Shear wave velocities to monitor curing evolution of soils treated with alkali activated binders

Sara Rios[#1], *Isabela* Caetano[1], *Claver* Pinheiro[1] and *António* Viana da Fonseca[1]

[1]*Construct, GEO, Faculdade de Engenharia da Universidade do Porto - FEUP, Porto, Portugal*
[#]*Corresponding author: sara.rios@fe.up.pt*

ABSTRACT

The use of alkali activated binders for soil improvement has been significantly increased in the past decade indicating a significant interest in the use of alternative low carbon binders. The optimisation and preliminary characterisation of the mixtures is usually addressed with destructive tests to evaluate strength such as unconfined compression strength tests. Although important and necessary, these tests require a significant number of specimens, especially when monitoring the strength evolution with time due to curing. Seismic wave velocity measurements, in particular S wave velocities, allow a fast evaluation of the elastic shear stiffness with time avoiding specimens' failure. This is even more important in soils stabilised with alkali activated binders, since its strength and stiffness evolution with time is generally very long, keeping increasing far beyond the 28 days mark usually assumed in soils improved with Portland cement. In this paper, the evolution of the elastic stiffness with time for different soils treated with alkali activated binders is presented. The compilation of the results for different soils and mixtures, allowed a clear definition of the typical trend and range.

Keywords: alkali activated binders; soil improvement; seismic wave velocity measurements; ultrasonic transducers.

1. Introduction

1.1. The need to study alternative binders

Most of ground improvement techniques use lime or Portland cement which have a significant carbon footprint. In fact, the cement industries are among those penalized by the Kyoto Protocol, signed in 1997 and more recently in the Glasgow Agreement, 2021, due to excessive greenhouse gas emissions. Moreover, the massive production of Portland cement is also causing other environmental impacts due to the use of clay and limestone that are becoming increasingly scarce. For this reason, a significant effort has been made by the research community in the last decades to develop more sustainable binders. More sustainable options include: clinker replacement by other industrial by-products such as slags or fly ash; energy efficiency techniques and use of alternative fuels for clinker production; binders without clinker such as alkali activated binders; CO2 cured concretes (Lehne & Preston, 2018). Most of these options have been heavily studied and improved, while some products have been recently released in the market. However, a long way of research is still needed in these materials to obtain optimised procedures, as exists currently for Portland cement. In this work, the use of seismic wave measurements is presented as a way to monitor the curing evolution of different soils treated with alkali activated binders. These binders have a long-term evolution beyond 28 days usually considered as time for the final Portland cement strength.

1.2. Alkali activated binders

Alkali activated cements can significantly reduce carbon dioxide emissions as well as the consumption of non-renewable natural resources in civil engineering applications, relatively to ordinary Portland cement, since industrial by-products and waste materials can be used instead of natural aggregates.

Alkaline activation can be described as a reaction between aluminosilicate materials (precursors) and alkali substances such as sodium (Na) or potassium (K), or an alkaline-earth ion, such as calcium (Ca). This technique is particularly adequate to create binders based on residues, such as fly ash or slag, which constitute very effective options due to their amorphous or vitreous aluminosilicate microstructure (Pinheiro *et al.*, 2020). When calcium is present in the mixture in significant amounts, the formation of a gel type C-A-S-H with 2D structure is favoured. However, in a material with low calcium content, the gel formed is an amorphous aluminosilicate gel (sometimes designated as N-A-S-H gel) with 3D structure (Provis and van Deventer, 2014). In some circumstances (for intermediate calcium contents and high pH values) both types of cementitious gels are present and interacting leading to structural and compositional changes in the process (Garcia-Lodeiro et al., 2011).

The curing conditions are one of the most influencing factors affecting the mechanical performance of alkali activated binders. Athira et al. (2021) reports that ambient cured slag-based alkali-activated binders exhibited better strength gain compared to ambient cured fly ash-based binders. Moreover, fly ash-based binders require high temperature to initiate the reaction, and higher temperature curing guaranteed higher early strength gain for both slag and fly ash-based binders.

1.3. Seismic wave measurements in stabilised soils

The use of seismic wave measurements to measure elastic properties is a well-known technique (Stokoe & Santamarina, 2000). The application of this technique has been successfully applied to stabilized soils (Azenha *et al.*, 2011; Consoli *et al.*, 2012; Viana da Fonseca *et al.*, 2014). In particular, the use of S waves is very useful to monitor the stiffness evolution with time (Rios *et al.*, 2017; Ferreira *et al.*, 2021) due to the non-destructive nature of this technique. There are several devices that can be used for this purpose: bender elements (Viana da Fonseca *et al.,* 2009), resonant column (Camacho-Tauta *et al.*, 2016), or ultrasound transducers (Amaral *et al.,* 2011; Khan *et al.,* 2011). These latter have the advantage of being a non-intrusive, 'plug-and-play' methodology which is very convenient for very stiff soils.

The standard procedure for ultrasonic testing consists on the use of an input frequency identical to the nominal frequency of the transducers. This procedure allows for an optimised performance of the transducers, providing a higher amplitude of the response signal. However, it is recognised that the best input frequency for seismic wave measurements depends on the stiffness of the material. For example, ASTM (2000) prescribes the ultrasonic analysis of rock within a frequency range from 75 kHz to 3 MHz. In order to monitor curing dependent materials in which the stiffness of the specimen is increasing with time, Ferreira *et al.* (2021) has proposed a procedure to assess the optimum ultrasonic frequency. In this case, the same ultrasonic transducers can be used from the very early ages to higher curing times, provided that the optimum ultrasonic frequency is selected at each curing time. In this work, this procedure was applied to different soils treated with alkali activated binders obtaining a trend of stiffness evolution with time.

2. Materials and Methods

2.1. Soils

Different types of silty sands were involved in this work. One of the soils is a well graded silty sand obtained after remoulding a residual soil from granite, known as Porto silty sand, and thoroughly studied in its natural conditions (Viana da Fonseca, 2003; Viana da Fonseca et al., 2006). It is an excellent material for earthworks with or without cementation (Amaral, 2009; Arroyo et al., 2013; Rios et al., 2014; Rios et al., 2017).

The other soils comprise a silty sand obtained after rock crushing in a quarry in Bogotá, Colombia, a dredged silty sand collected in Vila de Conde on the north of Portugal, and iron tailings from Minas Gerais, Brazil. Their grain size distribution curves are presented in Figure 1. Since the quarry silty sand is not a natural soil it has a gap graded curve due to the rock crushing process. Table 1 summarises the main physical parameters of the soils.

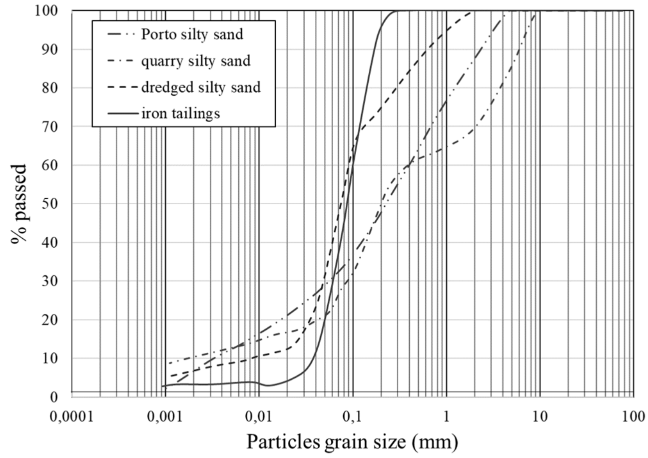

Figure 1. Soils grain size distribution curves.

Table 1. Physical parameters of the soils

Soil type	Iron tailings	Dredged silty sand	Porto silty sand	Quarry silty sand
γ_s (kN/m^3)	29.5	25.6	26.6	25.9
D_{50} (mm)	0.07	0.07	0.25	0.20
C_u	2.71	10.02	113	210
C_c	1.10	2.72	2.72	8.60
w_L	NP	NP	34	NP
w_P	NP	NP	31	NP

NP – Non-plastic

2.2. Precursors and activators for alkaline activation

The soils indicated in Figure 1 were stabilised with various alkali activated binders, most of them using a low calcium fly ash as precursor, which led to the formation of a gel type N-A-S-H gel with 3D structure. However, in the case of the dredged soil, a ladle steel slag rich in calcium was used with a much higher binder/soil ratio since the purpose was to cure the specimens in water. The properties of this ladle slag can be found in Pinheiro et al. (2020). In this case, the stabilisation with the alkali activated binder was compared with Portland cement.

In terms of the activator solution, all studied cases used sodium hydroxide (SH) with sodium silicate (SS) solution as activator, except for the iron tailings where only the sodium hydroxide was used in an attempt to reduce the number of components and the carbon footprint brought by the activator. A commercial SS solution was used while the SH solution was prepared to the desired concentration by dissolving sodium hydroxide pellets in water. The SS solution has a bulk density of 1.464 g/cm^3 at 20 °C, a SiO_2/Na_2O weight ratio of 2.0 (molar oxide ratio of 2.063) and a Na_2O concentration in the solution of 13.0%. The SH pellets have a specific gravity of 2.13 at 20 °C (99 wt%).

2.3. Specimen preparation

The specimen preparation procedures were very similar among the various specimens. First, the fly ash or slag is mixed in the dry soil and then the activator is added until a homogeneous mixture is obtained. In the particular case of the iron tailings part of the water was first added to the soil (to achieve a soil water content of 16%), which was then mixed with the fly ash. The remaining water was mixed with the activator and then joined to the mixture. This procedure tried to replicate the stabilisation of filtered tailings which are not dry.

All the specimens were moulded in three layers in moulds with sizes varying from 38 mm to 70 mm of diameter being the specimen height twice the diameter. After demoulding the specimens were left to cure in air or water. Table 2 summarises the binders used for each soil and corresponding curing conditions, while Table 3 indicates the composition of the mixtures by presenting the binder/soil ratio, the liquid/solid ratio, the SS/SH ratio, and the concentrations of SH.

The mixtures with iron tailings were cured at different temperatures to analyse its effect on stiffness and strength evolutions. Mixture II.24 and Mixture II.48 were cured for 24h and 48 h at 50ºC, respectively. The remaining curing time was at 20ºC. The idea is to analyse if the increase in curing temperature during a limited period accelerates the chemical reactions leading to the formation of the binding gel.

In the improvement of the dredged silty sand, two mixtures were prepared with alkali activated binders, and these were compared with a mixture with Portland cement. Soil + Slag (6.5) and Soil + Slag (8.0) were cured under water, but the former was cured in salt water (to simulate maritime conditions) while the latter was cured in distilled water. The main differences between these curing waters are their pH values: while the sea water has a pH of 7.34, the distilled water is acid with a pH of 5.4.

On Porto silty sand there are three mixtures stabilised with a low calcium alkali activated binder with different binder/soil ratios described in Rios et al. (2017), and two additional mixtures with Portland cement studied by Amaral (2009).

The stabilisation of the quarry silty sand was analysed by doing 10 different mixtures with two different binder/soil ratios and testing several activator compositions and concentrations.

Table 2. Binders and curing environment

Ref.	Soil type	Binder	Curing environment
This work	Iron tailings	FA + SH	Air – 20º/50ºC
	Dredged silty sand	Slag + SS + SH	Salt/distilled water - 20ºC
		Portland Cement	
Rios et al. (2017)	Porto silty sand	FA + SS + SH	Air – 20ºC
Amaral (2009)		Portland Cement	
Rios et al. (2019)	Quarry silty sand	FA + SS + SH	

Table 3. Characteristics of the binder in each specimen

Id	Binder /Soil	SS/ SH	Liquid /solid (%)	Concent ration	Soil
IIa	0.3	0	17	2.19	Iron tailings
IIa.24	0.3	0	17	2.19	
IIa.48	0.3	0	17	2.19	
Soil+Slag 6.5	1	1	14	6.5	Dredged silty sand
Soil+Slag 8.0	1	1	14	8	
Soil + CEM	6.5	-	26	-	
M1	0.15	0.5	11.7	7.5	Porto silty sand
M2	0.2	0.5	15.6	7.5	
M3	0.25	0.5	19.5	7.5	
5%	0.05	-	12%	-	
7%	0.07	-	12%	-	
A1C5	0.1	1	8	5	Quarry silty sand
A05C7	0.1	0.5	8	7.5	
A1C7	0.1	1	8	7.5	
A05C10	0.1	0.5	8	10	
A1C10	0.1	1	8	10	
A1C12	0.1	1	8	12.5	
B1C5	0.2	1	8.8	5	
B05C7	0.2	0.5	8.8	7.5	
B1C7	0.2	1	8.8	7.5	
B1C10	0.2	1	8.8	10	

2.4. Testing procedures

P and S seismic wave velocities were performed by ultrasonic transducers throughout the curing period from 3 days to 90 days depending on the specimen. In some cases, unconfined compression strength was also measured in the same specimen, up to 365 days.

The testing setup for seismic wave measurements (Figure 2) is commercially available and comprises: piezoelectric ultrasonic transducers (one pair for compression waves with nominal frequency of 82 kHz, and another pair for shear waves with nominal frequency of 100 kHz), as well as a pulse waveform generator and data acquisition unit equipped with an amplifier directly logged to a PC which has a specific software to operate as an oscilloscope.

An excitation voltage of 500 V and a pulse input signal frequency of 82 kHz were initially used, both for the P-wave and S-wave transducers. Then, a wider range of frequencies was used as input between 24 kHz and 200 kHz, so that the best input frequency could be selected, depending on the type of specimen and curing time, as proposed by Ferreira et al. (2021).

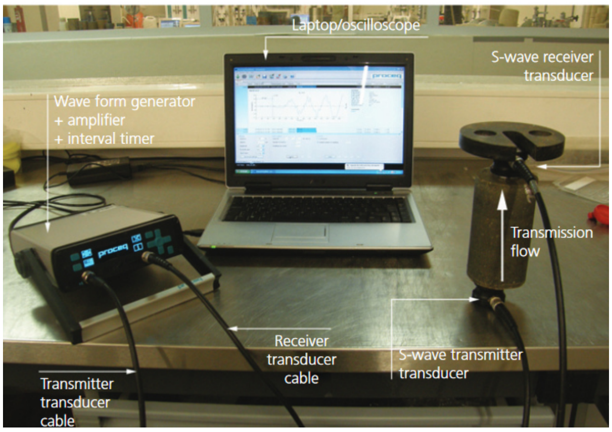

Figure 2. Setup for seismic wave measurement.

3. Results

3.1. Behaviour beyond the 28 days

The results of the elastic shear modulus or unconfined compression strength are herein presented in terms of normalised values because the focus of this study is the type of evolution with time and not the final strength/stiffness. Since the strength or stiffness values are divided by the corresponding value at 28 days, the differences between mixtures are not taken into account.

Figure 3 shows the normalised strength evolution with curing time for two mixtures with the quarry silty sand. A continuous increase up to 365 days is observed without the stabilisation at 28 days typical of Portland cement mixtures. Although, there are only two mixtures with data available up to such long period, other results of stiffness evolution with time confirm the trend of continuous evolution. Figure 4 shows the normalised stiffness evolution with time for three mixtures with Porto silty sand. Two of those mixtures (M1 and M3) show an almost linear increase up to 90 days while the other mixture (M2) shows a logarithmic trend.

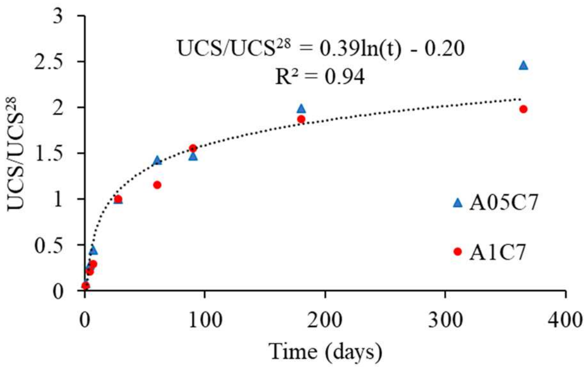

Figure 3. Strength evolution after 28 days.

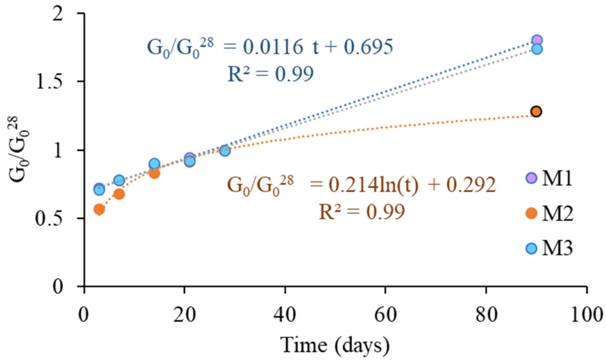

Figure 4. Stiffness evolution after 28 days.

3.2. Effect of curing temperature in stiffness and strength evolution

The evolution in time of the iron tailings stabilised with a low calcium alkali activated binder and cured at different temperatures, was evaluated. The elastic shear modulus was measured in these mixtures at 7, 14, 21 and 28 days of curing time and the specimens were failed in unconfined compression at 7 and 28 days.

The results are presented in Figure 5 indicating, as expected, a significant influence of the temperature on the stiffness increase. While the mixture cured at 20ºC shows a linear increase, in the mixtures cured with temperature the shape of the curve is different as the initial stiffness is much higher and then rapidly tends to stabilise. The higher the curing temperature the higher the initial stiffness, as temperature accelerates the chemical reactions involved in the material hardening.

The comparison of the 28 days unconfined compression strength to the 7 days strength indicates that the temperature may also affect the final strength, but further tests are required to confirm this.

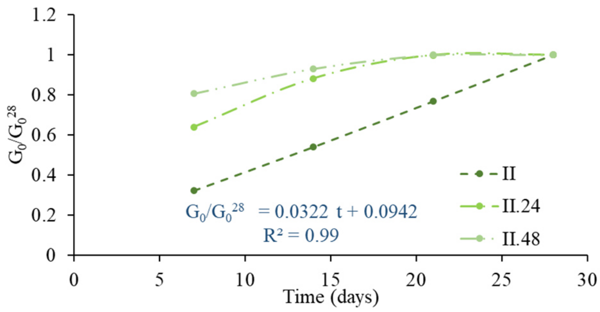

Figure 5. Effect of curing temperature on stiffness evolution
Figure 6.

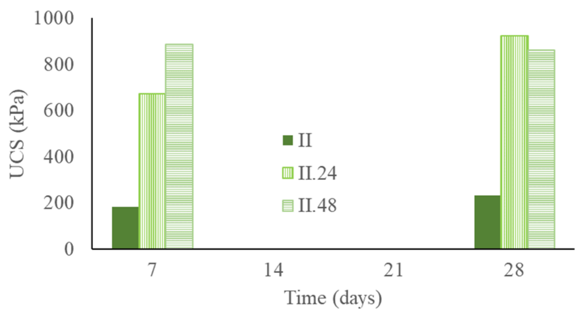

Figure 7. Effect of curing temperature on strength evolution.

3.3. Effect of curing of water in stiffness evolution

Considering that the development of the alkali activation reactions requires an alkaline medium, the presence of an acid water is not favourable. This was studied with the dredged silty sand where the specimens were submerged in sea water and distilled water. In these conditions, it was concluded that the mixture submerged in distilled water had to be more concentrated (Soil + Slag (8.0)), but even though, it has demonstrated lower stiffness. However, the normalised stiffness evolution is very similar between the two mixtures, in spite of the different curing waters. Figure 7 shows that their trend has an almost linear evolution, which can be explained by the high binder content, and also by the type of C-A-S-H binders, that typically have a faster curing rate at young ages (Athira et al., 2021).

In this same figure there is also a mixture of this soil with Portland cement as well as other mixtures of Porto silty sand with Portland cement. It is interesting to observe that all mixtures with Portland cement have the same normalized stiffness evolution in spite of different cement contents and soil types (Porto silty sand and dredged silty sand). As expected, the Portland cement mixtures have a logarithmic evolution that tends to stabilise when approaching 28 days of curing, conversely to what was observed in the soil stabilised with high calcium alkali activated binders.

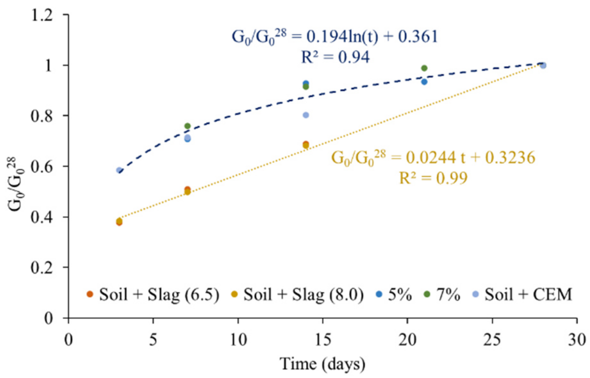

Figure 8. Trends of the normalised stiffness evolution for the dredged silty sand with high calcium alkali activated binders (Soil + Slag (6.5) and Soil + Slag (8.0)) and Portland cement mixtures (5%, 7%, Soil + CEM).

3.4. Comparison of the stiffness evolution for the studied mixtures

Figure 8 presents the normalised stiffness evolution with time for the mixtures with the quarry silty sand stabilised with low calcium alkali activated binders. A logarithmic evolution of the normalised stiffness within a narrow range (± 0.1) is observed which contrasts with the almost linear evolution observed in the high calcium mixtures of Figure 7.

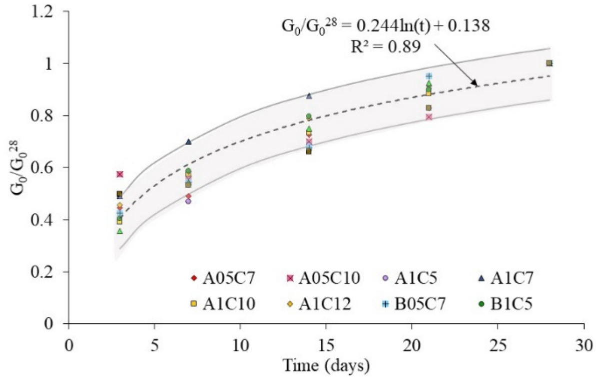

Figure 9. Trend of the normalised stiffness evolution for the quarry silty sand stabilised with low calcium alkali activated binders.

In summary, it can be concluded that mixtures M1 and M3 with Porto silty sand, Mixture II with Iron tailings and Soil+Slag (6.5) and Soil+Slag (8.0) with the dredged silty sand have a more linear trend (equation (1)), while the others have a logarithmic evolution with a higher evolution rate for young ages (equation (2)).

$$G_0 / G_0^{28} = A \ln(t) + B \quad (1)$$

$$G_0 / G_0^{28} = C \times t + D \quad (2)$$

However, the constant parameters of those equations are different as compared in Table 4. On the logarithmic trends, the mixtures with Portland cement have higher initial stiffness and less increase in time in comparison with the low calcium alkali activated binders. These equations are also different from the normalised unconfined compression strength relation present in Figure 3 where the initial strength is very small.

Table 4. Characteristics of the binder in each specimen

Logarithmic trends	A	B
Porto silty sand with low calcium AA binders (Figure 4)	0.214	0.292
Mixtures with Portland cement (Figure 7)	0.194	0.361
Quarry silty sand with low calcium AA binders (Figure 8)	0.244	0.138
Linear trends	**C**	**D**
Porto silty sand with low calcium AA binders (Figure 4)	0.0116	0.695
Iron tailings with low calcium AA binders (Figure 5)	0.0322	0.0942
Dredged silty sand with high calcium AA binders (Figure 7)	0.0244	0.3236

On the linear trends, it is interesting to notice that there are also mixtures with low calcium AA binders, which are usually associated to low initial strength rates. This means that there are still issues governing the behaviour of soils stabilised with alkali activated binders that are not fully understood.

4. Conclusions

This paper analyses the elastic shear stiffness evolution with time, when normalised by the corresponding value at 28 days. A comparison is performed for several soils stabilised with alkali activated binders. In some cases, a comparison with Portland cement binders is also presented. The effect of curing conditions was analysed first in terms of curing temperature for specimens cured at air, showing an acceleration of the stiffness evolution with the increase in curing temperature. For the specimens cured under water, the type of curing water had an important influence on the final strength or stiffness of the specimens, but it did not affect the rate of stiffness evolution with time, for the studied mixtures.

The different trends were organised in two groups: one following an almost linear evolution with time, and another following a logarithmic evolution with time. These different evolution trends depend on several factors: type of binder, type of soil, binder concentration, and curing conditions. However, it is very important to compile information from different mixtures to work on possible indexes that might explain the observed behaviour. This can be of significant importance for the design and dosage definition of these alternative binders. While Portland cement mixtures benefit from a very long experience acquired for many years, alkali activated binders are just in the first part of its life cycle (Provis, 2018).

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