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The Effect of Fly Ash and Alkali Activator Mass Ratio (FA/AA) and Curing Conditions on the Mechanical Properties of High Compressive Strength Geopolymer Mortar

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ABSTRACT

Cement production is not an environmentally friendly process, as it consumes a lot of energy. One of the cement production processes is the sintering of calcareous and clay materials which is responsible for CO_2 emissions into the atmosphere. Globally, Cement production is associated with excessive carbon dioxide emissions. Geopolymer is an environmentally friendly alternative to replace Portland cement. Geopolymer is synthesized by a chemical reaction (geopolymerization) between aluminosilicate materials (industrial by-products, such as blast furnace slag or fly ash) and alkali activators. Fly ash is an aluminosilicate material from coal combustion residue that can be used as a geopolymer material to make more environmentally friendly mortar. Geopolymer mortar with FA/AA variations with a FA/AA variation range of 1.75-3.25 has an optimal compressive strength, namely at a ratio of 2.75 with a value of 57.6 Mpa. Oven curing for 48 hours is the optimum value with a value of 56.2 MPa and the highest compressive strength value was obtained at curing at a temperature of 140℃, namely 55.2 MPa.

1. Introduction

Cement production is not an environmentally friendly process, as it consumes a lot of energy. One of the cement production processes is the sintering of calcareous and clay materials which is responsible for around 10% of CO_2 emissions into the atmosphere. Globally, Portland cement is produced more than 4 billion metric tons per year. Cement production is associated with excessive carbon dioxide emissions. OPC production is widely recognized as a major contributor to greenhouse gas emissions, accounting for 6-7% of all CO_2 emissions as documented by the International Energy Agency (IEA) [1]. In the construction sector, mortar is the most commonly used building material. Ordinary Portland cement (OPC) remains the main binder used to bind mortar composite components. OPC has been widely used as an effective binder in mortar and other building materials. The use of OPC will continue to increase along with the growth of infrastructure. Global demand for

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OPC will increase by almost 200% by 2050 [2]. Mortar has characteristics that are easy to process and its compressive strength value is planned for the load to be received. mortar also has a shape that is easy to make as desired. That is what makes mortar the main material in making buildings around us. However, the continuous use of cement also has a negative impact on the environment. That is why many efforts have been made to replace cement with other materials that have similar capabilities and are environmentally friendly. To reduce these emissions and the problems associated with them, a new type of environmentally friendly and alternative environmentally friendly material called geopolymers was realized. Geopolymers are an environmentally friendly alternative to replace Portland cement. Geopolymers are synthesized by a chemical reaction (geopolymerization) between aluminosilicate materials (industrial by-products, such as blast furnace slag or fly ash) and alkaline activators [3]. Geopolymer cement has been proposed as an alternative binder to conventional ordinary Portland cement (OPC) in producing sustainable and low-carbon mortars. Geopolymers can be produced through the alkaline activation of low-calcium solid aluminosilicates with alkaline solutions (e.g. sodium hydroxide and aqueous silicate) [4]. Commonly used aluminosilicate precursors include metakaolin produced from the calcination of kaolinite clay and pulverized fly ash from coal combustion by-products.

Geopolymerization is a chemical reaction similar to the organic polymer chain in the final product, forming an alumina-silica chain that has binding properties. The geopolymeric reaction occurs between silica and alumina in a strong base catalyst medium with a high pH. This reaction is also called alkali activation, which changes the composition of the source material into a solid compound material with strong binding properties [1].

The main ingredients in the manufacture of geopolymer materials are precursors and alkali activators. The precursor plays a role in replacing portland cement which is now commonly used as a binder for mortar mixtures and the alkali activator solution functions to activate the precursor so that the geopolymerization process occurs and produces a binder or binder that is in accordance with what is planned. The selection of aluminosilicate precursors and alkali activators is very important. Various aluminosilicate materials, such as kaolin, metakaolin, fly ash, bottom ash, slag, red mud, rice husk ash, and volcanic ash, have been widely explored for their potential as geopolymer precursors. Alkali activator, consisting of NaOH and Na_2SiO_3 which can affect the compressive strength of geopolymers [5].

As the main material of geopolymers, fly ash as a precursor and Na_2SiO_3 and NaOH as activators that affect the characteristics of geopolymer mortar have the optimum composition and ratio to obtain mortar with high compressive strength. According to Aliabdo *et al.,* [6] The compressive strength of geopolymers decreases with increasing FA/AA ratio to 2.5 and then reversed, while Joseph and Mathew [7] found that the compressive strength of geopolymers decreased to an FA/AA ratio of 1.82. Likewise, Shehab *et al.,* [8] found that the greater the ratio of FA/AA solution, the lower the compressive strength after the test specimens were 7 and 28 days old. However, several studies have shown that the compressive strength of geopolymers increases with increasing FA/AA ratio [9]. The inconsistent compressive strength results of the FA/AA ratio from previous studies require further study of the fly ash and alkali activator (FA/AA) ratio to obtain geopolymer mortar with high compressive strength.

It has been reported that geopolymers made from fly ash or metakaolin have difficulty in curing at normal temperature due to the slow reaction rate, therefore most of the experiments in the past were carried out at higher curing temperatures [10]. Due to the slow reaction rate at room temperature, most geopolymer mortars are made by precasting. Therefore, an in-depth study must be carried out to obtain the right curing method for geopolymer mortar.

2. Materials

2.1 Fly ash

The fly ash used in this study were obtained from PT. Pupuk Sriwidjaja Palembang and analyzed using XRF (X-Ray Fluorescence), XRD (X-Ray Diffraction), and SEM (scanning Electron Microscopes) [11-14]. The chemical composition of the fly ash is given in Table 1, and the results of SEM and XRD are shown in Figure 1 and 2, respectively.

Most fly ash granules are sphericalas shown in Figure 1, which is an SEM photo of fly ash at 10,000× magnification. The spherical shape of fly ash allows it to react rapidly with other particles when making geopolymer mixtures.

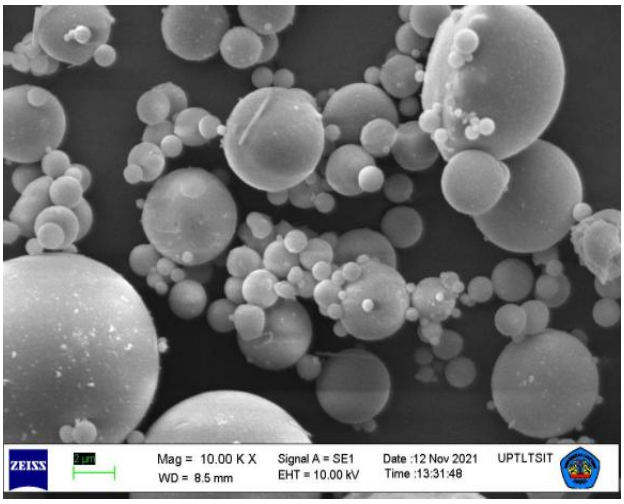

Fig. 1. SEM image of the fly ash used in this study

The fly ash can be classified as type C according to the ASTM 618 standard [14], because it contains 50% ≤SiO_2 + Al_2O_3 + Fe_2O_3 ≤ 70% based on Table 1.

Table 1
Chemical composition of the fly ash used in this study

Components	% in mass
MgO	0,2866
Al_2O_3	18,6427
SiO_2	36,3552
P_2O_5	0,2055
SO_3	0,2849
K_2O	0,7973
CaO	2,9143
TiO_2	0,9807
MnO	0,0932
Fe_2O_3	5,7413
CuO	0,0116
ZnO	0,0246
SrO	0,0931
Y_2O_3	0,0095
ZrO_2	0,0555
Balance	33,5039

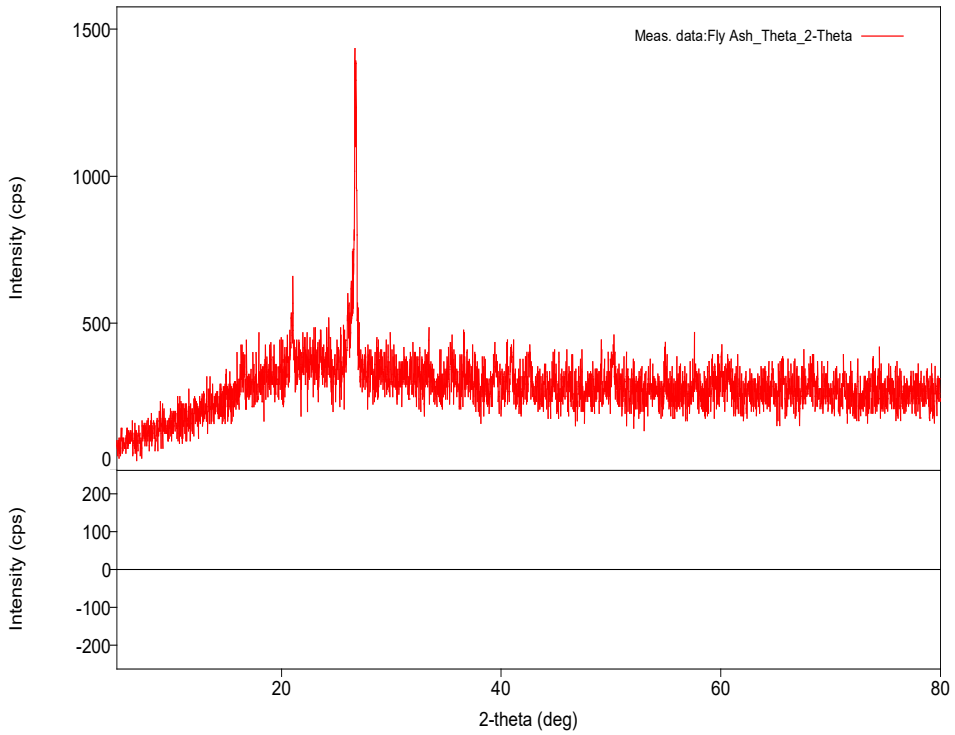

Fig. 2. XRD of the fly ash used in this study

The diffractogram obtained by conducting XRD analysis of the fly ash is shown in Fig. 2. It is observed that the crystalline peak of fly ash is very small, i.e., it occurs at a diffraction angle of 26.7° with an intensity of 1433,333 cps. This shows that the fly ash used in this study has an amorphous structure.

2.2 Alkali activator

Sodium hydroxide (NaOH) and sodium silicate (Na_2SiO_3) were used as alkali activators [15-17]. Activators are substances or elements that cause other elements to react [18-19]. The activator used contains NaOH and silica, which is a strong acid and hence will react with a strong base. Na_2SiO_3 has the function of accelerating the polymerization reaction. Meanwhile, NaOH reacts with the Si and Al in the fly ash to produce strong polymer bonds. To prepare the alkaline activator [20], an NaOH solution was mixed with a Na_2SiO_3 solution, and the mixture as allowed to stand for 1 day to achieve equilibrium before being used.

2.3 Sand

The Tanjung Raja area in South Sumatra province as a significant supplier of fine aggregate material utilized in this study. Located in a region known for its natural resources, Tanjung Raja provides high-quality aggregates that meet the requirements for various construction and research applications. The fine aggregates from this area are carefully selected for their consistency, particle size distribution, and suitability in different engineering projects, making them an essential component for the study's objectives.

3. Methods

This research focuses on various modifications of geopolymer mixture composition. The variations carried out are the ratio of fly ash mass to alkali activator (FA/AA), curing time in the oven, and changes in curing temperature in the oven. The test object used was a cube measuring 5cm x 5cm x 5cm. Geopolymer mortar test objects will be tested for the mechanical properties of the mortar when they are 7 days old. The mechanical properties test is in the form of compressive strength with a sample size of 3 for each variation. The compositions for test specimens with variations in the ratio of fly ash mass to alkali activator (FA/AA), curing time in the oven, and changes in curing temperature in the oven are shown in Table 2, Table 3, and Table 4, respectively.

Table 2
Chemical composition of the fly ash used in this study

Sample	Variation					
	FA/AA	Na_2SiO_3/NaOH	Sand/FA	NaOH molarity (M)	Oven curing temperature (℃)	Oven curing time (H)
FA 1,75	1,75	2,5	0,4	9	80	24
FA 2	2	2,5	0,4	9	80	24
FA 2,25	2,25	2,5	0,4	9	80	24
FA 2,5	2,5	2,5	0,4	9	80	24
FA 2,75	2,75	2,5	0,4	9	80	24
FA 3	3	2,5	0,4	9	80	24
FA 3,25	3,25	2,5	0,4	9	80	24

Table 3
Geopolymer Composition Variation of Oven Curing Time

Sample	Variation					
	FA/AA	Na_2SiO_3/NaOH	Sand/FA	NaOH molarity (M)	Oven curing temperature (℃)	Oven curing time (H)
T6	2,5	2,5	0,4	9	80	6
T12	2,5	2,5	0,4	9	80	12
T18	2,5	2,5	0,4	9	80	18
T24	2,5	2,5	0,4	9	80	24
T48	2,5	2,5	0,4	9	80	48
T72	2,5	2,5	0,4	9	80	72

Table 4
Geopolymer Composition Curing Oven Temperature

Sample	Variation					
	FA/AA	Na_2SiO_3/NaOH	Sand/FA	NaOH molarity (M)	Oven curing temperature (℃)	Oven curing time (H)
C50	2,5	2,5	0,4	9	50	24
C80	2,5	2,5	0,4	9	80	24
C110	2,5	2,5	0,4	9	110	24
C140	2,5	2,5	0,4	9	140	24

4. Results and Discussion

Discussion of the results of this study are as follows: results from laboratory tests of mortar geopolymer mechanical properties.

4.1 Geopolymer Composition Variation Fly Ash to Alkali Activator (FA/AA) Ratio

Figure 3 shows the details of the fluctuation of the FA/AA ratio. The compressive strength of geopolymer increases with the increase in the FA/AA ratio from 1.75 to 2.75 or from 43.6 MPa to 57.6 MPa. The optimal FA/AA mass ratio parameter value for geopolymer is 2.75. The FA/AA ratio has a positive impact on the properties of geopolymer mortar, including ease of workability, setting time, and compressive strength. Decreasing this ratio will increase ease of workability and delay setting time. Conversely, a higher FA/AA ratio produces higher compressive strength with a certain limit up to a ratio where the mixture has excess fly ash so that it does not react perfectly with the alkali activator [22].

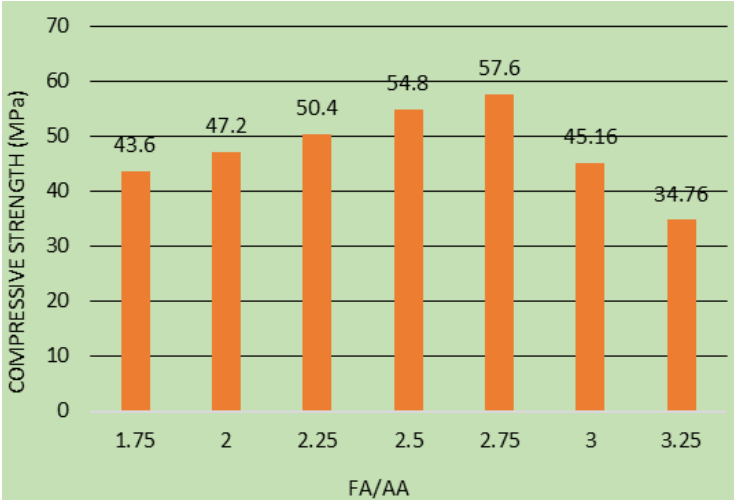

Fig. 3. Compressive strength of geopolymer mortar with variations in the ratio of fly ash and alkali activator (FA/AA)

According to Aliabdo *et al.,* [6] the compressive strength of geopolymers decreases with increasing FA/AA ratio up to 2.5 and then reverses direction, while Joseph and Mathew [7] found that the compressive strength of geopolymers decreases up to a FA/AA ratio of 1.82. Likewise, Shehab *et al.,* [8] found that with increasing FA/AA solution ratio, there was a decrease in compressive strength after the specimens were 7 and 28 days old. However, several studies have shown that the compressive strength of geopolymers increases with increasing FA/AA ratio [9].

The FA/AA ratio value is the comparison between the fly ash content and the alkali activator content. The higher the FA/AA ratio value, the lower the workability of the geopolymer made. The FA/AA ratio is a parameter that affects the compressive strength of the geopolymer, this is because the amount of fly ash and alkali activator used affects the geopolymerization reaction between the two materials. The right ratio of fly ash and alkali activator is needed to obtain the optimal compressive strength value.

This study investigates the fluctuation of FA/AA values to determine the optimum compressive strength value. The compressive strength of geopolymer increased with an increase in the FA/AA ratio value from 1.75 to 2.75, or from 43.6 MPa to 57.6 MPa. The ideal FA/AA mass ratio parameter value is 2.75 for geopolymer. This study looked at the fluctuation of FA/AA values to determine the optimal compressive strength value. The compressive strength of geopolymer increased with an increase in the FA/AA ratio value from 1.75 to 2.75, or from 43.6 MPa to 57.6 MPa. The optimal FA/AA mass ratio parameter value for geopolymer is 2.75.

4.2 Geopolymer Composition Variation Oven Curing Time

This study examines the difference in oven drying time with a time of 6 hours, 12 hours, 18 hours, 24 hours, 48 hours, and 72 hours. The compressive strength of geopolymers with variations in oven drying time can be seen in Figure 4.

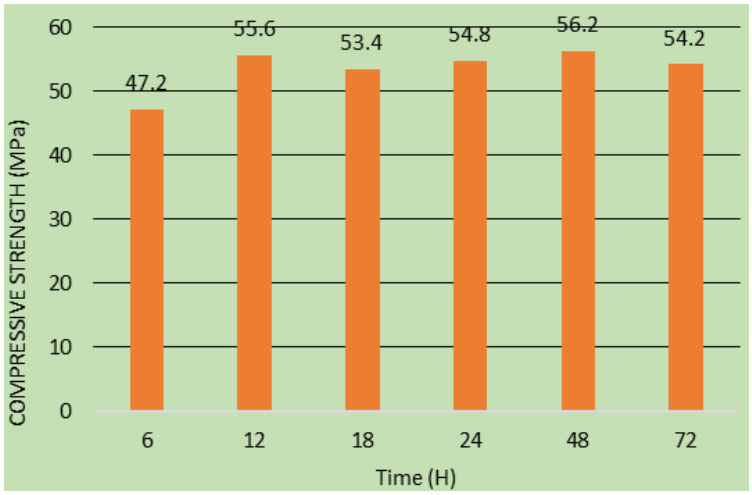

Fig. 4. Compressive strength of geopolymer mortar with variations Variation Oven Curing Time

Based on Figure 4, there is an increase in compressive strength during oven curing for 6 hours to 12 hours, with an oven curing time of 48 hours being the optimum value with a value of 56.2 MPa, and there is no significant increase or decrease in compressive strength between oven curing times of 12 hours to 72 hours. This insignificant compressive strength value is because the oven curing time of 12 hours can accelerate the geopolymerization process effectively so that the oven curing time does not greatly affect the compressive strength of the geopolymer and consumes large energy resources.

The compressive strength of fly ash-based geopolymer concrete increases with drying time, at least for 48 hours. The compressive strength value increases with increasing drying temperature. Drying is very important for the growth of concrete strength, be it geopolymer concrete or ordinary Portland cement concrete. The impact of drying time on the development of early strength in Geopolymer Concrete is very important. Drying has been shown to increase the polymerization process in Geopolymer Concrete, resulting in higher compressive strength [21].

This study examines the variation of oven drying time values to obtain optimal compressive strength values. The compressive strength values of geopolymers with variations in oven drying time can be seen in Figure 4. These geopolymer variations are cured at an oven temperature of 80℃.

This study examines the differences in oven curing time with a time of 6 hours, 12 hours, 18 hours, 24 hours, 48 hours, and 72 hours. There was an increase in compressive strength during oven curing for 6 hours to 12 hours, with 48 hours being the optimum value of the variation in oven curing time with a value of 56.2 MPa, and there was no significant increase or decrease in compressive strength between 12 hours and 72 hours.

4.3 Geopolymer Composition Variation Oven Curing Temprature

This study investigated the effect of different oven drying temperature values on the compressive strength. Figure 5 illustrates the compressive strength of geopolymers as a function of oven curing

temperature.

In the curing temperature variation, the oven uses temperatures of 50℃, 80℃, 110℃, and 140℃. There is a relatively high increase in compressive strength from 50℃ to 80℃, namely from 25.8 MPa to 54.8 MPa. Curing temperature is very important in increasing the strength of geopolymer materials. It is known that increasing the curing temperature results in an increase in strength. The highest compressive strength value was obtained at curing at a temperature of 140℃, which was 55.2 MPa. Then at a temperature of 80℃ to 140℃ there was no significant increase or decrease in compressive strength.

Increasing the curing temperature in the range of 30 to 90 °C increases the compressive strength of geopolymer concrete and longer curing time also increases the compressive strength. Several steps are involved during geopolymerization and the entire process is accelerated by molecular thermal agitation, which can be successfully provided by heating at 60–80 °C for a certain time interval. The strength continues to increase with time and the reaction proceeds to completion [22].

Curing conditions have a significant impact on the formation of alkali-activated materials because high temperatures accelerate the chemical reactions and solubilization of reactive species, enhancing the interaction between the aluminosilicate source and the alkali solution during geopolymer synthesis. Previous studies have shown that curing time and temperature have a substantial impact on the compressive strength of geopolymer concrete [22].

This study investigates the effect of different oven curing temperature values on the compressive strength. Figure 5 illustrates the compressive strength of geopolymers as a function of oven curing temperature.

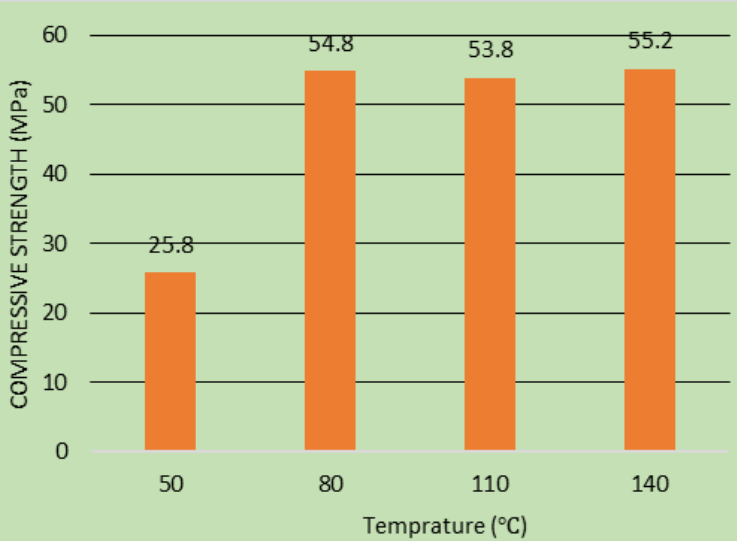

Fig. 5. Compressive strength of geopolymer mortar with variations Variation Oven Curing Temprature

In the curing temperature variation, the oven uses temperatures of 50℃, 80℃, 110℃, and 140℃. There is a relatively high increase in compressive strength from a temperature of 50℃ to 80℃, namely from 25.8 MPa to 54.8 MPa. The highest compressive strength value is obtained at curing at a temperature of 140℃, namely 55.2 MPa. Then, in the compressive strength from a temperature of 80℃ to 140℃, there is no significant increase or decrease in compressive strength.

5. Conclusions

Based on the results and analysis carried out, it can be concluded that:

i. Geopolymer mortar with FA/AA variation with FA/AA variation range 1.75-3.25 has optimum compressive strength at ratio 2.75 with value 57.6 Mpa. There is an increase in compressive strength value from FA/AA ratio 1.75 to 2.75 then the compressive strength value decreases to FA/AA ratio 3.25.
ii. The increase in compressive strength occurs during oven curing for 6 hours to 12 hours. Oven curing for 48 hours is the optimum value with a value of 56.2 MPa, and there is no significant increase or decrease in compressive strength between oven curing times for 12 hours to 72 hours.
iii. In the variation of curing oven temperature using temperatures of 50℃, 80℃, 110℃, and 140℃. There was a relatively high increase in compressive strength from 50℃ to 80℃, namely from 25.8 MPa to 54.8 MPa. The highest compressive strength value was obtained at curing with a temperature of 140℃, which was 55.2 MPa. Then in the compressive strength from a temperature of 80℃ to 140℃ there was no significant increase or decrease in compressive strength.

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