

## Utilization of Grits and Dreg Wastes for Ceramics Raw Material

### *Pemanfaatan Limbah Grits dan Dreg sebagai Bahan Baku Pembuatan Keramik*

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#### **Kata Kunci:**

Dreg; grits; keramik;  
ICP; sifat mekanik.

#### **Abstrak**

Penelitian ini memanfaatkan limbah dreg dan grits dari industri pulp dengan penambahan kaolin menjadi bahan baku keramik untuk mendapatkan keramik yang mempunyai sifat fisis dan mekanik yang baik. Dreg dan grits dipreparasi dengan cara dikeringkan pada oven 120°C selama 5 jam lalu dihancurkan dengan crusher dan diayak menggunakan ayakan ukuran 200 mesh, kemudian dikarakterisasi *Inductively Coupled Plasma* (ICP). Sampel keramik dicetak menggunakan *Compressive Gauge Testing* dengan kekuatan 15 kN selama 60 detik. Persentase kaolin divariasikan antara 20-80% diikuti pengeringan 48 jam di temperatur ruangan. Sampel keramik dibakar di furnace suhu bertahap hingga 1000°C dengan waktu tunggu selama 30 menit. Keramik di karakterisasi sifat fisisnya yaitu densitas, porositas, susut bakar, dan daya serap air sedangkan untuk sifat mekanik dilakukan pengujian kuat tekan. Hasil uji ICP berupa komposisi kandungan senyawa kimia pada dreg dan grit yaitu kandungan yang paling tinggi adalah CaO yaitu 36% dan 50%. Dosis optimum keramik yang dihasilkan pada penelitian ini adalah dengan komposisi kaolin 80%. Persentase campuran kaolin berbanding lurus dengan kuat tekan, densitas dan susut bakar, sedangkan untuk porositas dan daya serap air berbanding terbalik, diperoleh karakterisasi keramik dengan kekuatan tekan berkisar antara 4.48MPa- 11.98MPa.

#### **Keywords:**

Dreg; grits; ceramic;  
ICP; mechanical  
properties.

#### **Abstract**

*This research utilizes dreg and grits wastes from pulp industry by addition of kaolin as raw material in producing ceramics that have good physical and mechanical properties. Dreg and grits were prepared by drying in an oven at 120°C for 5 hours, then crushed and sieved using a 200 mesh sieve, then characterized by means of Inductively Coupled Plasma (ICP). Ceramic samples were printed using Compressive Gauge Testing with a strength of 15 kN for 60 seconds. The kaolin percentage was varied between 20-80% followed by drying for 48 hours at room temperature. The ceramic samples were burned in a stepwise furnace temperature of up to 1000°C with a holding time of 30 minutes. Their physical properties, i.e. density, porosity, fuel loss, and water absorption, were characterized, and their compressive strength was investigated. The ICP test showed that the highest chemical content in the dreg and grit is CaO, at 36% and 50%. The optimum dose of ceramics produced in this study is the composition of 80% kaolin. The percentage of kaolin mixture is directly proportional to the compressive strength, density and fuel shrinkage, while the porosity and water absorption are inversely proportional. The ceramic characterization was obtained with compressive strength ranging from 4.48 MPa to 11.98 MPa.*

## INTRODUCTION

Pulp and Paper Mills produce organic and inorganic residues with highly variable properties during the kraft process, such as dreg green liquor, grits slaker, biomass ash and lime sludge (Martínez-Lage et al., 2016). It was recorded that Indonesia's paper production capacity in 2017 was 12.98 million tons per year, while the pulp production capacity was 10.43 million tons per year, an increase from the previous year which was only 7.93 million tons/year. Data obtained from Indonesian Pulp and Paper from January to March 2021, the recausticizing process produced 16,264 tons of dreg, and 4,191 tons for grits.

Landfill can produce negative ecological impacts such as soil contamination, odor emission and leaching of toxic compounds causing water and soil pollution. In addition, the hoarding of this land is a waste of natural resources, because this waste has a high potential to be reused in productive processes as high-quality alternative raw materials. New proposals for waste management are needed to minimize landfill disposal and generate economic and social benefits (Simão et al., 2018).

Due to the high  $\text{CaCO}_3$  content, the main sources of lime can be replaced by dregs and grits, especially in building materials (Quina & Pinheiro, 2020). Slaker grits are also applied to cement mortar (Saeli et al., 2019), a mixture of bituminous and cement clinker (Santos, V. R. dos et al., 2019). Composition of dreg green liquor contains sodium carbonate ( $\text{Na}_2\text{CO}_3$ ), calcium carbonate ( $\text{CaCO}_3$ ) and sodium sulfide ( $\text{Na}_2\text{S}$ ), as well as several other insoluble solids in small amounts containing silicon, iron, aluminum, magnesium, manganese, and sulfides (Martins et al., 2007). Slaker grit is produced in the slaker lime unit which converts calcium oxide ( $\text{CaO}$ ) to calcium hydroxide ( $\text{Ca}(\text{OH})_2$ ) in its recovery process. It consists mainly of  $\text{CaCO}_3$ , but residual amounts of  $\text{CaO}$ ,  $\text{Ca}(\text{OH})_2$  and  $\text{Na}_2\text{CO}_3$  may also present (Machado et al., 2007). Metal ions such as barium, chromium, copper, nickel, and zinc can be found in very small amounts (Monte et al., 2009).

To improve the quality of ceramic products, it is necessary to engineer mechanical properties so as to minimize defects or damage during shipping as well as the durability of ceramic products such as ceramic compressive strength, internal structure in the form of porosity, ceramic density and also ceramic composition. Chemical composition of clay minerals-based ceramic, which is kaolinite, illite, and smectite (Ion et al., 2016). Pressureless sintering is one of the methods to produce kaolin-based ceramic (Amin et al., 2008). Research on ceramic compressive strength and porosity testing with different materials is still rarely done. Compressive strength and porosity tests can be used to determine ceramic quality (Setiawan et al., 2017).

This research was conducted in order to be able to utilize green liquor dregs and grits slaker as alternative raw materials so that they are not only dumped in landfills which can have an environmental impact but can contribute to greater sustainability by utilizing grits and dreg wastes with the addition of kaolin as raw material for ceramics.

## MATERIALS AND METHOD

The tools used in this research and testing include analytical scales, caliper, 200 mesh sieve, 50mm×50mm×50mm iron cube mold in accordance to ASTM C109, Testing gauge (5kN./DIV.), furnace, oven, microwave digestion, vessel, flask 100 ml, 250 ml flask, 250 ml erlenmeyer, 250 ml beaker, thermos scientific, filter paper no 42 and 93, crusher, mortar, heating flowing cooler, desiccator, krusibel, 500 ml measuring cup, digital compression machine (Kn), and Inductively Coupled Plasma (ICP). Materials used in this research include dreg, grits, kaolin, pure chemical  $\text{HNO}_3$ ,  $\text{HBF}_4$ , demineralized water, EDTA 0.05 M,  $(\text{NH}_4)_2\text{CO}_3$  10%, indicator MO (Methyl Orange), 0.5% HCl, 1:1 HCl, 1:9 HCl, 10% hydroxylamine chloride, triethanolamine 15%, KOH 20% and calcium indicator.

The initial stage is preparation by checking dry weight, combustible material, ash content, LOI (Loss Of Ignition). Then the dregs and grits were prepared by drying in an oven at 120°C for 5 hours then crushed with a crusher and sieved using a 200 mesh sieve, then characterized by metal content and chemical composition (ICP), checking the content of CaO and Total Titratable Alkali (TTA).

The formation of ceramic samples using the dry pressing method, which is the formation of a fine powder containing a small amount of water or the addition of organic matter by applying pressure that is limited by the mold into a strong solid product. In this method, the powder material is mixed with 15wt% of water. The casting of the test sample is carried out with a powder press which has been mixed evenly poured into a cube-shaped 50×50×50mm<sup>3</sup> mold then pressed with a load of 15 kN for 60 seconds using Compressive Gauge Testing, until solid according to Table 1. The density and porosity test were calculated conformed to ASTM C373, while the firing shrinkage of ceramic were measured according to ASTM C326.

**Table 1.** Mix Design of Dreg and Grits based Ceramic

Sample No.	Kaolin:Dreg:Grits (%)
1	20:40:40
2	30:35:35
3	40:30:30
4	50:25:25
5	60:20:20
6	70:15:15
7	80:20:20

## RESULTS AND DISCUSSION

### *Inductively Coupled Plasma Test Analysis*

The results of the raw materials testing have been presented in Table 2.

**Table 2.** ICP result of Dreg and Grits

Sample No.	Dreg (%)	Grits (%)
CaO	36.423	51.168
B <sub>2</sub> O <sub>3</sub>	14.903	17.872
Na <sub>2</sub> O	6.737	9.030
MgO	0.396	4.462
SiO <sub>2</sub>	2.515	3.379
Al <sub>2</sub> O <sub>3</sub>	1.644	3.199
Fe <sub>2</sub> O <sub>3</sub>	0.368	1.815
SrO	0.012	0.063
MnO	0.004	0.233
TTA	2.436	5.121

Testing of raw materials was carried out to determine the content of it (Sukojo et al., 2019). This content will affect the process and the resulting product. The results of the ICP test, CaO test, and TTA were in the form of the composition of chemical compounds in dreg and grits, namely the highest content was CaO, namely 36% and 50%, followed by B<sub>2</sub>O<sub>3</sub> 14% and 17%, Na<sub>2</sub>O 6% and 9%, MgO 0.3% and 4%, then the average for SiO<sub>2</sub>, Al<sub>2</sub>O<sub>3</sub>, Fe<sub>2</sub>O<sub>3</sub>, SrO, MnO were below 2% while TTA value of dreg and grits are 2% and 5%, respectively.

One of the functions of CaO in the manufacture of ceramics is as a flux or non-plastic melter which functions as a fuse to bind the filler or frame at high temperatures to form a ceramic bond. The use of this mineral is quite important for the manufacture of ceramics (Zhou et.al, 2017). The CaO content in dregs and grits is

very high, namely 36% and 50%, which will affect the strength and porosity of the resulting ceramic when too much of the weight of the material is used. Ca content in ceramic samples which melt at high temperatures and leave pore marks causes a decrease in compressive strength due to high porosity in hardened sample. The kaolin in this study cannot act as a binder and filler in ceramics when dreg and grits contents are high.

*Density measurement results*

Density is defined as mass per unit volume. The results of the raw materials testing have been presented in Table 3.

**Table 3.** Density measurement result

Sample No.	Average Density (g/cm <sup>3</sup> )
1	1.387
2	1.400
3	1.489
4	1.528
5	1.650
6	1.686
7	1.707

The increase in sintering temperature also affects the density of resulting sample, the silica content in the ceramic constituent material has undergone a glass phase and binds other elements contained in the ceramic body, so that the bonds formed between the ceramic constituent materials are getting stronger. A strong bond shows a high density between particles, so the density is higher. The resulting ceramic density with 20-80% kaolin percentage are ranged from 1.3-1.7 g/cm<sup>3</sup>. Of all these samples, the highest density value achieved at the 80% kaolin sample, with a density value of 1.6 g/cm<sup>3</sup>.

The sample were dried for 2x24 hours at room temperature then were burned in a stepwise furnace firing temperature of up to 1000°C with a holding time of 30 minutes. The density, porosity, burn-in shrinkage, water absorption, and compressive strength were evaluated. According to Mirna et al (2017), the value implied in the manufacture of ceramics is (1.6-2.0) g/cm<sup>3</sup>. The density value is in accordance with the density value produced in this study, with values above 1.6 gr/cm<sup>3</sup> in 60-80% kaolin content samples. The values can be said to have met the requirements used in ceramics manufacture. According to Xiao et.al (2020), the low density of ceramics is caused by the inhomogeneity of grain size and mixing of samples that are less homogeneous. This shows that the low density of ceramics which has the kaolin content of 20-50% were caused by the inhomogeneity of the grain size and the lack of homogeneity of the sample size when mixing the ceramic sample materials.

*Firing shrinkage measurement results*

Burning loss is a change in the dimensions or volume of the material that has been burned. One of the parameters that indicate the occurrence of the sintering process is shrunk due to changes in the microstructure (grain or grain boundaries). The results of the Burn Loss Test on Ceramics have been presented on Table 4.

**Table 4.** Firing shrinkage measurement result

Sample No.	Firing Shrinkage (%)
1	0.050
2	1.675
3	1.905
4	2.215
5	2.580
6	2.925
7	3.290

The highest firing shrinkage value was in the composition of the 80% kaolin sample, with a burn loss value of 3.290%. The composition consists of 80% of kaolin, 10% of dreg and 10% grits. Meanwhile, the lowest shrinkage achieved in the with 20% kaolin content, 40% of dreg and 40% of grits. It has the shrinkage value of 0.050%, where almost no shrinkage. Smaller grain of the material resulted in better sintering process takes place because the energy given to the densification or compaction process is greater where the grains were fused, then a very high-volume diffusion rate occurs so that the weight loss that occurs is greater and a higher density is obtained. From the calculation of the ceramic burn loss, it shows a close relationship with the ceramic density. This indicates that the shrinkage of ceramic deeply influenced by density of material. Higher particle density means higher shrinkage value.

#### *Porosity measurement results*

Porosity in a ceramic material is expressed in % voids or volume fraction of a cavity in the material, and the open pore volume to the overall volume of the object. The porosity test results of the Porosity Test of Ceramics were presented in Table 5. The obtained porosity values ranging from 24.76% - 50.18%. The lowest porosity achieved at 80% kaolin content, which resulted in 24.76%.

**Table 5.** Porosity measurement result

Sample No.	Porosity (%)
1	50.18
2	48.33
3	46.87
4	44.37
5	39.21
6	32.97
7	24.76

The percentage of kaolin content is inversely proportional to the porosity. When the percentage of kaolin is increased, the size of porosities is smaller, and vice versa. This high porosity is due to the Ca content which melts during the combustion process at high temperatures develops new porosities, which increases the

oxygen content in it. The Ca content in dreg and grits is very dominant so that it can increase the porosity of the ceramic. Judging from the composition of solid waste, dreg and grits are materials that are rich in CaO, so that when burning the sample, the composition of the waste is quite large while the SiO<sub>2</sub> content is small. A small percentage of kaolin can result in kaolin not being able to act as a binder. In addition to the composition, the combustion temperature also affects the formation of ceramics. The sintering temperature is not reached and the combustion time is too short prevents the sintering process from happening optimally. The size of the porosity value is influenced by the printing process (Setiawan et al., 2017). At the time of printing the pressure in this study was only 15 kN and the holding time was 60 seconds, if the compressive strength during printing is increased it can increase the density of the ceramic to reduce the porosity of the ceramic.

*Water absorption test results*

Water absorption shows the percentage of water absorption in ceramics after being soaked for 24 hours. Table 6 shows the water absorption test results of ceramic made with dreg and grits.

**Table 6.** Water absorption result

Sample No.	Water absorption (%)
1	36.01
2	34.36
3	31.32
4	28.89
5	23.66
6	19.46
7	14.43

The results obtained for the water absorption have different values. According to SNI standards, the maximum value of water absorption value is 20%. The sample with kaolin percentage of 70% and 80% meets the SNI standard with a value of 19.46% and 14.43%. To get a water absorption value of less than 20%, it is necessary to carry out further research with a longer sintering time of 2 hours (Mirna et al., 2017). The water absorption value obtained from sample with the percentage of kaolin 20-60% did not meet the standards set by SNI. This is due to the large number of pores formed in the sample so that water can easily enter the pores, this is because during the ceramic formation process, it has not yet reached the ceramic grain limit while the sintering time is only 30 minutes, so it has not reached the recommended limit, namely for 2 hours.

*Compressive test results*

The ceramic compressive strength test is carried out to see the strength or ability of a material to accept the pressure applied to the compression test equipment. Compressive strength test equipment in general has a pair of stepping plates (bottom) and a pressure tool (top) which are used to determine how much force (load) is needed to cause the test object to fracture (the material changes shape) when loaded with a certain compressive force produced. by a press machine. Table 7 shows the compressive strength value of dreg and grits-based ceramic.

**Table 7.** Compressive test result

Sample No.	Compressive Strength (MPa)
1	4.48
2	5.92
3	7.10
4	9.62
5	10.44
6	11.42
7	11.98

The compressive strength of 100% kaolin burned at 1000°C for 3 hours is 19.317 MPa. When compared to the ceramic composition that has been mixed with burnt dreg and grits with a temperature of 1000°C for 30 minutes, there is a difference of  $\pm 7.3$  MPa. The Ca content in ceramic samples which melts at high temperatures and leaves pore marks, thereby reducing the value of compressive strength. This decrease was caused by the content of lime in the sample with the addition of dregs and grits which burned at high temperature combustion and appearance of new pores.

The more additions of dregs and grits this will decrease the compressive strength value. The compressive strength value experienced a significant increase in the 40% kaolin composition, from 7.10 MPa to 9.62 MPa at 50% kaolin percentage, this was due to the influence of the combustion temperature which had not yet reached the sintering temperature, where at a temperature of 1000°C ceramics with a kaolin percentage below 40% has not undergone good sintering so that it does not experience large shrinkage and low compressive strength. Ceramics with a composition of 20-50% meets to SNI 16-2094-2000 class 50 solid red bricks, while ceramics with a kaolin composition of 60-80% meets to SNI 16-2094-2000 class 100 solid red bricks. The compressive strength of kaolin ceramics increases with increasing sintering temperature and compaction pressure. Because the temperature used in this study is 1000°C and does not vary the sintering temperature, it is not possible to ensure a good optimization of the sintering temperature for ceramics with kaolin, dreg and grits as raw materials. At the time of printing the pressure in this study was only 15 kN and the waiting time was 60 seconds. The percentage of waste is inversely proportional to the amount of pressure, that is, the more waste that is mixed, the lower the compressive strength, while the percentage of kaolin is directly proportional to the magnitude of the compressive strength, i.e., the more kaolin, the higher the compressive strength. Replacing lime with waste grits in the manufacture of wall tiles can be carried out in a range of up to 15% by weight, enabling the production of single fast-firing wall tiles (Group BIII – ISO 13006 standard) for a wide firing temperature range (1100-1180°C), with the addition of 15% quartz and 70% kaolin (Siqueira & Hollanda, 2018). They said that the addition of grits can be used as single fast-firing wall tiles. Grit waste can be used as ceramic building material. The use of higher concentrations above 20% by weight of the material is not recommended due to increased water absorption and decreased mechanical strength of the resulting ceramic. The compressive strength produced in this study is relatively small, the highest compressive strength obtained is 11.98 MPa with a composition of 10% dreg, 10% grits and 80% kaolin. The lowest compressive strength is 4.48 MPa with 40% dreg composition, grits 40% and kaolin 20%. The optimum dose of ceramics produced in this study was with a kaolin composition of 80% because it complied with SNI ceramic standards, namely density at 1.6-2.0 gr/cm<sup>3</sup>, burn loss below 12%, water absorption below 20%, and inlet compressive strength meets the minimum standard criteria of SNI 16-2094-2000 solid red brick class 100. To increase the compressive strength, highly amorphous silica content material can be added. Some of them are corn cob and bagasse ash, which contains 23% SiO<sub>2</sub> (Harmaji, 2019).



## CONCLUSION

Based on the analysis and discussion of the research data above, it can be concluded that the highest composition of chemical compounds in dregs and grits was CaO at 36% and 50%, followed by B<sub>2</sub>O<sub>3</sub> at 14% and 17%, Na<sub>2</sub>O at 6% and 9%, MgO at 0.3% and 4%, then for SiO<sub>2</sub>, Al<sub>2</sub>O<sub>3</sub>, Fe<sub>2</sub>O<sub>3</sub>, SrO, MnO on average below 2%. The percentage composition of dregs and grits mixed with kaolin greatly influences the characteristics of ceramics. Waste mixed with 30%-80% kaolin percentage can be used as a construction ceramic material with certain characteristics. The optimum dose of ceramic produced in this study was 80% kaolin composition because it met SNI ceramic standards, namely density at 1.6-2.0 gr/cm<sup>3</sup>, burn loss below 12%, water absorption below 20%, and inlet compressive strength. to class to SNI 16-2094-2000 solid red brick class 100. The percentage of kaolin mixture is directly proportional to the compressive strength, density and fuel shrinkage, while the porosity and water absorption are inversely proportional. Characterization of ceramics obtained with compressive strength ranging from 4.48 MPa-11.98 MPa, porosity 24.76%-50.18%, water absorption 14.4%-36.01%, burn loss 0.050%-3.290% and density 1.387g/cm<sup>3</sup>-1.707g/cm<sup>3</sup>.

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