

# **“The Effect of Sintering Time on Recycled Magnesia Brick from Kiln of the Cement Plant”**

Bramantyo Aji<sup>1</sup>, Diah Rosalina<sup>2</sup>, Azhar<sup>2</sup>, Muh. Amin<sup>1</sup>

1) Lembaga Ilmu Pengetahuan Indonesia (LIPI) UPT Balai Pengolahan Mineral Lampung  
Ir. Sutami Street, Km. 15, Tanjung Bintang Bandar Lampung 35361

2) Chemical Engineering Department, Chemical Engineering, University of Lampung  
Prof. Dr. Soemantri Brodjonegoro Street 1 Bandar Lampung 35145

Email: [diahrosalina93@gmail.com](mailto:diahrosalina93@gmail.com)

## **ABSTRACT**

Manufactured refractory brick from used refractory of kiln cement plant with the variation of sintering times had been done. The purpose of this research was to find the effect of sintering time toward physical properties of refractory, such as the apparent porosity, the bulk density, cold crushing strength and thermal conductivity. The used refractory which used on this research was magnesia with the addition of kaolin clay as adhesive. Total weight which used was 300 grams with the composition of 85% used refractory, 15% kaolin clay, and 12%wt of water was added. The larger aggregate was 40+80 mesh in amount of 65,6%, the smaller aggregate was -80 mesh in amount of 34,4% and kaolin clay was -100 mesh. The materials then mixed and stirred till it perfectly blended, and after that it was molded with the size of 5x5x5 cm using hydraulic press under a load of 10 tons so it formed greenbody. Greenbody then dried and sintered at 1200 °C with time variation of 2 hours, 4 hours, 6 hours, 8 hours and 10 hours. The recycle refractory brick had been made the compared with the used refractory before it was recycled and also new refractory. Based on characteristic test that had been conducted, it shows that the longer sintering time decrease of apparent porosity and increase of the bulk density, Cold Crushing Strength (CCS) and thermal conductivity.

**Key Words :** Refractory, sintering, sintering time, bulk density, apparent porosity, cold crushing strength, thermal conductivity.

## **1. INTRODUCTION**

Refractory is one of ceramics that able to hold its physical and chemical properties at high temperature. Generally, refractory is made from silicon oxide ( $\text{SiO}_2$ ), potassium oxide ( $\text{CaO}$ ), magnesium oxide ( $\text{MgO}$ ), aluminium oxide ( $\text{Al}_2\text{O}_3$ ), zirconium oxide and some other oxides such as carbide, nitride, boride, silica and graphite. In cement industry, refractory is used for isolator rotary kiln to protect kiln from high temperature, chemical reactions, and abrasions. In Indonesia, Wiharja (2006) had been studying that the replacement of refractory of kiln cement is frequently done for 3 to 4 times in a year [1]. That frequency increase the used refractory which can not be used because had been damaged. Actually, refractory is one of the most spent cost of cement plant operation and

made from rare material because of its limited availability. So, it needs more efforts to make it usable again.

This research was done to make refractory from kiln used refractory of cement plant with the purpose to improve the physical properties of used refractory. Refractory which used was Magnesia. Refractory was made by using general refractory producing method by crushing, sift, pressing and heating (sintering). The common refractory physical properties were the bulk density, the apparent porosity, cold crushing strength, and thermal conductivity.

The chemical composition of the used refractory and kaolin clay raw material is shown in Table 1.

Tabel 1. The chemical analysis of the raw materials used for the production recycled refractory bricks.

Component	wt% in Used refractory	wt% in Kaolin clay
MgO	86,83	0,35
Al <sub>2</sub> O <sub>3</sub>	1,59	28,90
SiO <sub>2</sub>	1,16	42,32
Fe <sub>2</sub> O <sub>3</sub>	0,34	1,76
CaO	2,76	1,89

Almost all refractory physical properties were formed in sintering process. Sintering is the heating process of solid powder into more compacted solid under its melting point. The influencing factors of sintering were temperature and sintering time, particle size, raw mix material composition and green body density [2]. This research specifically discuss about effect of sintering time toward refractory physical properties. The physical properties then compared to used refractory and new refractory.

## 2. MATERIAS AND METHODS

**2.1 Materials.** Material which used was magnesita used refractory of rotary kiln from cement plant in West Java, Indonesia. Kaolin clay was added as adhesive from Kemiling Kaolin clay Mine, Bandarlampung.

**2.2 Procedure for recycle refractory brick production.** Total composition a refractory recycle sample (Rr) was 300 grams with 85% of used refractory and 15% kaolin clay and water was added as much as 12%wt. The larger refractory size was -40+80 mesh in amount of 65,6%, the smaller refractory aggregate size was -80 mesh in amount of 34,4% and kaolin clay was size of 100 mesh. All materials then were mixed and stirred till it was perfectly blended, then molded in size 5x5x5 cm using hydraulic press under a load of 10 tons till greenbody formed. Greenbody then dried at room temperature for 2 days after that dried in oven at 110 °C for 24 hours. Sintering temperature in furnace was 1200°C and cooled in furnace till reach room temperature [3]–[6]. The sintering time were 2 hours, 4 hours, 6 hours, 8 hours and 10 hours. The refractory without kaolin clay was made as standard of comparison with composition was 100% of used refractory with addition water of 12%wt, 300 grams. Agregates were made in the same size. The larger refractory

size was -40+80 mesh in amount of 65,6%, the smaller refractory aggregate size was -80 mesh in amount of 34,4% then sintered at 1200 °C with 6 hours sintering time.

**2.3 Thermal Conductivity Test.** Thermal conductivity is the heat amount which pass through a material at a unit of time, per unit of temperatures gradient as long as stream direction per unit of area. Sample thermal conductivity is tested by using standard guarded hot plat method ASTM C177-04 [7]. Sample which tested were recycle refractories and used refractory.

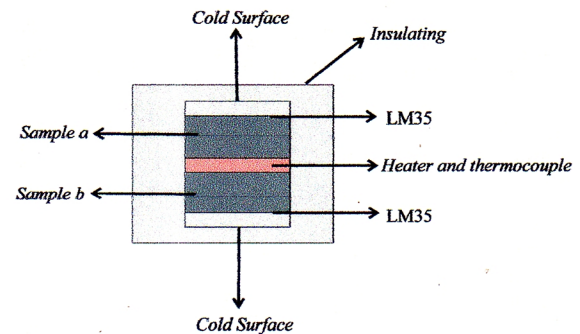


Figure 1. Illustration of thermal conductivity test

With absorbed energy equal to the released energy of heater, so:

$$Q = k \times A \times (\Delta T / \Delta x) \quad (1)$$

With Q (watt), k (watt/m.K), A sample surface area (m<sup>2</sup>), T temperature (K), and x material thickness (m).

The equation for conductivity (k) calculation, was as follows:

$$k = \frac{Q}{A[(\Delta T/x)_a + (\Delta T/x)_b]} \quad (2)$$

**2.4 Apparent Porosity and Bulk Porosity Test.** Apparent porosity and bulk density is tested by using standard method ASTM C20-00 [8].

**2.5 Cold Crushing Strength (CCS) Test.** Cold crushing strength or pressure strength is the maximum load when cracking occur or material destruction per unit surface area when pressured at room temperature [5]. Cold Crushing Strength test was based ASTM C133 [9].



### 3. RESULT AND DISCUSSION

Characteriation of recycle refractory with kaolin clay bricks after sintering:

**3.1 Apparent Porosity and Bulk Density.** Sintering time of refractory will affect to pores and density from refractory material which stated by apparent porosity and bulk density.

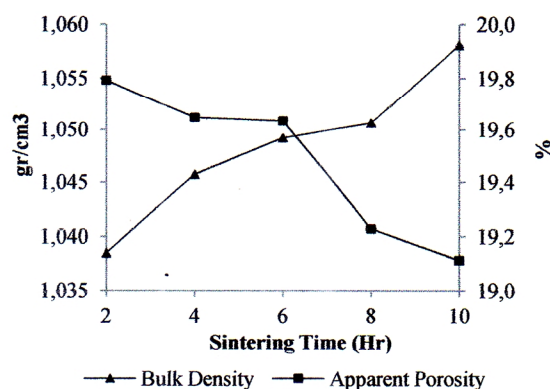


Figure 2. The effect of sintering time to apparent porosity and bulk density of refractory

Figure 2 presents that bulk density of recycle refractories increases and apparent porosity decreases with increasing the sintering time. Sintering was carried out at 1200 °C with variations of sintering time : 2 hours, 4 hours, 6 hours, 8 hours and 10 hours. The result of the bulk density are 1,038 gr/cm<sup>3</sup>; 1,046 gr/cm<sup>3</sup>; 1,049 gr/cm<sup>3</sup>; 1,051 gr/cm<sup>3</sup> and 1,058 gr/cm<sup>3</sup>. The apparent porosity are 19,79%; 19,65%; 19,63%; 19,23% and 19,11%.

Porosity of refractory was begun to form on molding process. On molding process, the fusion and compaction process occur cause of the given pressure so there will be empty spaces between particles. The smaller particle will penetrate to the empty spaces between the larger particle so the refractory will become more solid, is called as greenbody. Greenbody then be more solid and more powerful through sintering process.

Porosity of recycle refractories decreases with increases of sintering time. At sintering occur diffusion of atoms which move from particle surface to pores so there will be fusion one particle to another particle called as neck. The neck growth on specific temperature is depend on the sintering time. The neck growth will influence the shrinkage of material pores. The porosity can be formed because the

unbalanced diffusion and trapped gas in material [2]. The lower porosity of refractory bricks the higher material density of materials. This statement is displayed in Figure 4 .

**3.2. Thermal Conductivity.** There are several parameters influencing the thermal conductivity such as bulk density and apparent porosity.

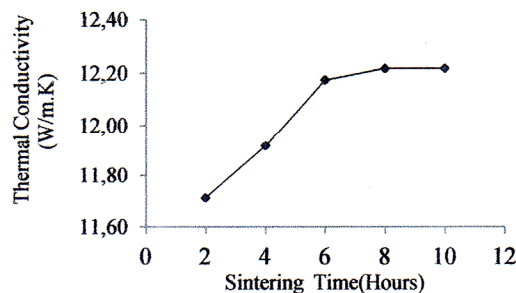


Figure 3. The effect of sintering time toward refractory thermal conductivity

Figure 3 present that thermal conductivity of recycle refractories increases with increasing sintering time. The result of conductivity at sintering time 2 hours, 4 hours, 6 hours, 8 hours and 10 hours are 11,7120 W/m.K; 11,9179 W/m.K; 12,1749 W/m.K; 12,2188 W/m.K; 12,2188 W/m.K.

Thermal conductivity is affected by its refractory density. The higher density the easier heat to pass through material because the restriction in form of porosity in getting lower. The low thermal conductivity is needed for refractory which used as isolator in industry, to hold the heat for it will not be released to environment and also protect equipments from the high temperature.

**3.3 Cold Crushing Strength.** Cold crushing strength (CCS) is standard of refractory which stated the strength of refractory to the given maximum load. CCS does not indicate the characteristic of refractory when it is used and more state the strength of refractory when it is transported. Refractory with the good strength is not easily crack or damage when it is transported. It is same with thermal conductivity, CCS also have relation with density and porosity of materials.

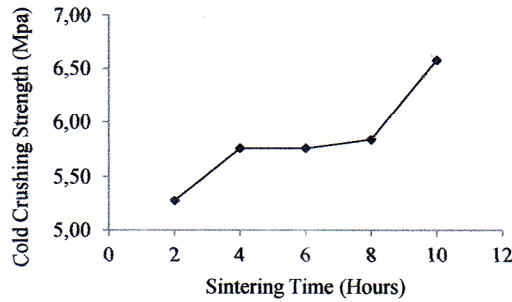


Figure 4. The effect of sintering time duration towards Refractory Cold Crushing Strength

Figure 4 present that cold crushing strength increases with increasing of sintering time. CCS at sintering time of 2 hours, 4 hours, 6 hours, 8 hours, 10 hours are 5,28 Mpa; 5,76 Mpa; 5,76 Mpa; 5,84 Mpa; 6,58 Mpa.

Refractory CCS has relation with its density and it porosity. The higher refractory density so the better strength of the refractory.

**3.4 The Effect of Kaolin clay Addition to Refractory Composition.** Kaolin clay is one of clay that has good plasticity when it is mixed with water [10]. Accordance with clay characteristic in common, kaolin clay will be flexible when it is mixed, it will be hard when it is dried and it will be solid and strong when it is burned.

Table 2. The comparison of recycle refractory with kaolin clay dan without kaolin clay.

Sample	CCS (Mpa)	Porosity (%)	Bulk Density gr/cm <sup>3</sup>	Conductivity (W/m.K)
Recycle + Kaolin clay	5,76	19,63	1,049	12,1749
Recycle without Kaolin clay	2,04	16,53	1.189	12,1835

According to Table 2, refractory with kaolin clay has the lower bulk density than refractory without kaolin clay, but has the the better strength, it is based on its cold crushing strength value.

Around temperature 450°C, the chemically combined water in clays is release and leaves a large pore within refractory body, resulting in the formation of metakaolin clay. As metakaolin clay is heated around 980°C, spinal phase is formed, an amorphous siliceous phase. As the temperature increases (at about

1100°C), the spinel is transformed into mullite and, simultaneously, SiO<sup>2</sup> is expelled in the form of glassy phase. Then glassy silica promotes sintering, which is identified by the third shrinkage phase found above 1200°C [6], [11]

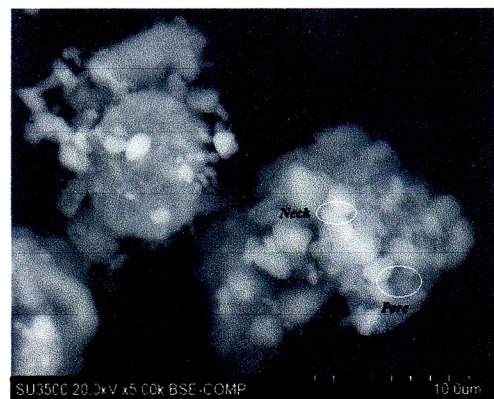
Mullite is considered as a binding phase in most of refractory brick and it has a high resistance to melting and minimum thermal expansion as well as low thermal conductivity [3].

**3.5 The Comparison of Recycle Refractory, Used refractory and New Refractory.** The comparison of recycle refractory, used refractory and new refractory is shown in table 3

Table 3. The comparison of recycle refractory, used refractory and new refractory

Sample	CCS (Mpa)	Porosity (%)	Bulk Density gr/cm <sup>3</sup>	Conductivity (W/m.K)
Kiln Used	48.38	5,49	2.82	13,5440
Recycle + Kaolin clay	6,58	19,11	1,058	12,2188
New Refractory [12]	34.47-55.16	15.5-19	2.84-2.9	10.56

The recycle refractory has not reach the best characteristic, which improve the used refractory characteristic resemble to new refractory characteristic.



a)





b)  
Figure 5. a) SEM from recycle refractory with kaolin clay addition with sintering at 1200 °C for 10 hours. b) SEM from kiln used refractory.

Figure 5 present the SEM result that recycle refractory sintering had occur with the presence of particle grains which united one to another (neck), but sintering occur imperfectly. It is proved by the growth of imperfect grains and its various size. The growth of too big grain will eliminate the bond between particles which located at the edge of grain borders (grainboundaries), therefore will decrease the strength of refractory, and it will cause the refractory to become fragile. Besides, the imperfect grains growth will affect untoward pores because the increasing of refractory porosity. It is different from SEM result of used refractory which looks more compact and united so it has the better physical properties.

#### 4. CONCLUSION

The manufacture of refractory from kiln used refractory from cement plant had been done. According to physical test that had been conduct, it shows that the longer sintering time cause the lower apparent porosity, bulk density, cold crushing strength (CCS) but it cause the higher thermal conductivity. Refractory with addition of kaolin clay has the bigger apparent porosity and lower bulk density, but it has the better cold crushing strength (CCS) and thermal conductivity than refractory with the absence of kaolin clay. The recycle refractory has not the characteristic as good as the used refractory.

Physical properties of recycle refractory is still not the same as new refractory, so it is not applicable yet. Even so, this research is very suitable to continue, it is because the

availability of kiln used refractory from cement plant that still can be used. There are some treatment that can be done to increase the recycle refractory properties. First, compaction pressure when molding can be increased in order to gain more solid greenbody. In industry, pressure which commonly used is 100 Mpa or in the sample of this research is about 25 tons. Second, sintering temperature is also can be raised, for magnesia refractory the temperatures are about 1500-1750 °C [5]. Heating rate in sintering process must also be a concern, it can not be too quick, so it will not cause fracture of refractory.

#### REFERENCES

- [1] Wiharja, "Perubahan Tipe Bata Tahan Api Pada Kiln Semen," *J. Tek. Lingkung.*, pp. 17–22, 2006.
- [2] M. N. . Rahaman, *Sintering of Ceramics*. New York: CRC Press, 2007.
- [3] M. Al-Amaireh, "Improving the physical and thermal properties of the fire clay refractory bricks produced from bauxite," *Journal of Applied Sciences*, vol. 6, no. 12, pp. 2605–2610, 2006.
- [4] S. Nugroho and Y. Umardhani, "Karakterisasi Material Refraktori Basa Berbahan Dasar Magnesia ( MgO ) Guna Lining Tungku Induksi Pengecoran Baja DI PT X KLATEN," *Pros. Semin. Nas. Sains dan Teknol. ke 2*, pp. 124–129, 2011.
- [5] C. A. Schacht, *Refractories Handbook*. New York: Marcel Dekker, Inc, 2004.
- [6] D. J. Duval, S. H. Risbud, and J. F. Shackelford, *Ceramic and Glass Materials*. 2008.
- [7] ASTM C177-04, "Standard Test Method for Steady-State Heat Flux Measurements and Thermal Transmission Properties by Means of the Guarded-Hot-Plate," *Annu. B. ASTM Stand*, vol. 552, pp. 1–23, 2015.
- [8] ASTM C20-00, "Standard Test Methods for Apparent Porosity , Water Absorption , Apparent Specific Gravity , and Bulk Density of Burned Refractory Brick and Shapes by Boiling Water," *Annu. B. ASTM Stand*, vol. 0, no. Reapproved 2015, pp. 1–3, 2015.
- [9] ASTM C133, "Standard test methods for cold crushing strength and modulus of rupture of refractories," *Annu. B. ASTM Stand*, vol. 97, no. Reapproved 2008, pp. 1–6, 2014.
- [10] Practical Action, "Clay as a binder," *Aurov. Earth Inst.*, vol. 44, no. 871954, 2015.
- [11] Process Engineering, "Sintering Behaviour of Clays for," vol. 84, no. 5, pp. 5–8, 2007.
- [12] Harbison & Walker, "Harbison-Walker Handbook of Refractory Practice," 2005.