



Effect of Thermal Activation on the Mineralogical Structure of Magnesium Slag

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Abstract:

There are many legal regulations and criteria regarding the disposal and management of industrial waste. These criteria are becoming more complex as industrial waste increases day by day. To reduce waste production and prevent the harmfulness of waste, it is necessary to develop and use clean technologies that use natural resources as little as possible. In cases where waste generation is inevitable, it is essential to recover waste or use it as an energy source through recycling, reusing, and other processes to obtain secondary raw materials. During the production of 1,000,000 tons of magnesium metal in the world in 2022, 8,500,000 tons of magnesium slag were produced annually. Much of the obtained magnesium slag is stored on land as waste without recycling. The increase in magnesium slag, along with increased production in the future, will bring about a global environmental problem. The production process of magnesium slag is similar to that of Portland cement. In addition, small amounts of clinker mineral phases are observed in the mineral structure of magnesium production slag. This study aimed to increase the number of structures similar to the clinker mineral phase by providing self-cooling due to sintering magnesium slag at different temperatures using the thermal activation method. Increasing the alite phase in the mineral structure of magnesium slag will pave the way for producing environmentally friendly magnesium slag-modified cement with high early strength at high concentrations.

1. Introduction

Since 2016, Turkey has begun producing indigenous magnesium metal from dolomite mines at its facility, integrating the process for the first time, even within Europe. The facility produces magnesium ingots with 99.80% purity and weighing 8-12 kg. At the facility, a blend of dolomite, ferrosilicate, and fluorite raw materials undergoes grinding, leading to the extraction of magnesium metal through thermochemical reduction in reduction furnaces. Annually, approximately 60,000 tons of slag material are generated as waste and stored near the facility without undergoing any treatment. These wastes generated in magnesium production facilities in other countries are used as additives in cement production [1-3]. However, scientific studies have yet to be able to take these wastes beyond the use of additives in cement production. Some reflections on the subject have revealed that magnesium production slag's (MPS) chemical and mineralogical structure is similar to Portland cement clinker but

has shown that the C_3S phases are lower and the C_2S phases are higher than the clinker [4-5].

It has been demonstrated that a suitable firing temperature and cooling conditions are required to obtain the appropriate proportions of mineral phases (C_3S , C_2S , C_3A and C_4AF) in Portland cement clinker [6-7]. It has been concluded that to produce a clinker similar to Portland cement clinker and a substitute for magnesium production slag, the slag remaining in the reduction furnaces due to Mg reduction must be brought to the sintering temperature and then self-cooling under atmospheric conditions. Thus, it is envisaged that magnesium-production slag clinkers with high C_3S content can be obtained by equalizing the process conditions of the clinker production process and the magnesium production slag. This application has been tried for the first time in our country and the world. With this application, magnesium metal production costs will also be reduced; it will contribute to the reduction of cement

production costs, it will enable more economical and long-lasting use of cement raw material resources and an environmentally friendly green product will be obtained as a result of the reduction in CO₂ emissions. More than 1,000,000 tons of magnesium metal were produced in the world in 2020, and 8,500,000 tons of magnesium slag were obtained during the production of this material [8]. The increase in the magnesium crisis during the pandemic period indicates that the magnesium production rate will increase in the coming years, and new production facilities will be opened. For this reason, converting magnesium slags into a product with high added value is critical for the environment, economy and even countries.

The primary purpose of this study is to demonstrate the possibility of obtaining a product closer to the mineral structure of Portland cement clinker by changing the mineral phase ratios and crystal structures of the obtained slag under optimum temperature and cooling conditions. Industrial tests and laboratory studies were first conducted to achieve this aim. In this study, a product with the mineralogical properties of Portland cement clinker was obtained by changing the temperature and cooling rate of magnesium slag.

2. Material and Methods

2.1 Preparation of raw materials for industrial tests

Industrial trial studies were conducted at the Primary Magnesium Production Facility of Kar Mining Company, located in the Emirdağ district of Afyonkarahisar Province. The facility boasts 16 reduction furnaces, each operating independently and facilitating thermochemical reactions. Within each reduction furnace, 58 retort tubes supply the necessary raw material for magnesium production. Specifically, 180 kg of pellets (a crude mixture akin to ground granulated almonds in size) are introduced into each retort tube. On average, approximately 15-16% of the pellets fed result in crown magnesium, while the remaining 84-85% are obtained as magnesium production slag (waste). Industrial trial studies were carried out in the reduction furnace no. 16, independently of the other furnaces. The operating temperature of reduction furnaces is 1300 °C. The reduction time is 12 hours. Temperature and reduction time can be increased or decreased according to the desired operating conditions in the furnaces. For this reason, a single furnace was selected for industrial trial runs so as not to disrupt the production process flow or affect production. In reduction, furnaces, 75-80% calcined dolomite, 10-

18% ferrosilica, and 1-2% raw material mixture (pellets) are fed. The obtained magnesium slag was optimized by changing the raw material mixture ratio according to clinker properties. The natural pellet mixture is provided at the beginning of the reduction period, as shown in Figure 1a. When the reduction process is completed after 12 hours, the magnesium in the raw mix is recovered as metal (crown magnesium). The remaining material (magnesium production slag) is retrieved in Figure 1b. It is stored as waste in an open stock area without being subjected to any processing.

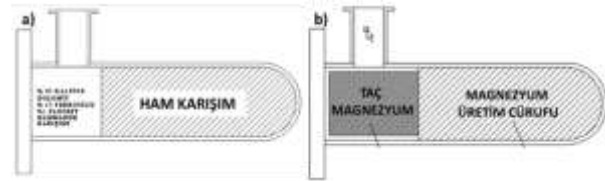


Figure 1. Retort tube feeding before the reduction process (a) and product acquisition after the reduction process (b)

Each reduction furnace also has two chambers. 16. One chamber of the reduction furnace was used for industrial trial runs. The chambers of reduction furnaces can operate independently of each other. There are 27 retort tubes in each chamber, allowing material to be fed separately from each other. Three different temperature ranges were determined in industrial trial studies. The first temperature range is 1200-1250 °C, the second is 1250-1300 °C, and the third is 1300-1350 °C. Magnesium slag will be produced at three different determined temperatures, and firstly, approximately 20 kg of the resulting products were taken separately and left to cool in the open air (Table 1). At the end of the cooling process, chemical analysis tests were carried out on the magnesium slag samples using the XRF method. The chemical analysis results calculated Portland cement clinker phases (C₃S, C₂S, C₃A and C₄AF) and their ratios according to the Bogue formula. According to the literature, the C₂S rate in the magnesium slag is expected to be around 60% due to self-cooling, and the C₃S rate is expected to be 0-10% [4,9]. After the cooling process, mineralogical studies were started on the products obtained.

3. Results and Discussions

3.1. Industrial trial studies

The use of mineralizers has become common in the last few years to facilitate and accelerate the slagging (clinkerization) process, reduce the fuel requirement of the kiln, improve cement properties and burn raw

material mixtures at much lower temperatures [10,11].

Table 1. Magnesium slag production plan

	Codes	Temperature Range (°C)	Cooling Methods
Industrial Tests	MPS1	1200-1250	Outdoor Self-Cooling
	MPS2	1250-1300	Outdoor Self-Cooling
	MPS3	1300-1350	Outdoor Self-Cooling

Mineralizers are inorganic substances that expedite the reaction process at the interface between the solid and liquid phases in both liquid and solid states. Various studies conducted in different laboratories have demonstrated that adding small quantities of these mineralizers to the raw material mixture considerably impacts the clinkerization of the combustion zone and the formation percentage of clinker compounds. Mineralizers play a significant role in determining the combustion zone and constructing clinker compounds. Furthermore, they can expedite and enhance the clinkerization process by reducing the temperature of the liquid phase, thereby conserving energy in clinker production and enhancing the reactivity of cement compounds. Additionally, they have the potential to improve the hydraulic properties of clinker. In producing magnesium metal and forming magnesium slag, calcium fluoride was added to the raw material mixture at the rates of 0.5%, 1%, 1.5%, 2%, 2.5% and 3% in different processes. The optimum calcium-fluoride ratio for magnesium metal production and magnesium slag formation was 1.8%. In this industrial test study, 1.8% calcium fluoride (CaF₂) was added to the raw mixture to benefit from the reducing effect of the slagging (clinkerization) temperatures of the pellet raw mixture without affecting the magnesium metal production process. An excess of MgO and CaF₂ leads to a decrease in C₃S (tricalcium silicate) and the emergence of unconventional cement phases, namely α -C₂S (alpha dicalcium silicate) and γ -C₂S (gamma dicalcium silicate) [1]. (Bouregba et al., 2013) used 2% calcium fluoride in the raw meal mixture [12]. Consequently, it was observed that this improvement enhanced the formation of clinker phases and the mechanical properties of the cement while simultaneously reducing the combustion temperature and free lime content. (Yamashita and Tanaka, 2012) managed to reduce the combustion temperature from below the current process (1450

°C) to 1350 °C by controlling the fluorine and sulfur trioxide in the clinker [13]. They also found that the setting and hardening properties of cement produced with clinker obtained at 1350 °C were equal to those of ordinary Portland cement. Using both industrial experience and in the light of the researched literature studies, the raw material mixture was adjusted at the rates of 81.9% calcined dolomite, 16.3% ferrosilicate and 1.8% calcium fluoride for industrial trial studies, was ground under 125 microns in the ball mill and then turned into pellets (Figure 2). Raw mixture prepared with 81.9% calcined dolomite, 16.3% ferrosilicate and 1.8% calcium fluoride for the first trial run (TR-1). The pellets were fed to the reduction furnace no. 16 at a temperature of 1200-1250 °C and were subjected to the sintering process to obtain magnesium production slag (MPS) (Figure 3). The first step of the industrial trial run (TR-1) was carried out in the reduction furnace no. 16 at a temperature of 1200-1250 °C. Industrial test studies monitored crown magnesium production and quality control parameters simultaneously. During the testing process in the temperature range of 1200-1250 °C, no problems were experienced in the production and quality of crown magnesium at the end of the reduction process. In the industrial trial study, the crown magnesium required for the show was taken into hives and sent to the refining unit for processing. The magnesium production slag obtained in the retort tubes as a result of sintering at a temperature of 1200-1250 °C in the reduction furnace no. 16 was taken into a high-temperature resistant bunker made of hard metal and allowed to cool down to approximately +65 °C in the atmospheric air in the area determined for the test run (Figure 4). Raw mixture prepared with 81.9% calcined dolomite, 16.3% ferrosilicate and 1.8% calcium fluoride were fed to the reduction furnace no. 16, this time at a temperature range of 1250-1300 °C (TR-2) (Figure 5a). As a result of the completion of the reduction process of pellets in the temperature range of 1250-1300 °C, no problems were experienced in the production and quality of crown magnesium. During the industrial trial run, the crown magnesium required for primary production were first taken into hives and sent to the refining unit for processing. The magnesium production slag obtained as a result of sintering at a temperature of 1250-1300 °C in the reduction furnace no. 16 was taken into a high-temperature resistant bunker made of hard metal and allowed to cool down to approximately +65 °C in the atmospheric air in the area determined for the test study (Figure 5b). In the third step of the industrial test study, the raw material mixture of 81.9% calcined dolomite, 16.3% ferrosilicate and 1.8%



Figure 2. pellet production (a) and pellet stock prepared for experimental studies (b)



Figure 3. Reduction furnace no. 16 pellet feed (a) and temperature image of 1200-1250 °C retorts (b)



Figure 4. Self-cooling of magnesium production slag in the temperature range of 1200-1250 °C

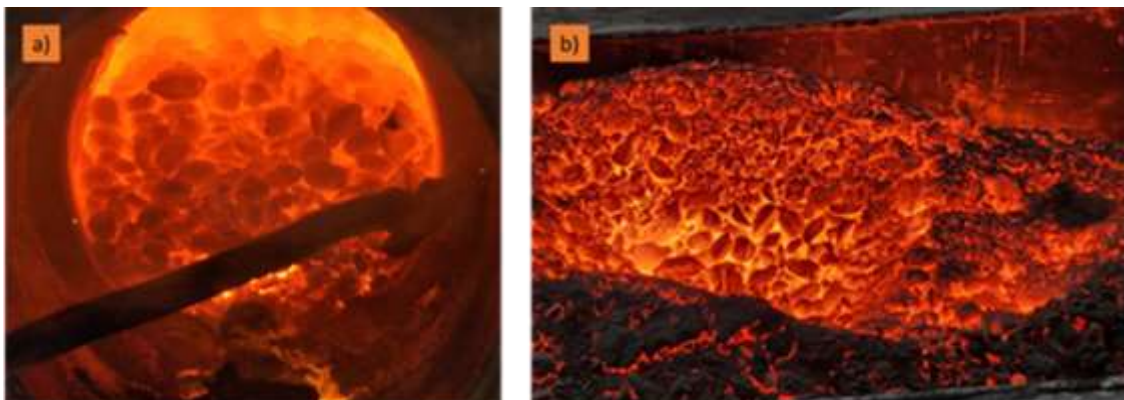


Figure 5. Clinkerization (a) and cooling (b) image of 1250-1300 °C magnesium production slags

calcium fluoride was fed to the reduction furnace no. 16 at a temperature range of 1300-1350 °C (TR-3). There were no problems in the production and quality of crown magnesium at the end of the reduction process in the 1300-1350 °C temperature range. After the crown magnesium required for primary production was taken into the hives, the magnesium production slag obtained as a result of sintering in the reduction furnace no. 16 was brought into the raw material bunker made of hard metal and allowed to cool down to approximately +65 °C in the atmospheric air in the area determined for the cooling test study (Figure 5). The heating and cooling rates and the type of furnace used also play a crucial role in shaping the microstructure of clinker [14]. The formation of clinker melt is an endotherm reaction, and as the clinker cools, most of the melt heat is released. For easy sintering, the melt must be formed at as low a temperature as possible, and its rate must be large enough. Clinkers come out of the rotary kiln at approximately 1300°C. The most commonly used method here is to supply pressurized outside air (atmospheric air) to the clinker grains that are slowly moving on the grid plates. The cooling rate should be controlled as it will affect the internal structure of the clinker. Cement clinker must rapidly cool after the sintering temperature to preserve as much C_3S as possible. Slow cooling can lead to the formation of C_2S and C_3A due to reactions between the clinker melt and a specific ratio of C_3S . Additionally, below 1250°C, C_3S is unstable and tends to decompose into C_2S and free CaO. In addition, rapid cooling is advantageous when the amount of MgO exceeds 2.5 wt%. Because of this, small periclases are formed. Clinker cooling conditions have a significant impact on the mineralogical composition of clinker [15,16]. According to various cooling degrees, The C_3S percentage reached 59.8% in slow cooling, 65.2% in rapid cooling and 70% in rapid cooling [15]. Clinker comprises oxides in its composition, which form calcium silicate and aluminium silicate compounds under specific stoichiometric values and kiln temperature conditions. The proportions of these compounds are influenced by the degree of cooling at the kiln exit. Limestone and clay, the primary raw materials of clinker, are typically not naturally found in completely pure forms, thus containing various foreign substances. Consequently, these foreign substances are in minor proportions in the raw materials, ultimately incorporated into the cement composition. Besides the main components such as CaO, SiO₂, Al₂O₃, and Fe₂O₃, which constitute around 90% of Portland cement, additional oxides

like MgO, SO₃, and alkali oxides are also present [17, 18]. During clinker formation, the main phases—alite, belite, aluminate, and ferrite—are generated at high temperatures. Collectively, these phases constitute approximately 90% of cement clinker compounds [19]. Cement compounds are typically quantified using Bogue models proposed in 1955. These models enable the calculation of potential phase amounts based on the chemical components of cement and the relative humidity (RH). The prerequisite for this calculation is the crystallization of the clinker melt in equilibrium with the solid phases. The stoichiometric phases are pure C_3S , C_2S , C_3A , and C_4AF . Bogue models provide a means to calculate the amounts of CaO, SiO₂, Al₂O₃, and Fe₂O₃ based on their initial raw oxide compositions, as defined in Table 2 [20]. Before calculating the Bogue formula, the chemical contents of magnesium production slag clinkers (MPS) produced under industrial conditions were analyzed by the XRF method and the results are shown in Table 2. The MPS phase quantities and modules were computed based on the Bogue model using the chemical analysis findings of magnesium production slag clinkers presented in Table 2, and the outcomes are provided in Table 3.

Three commonly utilized methods for determining the phase composition, particularly concerning the four main phases—alite, belite, aluminate, and ferrite—of Portland cement clinker are as follows:

- Quantitative X-ray diffraction analysis
- Optical microscopy utilizing point counting
- The Bogue Method

The Bogue Calculation, a standard technique reliant on the elemental composition of clinker, computes the potential phase composition and approximates the relative proportions of the four major clinker phases. However, it's important to note that the Bogue Method is not an analytical tool for directly measuring the actual phase composition of real clinker. Consequently, this calculation can yield misleading results in some instances because it assumes that equilibrium conditions are met during clinker production and that the composition of the four main phases in the clinker is C_3S , C_2S , C_3A , and C_4AF . The Bogue Method fails to consider the incorporation of foreign ions into the structures or forming different solid solutions. Previous research has indicated that the Bogue calculation typically underestimates the alite content and overestimates the oxide content compared to quantification by point counting and optical microscopy techniques [4, 15]. Quantitative X-ray diffraction techniques (QXRD) can yield results comparable to quantitative

Table 2. MPS chemical analysis results obtained after the industrial trial

Temperature Ranges (°C)	Cooling Type	Loi	SiO ₂	Al ₂ O ₃	Fe ₂ O ₃	CaO	MgO	SO ₃	Na ₂ O	F	Total
		%	%	%	%	%	%	%	%	%	%
1200-1250	Outdoor Self-Cooling	0.10	30.33	0.25	3.63	60.8	3.18	0	0.03	1.18	99.50
1250-1300	Outdoor Self-Cooling	0.12	29.49	0.32	3.47	62.49	2.45	0	0.03	1.12	99.49
1300-1350	Outdoor Self-Cooling	0.20	28.76	0.48	3.98	63.54	1.82	0	0.03	1.15	99.96

Table 3. MPS modules and phase percentages obtained after the industrial trial

Temperature Ranges (°C)	Cooling Type	LSF	SM	AM	HM	C ₃ S	C ₂ S	C ₃ A	C ₄ AF
1200-1250	Outdoor Self-Cooling	69.42	7.82	0.07	1.78	5.13	83.09	-5.48	11.05
1250-1300	Outdoor Self-Cooling	73.34	7.78	0.09	1.88	18.48	70.61	-5.02	10.56
1300-1350	Outdoor Self-Cooling	75.93	6.45	0.12	1.91	25.68	63.09	-5.46	12.11

optical microscopy for alite and belite phases. However, when measuring aluminate and ferrite phases, QXRD is generally more reliable due to its ability to overcome the resolution challenges encountered with optical microscopy. Standard QXRD techniques, such as internal standard field peak fitting, have limitations when dealing with complex multiphase materials. These limitations include preferential orientation and peak overlapping, which can result in significant errors. The latest development in Rietveld QXRD techniques addresses many of these challenges, eliminating or minimizing errors and enabling the quantification of clinker and cement phase compositions with good sensitivity and repeatability. [16-20]. A quantitative phase analysis was conducted on industrially produced magnesium slag clinker samples utilizing X-ray powder diffraction analysis, the Bruker D8 Advance Diffraction Meter, and the Rietveld method, as outlined in Table 4. Analysis of XRD graphs of magnesium production slag clinkers revealed notable trends: the C₃S phase, derived from the structure of MPS, exhibited an increase at high temperatures, while the C₂S phase demonstrated a decrease under similar conditions. Additionally, a small amount of C₄AF and free lime were observed. The XRD graphs and Rietveld analysis results further corroborated these observations. The utilization of the Rietveld method allowed for a comprehensive quantitative assessment of the phase composition of the magnesium slag clinker samples, providing valuable

insights into the relative proportions of various clinker phases present. Notably, the increase in the C₃S phase at high temperatures suggests a transformation within the clinker structure, potentially influencing its properties and behaviour during cement production processes. Conversely, the decrease in the C₂S phase indicates a shift in phase equilibrium, highlighting the dynamic nature of clinker formation under varying temperature conditions. Additionally, the presence of C₄AF and free lime underscores the complexity of the clinker composition, necessitating precise analytical techniques such as X-ray diffraction and Rietveld analysis for accurate characterization and understanding. The increase in the alite phase and decrease in the belite phase in magnesium slag clinker production can have significant implications for both the clinker and the Portland cement. Alite (C₃S) is the primary phase responsible for the early strength development of cementitious materials, making its increased presence advantageous in cement production. The higher proportion of alite in the clinker can lead to improved early-age strength characteristics of the resulting Portland cement. This enhancement in early strength is crucial for applications where rapid strength gain is required, such as in construction projects with tight deadlines or in cold weather conditions where concrete setting times are prolonged. Conversely, the reduction in the belite phase (C₂S) may impact the cement's long-term strength development and durability. Belite contributes to the later age strength of cementitious

materials and provides resistance against chemical attack and sulfate exposure. Therefore, a decrease in the belite phase could lead to slower strength development in the later stages of cement hydration and reduced durability of the final concrete product. However, the exact impact of this decrease would depend on the cement's specific composition and performance requirements [21-23]. Overall, the alterations in the alite and belite phases in magnesium slag clinker production highlight the importance of carefully controlling the clinker

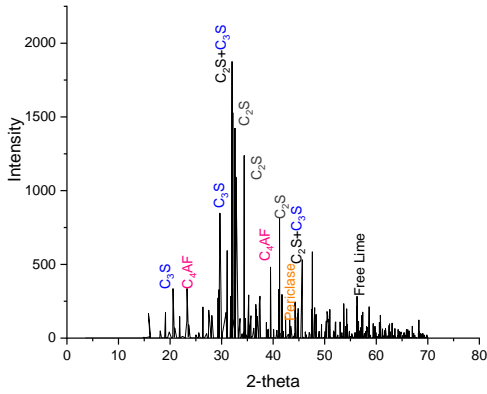


Figure 6. 1200-1250 °C, industrial MPS XRD chart produced as a result of self-cooling in the atmosphere.

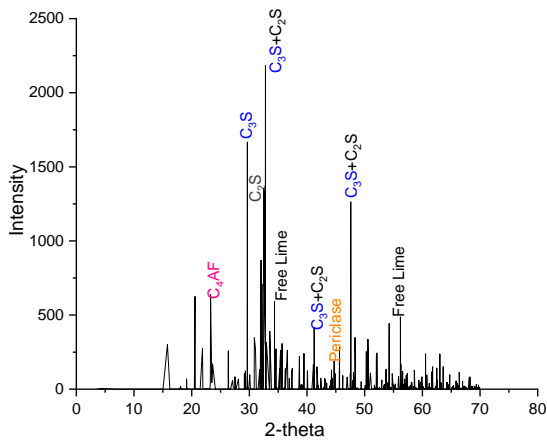


Figure 7. 1250-1300 °C, industrial MPS XRD chart produced as a result of self-cooling in the atmosphere.

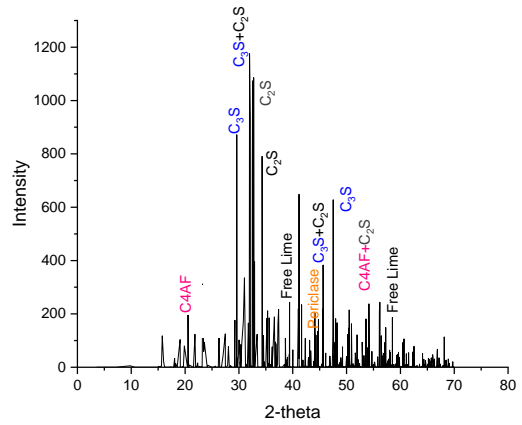


Figure 8. 1300-1350 °C, industrial MPS XRD chart produced as a result of self-cooling in the atmosphere.

composition to achieve the desired properties in the resulting Portland cement. Understanding the implications of these phase changes is crucial for optimizing cement production processes and ensuring the performance and durability of concrete structures in various applications. Further research and experimentation may be necessary to fully elucidate the effects of these phase alterations and develop strategies for mitigating any potential drawbacks while maximizing the benefits of cement production.

4. Conclusions

It has been observed that the mineral phase structure change due to thermal activation applied to magnesium slags. While the alite phase amount of magnesium slag was 5.13% at 1200-1200 oC, the alite phase amount of magnesium slag increased to 18.42% at 1250-1300 °C, and the alite phase amount of magnesium slag increased to 25.68% at 1300-1350 oC. has increased. It was observed that the amount of the belite phase of magnesium slag decreased due to thermal activation. While the belite phase amount of magnesium slag at 1200-1200 oC is 83.09%, at 1250-1300 oC, the belite phase amount of magnesium slag is 70.61% and at 1300-1350 oC

Table 4. Rietveld analysis results of MPS samples

Temperature Ranges	Cooling Type	C ₃ S	C ₂ S	C ₃ A+C ₄ AF+Free CaO+Alkali Sulfate	Periclase
1200-1250 °C	Outdoor Self-Cooling	4.87	80.75	9.69	3.86
1250-1300 °C	Outdoor Self-Cooling	17.26	68.49	10.85	3.01
1300-1350 °C	Outdoor Self-Cooling	23.22	61.98	13.19	1.47

the belite phase amount of magnesium slag is 63.09%. has fallen. It was also observed that the amount of MgO in the magnesium slag decreased due to high temperature. Rietveld analysis results also confirm the increase in alite phase and the decrease in belite phase in magnesium slag due to high temperature. Approaching the clinker phase amount of Portland cement due to thermal activation will also increase the clinker substitute used in Portland cement, which can be produced with magnesium slag additives. Thus, more environmentally friendly cement can be made from recycled waste using magnesium slag from thermal activation.

Author Statements:

- **Ethical approval:** The conducted research is not related to either human or animal use.
- **Conflict of interest:** The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper
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