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Research Article

Investigation of The Use of Calcium Naphthalene Sulfonate as A Binder in Magnesite Spinel Brick Production and Determination of Optimum Conditions

Özkan Küçük*[1], Onur Aşaman[2]

[1]*Bilecik Şeyh Edebali University, Faculty of Engineering, Department of Metallurgical and Materials Engineering, Bilecik, Turkey*
[2]*Bilecik Şeyh Edebali University, Graduate Education Institute, Bilecik, Türkiye.*

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Taguchi Method.

Abstract

Refractory materials are indispensable for industries working with high temperatures. Magnesite spinel refractory bricks are used in the cement industry, and calcium lignosulfonate is used as a binder in their production. Due to forest fires and similar reasons in recent years, there is a problem in the supply of calcium lignosulfonate raw materials. In this study, research was conducted on alternative binders to be used in the production of magnesite spinel bricks and the optimum conditions for production with the selected binder were determined. Some of the binders are calcium naphthalene sulfonate, sodium naphthalene sulfonate, magnesium oxide, magnesium sulfate, and molasses. As a result of preliminary tests, it was observed that the most successful results were obtained with calcium naphthalene sulfonate. The optimum conditions for the production of magnesite spinel refractory bricks using calcium naphthalene sulfonate were found by the Taguchi method. As parameters, seawater sintered magnesite in 4 different fractions (A: 3-5 mm, B: 1-3 mm, C: 0-1 mm and D: Powder) and Sinter Spinel in 2 different fractions (E: 3-5 mm and F: 1-3 mm) in total, 6 parameters were selected and an L16 (44x22) Taguchi orthogonal array design was created for this. Volume weight, water absorption, porosity, and strength tests were performed on the samples obtained. Accordingly, taking into account the strength values, the optimum conditions were determined as A1, B1, C2, D4, E1 and F2. Under these conditions, the estimated strength value was calculated as 84.24 N/mm2 and the experimental value was 83.85 N/mm2.

1. Introduction

When we look at the word meaning of refractory, it is defined as "stubborn". When defined technologically, it is defined as "resistant to the chemical and physical effects of solid, liquid, and gaseous substances at high temperatures". In line with these definitions, refractory is used in many materials that we see around us, which have a thermal process.

Refractory can be classified according to many parameters. Traditionally; When designing furnaces and similar units containing high temperatures, composites that can withstand corrosive and melting effects as well as chemical and physical effects are called Refractory Materials [1].

Bricks with high porosity are preferred for the transition zone in cement rotary kilns where refractories are consumed a lot. To increase the number of pores, experiments have been carried out with additive materials such as MgOH, electrode shards, activated carbon, polyvinyl alcohol, coke, calcined magnesite, and SiC as additives to the magnesite spinel brick structure [2].

Although the most ideal product in cement rotary kilns is magnesite chrome bricks containing Cr+6, they are not used due to their environmental damage and toxic effects. Instead of these bricks, magnesite hercynite and magnesite spinel bricks are preferred. In a study, a comparison was made about the advantages and disadvantages of magnesium chrome, magnesium spinel, and magnesium hercynite bricks [3].

The problem encountered on the ground of construction sites can be solved in two ways. Firstly, improving the ground and secondly, changing the field is preferred. Experimental tests were carried out by taking samples from the problem area on the ground. In order to increase the strength values, the necessary tests were carried out by adding different amounts of ligno sulfonate and it was stated that there was an increase in the strength values and that this material could be used for the purpose of improving the ground [4].

Lignosulfonates are produced as by-products in paper production. The effects of lignosulfonates containing metal cations, tricalcium aluminate, tetra calcium aluminoferrite, and Ca(SO4)2H2O cements on concrete mixtures were examined. It has been determined that the SO3 content of cement reduces the water need. It has been determined that lignosulfonate has a positive effect on compressive strength in cement [5].

In building materials, mixing ratios of concrete and water are important. In cases where more than the desired amount of water is given due to human error, it is possible that the strength and performance will decrease. In this investigation, lignosulfonate and naphthalene sulfonate were added to the structure at different rates as water reducers. According to the data obtained, it has been determined that water reducers, when used in excessive amounts, have a negative effect on mechanical and physical values and should be used in ideal proportions [6].

Hydration effects were examined in Portland cement using sodium lignosulfonate and naphthalene sulfonate formaldehyde plasticizers. During the tests, it was observed that lower amounts of water were used than the standard amount of water used. This desire to reduce

*Corresponding Author: ozkan.kucuk@bilecik.edu.tr
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water leads to an improvement in the mechanical value of cement. When two plasticizers were compared, naphthalene sulfonate formaldehyde was found to provide a higher mechanical improvement [7].

The procedure from mineral to raw material used in refractories is achieved by synthesizing magnesia-based refractories from magnesite. Wang et al. An attempt was made to obtain magnesite and MgAlON refractories by reaction in a carbon-embedded atmosphere. Studies were carried out on sintering and mechanical properties by making changes in the obtaining temperature and magnesite ratios. It has been observed that when working with extra high amounts of magnesite, it accelerates densification and sintering as a result of expansion in volume. It has been determined that double-layer MgAlON refractories are better against corrosion caused by slag. It is a cost-effective and easy-to-obtain material [8].

In a study on corrosion in cement rotary kilns, microstructured magnesia aggregates were preferred in size and periclase-magnesium aluminate (LPSR) refractories with good mechanical properties against corrosion were produced. This synthesized microstructured product was compared with classical dense periclase-magnesium aluminate spinel refractories (DPSR). It was determined that the visible porosity microstructure was higher than the dense one. Examinations made with an electron microscope showed that the microstructure had a better interface, which affected the strength and thermal shock resistance. Studies on slag have shown that the microstructure was better than one dense [9].

The effects of alumina (HA), which can be hydrated in Calcium Aluminate cement (CAC), have shown variations in magnesia retention in aqueous suspensions. Change in binder amount and content; The effects of pH, volumetric expansion, strength, and void amount on hydration-dehydration tests were investigated. The choice of binder has been found to have an impact on minimizing the negative effects of hydration [10].

An investigation was made on the bricks used in cement rotary kilns. The event that affects the end of the life of bricks is corrosion. Quantitative and qualitative analyses were carried out on samples taken from the sintering area of the furnace. When the mineralogical examinations were combined with the Rietveld method, it was determined that the reason for the deterioration of the refractory brick was sulfur content and high temperature (>1600 °C). It has also been observed that sulfur deteriorates the elasticity of the brick [11].

In another study conducted by Zhou et al., some samples were taken and examined from the area called the upper pass in cement rotary kilns. These reviews have been about dense periclase-magnesium aluminate spinel refractory bricks. The reason for the deformed fragmentation of the bricks after use is the corrosion of the clinker, alkaline salts and instantly changing operating temperature conditions, which effectively caused cracks, ruptures and expansions in the brick as a result of thermal stress. While high operating temperatures cause the clinker to pass into the liquid phase, it also causes the microstructure of the brick to deteriorate as a result of the interaction between the clinker in the liquid phase and the brick. Especially temperature changes cause the refractory materials in the furnace to deteriorate and disintegrate [12].

In spinel bricks used in rotary kilns, the presence of spinel and its pore properties affect the corrosion formation in the brick and the desire of the clinker in the kiln to adhere to the brick. Additionally, increasing the spinel ratio causes the glass phase to increase as a result of the reaction between the brick and the clinker. This is because the dissolution rate of actual spinel is higher than that of periclase. The amount of glass phase is the binder between cement clinker and aggregate. When used between 15-40% of spinel, it provides good adhesion as well as high clinker resistance. However, when used at 50% levels, cement clinker resistance will decrease [13].

In this study, an alternative binder raw material was sought due to reasons such as the unavailability of the raw material used as a binder in the production of magnesite spinel bricks preferred in cement rotary kilns in recent years. The closest alternative product to the currently used product was identified and the ideal product recipe was aimed to be found by using the Taguchi method.

2. Material and Method

2.1. Preparation of Materials

Seawater sintered magnesite and Sinter Spinel used in this study were obtained from Sörmaş Söğüt Refrakter Malzemeleri A.Ş.

In the optimization, seawater sinter magnesite in 3 different fractions and sinter spinel in 2 different fractions were selected as parameters, and the binder ratio was kept constant. The seawater sinter used was imported from abroad in sizes of 0-40 mm. This material is passed through crushers and screens and separated into fractions. The resulting fraction size was chosen as 3-5 mm, 1-3 mm, 0-1 mm and powder (63μm) (Figure 1).

Figure 1. Views of seawater sintered magnesite fractions (3-5 mm, 1-3 mm, 0-1 mm, Powder)

Sinter Spinel was imported from abroad in sizes 1-3 mm and 0-1 mm. It was used in its current dimensions without the need for crushing and sieving for use in production (Figure 2).

Figure 2. Views of Sinter Spinel fractions (1-3 mm and 0-1 mm)

The process flow chart of the study is given in Figure 3.

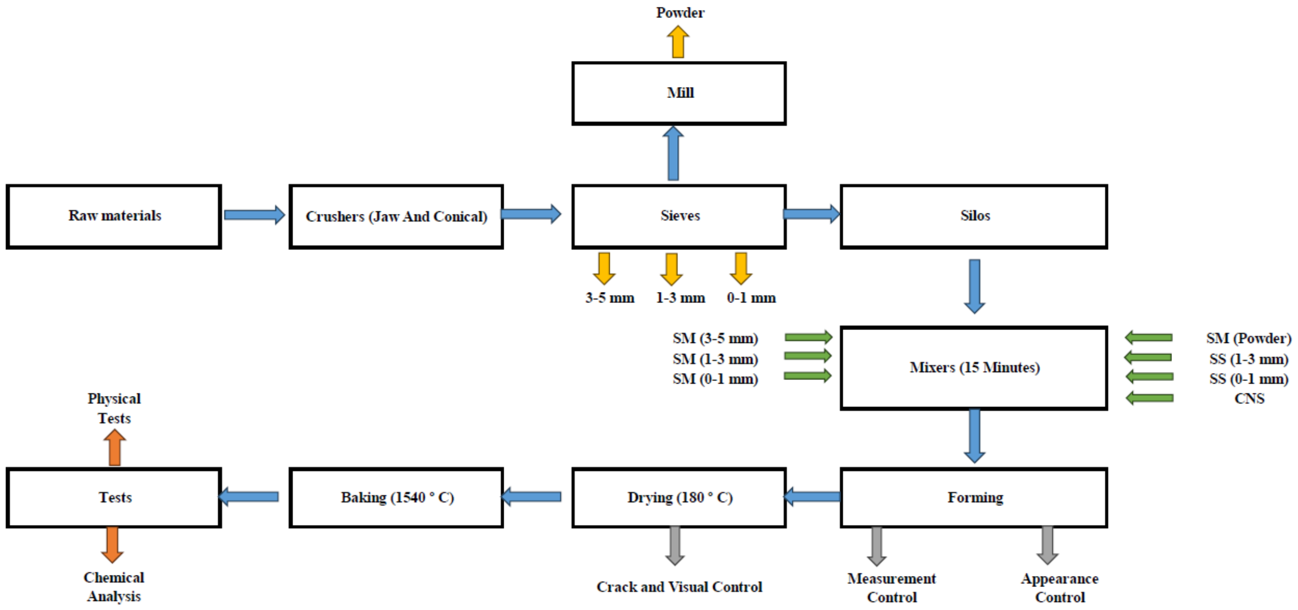

Figure 3. Process Flow Diagram of the Study

Mixing is done in mixers according to the desired composition. While mixing; First, the coarse grains were added, the liquid was added, and the mixing process was completed by adding the powder last.

While creating the composition, the targeted volume value is calculated according to the brick dimensions. After the resting process, the mixed blend was dry-pressed with a constant pressure of 1500kg/cm2.

After the density and measurement values were taken after pressing, the sintering process was carried out in a tunnel kiln at 1540˚C, which is the ideal firing temperature of magnesite spinel bricks. The furnace length is 110 meters. The bricks remained in the 1540˚C fire zone for 2 hours and were removed from the kiln after approximately 70 hours or 3 days in total. The tests of the samples taken out of the oven were carried out according to certain standards, and Table 1 shows which test was carried out according to which standard.

Table 1. Test Performed - Standard Used

Test Performed	Standard Used
Apparent Porosity, Density and Water Absorption	ASTM C 830
Compressive Strength in Cold	ASTM C 133
Sieve Analysis	ASTM E11-70
Size Change	BS EN 993-10
Thermal Shock	DIN EN 993-11
Chemical analysis	ISO 12677

In evaluating the optimum conditions, MSP85S, one of the products of Sörmaş company, was chosen as the reference material. The XRF analysis result and mechanical and physical test results of the reference material are given in Tables 2 and 3.

Table 2. XRF analysis result of the reference material

	SiO_2%	Al_2O_3%	Fe_2O_3%	CaO%	MgO%
Reference Sample	0,20	9,87	0,40	0,90	88,54

Table 3. Mechanical and physical test results of the reference material

Specifications	Units	Reference Material
Forming Pressure	kg/cm^2	1500
Raw Density	gr/cm^3	3,00-3,05
Baking Temperature	^0C	1520- 1540
Volumetric Density After Firing	gr/cm^3	2,94
Water Absorption After Firing	%	5,40
Visible Porosity After Firing	%	15,86
Strength After Firing	N/mm^2	65

2.2. Taguchi Method

In experimental studies, results should generally be close to ideal working conditions, different environments, or times. If the results are close, the optimization criterion should be able to give Taguchi appropriate performance values. In determining the optimum conditions with the Taguchi method, the result is reached with performance statistics formulas [14].

Bigger is better;

$$SN_L = -10\log\left(\frac{1}{n}\sum_{n=1}^{n}\frac{1}{y^2}\right) \quad (1)$$

$$SN_S = -10\log\left(\frac{1}{n}\sum_{n=1}^{n}y^2\right) \quad (2)$$

Here;

n: number of experiment repetitions, y: is the experimentally found value of the parameter (where y: strength).

If the aim is to find the highest value in the experimentally created composition, the parameter levels that maximize the SNL value are optimum. If the aim is to find the lowest value, parameter levels that maximize SNS are optimum [15].

The most ideal performance values achieved by the Taguchi method are assumed with the following equation:

Yit= µ+Xi + ei (3)

Here;

Yit: Predicted performance value of experiment i, Xi: Total effectiveness size of the parameter levels used in experiment i, μ: mean and ei: experimental error.

Equation 3 is an assumption calculated using experimental data to determine whether the extra model is appropriate. Therefore, confidence limits for assumption error must be determined.

Estimated error is the difference between observed Yit and predicted Yit. The confidence interval (Se) for the estimated error is found with the help of the following equation.

$$S_e = \pm 2\sqrt{\frac{1}{n_0}\sigma_e{}^2 + \frac{1}{n_r}\sigma_e{}^2} \quad (4)$$

$$\sigma_e{}^2 = \frac{\text{error sum of squares}}{\text{degrees of freedom for error}} \quad (5)$$

$$\frac{1}{n_o} = \frac{1}{n} + \left[\frac{1}{n_{A_i}} - \frac{1}{n}\right] + \left[\frac{1}{n_{B_i}} - \frac{1}{n}\right] + \left[\frac{1}{n_{C_i}} - \frac{1}{n}\right] + \cdots \quad (6)$$

Here;

Se: confidence interval, n: total number of experiments performed, nr: number of repetitions of the confirmation experiment, and nAi, nBi, nCi,... is the number of i.levels of A, B, C parameters. If the estimated error is outside these limits, the model is considered unfit.

The validation experiment is a powerful tool to detect the existence of interactions between control parameters. If the predicted value under optimal conditions does not match the observed value, it means that the interactions are significant. If the predicted response matches the observed response, it means that the interactions are probably not significant and the additive model is a good approximation [16].

2.3. Effect of parameters on physical test results

The use of different binders in the refractory brick production recipe has been examined, these are molasses, magnesium sulfate, and naphthalene calcium sulfonate. As a result of the experiments, the closest results to the main product were obtained with calcium naphthalene sulfonate (liquid) as an alternative. Therefore, calcium naphthalene sulfonate (CNS) was chosen as the alternative binder. To determine the conditions under which production is optimum, the best physical values were tried to be obtained by using the Taguchi method. While the best conditions for physical properties were sought, it was aimed to ensure that there was no change in the chemical structure.

In the optimization, an L16(44x22) Taguchi orthogonal array design was created (table 4). The proportion of binder was kept constant. The reason it is kept fixed is related to the pressing. The amount of binder is not included in the total weight calculation.

The experimental plan was created considering the parameters and levels in Table 4 and the data obtained as a result of the experiments were given in Table 5.

3. Results and Discussion

3.1. Features of Produced Recipe

As a result of the studies, chemical analysis and physical tests of the refractory brick obtained under optimum conditions were carried out, and the physical test results are given in Table 6 and the chemical analysis results are given in Table 7. Optimum conditions are examined in detail in the statistical calculations section.

Table 4. Parameters and levels of design

Parameters		Levels			
		1	2	3	4
A	Seawater Sintered Magnesite 99, Fraction 3-5mm, g (SM 3-5)	16	20	25	32
B	Seawater Sintered Magnesite 99, Fraction 1-3mm, g (SM 1-3)	16	20	25	32
C	Seawater Sintered Magnesite 99, Fraction 0-1mm, g (SM 0-1)	16	20	25	32
D	Seawater Sintered Magnesite 99, Fraction Powder, gr (SM Powder)	27	29	32	35
E	Sinter Spinel MAS 66, Fraction 1-3mm, gr (SS 1-3)	7	8	-	-
F	Sinter Spinel MAS 66, Fraction 0-1mm, gr (SS 0-1)	7	8	-	-
Binding Quantity (calcium naphthalene sulfonate),g (CNS)		3,5			

Table 5. Experimental plan and results of the design

Exp. No	SM 3-5	SM 1-3	SM 0-1	SM Toz	SS 1-3	SS 0-1	CNS	Volume Weight (g/cm^3)	Water Absorption (%)	Porosity (%)	Resistance (N/mm^2)
1	1	1	1	1	1	1	1	2.94	5.39	15.83	73.55
2	1	2	2	2	1	2	1	2.95	5.28	15.59	72.41
3	1	3	3	3	2	1	1	2.95	5.26	15.52	65.25
4	1	4	4	4	2	2	1	2.97	5.01	14.89	79.45
5	2	1	2	3	2	2	1	2.98	4.90	14.62	74.68
6	2	2	1	4	2	1	1	2.94	5.46	16.01	71.66
7	2	3	4	1	1	2	1	2.97	5.00	14.84	64.87
8	2	4	3	2	1	1	1	2.96	5.10	15.09	61.99
9	3	1	3	4	1	2	1	2.98	4.82	14.37	69.02
10	3	2	4	3	1	1	1	2.97	4.95	14.70	62.99
11	3	3	1	2	2	2	1	2.98	4.85	14.44	58.33

12	3	4	2	1	2	1	1	2.98	4.83	14.38	64.84
13	4	1	4	2	2	1	1	2.97	5.03	14.95	65.63
14	4	2	3	1	2	2	1	2.98	4.82	14.38	60.75
15	4	3	2	4	1	1	1	2.96	5.22	15.43	69.55
16	4	4	1	3	1	2	1	2.97	4.95	14.71	64.06

Table 6. Physical Test Results of the Prescription Recommended as the Most Ideal by the Taguchi Method

Ideal Composition	Volume Weight(g/cm³)	Water absorption (%)	Porosity (%)	Resistance (N/mm^2)
1	2.98	4.76	14.15	83.60
2	2.98	4.98	14.85	84.09

Table 7. Chemical Analysis Results of the Recipe Recommended as the Most Ideal by the Taguchi Method (XRF Analysis Results)

Ideal Composition	SiO_2 %	Al_2O_3 %	Fe_2O_3 %	CaO %	MgO %
Experiment1	0.18	9.62	0.38	0.90	88.83
Experiment2	0.12	9.83	0.38	0.72	88.87

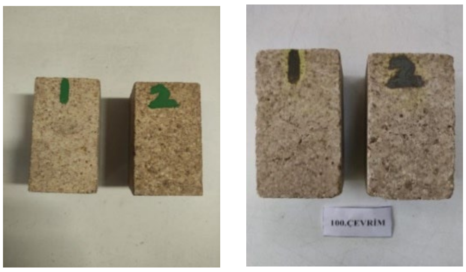

Figure 4. Brick images before and after thermal shock test (Number 1 Trial Brick, Number 2 Standard Product)

3.2. Statistical Calculations

MINITAB 13 package program was used to perform statistical calculations and analyses.

Equation (2) was used to find the parameter levels that maximize the post-firing strength of spinel bricks containing different additives. To determine the parameter levels that maximize the strength, SNL values were found from equation (2) using the values in Table 5. With the help of these SNL values, marginal average performance statistics values (SNort) were calculated for the parameter levels and the results are presented graphically in Figure 5. Equation (4) was used to estimate the performance value of the parameter levels that maximize the SNort value.

When Figure 5 is examined, it is seen that the parameter levels that maximize the SNort value are A1, B1, C2, D4, E2, and F2. Therefore, the parameter values that maximize the strength value will be A1, B1, C2, D4, E2, and F2. Accordingly, the optimum conditions were determined as 16 g for SM 3-5 fraction, 16 g for SM 1-3 fraction, 20 g for SM 0-1 fraction, 35 g for SM Powder fraction, 8 g for SS 1-3 fraction and 8 g for SS 0-1 fraction. Under optimum conditions, the estimated strength value is 84.24 N/mm2 and the experimentally found strength value is 83.85 N/mm2.

As can be seen in Figure 5, since the first and second levels for the E parameter are almost the same and very similar results can be achieved with less raw material at the first level, we can take the parameter values that maximize the strength value as A1, B1, C2, D4, E1, and F2. Accordingly, we determined the optimum conditions as 16 g for SM 3-5 fraction, 16 g for SM 1-3 fraction, 20 g for SM 0-1 fraction, 35 g for SM Powder fraction, 7 g for SS 1-3 fraction and 8 g for SS 0-1 fraction.

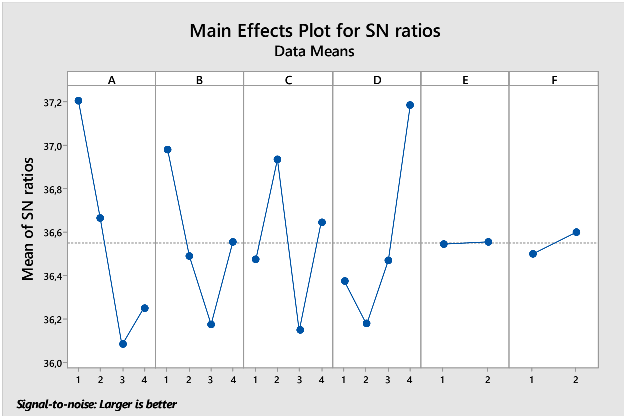

Figure 5. SNort values according to parameter levels for the Strength value of Magnesite spinel brick produced under optimum conditions

Table 8. Analysis of variance for optimization experiment

Parameters	SDi	SSi	MSi	F_D	% Activity ($F/\Sigma F$)x100	
A:SM 3-5	3	378.350	126.117	1380.92	37.72	Effective
B:SM 1-3	3	157.13	52.375	573.49	15.66	Effective
C:SM 0-1	3	157.46	52.488	574.72	15.70	Effective
D:SM Toz	3	283.76	94.587	1035.68	28.29	Effective
E:SS 1-3	1	0.575	0.575	6.30	0.17	Effective
F:SS 0-1	1	8.237	8.237	90.19	2.46	Effective
Error	17	1.553	0.091			
Total	31	987.07				

$F_{Tablo}(3.17)_{0.95}$=3,20,$F_{Tablo}(1.17)_{0.95}$=4,45,
$F_{Tablo}(3.17)_{0.99}$=5,19, $F_{Tablo}(1.17)_{0.99}$=8,40

The confidence interval was calculated from equations 4, 5, and 6, and the optimum operating conditions, observed and predicted values in the experiments are given in Table 9.

Table 9. Optimum operating conditions, observed and predicted values in the experiments

	Case 1		Case 2*	
Parameters	Value	Level	Value	Level
A:SM 3-5	16	1	16	1
B:SM 1-3	16	1	16	1
C:SM 0-1	20	2	20	2
D:SM Toz	35	4	35	4
E:SS 1-3	8	2	7	1
F:SS 0-1	8	2	8	2
Observed value (%)	84.09		83.85	
Predicted value (%)	84.50		84.24	
confidence range(%),Se	±2.33 (82.17-86.83)		±2.33 (81.91-86.57)	

*Alternative and selected optimum conditions

Whether the parameters are effective or not was determined by comparing the FExperimental value found experimentally with the FTable values. If FExperimental>FTable, the relevant parameter is effective on the performance value. If FExperimental<FTable, the parameter is not effective on the performance value. Variance analysis performed for the design experiments is given in Table 8.

When Table 8 is examined by comparing the FExperimental values with the FTable values, it was seen that the most effective parameters at the 95% confidence level in the experiments were SM 3-5 and SM Powder, SS 1-3 is almost ineffective, and SS 0-1 was very little effective.

4. Conclusion

The main conclusions from the current study are:

The usability of different binders in the recipe for the production of magnesite spinel bricks was examined. Unsuccessful results were obtained with Sugar Molasses and Magnesium sulfate additives. Successful results have been obtained with calcium naphthalene sulfonate.

In order to increase the physical values of the existing product, especially the compressive strength value in cold, the ideal amount of calcium naphthalene sulfonate usage rate was found to be 3.5%. Calcium naphthalene sulfonate is supplied as liquid. Preliminary trials were carried out at 3-3.5% without making any changes to the main recipe. However, printable consistency could not be achieved with 3%. Due to its weakness, the rate was increased to 3.5%. Since it was observed that the hardening of the sample produced after pressing was good, the ratio was chosen as 3.5%.

According to the optimization created, 16 bricks were prepared, shaped, and fired. According to these results, the average strength according to the recipe determined under optimum conditions was found to be 83.85 N/mm2. No change was observed as a result of chemical analysis.

A thermal shock test was carried out for the produced brick and reference brick. A thermal shock test was completed by applying load every 5 cycles. In the 5th cycle, hairline cracks appeared in both bricks. In the 50th cycle, it was observed that hairline cracks opened. The cracks have become more evident in recent cycles. The test was completed by performing 100 cycles. As a result of 100 cycles, thermal shock samples showed similar deformation and crack formation.

The predicted and observed values are very close to each other and it can be concluded that the additive model is sufficient to explain the dependence of the production process on various parameters.

The optimum conditions determined by the Taguchi method have been realized on an industrial scale in real production environments.

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Declaration of Conflict of Interests

The authors declare that there is no conflict of interest. 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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