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Utilization of iron ore slag in the manufacture of calcium silicate boards

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ABSTRACT

This study aims to determine the iron ore slag effect as an additive in particleboard based on the SNI 7705:2011 standard. Iron ore slag comes from the waste processing of iron ore into sponge iron. The iron ore slag is reduced to a size of 200 mesh. Particleboard made with the composition of slag and silica is 0:40, 8:32, 16:24, 20:20, 24:16, 32:8, and 40:0 wt%. Meanwhile, other materials were made permanent, namely PCC cement and lime 16 wt%, coconut fiber 3wt%, and water 3 wt%. They are pressed with 3 tons of pressure for 1 hour using a hydraulic press. Drying at room temperature for one day, under the hot sun for two days, then in an oven at 110 °C for 8 hrs. Analysis of the chemical composition of X-ray fluorescence and X-ray diffraction crystalline phase, SEM-EDS micro-photographs, physical tests including density and porosity, and mechanical compressive strength tests. The dominant composition of SiO_2 and CaO affects the formation of silicon dioxide (SiO_2), calcium silicate ($CaSiO_3$), and dicalcium silicate (Ca_2SiO_4) phases. Silica has a positive effect on the compressive strength of particleboard but is different from Ca, which has an impact on reducing the compressive strength. The sem morphology shows that coconut fiber cannot withstand heating at 190 °C and results in agglomeration. The addition of 20% ore slag and silica has met the calcium silicate board SNI 7705-2011. These results can be used to develop slag waste from iron ore processing into much more useful objects.

1. INTRODUCTION

The metal industry in Indonesia continues to increase in line with the development and industry developments. According to data from the Indonesian Ministry of Industry, the population of the Metal industry continues to grow with the addition of 142 sectors in 2019 (kemenperin.go.id). Metal production produces various products, especially iron/steel, nickel, and aluminum. The production process of metal smelting from iron ore produces solid waste known as slag (Siradjuddin, 2011). The management of steel slag waste in practice has challenges and the increase in the steel industry (Yang, 2017). The slag content of iron ore contains calcite oxide compounds (CaO), silica (SiO_2), alumina (Al_2O_3), and magnesia (MgO) (Feng et al., 2013). Iron ore slag contains gehlenite ($2CaO.Al_2O_3.SiO_2$) and akermanite (2CaO.MgO.2SiO) (Puertas and Jimenez, 2003). Technically, steel slag can be used as an aggregate material in civil works, such as cement mixture in concrete (Pane et al., 2021), road aggregate (Theresia & Susanti, 2017), to use as fertilizer (Rosidah et al., 2018). The dominant oxide compounds in iron ore slag in CaO and SiO_2 are very close to calcium silicate compounds. Calcium silicate is the result

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of the reaction of calcium carbonate (CaO) and silicon dioxide (SiO_2) using a reliable method to form $CaSiO_3$ (Jacob, 1976) and Ca_2SiO_4 (Budiman and Asmi, 2013). The response can be seen as follows:

$$CaCO_3(s) + SiO_2(s) \longrightarrow CO_2(g) + CaO(s) + SiO_2(s) \longrightarrow CaSiO_3(s) \quad (1)$$

$$CaSiO_3(s) + CaO(s) \longrightarrow Ca_2SiO_4(s) \quad (2)$$

Calcium silicate contains calcium (Ca), silica (Si), and oxygen (O_2) (Newport, 2006). Calcium silicate can be made from natural and artificial minerals. Calcium silicate contains calcium (Ca), silica (Si), and oxygen (O_2) (Newport, 2006). Calcium silicate can be made from natural and artificial minerals. Minerals derived from nature are classified as impure because they still contain iron, magnesium, manganese, sodium, and potassium, so it is necessary to carry out a purification process to obtain pure calcium silicate. Iron ore slag from industrial waste also still contains many other chemical compounds, which are impure calcium silicates, especially the content of hematite (Fe_2O_3). The average silica content in iron ore slag is 41.54% (Syarif, 2010). The advantage of using iron ore slag with a high silica content is that its ability to provide a strong bond between cement and aggregate (Anggraeni, 2017). The content of iron ore slag is close to that of cement and natural sand. Iron ore slag can be used as a substitute for cement with alternative materials such as silica, portland cement, coconut fiber, and water. Based on (SNI 7705: 2011), cement functions as a binder, cellulose for flexibility, silica sand as a space provider or matrix, aluminum hydroxide or clay media for fire resistance (Hariadi, 2010). Types of sheet boards include magnesium, calcium silicate, and gypsum boards (Hariadi, 2010). This type of calcium silicate board has better advantages over other sheet materials. The weakness of the calcium silicate board is that it is not strong enough to be applied as a structural wall and has not been able to absorb sound waves (Asmi et al., 2011).

Standards for determining the percentage of the chemical composition of calcium silicate boards are not yet available. This study examines calcium silicate boards based on their chemical composition according to SNI 7705:2011. The standard is about a building material made of silica sand, portland cement, cellulose, and water with or without additives if needed, which is formed into a flat shape. The chemical reaction occurs at temperature and pressure between the elements of silica and calcium. So that the dominant chemical compounds on the calcium silicate board are SiO_2 and CaO, based on SNI 7705:2011, particleboard is grouped into five classes based on flexural strength. Classification of flat sheets for the flexural strength of calcium silicate boards is shown in Table 1.

Tabel 1. Classification of calcium silicate board

No	Class	Type A (Mpa)	Type B (Mpa)
1	Class 1	4	4
2	Class 2	7	7
3	Class 3	13	10
4	Class 4	18	16
5	Class 5	24	22

Category A is the saturated condition for products that are used outdoors. Category B is the ambient condition intended for indoor (interior) (SNI 7705-2011).

This research aims to make calcium silicate boards from iron ore slag. Flexural strength test based on SNI 7705-2011 standard. Chemical composition, crystal phase, SEM morphology, and physical examinations were used to support the properties analysis of the calcium boards made.

2. METHOD

Slag obtained from processing iron ore is cleaned of other impurities. Subsequently, the slag and silica materials were milling used a ball bill machine to get a powder. The powder of iron ore slag and silica are then sieved through an ASTM 200 mesh sieve. The coconut fiber used has a size of 40 mesh. The parameters for the manufacture of calcium silicate boards are shown in Table 2.

The process of mixing all the ingredients using a mixing machine is then formed in a mold measuring 200 x 50 x 20 mm using the hand layup method. Three-ton compaction to molds used the pressure press machine for 6hrs. The samples were then left at room temperature for 24hrs. Drying in the sun for two days and continued using the oven at 110 °C for 8hrs. Flexural strength test using UTM HT 2404 100kN. Physical examinations include density, porosity, absorption, and water content based on the ASTM C 642-90 test standard, using equations (3) to (5).

$$\text{Porosity (\%)} = \frac{W2-W1}{W2-W3} \text{ x } 100\% \quad (3)$$

$$\text{Absorption (\%)} = \frac{W2-W1}{W1} \text{ x } 100\% \quad (4)$$

$$\text{Porositas (\%)} = \frac{W1}{W2-W3} \text{ x } \rho\text{air} \quad (5)$$

Table 2. The composition of calcium silicate board

Compotitions (wt%)	TS1	TS2	TS3	TS4	TS5	TS6	TS7
Iron ore Slag	0	10	15	20	25	30	40
Silica	40	30	25	20	15	10	0
PCC cement	Fixed 15						
Lime	Fixed 15						
Aquades	Fixed 25						
Coconut fiber	Fixed 5						

The chemical compositions of the raw material were characterized by Epsilon 4 XRF Spectrometer from Malvern Panalytical with operating at 50 kV and three mA. The board's crystallinity and phase were measured by Panalytical Xpert 3 Powder X-RD with a Cu-Kα as a source of X-ray operating at 40 kV and 30 mA. The sample was scanned in the range 2θ of 0-80. Meanwhile, to determine the morphology used Nixon Eclipse MA100.

3. RESULT AND DISCUSSION

Results on the chemical composition test using X-ray fluorescence obtained from chemical compounds, as shown in Table 3.

Slag from iron ore processing has a dominant chemical composition of 38.73% SiO_2, 24.36% CaO, 16.31% Fe_2O_3, 8.64% Al_2O_3, and 6.19% SO_3. At the same time, other chemical compounds are less than 2%. The high CaO content in the slag is influenced by lime ($CaCO_3$) during the iron ore reduction process. In the iron ore smelting process, limestone serves to bind iron ore impurities and coke ash (C), including silicon, phosphorus, and manganese (Syarif, 2010).

The silica has a composition of 95.26% SiO_2, while other compounds such as Al_2O_3, CaO, P_2O_5, and Fe_2O_3 are less than 2%. SiO_2 concentrations above 90% are good silica. Lime contains 95.91% CaO, while MgO, Al_2O_3, SiO_2, K_2O, TiO_2, Cr_2O_3, MnO, and Fe_2O_3 are below 2%. Based on SNI 15 7064 2004 standard, PCC cement contains 60-67% CaO, 17-25% SiO_2, 3-8% Al_2O_3, and a maximum of 4.0% SO_3.

The chemical composition of the calcium silicate board can be seen in Table 4.

Tabel 3. Chemical composition for raw materials

No	Compound	Iron ore slag (%)	Silicate (%)	Lime (%)
1	MgO	1.30	-	0.30
2	Al_2O_3	8.64	0.70	0.78
3	SO_3	6.19	-	-
4	SiO_2	38.73	95.26	1.93
5	K_2O	1.89	-	-
6	CaO	24.36	0.98	95.91
7	TiO_2	0.59	-	-
8	MnO	1.44	-	-
9	Fe_2O_3	16.31	2.12	0.73
10	P_2O_5	-	0.74	-
11	PbO	0.14	-	

Tabel 4 . The chemical composition of the calcium silicate board

Compound	TS1	TS2	TS3	TS4	TS5	TS6	TS7
MgO	0.206	0.206	0.996	1.156	1.241	1.831	2.065
Al_2O_3	1.399	1.411	3.157	4.453	3.470	4.644	5.028
SO_3	0.288	0.311	0.473	0.374	0.229	0.759	0.366
SiO_2	40.468	29.364	47.069	33.395	29.307	28.667	21.04
K_2O	0.479	0.950	0.861	1.325	1.322	1.422	2.038
CaO	53.193	62.499	42.613	55.243	59.666	57.477	63.094
TiO_2	0.137	0.224	0.245	0.303	0.270	0.335	0.372
MnO	0.134	0.389	0.512	0.741	0.729	0.934	1.288
Fe_2O_3	1.524	4.399	3.825	3.758	3.513	3.639	3.620
SrO	0.111	0.149	0.131	0.134	0.140	0.193	0.162

Tabel 5. The physical and mechanical tests of the calcium silicate board

Sample	Physical				Mechanical
	Porosity (%)	. Absorbtion (%)	Density (g/cm³)	Water content (%)	Flexural content (Mpa)
Standard	-	≤60 (oversea, 1987)	≥1,3 (overseas, 1987) dan ≥ 0,8 (SNI 7705-2011)	5-15 % (JIS A 5905-2003)	4 (1A/B) 7 (2 A/B) 16 (4B) 18 (4A) 22 (4B) 24 (4A)
TS1	47.895	41.264	1.160		2.50
TS2	41.309	30.809	1.340	Average: 0.53-5.32	2.50
TS3	39.572	28.522	1.388		2.00
TS4	37.586	27.199	1.382		4.00
TS5	38.204	26.466	1.367		3.00
TS6	42.499	32.173	1.320		3.50
TS7	43.011	32997	1.308		3.25

Based on SNI 7705:2011 calcium silicate board in terms of SiO_2 and CaO elements. From the study results, the highest SiO_2 was found in samples of 15% slag and 25% silica, which was 47.069%. SiO_2 content is influenced by the use of slag but does not dominantly change the composition of the calcium silicate board. The CaO content affects high CaO compounds in limestone, and cement is more dominant. 40 wt% slag without the addition of silica has made the CaO content of calcium silicate board high.

Other chemicals did not significantly change based on XRF analysis, even with added iron ore slag variations. The total content of Fe_2O_3 itself is an average of 2% so that Fe_2O_3 is not too influential. Calcium silicate boards with a small iron oxide content are particularly advantageous. More Fe can cause a decrease if it reacts with water (Cahyono & Kusdarini, 2019). The physical and mechanical tests of the calcium silicate sample board can be seen in Table 5.

The density of the average calcium silicate board is 1.323 g/cm³. The most negligible density is obtained when the panels are made without slag. At the same time, the addition of 10-40 wt% slag has a density that is not much different, which is around 1.3 g/cm^3. The addition of slag and silica with concentrations of 15/25, 20/20, and 25/15 wt% gave higher board density than the other percentages.

The relationship between the porosity and density of the calcium silicate board in this study is shown in Figure 1.

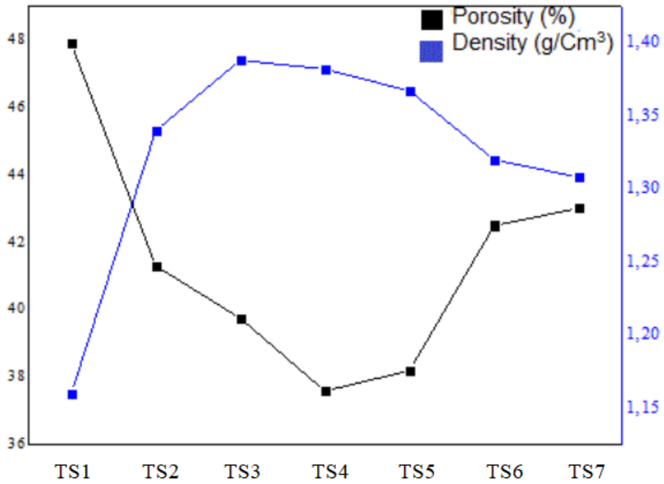


Figure 1. The relationship between porosity and specimen density

The board with the percentage of slag and silica 0/40 wt% showed the highest porosity value and the lowest density. Based on the XRF results, the CaO, SiO_2, Al_2O_3, Fe_2O_3, and MgO compounds were less. The density of iron ore slag is 3.5 g/cm^3. The sample board with 0/40 wt% slag and silica has the lowest density value because it uses the least iron ore slag. The more iron ore slag used, the higher the density value and the porosity decreases until the optimum point is added to 40/40 wt% slag and silica. The high porosity is due to the dominant composition of iron ore slag and higher CaO content. The heating process affects the particle size and surface area of CaO. Heating limestone for too long will result in the agglomeration of CaO particles so that the CaO surface increases significantly and causes porosity (Khaira, 2011). Overall, calcium silicate boards with the addition of iron ore slag showed an average density of 1.3 g/cm^3, following those on the market (Asmi et al., 2011). The specific gravity of iron ore slag is heavier than other materials and affects density (Amin et al., 2018). The board density value based on SNI 7705:11 has met the density standard, a minimum of 0.8 g/cm3.

The relationship between porosity and absorption is shown in Figure 2.

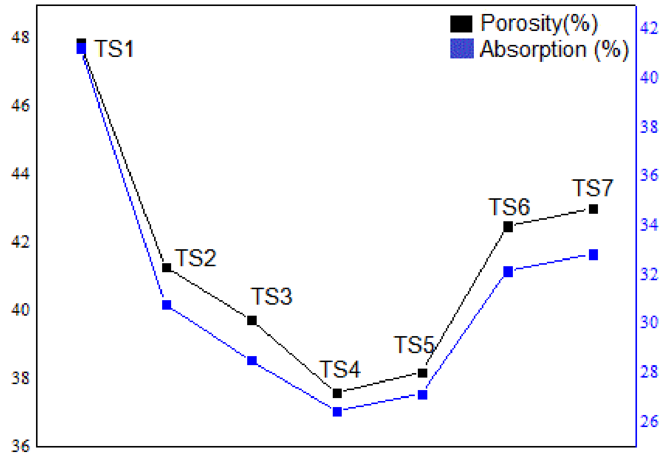


Figure 2. The relationship between porosity and absorption

The composition of iron ore slag and silica affects the porosity and absorption of the calcium silicate sample board so that the porosity and absorption values are directly proportional. The absorption function determines the absorption of the sample to water (Sagel et al., 1997). The absorption value is directly proportional to the porosity value. The higher the percentage of iron ore slag and the lower the percentage of silica, the lower the porosity value. In addition, the specimen contains coconut fiber. In natural fibers, cellulose and lignin are the most significant composition. These components are insoluble in water, but cellulose can absorb water (Pohan and Setiawan, 2000). Drying at high temperatures will result in the loss of some of the swelling properties. Heating at a temperature of about 100°C will reduce the ability to expand by about 50% (Putera, 2012). In addition, due to the heating process at 110°C, the coconut fiber changes its structure and mass. As a result, the board shrinks, and pores are formed. The water absorption capacity of the board is also affected by the high silica content (Sagel et al., 1997). The lowest water absorption was a sample of slag and silica 40/40 wt% with SiO_2 of 33.395%. SiO_2 can still fill the empty spaces on the board surface to reduce the water absorbed. The moisture content of the calcium silicate board is between 0.53%-5.32%.

Figure 3 shows the moisture content of the calcium silicate board. The higher the heating temperature of a material, the higher the percentage loss of water content. When heated in a temperature range of 110 to 190°C, the fiber and CaO will be lost. It makes a significant change in water content.

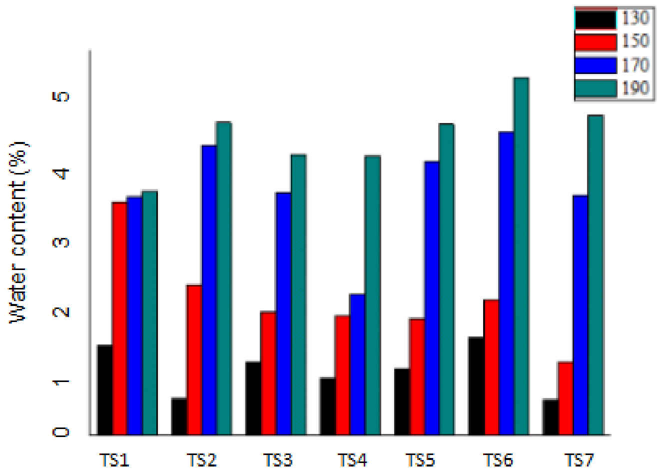


Figure 3. Water content of calcium silicate boards.

The results of the flexural strength test of calcium silicate board are shown in Figure 4.

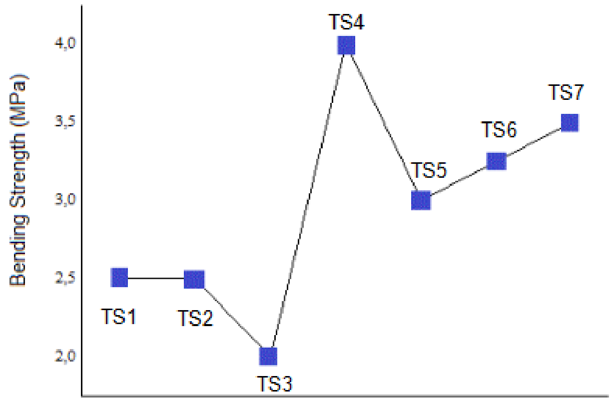


Figure 4. The flexural strength test of calcium silicate board

The highest flexural strength was found in the 40/40 wt% silica slag sample board at 4.0 MPa with the silica and ore slag percentage each of 20.16%. The addition of the slag percentage will increase the compressive strength, tensile strength, and flexural strength (Stiati & Halim, 2018). In addition, the acquisition of the elastic properties of coco fiber affects the flexural strength of the sample. Judging from the percentage of slag used, the piece with the best flexural strength was slag - silica 0/40wt%. However, this board has a high CaO compound. The nature of CaO, along with the increase in calcium dioxide, will decrease its compressive strength and flexural strength. Silica properties, the higher SiO_2 can increase the compressive strength and flexural strength of calcium silicate (Astuti et al., 2012). The mixing of materials in the manufacturing process also affects the flexural strength of the sample, for inhomogeneous mixing will cause the flexural strength graph to be formed imperfectly (Kanti, 2014).

The results of the XRD analysis are shown in Figure 5.

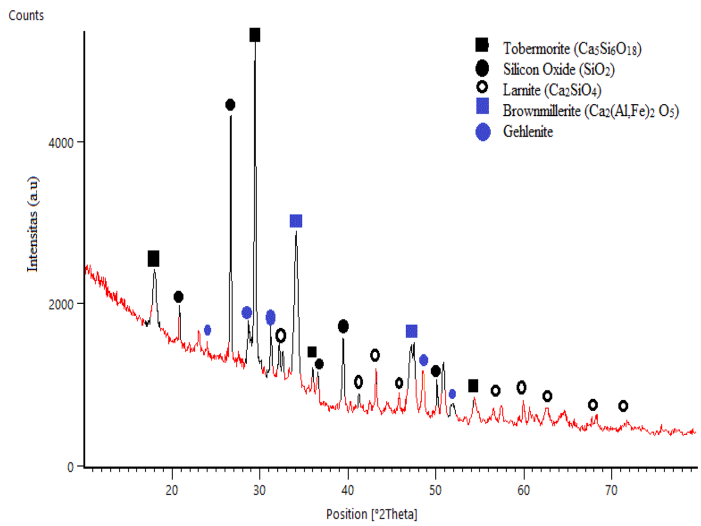
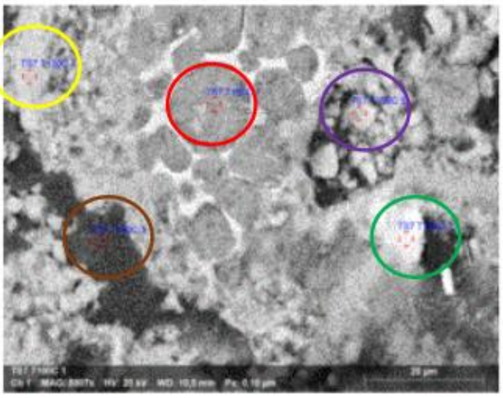


Figure 5. The results of the XRD analysis

Calcium silicate board phases include silicon dioxide, wollastonite, brownmillerite, gehlenite, and tobermorite. The silicon oxide (SiO_2) phase has a hexagonal crystal structure and is the highest peak on the graph. Wollastonite ($CaSiO_3$) phase with the monoclinic crystal structure. Brownmillerite ($Ca_2(AlFe)_2O_5$) phase with orthorhombic crystal structure and gehlenite phase. The diffraction peak in calcium silicate is a typical peak for dicalcium silicate. High CaO tends to produce calcium silicate. This silicate is a type of dicalcium silicate (Ca_2SiO_4). Dicalcium silicate (Ca_2SiO_4) is optimum at a ratio of SiO_2/CaO 4:6 (Haryono et al., 2018).

Figure 6 depicts the morphology of the particle size with a magnification of 5000X.



Gambar 6. The morphology of the particle size at 190 °C

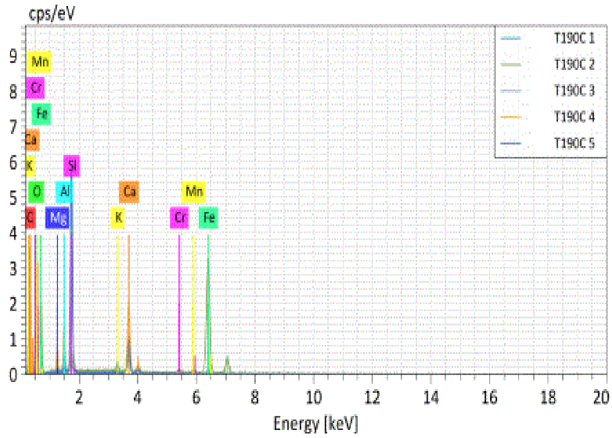


Figure 7. The results of SEM-EDS at 190ºC

The distribution of the constituent elements is shown using colored circles. Colored circles of red, green, orange, yellow, purple are indicated in order. In SEM-EDS results, the area with purple color displays the distribution of calcium silicate minerals ($CaSiO_3$) as evidenced by the absence of other elements except Ca and Si with mass percentages of 11.20% 11.12%, respectively.

The constituent materials are still visible, pores are formed in the board, not yet fully integrated, and some grains have accumulated (agglomerated). The phenomenon caused by the minerals that make up the sample, such as fiber, has melted at 190ºC, so there are still some inhomogeneous constituent minerals.

The results of SEM-EDS are shown in Figure 7. Wherein In the energy spectrum of 0-2 KeV, there are the elements of carbon (C), oxygen (O), potassium (K), iron (Fe), chromium (Cr), manganese (Mn), magnesium (Mg), aluminum (Al) and silicon (Si) are present. In the energy spectrum 2-4 KeV, there are elements of potassium (k) and calcium (Ca) detected, and in the energy spectrum 4-7 KeV, there are elements of chromium (Cr), manganese (Mn), and iron (Fe) observed.

4. CONCLUSION

The manufacture of calcium board from iron ore slag and silica with a percentage of 20.16% of each material on a 40/40 wt% slag-silica sample board has met the class 1 standard of SNI 7705:2011. The phases formed in the sample are silicon dioxide (SiO_2), calcium silicate ($CaSiO_3$), and dicalcium silicate (Ca_2SiO_4). The highest water content in the test object is 4.76, with a heating temperature of 190ºC. SEM morphology shows that the constituent minerals and pores can still be seen clearly, but not yet wholly mixed, and some grains accumulate (agglomerate). The agglomerates caused by the fibers have undergone melting at 190ºC. The EDS results show the formation of a distribution of calcium silicate ($CaSiO_3$) minerals as evidenced by the absence of other elements except Ca and Si with mass percentages of 11.20% and 11.12%, respectively. This research shows that iron ore waste slag can be developed as calcium silica board manufacture.

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