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Study of using Calcium Carbide Slag to Prepare Calcium Oxide Briquettes by Molding and Calcination Processes through Taguchi Method

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Abstract

As the disposal of calcium carbide slag (CCS) obtained a by-product during an acetylene gas process creates an environmental problem, this study aimed to use of CCS to prepare calcium oxide (CaO) briquettes for re-use in the production of calcium carbide (CaC_2). The influence of binder types (phosphoric acid (H_3PO_4), molasses, and corn syrup), binder amount (1, 3, and 5%), briquetting pressure (20, 28, and 36 MPa), calcination temperature (800, 900, and 1000 ^oC), and calcination time (30, 45, and 60 min) on the strength value of CaO briquettes was investigated using the Taguchi approach. The highest compressive strength value of CaO briquettes was found to be 4.05 MPa. The stability and friability values of the final product were 92% and 8%, respectively. ANOVA analysis revealed that the contribution rate of production parameters on the strength value of CaO briquettes were as follows: (i) binder amount, (ii) binder type, (iii) briquetting pressure, (iv) calcination temperature, and (v) calcination time. The optimal production conditions were determined as follows: the amount of binder: 5%, briquetting pressure: 28 MPa, calcination temperature: 900 ^oC, and calcination time: 60 min. The obtained CaO briquettes provide a required strength value for re-use in the production of CaC_2.

Keywords: Calcium carbide slag, CaO briquette, Corn syrup, Molasses, H_3PO_4

Taguchi Metodu ile Kalsiyum Karpit Cürufundan Kalıplama ve Kalsinasyon Yöntemleri Uygulanarak Kalsiyum Oksit Briketlerinin Hazırlanması

Abstract

Asetilen gazı üretimi sırasında elde edilen kalsiyum karpit cürufunun (KKC) depolanması çevre açısından sorun yaratması nedeniyle, bu çalışmada KKC'den üretilen kalsiyum oksit (CaO) briketlerinin tekrar kalsiyum karpit (CaC_2) üretiminde kullanılması amaçlanmıştır. Taguchi yaklaşımı ile bağlayıcı tipinin (fosforik asit (H_3PO_4), melas ve mısır şurubu), bağlayıcı miktarının (%1, %3 ve %5), briketleme basıncının (20, 28 ve 36 MPa), kalsinasyon sıcaklığının (800, 900 ve 1000 ^oC) ve kalsinasyon süresinin (30, 45 ve 60 dk) üretilen CaO briketlerinin dayanımına olan etkileri araştırılmıştır. En yüksek dayanım değeri 4,05 MPa olarak bulunmuştur. En yüksek dayanıma sahip briketin dayanıklık ve kırılganlık

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değerleri sırasıyla %92 ve %8 olarak belirlenmiştir. ANOVA analizine göre, üretim parametrelerinin CaO dayanımına olan etkisi sırasıyla (i) bağlayıcı miktarı, (ii) bağlayıcı tipi, (iii) briketleme basıncı, (iv) kalsinasyon sıcaklığı ve (v) kalsinasyon süresi olarak belirlenmiştir. Elde edilen optimum deney şartları ise; %5 bağlayıcı miktarı, 28 MPa briketleme basıncı, 900 ^{o}C kalsinasyon sıcaklığı ve 60 dakika kalsinasyon süresi olarak belirlenmiştir. Elde edilen CaO briketleri CaC_2 üretiminde tekrar kullanılabilecek dayanımı değerini sağlamaktadır.

Anahtar Kelimeler: Kalsiyum karpit cürufu, CaO briketi, Mısır şurubu, Melas, H_3PO_4

1. INTRODUCTION

Millions of calcium carbide slag (CCS) containing a high amount of calcium hydroxide and small amounts of organic and inorganic materials (Al_2O_3, Fe_2O_3, unburned carbon, and etc.) is obtained as a by-product during the production of acetylene gas, in which the required CaO for this process was obtained as a result of the calcination of limestone rocks [1-2].

However, the disposal of CCS pollutes the water resources and soil due to its chemical composition. In order to minimize its harmful effect on an environment, the use of CCS as a binder in concrete have been investigated [3-7]. Furthermore, it has been used in a preparation of organic [8] and inorganic materials (xonotlite) [9-11], flue gas desulfurization [12], CO_2 capture [13,14]. However, the use of CCS in those of studies processes in the industrial applications was not preferred due to its lower economic efficiency.

Wang et al. [15] suggested the re-use of CCS in the production of CaC_2. For this purpose, the combination of briquetting and calcination processes was carried out to obtain CaO briquettes from CCS and therefore the recycling of the industrial waste was successfully performed. In the briquetting process, the CCS was mixed with a phosphoric acid (H_3PO_4, as a binder), and pressed to obtain a specimen prior to the calcination process. It was revealed that an increase in an amount of H_3PO_4 led to an increase in the strength value of CaO briquettes [16]. Unfortunately, the presence of phosphor (P) in the CaO briquettes maybe harmful material for the production of CaC_2. It was thought that the use of different binders for the CaO production may be useful.

As aforementioned above, the disposal of CCS generated during a production of acetylene gas is a problematical issue, as it pollutes an environment and soil due to the fact that it contains $Ca(OH)_2$ that leads to an increase pH values of the soil. Therefore, this study suggests a new perspective for the minimization of those tailings' effect on environment. It was aimed that calcium oxide (CaO) briquettes were prepared from CCS by a molding and calcination process in order to re-use in the production of calcium carbide (CaC_2) that reacts with water to produce acetylene gas. Herein, different binders (corn syrup and molasses) were used to prepare high strength CaO briquettes. In addition, CaO briquettes from CCS were produced using H_3PO_4 as a binder for the comparison. The effects of binder types (corn syrup, molasses, and H_3PO_4), binder amount (1, 3, and 5%), briquetting pressure (20, 28, and 36 MPa), calcination temperature (800, 900, and 1000 ^{o}C), and calcination time (30, 45, and 60 min) on the strength value of CaO briquettes were evaluated based on the Taguchi approach (L_{27}). The stability and friability values of the final product with the highest strength value were determined using a shatter test. This study not only reveals the usability of various binders for the production of CaO briquettes that are not harmful or the production of CaC_2 but also recommends mitigation ways to decrease the harmful effects of CCS on environment.

2. EXPERIMENTAL PROCEDURE

2.1. Material and Method

The calcium carbide slag was provided from a factory of acetylene gas production in Adana, Turkey. The sample was dried at an ambient temperature for 5 days in order to decrease the moisture content. The dried sample was crushed and ground to a value below <125 µm by using a jaw crusher and ball mill. The chemical composition of the sample is shown in Table 1.

Table 1. Chemical composition of the sample

Element	(%)	Element	(%)
MgO	0.16	SO_3	0.20
Al_2O_3	0.22	CaO	71.61
SiO_2	0.68	Fe_2O_3	0.24
C	4	LOI	23.03

X-ray diffraction analysis (XRD) given in Figure 1 shows that the sample is mainly composed of portlandite mineral (94.64%). Corn syrup ($C_6H_{14}O_7$), molasses, and phosphoric acid (H_3PO_4, analytical grade) were used as a binder. To the best of the author's knowledge, it was the first study that used corn syrup as a binder, whereas molasses and phosphoric acid were used as binders for the briquetting of coal [17] and CaO [16], respectively.

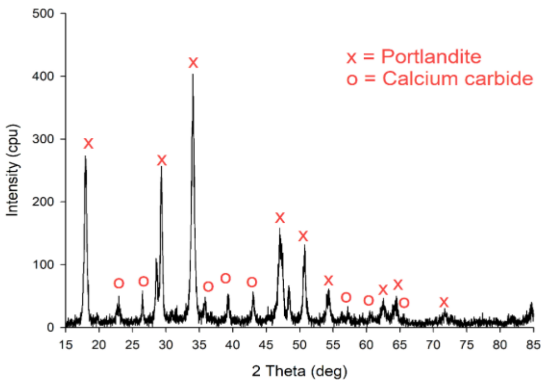

Figure 1. XRD pattern for calcium carbide slag (CCS) (Portlandite PDF Card No: 077-3842, Calcium carbide PDF Card No: 001-0917)

The binder solutions were prepared with 10 wt. % concentration for use in each briquetting test. The experimental procedure was carried out as follows:

(i) 20 g of the sample was mixed with binder and water to prepare calcium carbide briquettes prior to the calcination process.

(ii) The specimen was casted into a stainless steel mold (diameter: 40 mm) and various pressures were applied.

(iii) Each prepared calcium carbide briquettes was calcined at various temperatures in a range of 800-1000 ^{o}C in an ash furnace for the conversion of calcium carbide briquette into calcium oxide (CaO) briquettes.

2.2. Mechanical Properties of CaO Briquettes

The compressive strength value of CaO briquettes was determined in triplicates using a LT-SP1000 machine. The average value of three experiments was used to evaluate each production parameter on the properties of CaO briquettes. In addition, the stability and friability values of the final product were determined using a drop shatter test that has been used to determine the stability of coal briquettes according to the ASTM D440-86 [17].

The final product with a high strength value was dropped six times from the height of 100 cm. Thereafter, the particle size distribution of the product was determined by a dry sieve using the size fractions as follows: +40 mm, -40+31.5 mm, -31.5+20 mm, -20+10 mm, -10+8 mm, -8+5 mm, and -5 mm. The percentage of CaO briquettes before and after the shatter test was determined after weighing. The stability and friability values of the final product was determined by Equations (1) and (2) given below:

$$X = \frac{100 \times s}{S} \times 100 \quad (1)$$

$$F = 100 - X \quad (2)$$

where; X is the stability of CaO briquette (%), s is the sum of product after shatter testing (g), S is the total amount of the sample (g), F is the friability of CaO briquette (%).

The chemical composition of each product was determined using a standardless X-ray fluorescence (XRF, Panalytical MiniPal4). The phase composition of each product was determined by X-ray diffractometer (XRD, Rigaku Miniflex II)) with Cu Kα (k = 0.15406 nm) radiation over the 2θ range of 5°–85°.

2.3. Experimental Design

The number of experiments conducted to obtain CaO briquettes with a high strength from CCS was determined based on Taguchi method, which has been widely used in many engineering applications. This method makes the reduction in number of experiments [19]. The production parameters and their levels are listed in Table 2. Table 3 shows the selected orthogonal array ($L_{27}(3^5)$) for independent variables and levels. The influences of each production parameter on the strength value of CaO briquettes were determined. The aim of this study was to obtain CaO briquettes having high strength values. Therefore, the-larger-the-better quality characteristic was used as shown in Equation 3. The obtained experimental findings were evaluated using the Minitab 14 software.

$$S/N_s = -10\log\left[\frac{1}{n}\sum_{i=0}^{n} 1/y_i^2\right] \quad (3)$$

Table 2. Production parameters and their levels

Parameters	Symbol	Level 1	Level 2	Level 3
Binder type	**A**	Corn syrup (CS)	Molasses (M)	Phosphoric acid (P)
Binder Amount (%)	**B**	1	3	5
Pressure (MPa)	**C**	20	28	36
Calcination Temperature (°C)	**D**	800	900	1000
Calcination Time (min)	**E**	30	45	60

Table 3. Full factorial design with an orthogonal array of Taguchi $L_{27}(3^5)$

Experiment No	A		B		C		D		E	
1	1	**CS**	1	**1**	1	**20**	1	**800**	1	**30**
2	1	**CS**	1	**1**	1	**20**	1	**800**	2	**45**
3	1	**CS**	1	**1**	1	**20**	1	**800**	3	**60**
4	1	**CS**	2	**3**	2	**28**	2	**900**	1	**30**
5	1	**CS**	2	**3**	2	**28**	2	**900**	2	**45**
6	1	**CS**	2	**3**	2	**28**	2	**900**	3	**60**
7	1	**CS**	3	**5**	3	**36**	3	**1000**	1	**30**
8	1	**CS**	3	**5**	3	**36**	3	**1000**	2	**45**
9	1	**CS**	3	**5**	3	**36**	3	**1000**	3	**60**
10	2	**M**	1	**1**	2	**28**	3	**1000**	1	**30**
11	2	**M**	1	**1**	2	**28**	3	**1000**	2	**45**
12	2	**M**	1	**1**	2	**28**	3	**1000**	3	**60**
13	2	**M**	2	**3**	3	**36**	1	**800**	1	**30**
14	2	**M**	2	**3**	3	**36**	1	**800**	2	**45**
15	2	**M**	2	**3**	3	**36**	1	**800**	3	**60**
16	2	**M**	3	**5**	1	**20**	2	**900**	1	**30**
17	2	**M**	3	**5**	1	**20**	2	**900**	2	**45**
18	2	**M**	3	**5**	1	**20**	2	**900**	3	**60**
19	3	**P**	1	**1**	3	**36**	2	**900**	1	**30**
20	3	**P**	1	**1**	3	**36**	2	**900**	2	**45**
21	3	**P**	1	**1**	3	**36**	2	**900**	3	**60**
22	3	**P**	2	**3**	1	**20**	3	**1000**	1	**30**
23	3	**P**	2	**3**	1	**20**	3	**1000**	2	**45**
24	3	**P**	2	**3**	1	**20**	3	**1000**	3	**60**
25	3	**P**	3	**5**	2	**28**	1	**800**	1	**30**
26	3	**P**	3	**5**	2	**28**	1	**800**	2	**45**
27	3	**P**	3	**5**	2	**28**	1	**800**	3	**60**

3. RESULTS AND DISCUSSION

3.1. Evaluation of Experimental Findings

The highest strength value of CaO briquettes prepared from CCS is curicial for the quality of product that is re-use in the production of CaC_2 production. For this reason, the "**larger-the-better**" equation was used to calculate the S/N ratio. The photographs of prepared CaO briquettes before and after the calcination process are shown in Figure 2. Table 4 gives the values of the S/N ratio depending on the strength value of each CaO briquettes.

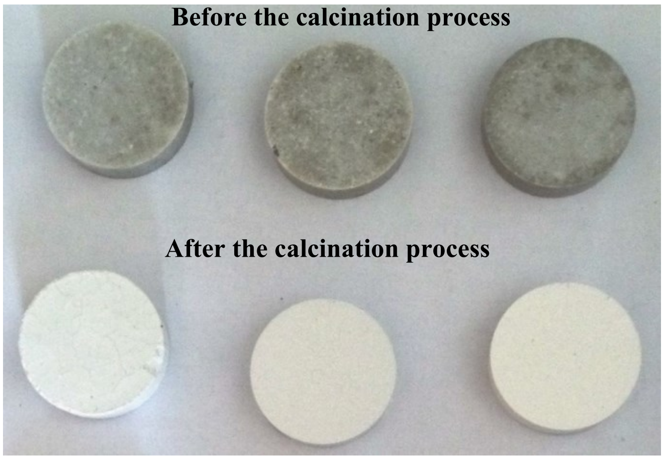

Figure 2. Photographs of prepared briquettes before and after the calcination process (calcination temperature: 700 ^{o}C, calcination time: 60 min, briquetting pressure: 28 MPa, binder amount: 5%, binder type: melasses)

The effects of each control factor (binder type, binder amount, briquetting pressure, calcination temperature, and calcination time) were analyzed through the S/N response table (Table 5). The highest S/N ratio shows the best level for the production condition of CaO briquettes (Figure 3). According to the Figure 3, the experimental conditions for the production of CaO briquettes with the highest strength from CCS should be as follows: phosphoric acid as binder, the amount of binder: 5%, briquetting pressure: 28 MPa, calcination temperature: 900 ^{o}C, and calcination time: 60 min.

Table 4. Experimental results and calculated S/N ratio in this study

No	Strength Value (MPa)*	S/N ratio	No	Strength Value (MPa)	S/N ratio	No	Strength Value (MPa)	S/N ratio
1	1.708	4.725	**10**	1.725	4.813	**19**	2.300	7.029
2	1.743	4.819	**11**	1.760	4.907	**20**	2.335	7.123
3	1.813	5.097	**12**	1.830	5.185	**21**	2.405	7.400
4	1.956	5.871	**13**	1.909	5.656	**22**	1.822	5.275
5	1.991	5.965	**14**	1.944	5.750	**23**	1.857	5.369
6	2.061	6.243	**15**	2.014	6.028	**24**	1.927	5.647
7	1.945	5.798	**16**	1.949	5.842	**25**	3.954	11.879
8	1.979	5.892	**17**	1.984	5.937	**26**	3.988	11.973
9	2.049	6.170	**18**	2.054	6.214	**27**	4.058	12.250

Table 5. The results of S/N ratio values for the production of CaO briquettes

Level	Control Factors				
	A	B	C	D	E
Level 1	5.630	5.751	5.436	**7.582**	6.310
Level 2	5.596	5.761	**7.679**	6.459	6.459
Level 3	**8.292**	**8.007**	6.404	5.458	**6.750**
Delta	2.696	2.225	2.242	2.124	0.440

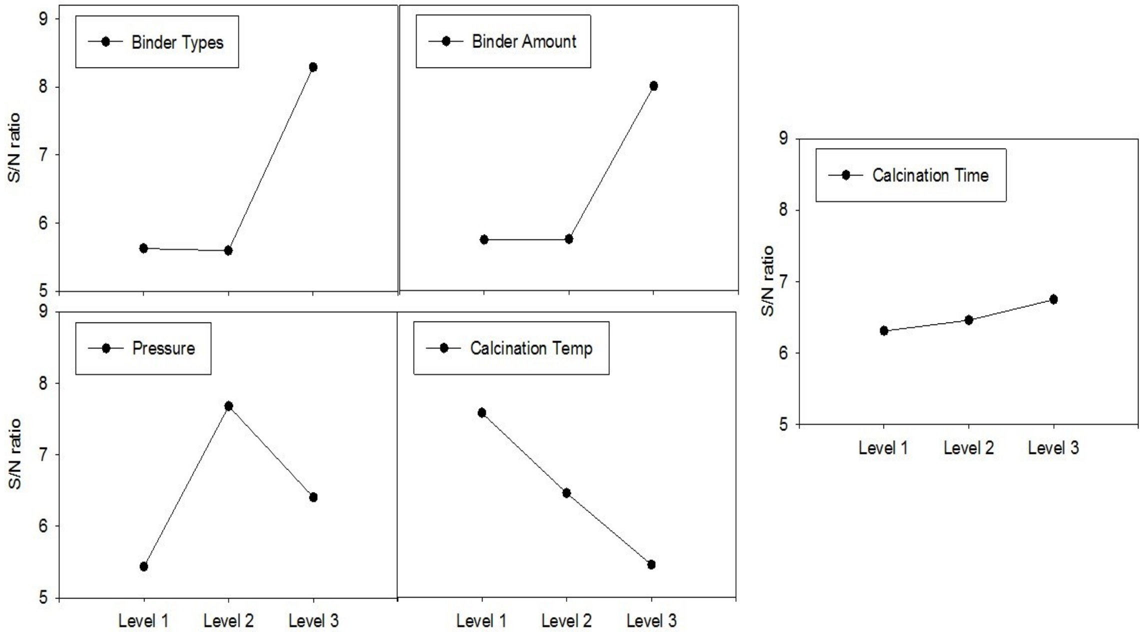

Figure 3. S/N response values for the strength value of CaO briquette

3.2. ANOVA Method

The individual interactions of the production parameters were evaluated by the ANOVA method. This analysis was performed with a 5% significance level and 95% confidence level. The influences of binder type and amount, briquetting pressure, calcination temperature and time on the strength properties of CaO briquettes were analyzed. Table 6 lists the ANOVA results for the strength value of CaO briquettes.

The significance of each production parameter was determined using F-values of each parameter. As given in Table 6, the contribution rate of each parameter was found to be 23.06% for factor A (binder type), 43.21% for factor B (binder amount), 17.79% for factor C (briquetting pressure), 8.12% for factor D (calcination temperature). However, the contribution rate of factor E (calcination time) was quite low, revealing that the strength value of CaO briquettes was not influenced by the calcination time. These rates indicated that the most effective factor on the strength value of CaO briquettes was binder amount (factor B, 43.21). The percent of error was 7.71% which was negligible. The relationship between binder types and other production parameters were examined as shown in Figure 4.

Table 6. Results of ANOVA for strength value of CaO briquettes

Variance Source	Degree of freedom (DoF)	Sum of squares (SS)	Mean squares (MS)	F-Value	Contribution rate (%)
A	2	3.1935	1.5976	23.93	23.06
B	2	5.9819	2.9909	44.82	43.21
C	2	2.4629	1.2314	18.46	17.79
D	2	1.1242	0.5621	8.42	8.12
E	2	0.0151	0.0075	0.11	0.11
Error	16	1.0676	0.0667		7.71
Total	26	13.8453			
Model → R= 92.29%, R-sq = 87.48%, R-sq(pred)= 78.04%					

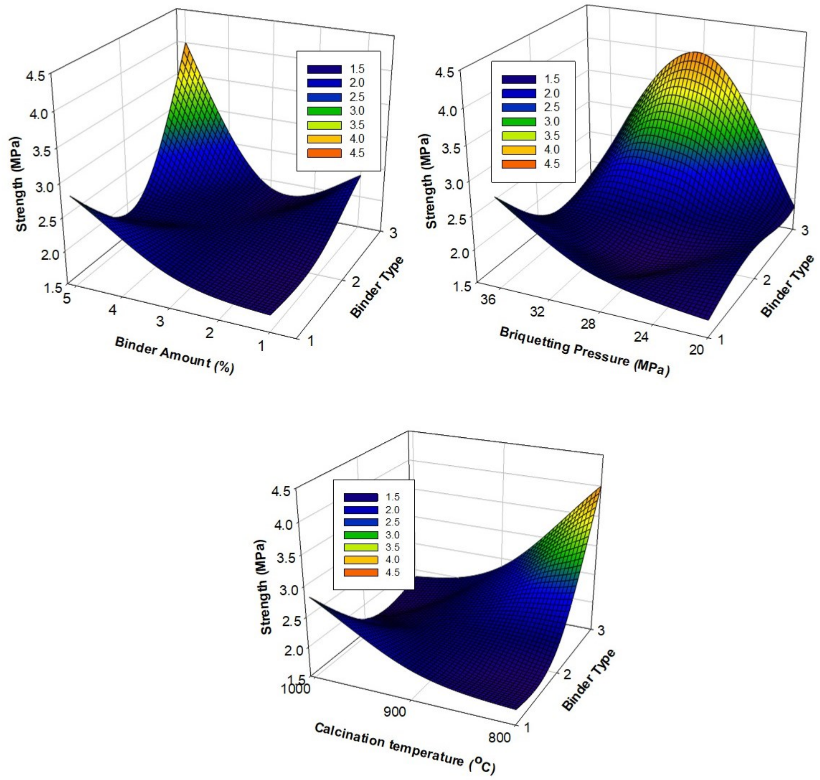

Figure 4. Effects on production conditions on the strength value of CaO briquettes (1: corn syrup, 2: molasses, 3: H_3PO_4)

It is obvious that the use of H_3PO_4 acid as a binder for the production of CaO briquette gave satisfactory results in comparison with the other binders (molasses and corn syrup). Furthermore, the increase of binder amount in the CaO briquettes led to an increase its strength value. The highest strength value of CaO briquettes was found to be 4.05 MPa that is higher than that of the previous study [14]. The stability and friability values of CaO briquettes were further determined using the shatter test. The experimental findings are listed in Table 7. These values revealed that the final product provides the necessary strength for use in the production of CaC_2.

Table 7. The shatter test result for the CaO briquette having the highest strength value

Particle size (mm)	Weight (%)	Normalizing Fac.	Size Stability (%)
Before Test			
-50 + 40 mm	100	-	-
After Test			
-50+40 mm	83.33	1	83.33
-40+31.5 mm	4.17	0.79	3.31
-31.5+20 mm	6.25	0.57	3.58
-20+10 mm	4.17	0.33	1.39
-10+5 mm	2.08	0.17	0.35
Size stability (S) = 91.96 ~ 92 Friability (F) = 100 – 92 = 8			**91.96**

The briquette obtained at optimal conditions was mainly composed of CaO (lime) as shown in Figure 5 and it contained 96.18% CaO, 1.4% Al_2O_3, 1.9% SiO_2, 0.25% Fe_2O_3, and 0.27% others.

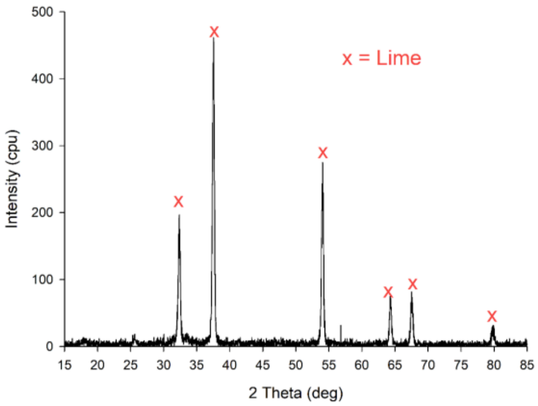

Figure 5. XRD pattern of the final product (Lime PDF Card No: 082-1691)

4. CONCLUSIONS

This study reported the results on the preparation of calcium oxide (CaO) briquettes from calcium carbide slag (CCS) for the re-use in the production of CaC_2 using briquetting and calcination processes. By this, the disposal problem of CCS was minimized.

The experimental findings were evaluated through Taguchi approach which decreases the number of experiments. The effects of production parameters were ordered (from the highest to the lowest) as follows: (i) binder types, (ii) binder amount, (iii) briquetting pressure, (vi) calcination temperature, and (v) calcination time, respectively. Besides H_3PO_4, molasses or corn syrup can be used as a binder for the briquetting of CaO as its strength value was in line with that of the previous study. This study presents an alternative to minimize the disposal problem of industrial tailings.

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