instability. These future efforts will ensure a deeper understanding of the long-term performance, dimensional stability, and practical viability of high-purity M3-alite in advanced cement formulations.

Furthermore, other aspects which may be improved include using different cooling rates, different raw materials, or adjusting the starting materials range of particle size distribution. To fully test the practicality of the synthesized M3-alite, it is imperative that in-depth studies on the kinetics of hydration at different curing temperatures should be performed [34, 35]. These studies need to be coupled with in-depth examinations on the mechanical properties of the material which was synthesized. The outcomes which are received from these analyses should be rigorously benchmarked against the industrial clinkers which are well known to be used in the cement industry. Through these studies, researchers will be able to confirm the differences and similarities of the synthesized M3-alite and industrial clinkers, in addition to the hydration behavior, strength development, and other essential comparison properties. This validation study is important in reflecting whether the synthesized material is ideal for modeling cement hydration processes and evaluating the performance of cementitious composites for different scenarios.

Author Contributions

Conceptualization, methodology, funding acquisition, data curation, investigation, visualization, formal analysis, writing–original draft, writing–review, and editing, R.K.; supervision, conceptualization, methodology, investigation, writing, review, and editing, S.B.; supervision, writing–review, and editing, N.G. All authors have read and agreed to the published version of the manuscript.

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Not applicable.

Data Availability Statement

The authors declare that data will be available on a request.

Conflicts of Interest

The authors declare no conflict of interest.

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