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Physical and Mechanical Properties of Mortar Mixed with Paper Mill Waste

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ABSTRACT

The industrial and construction sectors are experiencing continuous growth each year. Concrete, which is used as the main material in construction, is composed of cement, aggregate and water. Research on mortar and aggregate is carried out to create concrete according to the required specifications of certain buildings. However, 8% of global carbon dioxide emissions come from the cement manufacturing process which is one of the main materials in mortar. In the industrial sector, the paper industry is one of the top producers of waste, approximately 48 tonnes for every 100 tonnes of pulp produced. The main wastes of this industry include slaker grits, dregs, lime mud and fly ash which only end up in landfill. To address this problem, this research investigates the modification of mortar mix materials by utilising paper mill waste. Specifically, fly ash and grits are used as substitute for cement and fine aggregate, respectively. Results indicate that the density value of concrete decreased with the increase of fly ash mixture, and the compressive strength value of mortar with varying additions of 0%, 10%, 20%, 30%, 40% and 50% fly ash reached 7.42, 6.68, 6.18, 4.97, 3.93 and 2.97 MPa. The decrease in density and compressive strength with increasing fly ash content can be attributed to the lower adhesive power of waste compared to cement owing to its lower lime oxide content. The value of compressive strength samples 1, 2 and 3 belong to the N type, which can be used as load-bearing walls, while samples 4, 5 and 6 fall into the O type, which can be used as non-load-bearing and decorative walls in accordance with SNI 03-6882-2014 standards.

1. Introduction

In recent years, the growth of the industrial and construction sectors has paralleled the rising needs of society. In construction, concrete is commonly used due to its accessibility compared to other materials, ability to withstand compressive forces, cost-effectiveness and ease of use for various project. By definition, concrete is a mixture of Portland cement or other hydraulic cement, fine aggregate, coarse aggregate and water, with or without additional ingredients (admixture) [1], while mortar is a mixture only of Portland cement, fine aggregate and water.

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The development and innovations of concrete or mortar are essential to align needs without causing significant damage to the environment. However, according to the Chatham House Research Institute, 8% of carbon dioxide (CO_2) emissions come from the cement industry [2], a figure that will continue to increase along with rising construction needs [3]. Other materials, such as coarse aggregate and fine aggregate/sand in large production, can also threaten environmental stability because they are non-renewable.

The paper industry is one of the largest industries in the world. According to data from the Ministry of Industry of the Republic of Indonesia in 2021, the national pulp industry has a total installed capacity of 11,8 million tonnes per year [4]. Waste products from this industry, approximately 48 tonnes for every 100 tonnes of pulp produced, include solid waste consisting of dregs, bio sludge and grits from wastewater treatment plants and fly ash generated from combustion in substantial quantities [5]. In accordance with Government Regulation No. 101 of 2014, waste from the paper and ink processing industries is classified as hazardous and toxic material (B3), which, if buried in the ground, will cause environmental pollution. Currently, there are many ways to recycle paper waste, such as processing waste into recycled paper or crafts. Unfortunately, most of paper mill waste is disposed in landfills [6]. However, with many communities rejecting landfills located around settlements and environmental agencies demanding improved waste management processes, alternatives are needed that are consistent with environmental needs at rational costs.

To overcome the issue of sustainable development in the industry, modification research has been done that utilizes waste materials as substitutes for natural materials. In a research journal written by Capela *et al.,* [7] regarding concrete and mortar mixed with paper industry waste in the form of biomass ash (fly ash) and dregs, fly ash and dregs were used as cement substitutes. The research results showed that the compressive strength value of mortar mixed with biomass ash was relatively greater than the reference mortar without waste material substitution. Similarly for concrete, the compressive strength value increases by up to 8% with fly ash substitution variations of 10% of the cement weight.

Saeli *et al.,* [8] evaluated the innovation of waste grits from the paper industry as aggregate by making substitution variations of mortar based on sand, grits and a combination of both. After testing, all mixtures had a uniaxial compressive strength of > 10 MPa, which meets the minimum requirements for use in structural buildings. A mixture of grits and sand in mortar shows a higher compressive strength value compared to the compressive strength of mortar with sand only. Mo *et al.,* [9] conducted research of the effects of biomass ash and dregs on mortars. The study showed that by replacing 10% of the cement in the mortar cube with biomass ash, the highest compressive strength of 54.5 MPa could be achieved. This proved that the replacement of biomass ash can enhancing its compressive strength.

From the current study, researcher created proportions for mortar mixed with paper mill waste. Fly ash and grits were used respectively as a partial substitute for cement and a substitute for fine aggregate. The addition of fly ash and grits components as basic ingredients for mortar is expected to improve the mechanical properties of the mortar. The additional materials were obtained from PT OKI Pulp and Paper located in Palembang, South Sumatra. Based on the description above, it is important to research on the engineering performance of mortar cubes with respect to their admixtures to come out with suitable mix ratios that can be applied in the construction industry [10]. Therefore, this study was carried out regarding the physical and mechanical properties of mortar with a mixture of pulp and paper waste.

From the background presented regarding the problem of paper industry waste and innovation in the construction sector, a problem formulation was obtained: how does the addition of pulp and paper waste affect the physical and mechanical properties of mortar. Accordingly, the aim of this research was to determine the physical and mechanical properties of mortar mixed with paper mill waste.

2. Methodology

2.1 Materials and Methodology

According to SNI 03-6882-2014, mortar is defined as a mixture of materials in the form of fine aggregate, adhesive (portland cement) and water with a certain composition. Based on the binding material, mortar is grouped into several types, namely mud mortar, cement mortar, lime mortar and special mortar [11]. These types of mortar used accordingly to particular needs of the concrete.

In ASTM C 270 and SNI 03-6882-2014, mortar is classified into four types, M, S, N and O, based on the proportion of materials by volume (proportion specifications) and mortar property specifications. It is necessary to know the classification of mortar based on its compressive strength as a reference for determining the minimum compressive strength of modified mortar to be made. These classifications can be found in Table 1 [12].

The mix proportion and amount of mortar that will be made in this study follows ASTM C 109 that specifies the compressive strength testing procedure for hydraulic cement mortar with 50 mm cube specimens. The standard determines the proportion of material mixtures for making mortar in multiples of 6, 9 and 12 in one mixing time. The mixture proportions for one stirring are provided in Table 2 [13].

Table 1
Property specification requirements (SNI 03-6882-2014)

Mortar	Type	Average compressive strength at 28 days, MPa (psi)	Aggregate ratio
Cement mortar	M	17.2 (2500)	Not less than 2 1/4 and not more than 3 1/2 the amount of the separate volume of cementitious material
	S	12.4 (1800)	
	N	5.2 (750)	
	O	2.4 (350)	

Table 2
Mix proportions in one mortar mixing

Number of specimens	6	9	12
Cement (gr)	500	740	1060
Sand (gr)	1375	2035	2915
Water (mL)	242	359	514

2.2 Mortar Composition

In general, mortar consists of cement, fine aggregate and water mixed until homogeneous. The ingredients that make up mortar are as follows:

i. Cement: In mortar, cement acts as a binding agent between fine aggregates. Cement is composed of lime oxide, which acts as a binder, silica oxide (SO_2) and alumina oxide (Al_2O_3), which function as fillers, and iron oxide (Fe_2O_3), which serves as a stabilising agent. The cement used in this study is type I PCC (Portland Composite Cement). This cement has a relatively lower heat of hydration so the results do not crack easily.
ii. Fine aggregate: In mortar, cement acts as a binding agent between fine aggregates. Cement is composed of lime oxide, which acts as a binder, silica oxide (SO_2) and alumina oxide (Al_2O_3), which function as fillers, and iron oxide (Fe_2O_3), which serves as a stabilising

agent. The cement used in this study is type I PCC (Portland Composite Cement). This cement has a relatively lower heat of hydration so the results do not crack easily.

iii. Water: In mortar mixtures, water functions as a binder between aggregate particles to stimulate the mortar mixing process and support the ease of working with the concrete. The composition of the water must be adjusted to the amount of cement or what is usually called the water per cement ratio (w/c). The more water in the concrete mixture, the easier it is to work with.

iv. Admixture: Admixture is an additional material that is mixed into concrete according to needs. Concrete admixture consists of types A, B, C, D, E, F and G.

2.3 Paper Mill Waste

According the data from The Ministry of Industry of the Republic of Indonesia, paper and pulp industry produces around 48 tonnes of waste for every 100 tonnes of pulp produced. The main wastes from this process are dregs, grits and lime mud. Fly ash biomass, which is generated from combustion, is classified as non-B3 waste [14].

i. Fly ash: Fly ash is a by-product of combustion, typically in the form of fine and lightweight granules. It exhibits pozzolanic properties due to its high silica and alumina content. Substituting as much as 10% of fly ash can increase the compressive strength of mortar, but a higher percentage diminishes this quality [15]. This improvement in compressive strength is caused by the reaction that occurs in fly ash and lime during the hydration process between fly ash and water, which forms calcium silicate compounds.

ii. Grits: Slaker grits, also known simply as grits, are granular alkaline waste produced during the wood degradation and recausticising process, namely the re-purification of inorganic chemicals resulting from combustion in the recovery boiler. Grits are one of the main wastes produced from pulp and paper production. They contain calcite ($CaCO_3$), making them suitable for reuse, especially in construction [16]. Grits have a particle size distribution ranging from 1 mm to 12.5 mm, with a small percentage of dust, and can be used as aggregate. Before use, grits must be dried in an oven at a temperature of 60°C or dried naturally at the grinding location.

2.4 Microstructure Testing of Material

i. X-Ray fluorescence: X-Ray fluorescence (XRF) is a tool that analyses the chemical composition and percentage of elements in a sample of substances using spectroscopic methods. This method analyses matter and its attributes based on the light or sound emitted and then absorbed or reflected by the material.

ii. X-Ray diffraction: X-Ray diffraction (XRD) is a method to analyse the composition of a compound or the crystalline phase of a compound. The XRD inspection system works by determining the different types of atomic structures in the crystal so that the reactivity of the material being tested is obtained.

iii. Scanning electron microscope: Scanning electron microscope (SEM) is used to observe the surface of a substance sample directly. SEM magnification ranges from 10 to 3,000,000x, with a depth of field between 0.4 and 4 mm and a resolution of 1-10 nm. The combination of these three SEM characteristics is the basis for determining the shape, size and structure of materials [17].

2.5 Mix Proportion

In this research, mix proportion was made based on research conducted by Martinez *et al.,* [5] which modifying the mix design by adding variations in the composition of the use of cement substituted by fly ash and grits as a substitute for fine aggregate. The test object used in accordance with ASTM C109-13 is a cube with dimensions of 5 x 5 x 5 cm^3. The mix proportion of the mixture used can be seen in Table 3.

Table 3
Mix proportion of the mortar

Name	Cement (gr)	Fly ash (gr)	Grits (gr)	Water (gr)
1	466.7	-	1400.0	233.3
2	420.0	46.7	1400.0	233.3
3	373.3	93.3	1400.0	233.3
4	326.7	140.0	1400.0	233.3
5	280	186.7	1400.0	233.3
6	233.3	233.3	1400.0	233.3

2.6 Concrete Treatment (Curing)

Curing is a treatment method to maintain concrete at its maximum strength. Concrete maintenance needs to be done when the concrete has entered its final setting time or after it has completely hardened. Curing can be carried out by soaking the concrete for 24 hours and wrapping it in plastic wrapping for 28 days until it is ready to be tested for compressive strength [18].

2.7 Concrete Testing

i. Slump flow test: Slump flow was measured according to ASTM C1437, to determine the consistency of mortar, a melt test (flow) method is carried out by measuring the average diameter of fresh mortar spread on a melting table, typically at a minimum of four points [19].
ii. Concrete density: Density is the ratio of an object's mass to its volume. Based on ASTM C39-05, concrete density testing is carried out at 28 days by weighing the concrete and dividing it by its volume.
iii. Compressive strength: The compressive strength of concrete represented by fc' is the maximum compressive stress obtained through standard test procedures [19]. In accordance with SNI 03-6825-2002, the method for testing the compressive strength of mortar is to apply a graduated load at a certain speed to a cube sample using a compressive strength testing machine until the mortar breaks. At the time of rupture, the mortar experiences maximum compressive force. Mortar strength can be calculated by the following formula:

$$\sigma m = \frac{P\ maks}{A} \tag{1}$$

Flowchart showing the research flow process for the study is depicted in Figure 1.

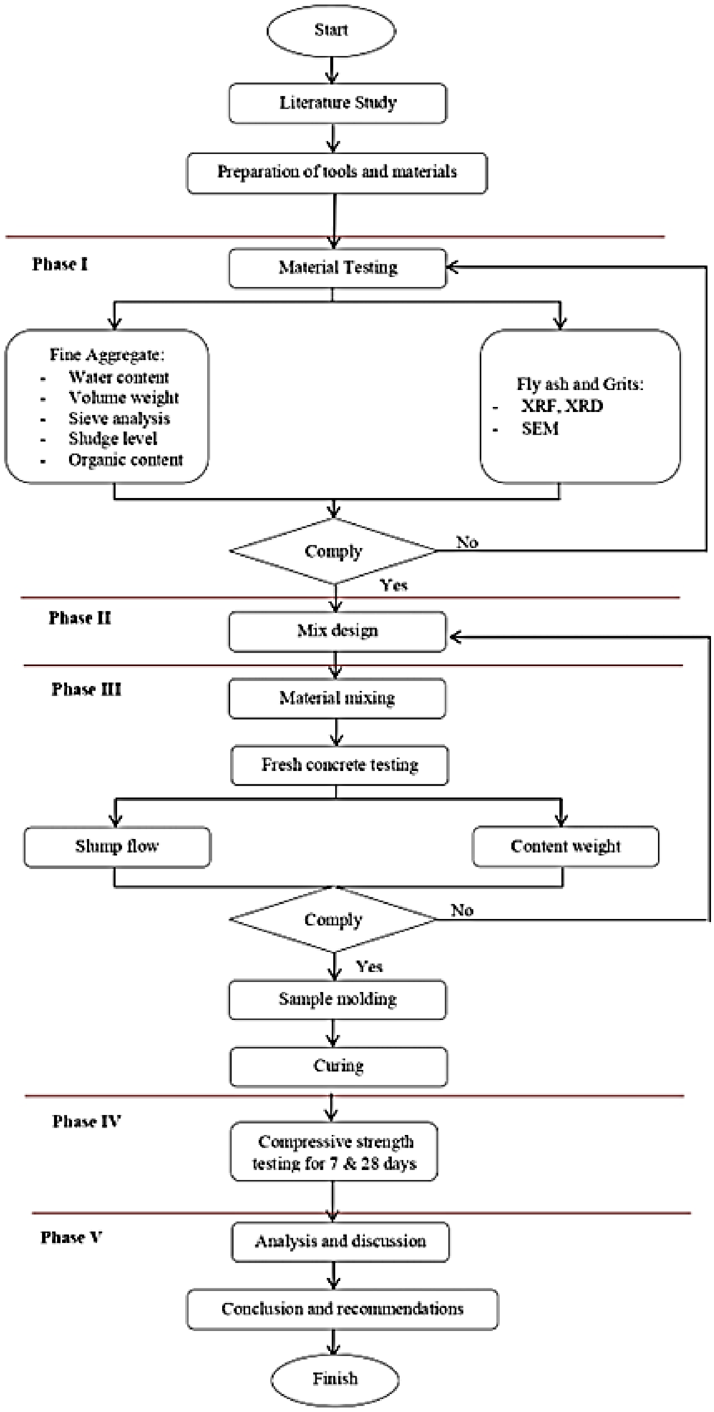


Fig. 1. Flowchart of research methodology

3. Results

3.1 Material Testing Result

Tests carried out on materials include testing of fine aggregate material or grits and testing of fly ash material. Material testing is carried out to determine the properties of grits and fly ash by comparing them to ASTM and SNI standards.

Table 4
Properties test results

Testing	Test result	Testing standards
Water content	12.52%	ASTM C 566
Volume weight	Solid= 1.13 kg/L Loose= 1.04 kg/L	ASTM C 29
Sieve analysis	No. 1	ASTM C 136
Sludge levels	17.41%	ASTM C 33
Organic content	No. 1	ASTM C 40

3.2 Water Content

The water content of fine aggregate in the form of grits is tested to determine the absorption capacity of the material. Based on the test results, the water content value that can be absorbed by grits is 12.52%, which is very high compared to existing standards. This is caused by weather factors and the condition of grits, which are paper production waste. Therefore, before their use as a mortar mixture, grits must first be made in a saturated dry condition or saturated dry surface (SDS).

3.3 Volume Weight

Volume weight testing aims to determine the weight of an aggregate per unit volume. Based on calculations from the volume weight test data, the volume weight value in the solid state was 1.133 Kg/L, and the volume weight of sand in the loose state was 1.044 Kg/L. The volume weight value is obtained by dividing the weight of the test object (W3) by the volume weight of the container (V1) in both solid and loose states. The volume weight in the solid condition is greater than in the loose condition because puncture occurs, reducing the sizes of the voids or pores between the aggregates and increasing the capacity of the container.

3.4 Sieve Analysis

Sieve analysis tests were carried out on grits material using a sieve in sequence based on ASTM C 136 standards to determine the size distribution of aggregate particles (Table 5). Based on the cumulative percentage value of aggregate retained in the sieve analysis, a fineness modulus value of 3.1% was obtained. This value is in accordance with the ASTM C 33 standard, which requires a fineness modulus value for fine aggregate in the range of 2.3%-3.1%. The fine aggregate gradation graph in Figure 2 shows that the fine aggregate area is included in gradation number 1. This means that the type of fine aggregate used has a coarse grain gradation

Table 5
Sieve analysis (ASTM C 136)

Filter size (mm)	Suspended weight (kg)	Retained amount (kg)	Total percentage (%)	
			Stuck	Past
4.75	0	0	0	100
2.36	0.058	0.058	11.69	88.31
1.18	0.144	0.202	40.73	59.27
0.60	0.154	0.356	71.77	28.23
0.30	0.100	0.456	91.94	8.06
0.15	0.040	0.496	100	0
Pan	0	0.496	100	0

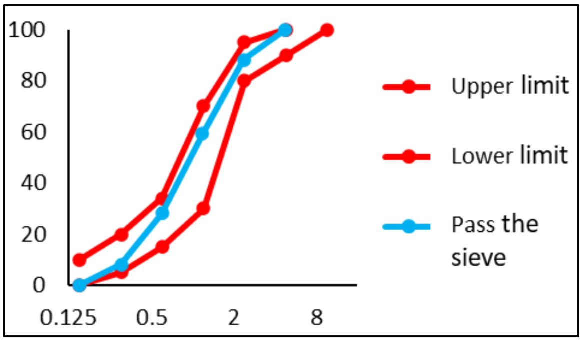


Fig. 2. Gradation filter analysis 1

3.5 Sludge Levels

Fine aggregate that is suitable for use as a concrete mixture must have a mud content of less than 5% in accordance with ASTM C 33 standards. Based on the test results, the mud content in grits was 17.14%. This means that before being used as a concrete mixture, the grits must be washed first so that they do not interfere with the bonding process between the materials when mixing.

3.6 Organic Content

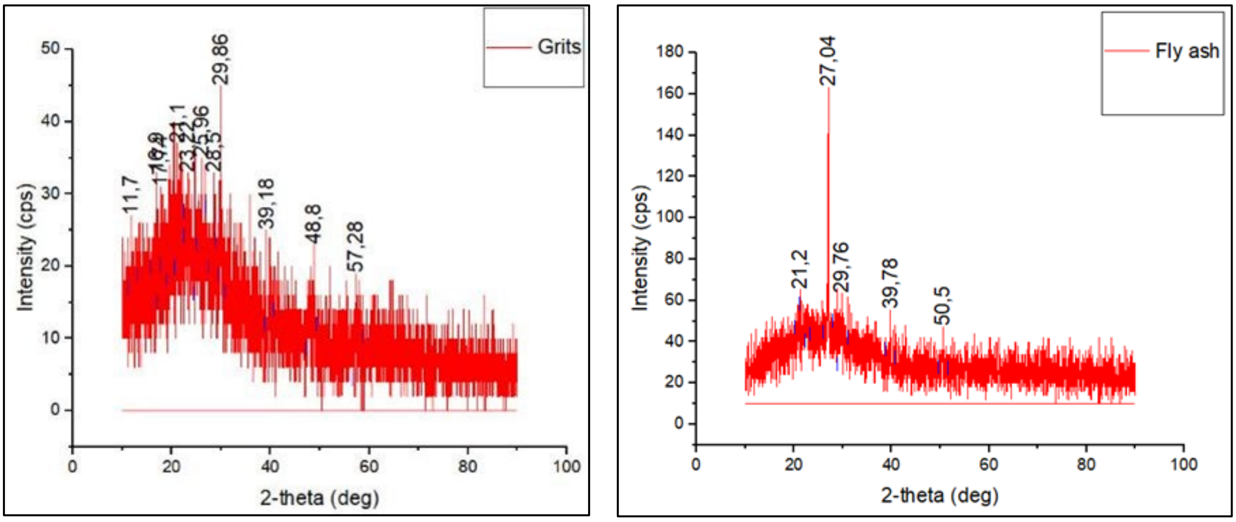
Fine aggregate is said to be suitable for use as a concrete mixture if the organic content has a maximum colour of No. 2 (ASTM C 40). High levels of organic substances will be detrimental to the quality of mortar or concrete because it slows down the cement binding process. Based on the test results, the organic content was obtained with colour number 1 on the organic plate, which means that the grits contain a small amount of organic substances and can be used in concrete mixtures.

3.7 X-Ray Diffraction

XRD examination was carried out at the Integrated Laboratory of Diponegoro University. Data from the test results were then processed using Origin 2021b software to obtain the crystal and amorphous phases in the material. The crystal phase was obtained by calculating the crystal area using the formula:

$$Crystal = \frac{\text{Total area crystal}}{\text{Total Area}} \times 100\% \quad (2)$$

The XRD test results on grits and fly ash can be seen in Figure 3. From the Figure 3 can be seen the Ipeak line, which indicates the surface is crystalline. The sharp peaks on the graph indicate the crystalline phase, which is unreactive. In XRD testing of the fly ash material, the crystalline percentage value was 18.15% and the amorphous percentage was 81.85%, as shown in Table 6. Meanwhile, for the grits material, the percentage of crystalline phase was 41.64% and the amorphous phase was 58.36%. Samples that have a high amorphous percentage are suitable for use as precursors because they are reactive. Meanwhile, samples with low amorphous and high crystalline values can be used as fillers.



(a) (b)

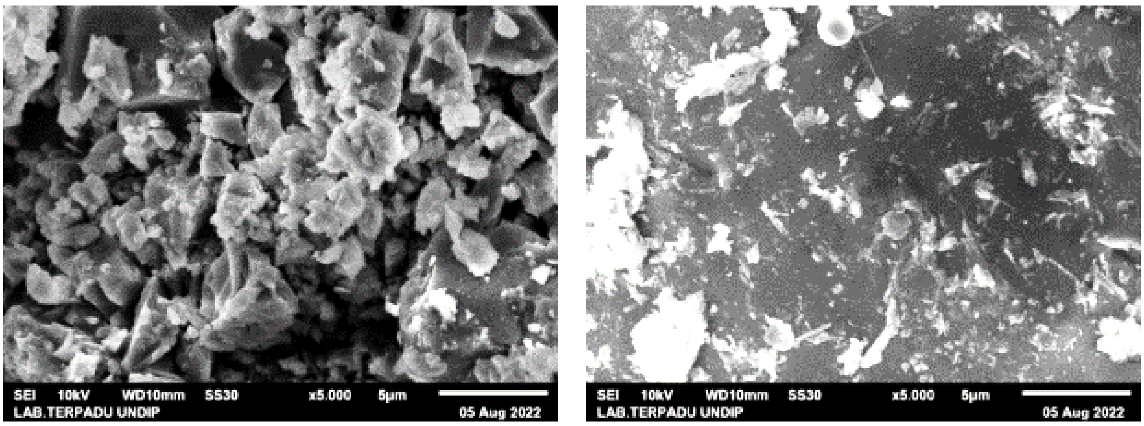
Fig. 3. XRD test results (a) Grits (b) Fly ash

Table 6
XRD test results on grits and fly ash

Sample	Crystal percentage (%)	Amorphous percentage (%)
Grits	41.64	58.36
Fly ash	18.15	81.85

3.8 Scanning Electron Microscope

Material testing using SEM aims to determine the shape, size and structure of the material according to the desired level of magnification. Figure 4(a) reveals the surface morphology of the grits at 5000x magnification. It shows that the shape of the grit particles is dense and sharp in accordance with the surface characteristics of fine aggregate. The rough surface allows the grit particles to absorb and increase the need for water, which can reduce the quality of the resulting concrete mortar. In Figure 4(b), the 5000x magnification of the fly ash does not show the fly ash surface clearly, such that the surface shape cannot be seen specifically.



(a) (b)

Fig. 4. SEM test results (a) Grits (b) Fly ash

3.9 X-Ray Fluorescence

XRF examination is carried out to analyse the elements contained in a material. Based on the XRF test in Table 7, the main content contained in fly ash is similar to the content in cement, namely 25.8% SiO_2, 9.17% Al_2O_3, 7% Fe_2O_3 and 8.88% CaO. The results of XRF testing on grits material are presented in Table 8. Based on the XRF test in Table 8, the main element contained in the grits is CaO at 40.9%. The CaO compound acts as a binder in the concrete mixture. Meanwhile, the 2.43% SiO_2 and Al_2O_3 compounds act as fillers.

Table 7
X-ray Fluorescence fly ash

No.	Test parameters	Unit	Test result
1	MgO	0.587	% heavy
2	Al_2O_3	9.17	% heavy
3	SiO_2	25.8	% heavy
4	P2O5	0.781	weight %
5	SO_3	1.49	weight %
6	Cl	0.114	weight %
7	K_2O	4.06	weight %
8	CaO	8.88	weight %
9	TiO2	0.693	weight %
10	MnO	0.164	weight %
11	Fe_2O_3	7.00	weight %
12	ZnO	0.0125	weight %
13	Rb_2O	0.0184	weight %
14	SrO	0.0435	weight %
15	Y_2O_3	0.0034	weight %
16	ZrO_2 _	0.0270	weight %
17	Balance	41,2	% heavy

Table 8
X-ray Fluorescence grits

No.	Test parameters	Unit	Test result
1	Na_2O	1.13	% heavy
2	Al_2O_3	0.106	% heavy
3	SiO_2	2.33	weight %
4	P_2O_5	0.353	weight %
5	SO_3	2.73	weight %
6	Cl	0.053	weight %
7	K_2O	0.365	weight %
8	CaO	40.9	weight %
9	Fe_2O_3	0.185	weight %
10	SrO	0.0129	% heavy
11	Balance	51.8	% heavy

3.10 Concrete Test Result

Tests carried out on concrete include slump flow testing, unit weight and compressive strength in accordance with ASTM and SNI standards.

3.11 Slump Flow Test

Mortar consistency/workability testing is carried out using a flow table to determine the diameter of the mortar on the melting table at a minimum of four different places to obtain the average diameter (d) of the mortar. From the research results, an average diameter consistency value of 20.5 cm was obtained in accordance with the consistency standard of 18-22 cm.

3.12 Concrete Density

Concrete density testing aims to determine the weight of concrete per unit volume. This test is carried out by weighing the test object and dividing it by the volume of the cube. The results of concrete density can be seen in Table 9. Based on the specific gravity test results presented in Figure 5, the highest density value (2.18 gr/cm^3) was found in sample 1, namely the sample without fly ash. Meanwhile, the lowest density value (1.72 gr/cm^3) was found in sample 6. The graph in Figure 5 shows that the addition of fly ash is inversely proportional to the density of the mortar produced. The more fly ash is used, the smaller the density of the mortar. This is caused by an inhomogeneous mixture between fine aggregate and fly ash paste.

Table 9
Density of concrete

Sample	Density (grams/cm ^{3})			Average
	A	B	C	
1	2.2	2.15	2.2	2.18
2	2.12	2.12	2.10	2.11
3	2.02	1.97	1.98	1.99
4	1.98	1.97	1.97	1.97
5	1.83	1.90	1.87	1.87
6	1.75	1.68	1.72	1.72

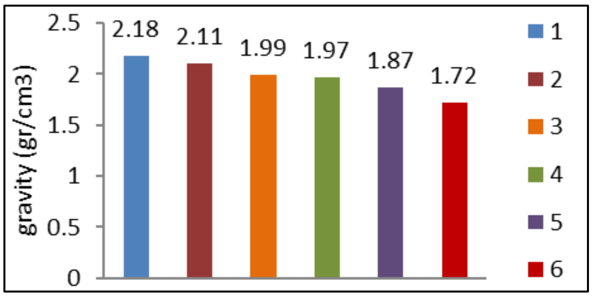


Fig. 5. Concrete density

3.13 Compressive Strength Test

Mortar compressive strength is the maximum force per unit area acting on the mortar test object (N/mm2 or MPa) (Table 10). Figure 6 shows that the addition of fly ash has an effect on the strength of the concrete. The more fly ash is added, the lower the compressive strength produced. The decrease in compressive strength with the addition of 10% fly ash reached 9.98% of the compressive strength of concrete without the addition of fly ash.

The increase in solid waste in the mixture is inversely proportional to the compressive strength of the resulting mortar. This is because during the casting process, the material is stirred in such a way that the solid waste grits and fly ash are mixed with the cement. The adhesion power of waste is smaller than the adhesion power of cement, causing an increase in fly ash to reduce the binding force of the aggregate grains. The results of the mortar compressive strength values produced in mortar with additional variations of fly ash at 0%, 10%, 20%, 30%, 40% and 50% are 7.42, 6.68, 6.18, 4.97, 3.93 and 2.79 MPa, respectively. These figures indicate that samples 1, 2 and 3 fall into type N and samples 4, 5 and 6 fall into type O according to the SNI 03-6882-2014 standard. Type N can be used in building parts, such as load-bearing walls located on the outside and inside. Meanwhile, type O mortar can be used in building parts such as non-load-bearing walls and non-load-bearing partitions and in exterior and interior decorations.

Table 10
Compressive strength of mortar

Sample	Compressive strength (kN)			Average	fc' (MPa)
	A	B	C		
1	18.23	17.69	17.53	17.82	7.42
2	16.31	15.46	16.33	16.03	6.68
3	15.27	14.31	14.93	14.84	6.18
4	11.41	12.53	11.87	11.94	4.97
5	9.64	9.73	8.93	9.43	3.93
6	6.19	6.74	7.14	6.69	2.79

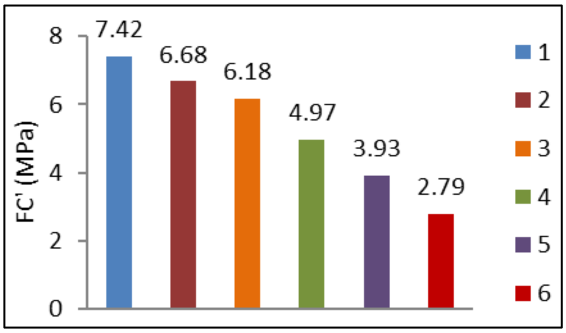


Fig. 6. Compressive strength of concrete

4. Conclusions

By using grits-based mortar as a substitute for fine aggregate and incorporating fly ash as a partial cement substitute, it can be concluded that the physical properties of the mortar samples meet the standards. For instance, when cast in a 5 x 5 x 5 cm^3 cube, the average mortar flow of 20.5 cm corresponds to the standard flow of 18-22 cm. Moreover, in the density test, there was a decrease in the density value of the concrete to 0.46 g/cm^3 or 21.1% of the density of mortar without additional fly ash. In the testing of mechanical properties, there was a decrease in the compressive strength value with the addition of 10% fly ash, amounting to 9.98% of the compressive strength of concrete without the addition of fly ash.

This result is attributed to the addition of the amount of fly ash waste in the mixture, which is inversely proportional to the density and compressive strength of the mortar produced. Specifically, the density value is directly proportional to the compressive strength, where the greater the density value, the denser the concrete produced. The sample mortar is categorised as type N mortar, which can be used as load-bearing walls, and type O, which can be used as non-load-bearing and decorative walls. Compressive strength results that are not optimal are influenced by several factors, namely:

i. Grits and fly ash waste materials from the paper industry contain different chemical compositions and do not comply with the required material content standards.
ii. The grit particle size falls into gradation no. 1 or has coarse grains, so it does not completely fill the voids/pores in the concrete.
iii. The mortar sample has voids in several parts, which affects the density and compressive strength of the concrete.

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