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Mechanical and Physical Properties of Cement Mortar with Recausticizing Solid Byproduct

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Abstract
The Kraft process is a method used to make paper pulp. This method produces cooking residue that can be recycled again. The recausticizing process produces large amounts of by-products.This research utilizes solid waste from causticizing from the pulp industry as a partial substitute for Portland cement for mortar raw material to obtain a material with good mechanical properties. The solid waste was pulverized, then characterization of Loss on Ignition (LOI), Inductively Coupled Plasma (ICP), Titration, and Total Titrable Alkali was carried out. The percentage of solid waste resulting from recausticizing as a substitute for cement is 0-100%, with a water-cement ratio (w/c) of 0.3. The mixed material is then printed into a 50x50x50mm mold followed by drying using the moist curing method. The hardened samples were tested for Density, Porosity, Water Absorption, and Compressive Strength. Mortar with partial cement replacement with 20-100% solid waste recousticizing produces a compressive strength of 1.3-22.6 MPa. The resulting water absorption ranges from 14.59-31.35%.

INTRODUCTIONS

The number of sizeable medium-scale manufacturing companies reached 30,381 companies in Indonesia according to Central Bureau of Statistics, 2020. This plays a very important role in national economic growth, as evidenced by the achievement of Indonesia as the 5th largest manufacturing industrial country. Currently, the manufacturing industry can contribute to the national Gross Domestic Product (GDP) of 20%. From this 20% achievement, Indonesia ranks fifth among the G20 countries. Indonesia's position is after China, with the contribution of its manufacturing industry reaching 29.3 percent. Then, followed by South Korea (27.6%), Japan (21%) and Germany (20.7%). Indonesia became the first Asean country to be trusted as an official partner in organizing the largest manufacturing technology exhibition in the world. This is a form of recognition for Indonesia, which is increasingly establishing itself as one of the world's industrial powers.

The larger the manufacturing industry, the greater the waste generated in each of its production processes, so the government is also tightening regulations for the disposal of industrial waste. In line with Government Regulation No. 12 of 1995 concerning Hazardous Waste Management, the pulp and paper industry has implemented a Zero Waste Policy. In accordance with this Policy, their industries strive to reuse, recover, and recycle its production waste. The purpose of implementing the policy is to encourage industrial players in the country to run their businesses more responsibly.

One of the pulp and paper industry utilizes acacia wood as the raw material for making paper pulp. Supported by sophisticated and up-to-date technology, the Kraft method was used as a method used in the process of making paper pulp. This Kraft method uses sodium hydroxide (NaOH) and sodium sulfide (Na_2S) as a cooking solution (Li, 2018). One of the advantages of this kraft process compared to other processes is that the cooking residue can be recycled. The rest of the cooking is in the form of black liquor which is recycled back into white liquor (NaOH and Na_2S). In the recovery process from recausticizing, many by-products are produced including Lime mud, Dregs and Grits (Han, 2021; Quina & Pinheiro, 2020).

Grits are oversized material and unreacted lime particles that have settled on the bottom of the lime slaker classifier and have been taken out for disposal. The number of grits varies from mill to mill depending on impurities and lime reactivity (du Plessis et al., 2021). Dregs are fine particles extracted from green liquor (green hard liquid) which is deposited in a large separator pond. Green liquor is obtained by adding water to inorganic substances after the wood organic components of the cooking liquid are burned in the cooking stove (AL-Kaabi, 2018). Grits and dregs are categorized as hazardous waste, which has a non-acute (delayed) effect and indirectly impacts humans and the environment. This waste category has a toxicity that tends to be sub-chronic or chronic/long term (Simão et al., 2019). So far, grits have only been used as additional material to make fertilizer, the rest will be directly disposed of in landfills. Theoretically, grits and dregs contain high levels of sodium carbonate (Na_2CO_3), calcium carbonate ($CaCO_3$) and sodium sulfide (Na_2S). This can be used as the basis for the theory that causticizing solid waste can be used as cement replacement in cement-based mortar because they have a high content of lime (CaO). Based on the above background, this study was conducted in carrying out an innovation in using causticizing solid byproducts (RSB) as a partial replacement in cement mortar.

MATERIALS AND METHODS

This research is divided into three stages. The first stage is the preparation stage. At this stage, preparations start from preparing raw materials and additional chemicals. The second stage is the manufacturing stage; at this stage the process of making mortars is carried out. The last stage is the testing stage, which tests the mortar samples produced following the criteria.

Preparation stage

The LOI, ICP, titration, and total titrable alkali measurements were conducted to measure the properties of RSB. Samples of dreg and grits in powder were weighed using an analytical balance for ±20 grams. LOI measurement begins with drying RSB in an oven at a temperature of 100°C for 24 hours, then their weight was measured. The weighed dreg and grits samples were burned in a furnace at 1000°C and held for 2 hours. After being cooled, it is then weighed again with an analytical balance. The weight of the dreg and grit samples that have been burned are referred to as combustible materials. To calculate the ash content. Samples of dreg and grits previously burned with a mass are then burned to a temperature of 1200°C using a furnace and then cooled again to room temperature. The mass of the sample that has been burned at a temperature of 1200°C is then weighed to determine the value of Loss of Ignition (LOI).

ICP Characterization was conducted by weighing RSB ±0.5 gram, then putting it into the vessel and adding 8 ml of HNO_3 and HBF_4 to the sample. Insert the vessel into the Microwave Digestion tool to prepare the sample which was initially a solid into a fluid before checking with ICP. The sample was transferred to a 100 ml flask, and then added denim water to the boundary line of the flask. Then the sample was homogenized. The sample was transferred to a test tube and perform the test using Inductively Coupled Plasma (ICP).

CaO measurement by titration was conducted by preparing RSB in wet and dry conditions as much as 0.5 grams. Add 30 ml of 1:1 HCl and heat until dry. After drying, add 25 ml of 1:9 HCl. Add 100 ml of demin water and heat for 10 minutes. Then wait until the temperature decreases. After cooling, filter using 42 filter paper and put it in a 250 ml flask. Dilute in a 250 ml flask by adding demineralized water to 250 ml and take a sample of 20 ml and then put it in an Erlenmeyer. Add 100 ml of demin/milli Q water. Add 5 ml of 10% hydroxylamine chloride, 15% thrietanolamine, 15 ml of 20% KOH, and 2-3 drops of calcin indicator. Titrate with 0.05 M EDTA with a color change from purple to blue.

Total Titrable Alkali was performed by weighing the sample ± 6 grams and putting it in a round flask. Add 250 ml of demineralized water and 10 ml of 10% $(NH_4)_2CO_3$ solution. Attach the round flask to the condenser running the cooling water and heat it until it boils for ± 2 hours. Filter and collect the filtrate with filter paper 93 on Erlenmeyer. Add enough MO indicator to the filtrate, then titrate with HCl solution. Record the volume of HCl used then calculate the Total Titratable Alkali.

Manufacturing stage

The variations of cement and RSB are shown in Table 1. The water for diluting dry material was kept at 0.3 water per cement (w/c) ratio.

Table 1. *Mix Design of RSB as partial replacement of cement mortar*

No	Cement:RSB (%)
1	100:0
2	80:20
3	60:40
4	40:60
5	20:80
6	0:100

Test sample casting is done with a press/mash tool. The dough that has been mixed evenly is poured into a 50x50x50mm cube-shaped mould and then pressed/pounded with a fist until dense. The drying process is carried out by leaving it at room temperature and not exposed to direct sunlight for 1 day and then releasing it from the mold. Treatment of the test object was carried out for 28 days by rinsing with water twice a day to maintain the water content in the mortar with the aim of avoiding cracks during curing time.

Testing stage

The testing of materials was conducted by density, porosity, water absorption, and compressive strength. The density of material (ρ) was measured by dividing the dry mass (m) and volume (V) of the mortar. The porosity test was done by immersing test objects in water until it is saturated for 24 hours. The weight in a wet state was weighed using a scale with an accuracy of 0.5 grams. Dry in the oven at a temperature of ± 105°C for 24 hours. The test object is weighed in an oven dry state. Weighing is done to determine the difference in weight between the test object that has been soaked in water for 24 hours and the test object that has been dried for 24 hours. The water absorption test was done by immersing the test object in water until it is saturated for 24 hours. The weight of sample in a wet state was weighed using a scale with an accuracy of 0.5 grams. Dry in the oven at a temperature of ± 105°C for 24 hours. The test object is weighed in an oven dry state. Weighing is done to determine the difference in weight between the test object that has been soaked in water for 24 hours and the test object that has been dried for 24 hours. Compressive Strength (F) was measured using compressive test machine by dividing maximum load (F) with area (A).

RESULT AND DISCUSSION

Loss of Ignition Test Analysis

The results of testing the raw materials used will be presented in Table 2. This test is carried out with the aim of knowing the nature of the raw material because dry mass, combustible mass, ash content and loss of ignition will affect the resulting mortars. This dry weight test was carried out with the aim of knowing the solids content contained in the dregs and grits.

Table 2. *Loss of Ignition Test Result of RSB*

Sample	Dry Mass (%)	Combustible Mass (%)	Ash Content (%)	Loss of Ignition (%)
Dregs	57.47	62.60	37.85	1.85
Grits	85.19	48.35	55.36	1.51

In the table 2, it is known that the dry weight of grits is more than dregs. This is due to the presence of a lot of weak wash liquor in the dregs. In its formation, dregs are produced in the process of separating solid (dregs) and liquor (green liquor) in optical clear, which uses a polymer to bind small particles to form lumps called dregs.

The burning material and ash content were tested by burning the sample at a temperature of up to 1000°C for 2 hours. The amount of burnt material was obtained at about 62% dregs and 48% grits. Meanwhile, the ash content in dregs is 37% and grits is 55%. The LOI (Loss on Ignition) test is carried out with the aim of knowing the percentage of substance content that is lost from the sample within a certain time and temperature. In this test, it is carried out by burning at a temperature of 1200%. The results of the LOI obtained 1.8% dregs and 1.5% grits

Inductively Coupled Plasma Test Analysis

The results of testing the dry raw materials RSB were presented in Table 3.

Table 3. *ICP result of Dreg and Grits*

Element	Dreg (%)	Grits (%)
Ag	0.0071	0.0004
Al	100.338	20.2371
B	810.5142	784.7686
Ba	306.7843	128.5977
Bi	328.9972	327.6378
Cd	0.0068326	0.002715
Co	0.0088	0.0019
Cr	0.2079856	0.0381107
Cu	1.0045795	0.0170538
Fe	37.5879	7.3295
Ga	0.2502	0.088
In	0.0472	0.0249
K	16.80851	8.71212
Li	0.0388	0.003
Mg	92.2533	22.0941
Mn	8.9575	0.1585
Na	118.0004	120.5947
Ni	0.3306828	0.1424366
Pb	0.0231	0.009
Si	75.0503	51.8488
Sr	2.9943	0.6885
Ti	0.0328	0.029

Table 3 shows that all elements contained in dregs and grits after drying for 5 hours at a temperature of 120°C. The wet sample still contains water contained in the dregs and grits. The drying mechanism will experience a dehydration reaction (evaporation), so that if the ICP test is carried out, the data that is read is truly derived from dregs and grits.

The most dominant elements in dregs and grits are B (Boron), Na (Sodium), Al (Aluminum) and Si (Silicon). Due to the addition of HBF_4 during the digestion process, the B (Boron) value cannot be calculated. As a result of the Ca (calcium) content being too high, the ICP device could not read the content of these compounds. To overcome this, a check is carried out using the titration method.

The data in Table 4 were the chemical compounds of dregs and grits that have been calculated from ppm to percent.

Table 4. *Loss of Ignition Test Result of RSB*

Sample	SiO_2 (%)	Fe_2O_3 (%)	Al_2O_3 (%)
Dregs	2.89	1.93	6.81
Grits	2.21	0.42	1.52

From table 4, the chemical compounds of SiO_2, Fe_2O_3 and Al_2O_3 which have been calculated as percent, have increased on average. In the manufacture of mortar, the raw materials used are cement, sand and water with the compositions contained therein, among others, SiO_2, Fe_2O_3, Al_2O_3, CaO and H_2O.

SiO_2 or Silicon dioxide is a crystalline compound that is insoluble in water and has a high tensile strength (Linec & Mušič, 2019). In the manufacture of mortar, SiO_2 plays an important role in the resulting product, among others, to reduce cracks in the drying process, increase the strength of the mortars by binding force and filling the empty spaces formed due to the drying process so that the resulting mortars are more tightly packed and has good resistance (Liu et al., 2020). Table 4 shows that the SiO_2 content in dregs and grits only reaches 1-3%, so other raw materials are needed as an addition to the manufacture of mortars.

Aluminum Oxide (Al_2O_3) is a compound commonly used in the use of aluminum. In the manufacture of cementitious material, Al_2O_3 acts as a catalyst that accelerates the hardening and drying process. Al_2O_3 content contained in dregs and grits reaches 6%. In the manufacture of mortars, the content of dregs and grits is more than sufficient so that no additional materials are needed in the manufacturing process.

Iron(III) oxide (Fe_2O_3) is an oxide compound of iron which has paramagnetic properties. In the manufacture of mortars, this Fe_2O_3 acts as a paving block compactor but this compound does not affect the properties and strength of mortars (Yang, 2019). The content of dregs and grits has met the standard so that no additional materials are needed in the manufacturing process.

CaO measurement by titration Test Analysis

Due to the Ca (Calcium) content being too high, the ICP could not read the content, so the CaO content was retested using the Titration method. Table 5 shows the results of the CaO content in dregs and grits.

Table 5. *Titration Test Result of RSB*

Sample	CaO (%)
Dregs	36.44
Grits	48.11

In the manufacture of hardened cementitious materials, CaO becomes a framework-forming compound or body. CaO has good hygroscopic properties. It reacts with water, producing a binding force that binds the materials, forming the hardened sample so that the mixture of materials will be easier to shape and mold. In this test, the CaO content in dregs and grits was 36% and 48%, respectively. The CaO content of both is more than sufficient. The content of CaO itself will greatly affect the strength and porosity of the resulting paving block.

High CaO content in cementitious material will leave pores, which cause a decrease in the compressive strength value and increase the porosity value; therefore, to minimize this, cement containing SiO2 is needed, which functions as a filler so that the quality of the hardened material will increase.

Total Titrable Alkali Test Analysis

According to the Government Regulation of the Republic of Indonesia No. 101 concerning the management of hazardous and toxic waste. Dregs and grits are classified as hazardous waste (Category II). This category of waste has a delayed effect, meaning that it has an indirect impact on humans and the environment and has chronic or sub-chronic toxicity, therefore it is necessary to test the content of Total Titratable Alkali (Table 6) on dregs and grits to determine the dangers of dregs and grits. This is if used as raw material for making cement mortar.

Table 6. *Total Titrable Alkali (TTA) Test Result of RSB*

Sample	Total Titrable Alkali (%)	
	Before Drying	**After Drying**
Dregs	13.36	2.43
Grits	14.09	4.87

The table 6 shows the test results for the TTA content of dregs and grits after drying. The results showed a decrease in dregs, from 13% down to 2%. Likewise, with grits, grits with an initial content of 14% decreased to 5%. The decrease in the TTA value was due to the evaporation of the Na content due to the drying of the sample at a temperature of 105°C for 5 hours.

Density Measurement

Density is a measurement of a material which is defined as mass per unit volume. The results of testing the raw materials used will be presented in Figure 1. The density test results were obtained at the percentage of cement 0%, 20%, 40%, 60%, 80% and 100%. The results obtained successively also are 1.67, 1.74, 1.78, 1.86, 1.91 and 2.18. The highest density value was obtained at a 100% cement percentage of 2.18, while the lowest density value was obtained at a 0% cement percentage of 1.67%. The reason for the increase in the density value is due to the higher cement density than grits and dregs. The density of cement is around 1.44 g/cm3, while for grits and dregs, it is only around 0.76 g/cm^3. In addition to the density value of the raw material, the density value is also influenced by the mass of the resulting paving block. The heavier the mass, the fewer cavities contained in the paving block. This cavity is formed because there is an exothermic reaction between CaO and SiO_2 which will cause heat and gas waves formed during molding. At the time of hardening these gas bubbles will decompose to form cavities (Dhasindrakrishna et al., 2021).

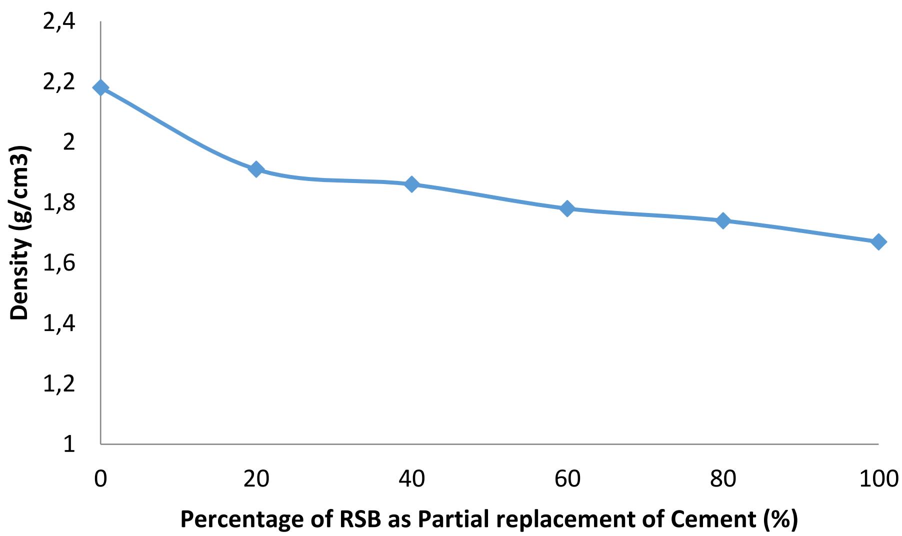

Figure 1. *Density of mortar with RSB as partial replacement of cement*

Porosity Test results

Porosity or pore is the empty space between the cement mortar. The porosity of mortar is expressed as a percentage (%) and is the ratio between the total volume of the pores and the total volume of mortars, which shown in Figure 2.

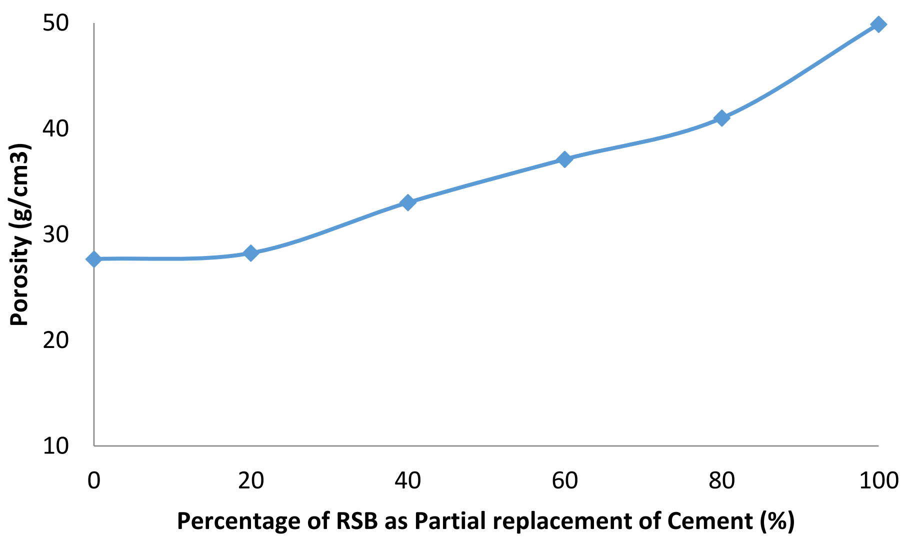

Figure 2. *Porosity of mortar with RSB as partial replacement of cement*

From the figure above, the results of porosity testing are obtained at the percentage of cement 0%, 20%, 40%, 60%, 80% and 100%. Successively participating results were 49.88%, 41%, 37.11%, 33.01%, 28.24% and 27.67%. The highest porosity value was obtained at 0% cement percentage of 49.88% while the lowest porosity value was obtained at 100% cement percentage of 27.67%. This shows that the more cement mixture in the manufacture of mortar, the lower the porosity produced.

High porosity or cavities are caused by the inaccurate quality and composition of the constituent materials, the influence of a ratio between cement and water that is too large can cause the formation of pores/cavities, because there is unreacted water then evaporates and leaves the cavity.

When viewed from the chemical content of the dregs and grits, the CaO content in both wastes was 70% while SiO_2 was only 3%. This excess CaO which when reacted with water will leave cavities during drying. The small percentage of cement causes the cement to be unable to bind and fill the cavities that are formed.

The casting process also influences the size of the porosity value. During the moulding process, the researcher only used a long iron stick as a compactor of dough into the mould (manually), so it was likely that the decrease in dough density would increase the porosity of the paving block.

Water absorption results

The water absorption test was carried out to determine the percentage of the ratio of the wet mass of the paving block that was soaked for 24 hours and the dry mass of the paving block after being baked for 24 hours was shown in Figure 3.

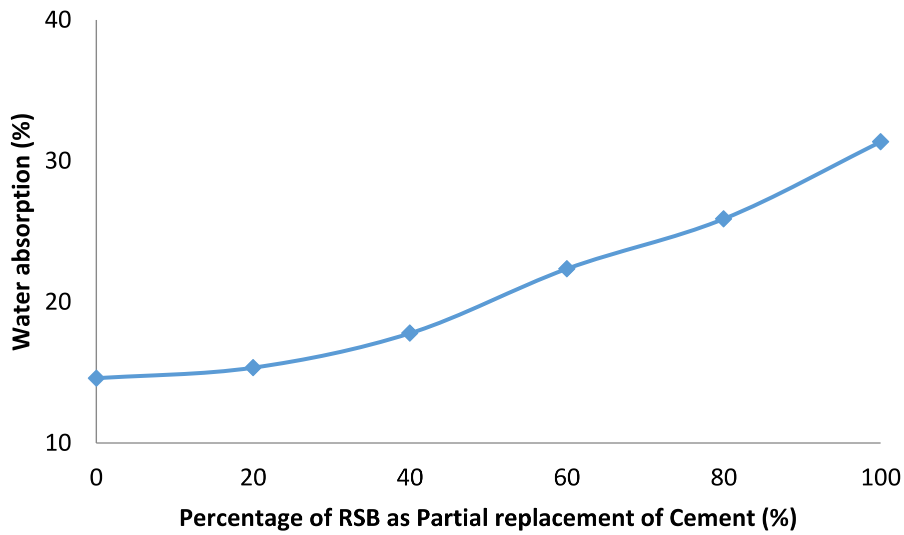

Figure 3. *Water absorption of mortar with RSB as partial replacement of cement*

From the test results, obtained the value of water absorption at the percentage of cement 0%, 20%, 40%, 60%, 80% and 100%. The results obtained were 31.35%, 25.89%, 22.35%, 17.78%, 15.34% and 14.59%, respectively. The highest absorption value was obtained at 0% cement percentage of 31.35% while the lowest absorption value was obtained at 100% cement percentage of 14.59%. This shows that the percentage of cement mixture is inversely proportional to the resulting absorption. If the percentage of cement increases, the resulting absorption will be smaller, and vice versa.

Figure 3 shows that water absorption value of mortars is found to be in the range of 14.59% - 31.35%. Excess CaO or Lime can cause water absorption to increase. This is due to the hygroscopic nature of CaO (Mulyasih, 2010) or absorbs water. In addition, the lack of SiO_2 contained in the dregs and grits makes the air cavities that are formed are not filled so that the density of the mortar decreases. The denser the mortar, the more difficult it is to absorb water.

Compressive test results

The compressive strength test is carried out to find out how much the mortars capacity to receive load. Testing the compressive strength of mortars is analogous to the compressive strength of concrete, which is the magnitude of the load per unit the area that causes the concrete test object to crumble when it is subjected to a pressure force by Compression Machine. The compressive strength of mortars was shown in Figure 4.

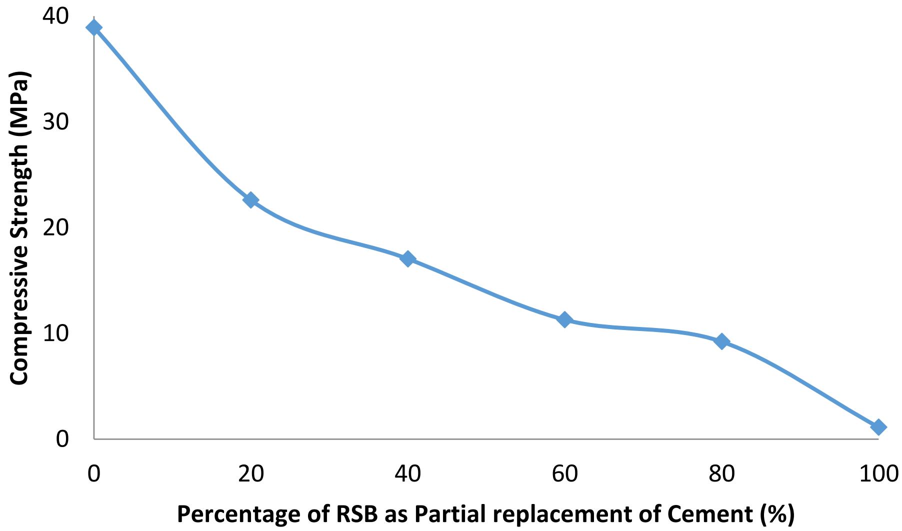

Figure 4. *Compressive strength of mortar with RSB as partial replacement of cement*

The test results obtained compressive strength values at the percentage of cement 0%, 20%, 40%, 60%, 80% and 100%. The results obtained were 1.13, 9.22, 11.29, 17.05, 22.60 and 38.93 Mpa, respectively. If you look at the graph above, the compressive strength value has increased. This shows that the higher the percentage of cement, the higher the compressive strength value produced (Sembiring, 2017). This increase was due to the SiO_2 content in the cement, which helps the binding of CaO and fills the cavities formed by evaporating water so that the resulting mortars are denser and stronger. In addition to the amount of cement, several factors affect the compressive strength value, namely the water-cement factor and density, the concrete's age and the aggregate's nature (Jurczak et al., 2021).

CONCLUSION

From the results of the analysis carried out in this final project, several conclusions were as follows: (1) The content of chemical compounds in dregs and grits that can be used as raw materials to manufacture mortars contained several elements: CaO at 34% and 50% followed by Na_2O at 5% and 7%. Then, Al2O3 was 6% and 1% for other compounds, SiO_2 was 2% and Fe_2O_3 was 2% and 0.5%, respectively. (2) The addition of cement to the manufacture of grits and dregs in the experiment of making mortars resulted in the answer that dregs and grits mixed with cement will greatly affect the characteristics of mortars. The percentage of cement mixture is directly proportional to the density test and compressive strength, while the porosity and water absorption are inversely proportional. (3) Paving block products produced from raw materials of dregs, grits and cement obtained the results of compressive strength testing, mortars with cement percentages of 20%, 40%, 60%, 80% and 100% met the classification of SNI-03-0691-1996 with different quality. Meanwhile, the results of the water absorption test ranged from 14.3% - 31.35%, meaning that the mortars produced did not meet SNI, so it is highly recommended to use mortars in areas or parks that are not submerged in water.

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