

Article

Use of a Piezoelectric Bender Element for the Determination of Initial and Final Setting Times of Metakaolin Geopolymer Pastes, with Applications to Laterite Soils

Janjit Iamchaturapatr ¹, Keeratikan Piriyakul ^{1,*}  and Aruz Petcherdchoo ²

¹ Center of Excellence in Structural Dynamics and Urban Management, Department of Civil and Environmental Engineering Technology, College of Industrial Technology, King Mongkut's University of Technology North Bangkok, 1518 Pracharat 1 Road, Wongsawang, Bangsue, Bangkok 10800, Thailand; janjit.i@cit.kmutnb.ac.th

² Department of Civil Engineering, Faculty of Engineering, King Mongkut's University of Technology North Bangkok, 1518 Pracharat 1 Road, Wongsawang, Bangsue, Bangkok 10800, Thailand; aruz.p@eng.kmutnb.ac.th

* Correspondence: keeratikan.p@cit.kmutnb.ac.th; Tel.: +66-2-555-2000 (ext. 6511)

Abstract: This study proposes the use of a non-destructive testing technique, based on piezoelectric bender element tests, to determine the initial and final setting times of metakaolin geopolymer pastes. (1) Background: Metakaolin geopolymer is a new eco-friendly building material that develops strength rapidly and is high in compressive strength. (2) Methods: The initial and the final setting times were investigated via bender element and Vicat needle tests. Metakaolin powder was prepared by treating kaolin at 0, 200, 800, 1000, and 1200 °C. All metakaolin powder samples were then mixed with geopolymer solution at different mixing ratios of 0.8:1.0, 1.0:1.0, 1.2:1.0, and 1.5:1.0. The geopolymer solution was prepared by adding 10 normal concentrations of sodium hydroxide (10 N NaOH) to sodium silicate (Na₂SiO₃) at various solution ratios of 1.0:1.0, 1.0:1.2, 1.0:1.5, 1.0:2.0, 1.2:1.0, 1.5:1.0 and 2.0:1.0. (3) Results: The optimum temperature for treating metakaolin is established at 1000 °C, with a mixing ratio between the metakaolin powder and the geopolymer solution of 1.0:1.0, as well as a solution ratio between NaOH and Na₂SiO₃ of 2.0:1.0. (4) Conclusions: The use of piezoelectric bender elements to determine the initial and final setting times of metakaolin geopolymer pastes is a useful method by which to detect geopolymerization by shear wave velocity in a real-time manner. Moreover, the penetration of the Vicat apparatus can confirm the setting times at specific intervals. The relationships between the shear wave velocity and the Vicat penetration appear to be linear, with an initial setting time of 168 m/s and a final setting time of 187 m/s. Finally, the optimum metakaolin geopolymer pastes are applied to improve laterite soils, as measured by CBR tests.

Keywords: metakaolin; geopolymer; shear wave velocity; setting time; laterite soil



Citation: Iamchaturapatr, J.; Piriyakul, K.; Petcherdchoo, A. Use of a Piezoelectric Bender Element for the Determination of Initial and Final Setting Times of Metakaolin Geopolymer Pastes, with Applications to Laterite Soils. *Sensors* **2022**, *22*, 1267. <https://doi.org/10.3390/s22031267>

Received: 30 December 2021

Accepted: 1 February 2022

Published: 7 February 2022

Publisher's Note: MDPI stays neutral with regard to jurisdictional claims in published maps and institutional affiliations.



Copyright: © 2022 by the authors. Licensee MDPI, Basel, Switzerland. This article is an open access article distributed under the terms and conditions of the Creative Commons Attribution (CC BY) license (<https://creativecommons.org/licenses/by/4.0/>).

1. Introduction

Various kinds of techniques are available in the marketplace for determining the properties of materials. The criterion by which to select a suitable technique depends on the properties to be determined. The bender element test is considered to be one of the outstanding non-destructive tests for numerous reasons, such as its capacity for real-time monitoring [1]. In combination with piezoelectric sensors, the bender element can effectively determine the shear wave velocity and the initial shear modulus of materials in studies [2–6]. As an example, the bender element test was applied to studying soil characteristics, e.g., thawing and stress restoration in artificial frozen sandy soils [7], transition evaluation of fine sand from capillarity to cementation [8], expansion-induced crack propagation monitoring in rocks [9], response monitoring of transitional mixtures retaining

the memory of in-situ overburden pressure [10], etc. For concrete materials, various studies can also be carried out using the bender element, e.g., the characterization of recycled concrete aggregate [11], evaluation of damage induced by alkali-silica reaction [12], etc.

Other than the aforementioned applications in terms of soil and concrete, the piezoelectric bender element can be used to determine the properties of those cement materials that are popular in construction due to the fact that they are both common in the marketplace and cost-effective. One of their essential properties to be determined is their setting time. As an example, Zhu et al. [13] utilized the bender element technique to determine the cement mortar setting times, using shear wave velocity evaluation curves. In their study, specimen geometry and the bender element were set to obtain shear wave velocity evaluation curves at varying times, as well as for different water-to-cement ratios and chemical admixtures. Accordingly, derivative methods were proposed by which to obtain the initial and the final setting times. It was found that the final setting time was correlated with the first-order derivative curves, while the initial setting time corresponded to the second-order derivative curves. Liu et al. [14] used piezoceramic bender elements in combination with ultrasonic shear waves to monitor the setting process of mortar and concrete. It was observed that the mortar, which was tested using different water-to-cement ratios, showed a clear relationship between shear wave velocity and penetration resistance. Moreover, the shear wave was more reliable than the P-wave for monitoring the setting process of cementitious materials. Reinhardt and Grosse [15] developed a device producing ultrasound waves, in order to continuously monitor the setting and hardening of cementitious materials. It was found that the beginning of setting could be determined from the relationship between velocity and the age of mortar, using a mathematical procedure. However, the final setting still relied on empirical experience. This method was considered to be applicable for concrete, gypsum, lime, starch, and other stiffening materials. It could also be used for quality control in the production of admixtures, as well as new binders, and for control of the consistency of concrete production.

In spite of its aforementioned advantages, however, cement production requires high energy input, leading to environmental concerns [16,17]. Hence, a new alternative building material is required for civil engineering works. As such, metakaolin, which is basically made from kaolin (a type of clay) treated using a high calcination temperature [18,19], is considered to be one of the possible replacement materials. In spite of the calcination process necessary in metakaolin production, Sullivan et al. [20], who reviewed the study by Selmani et al. [21], stated that the manufacture of metakaolin involved much lower calcining temperatures and emitted less carbon dioxide than Portland cement. Therefore, metakaolin is gaining popularity in various construction applications, as mentioned in other studies [22,23]. As one example of its applications, Attanasio et al. [24] used metakaolin, fly ash and furnace slag to produce alkali-activated mortars, in order to study the effect of binder compositions by focusing on workability and compressive strength. It was revealed that such a cement-free binder had the potential for using it as masonry mortar. Nas and Kurbetci [25] investigated the durability properties of concrete containing metakaolin. It was found that the optimum metakaolin substitution ratio was 20%. Moreover, the use of metakaolin in concrete improved its durability properties, such as sorptivity, permeability, and durability.

Another application of metakaolin is its use in producing geopolymer, which was considered as one of the possible products due to its advantages in economic and environmental terms, as well as its rapid strength development, etc. [26]. It is observed that although the geopolymer can fundamentally be made using different materials, such as fly ash, limestone powder, rice husk ash, water treatment plant sludge, blast-furnace waste, silica fume, etc., much of the research still focuses on metakaolin geopolymer due to its availability in many places. As an example, Elimbi et al. [27] produced geopolymer from a dehydroxylated form of natural kaolin with a calcination temperature of between 450 and 800 °C. Accordingly, it was reported that the most suitable temperature for kaolin calcination was around 700 °C. Alaskar et al. [28] stated that the metakaolin geopolymer

could be activated by heating clay in a furnace chamber at 600, 700, and 800 °C for 1, 2, and 3 h, respectively, or at 900 and 1000 °C for 1 and 2 h, respectively. Moreover, the maximum compressive strength in terms of calcination was found at 700 °C for 1 h. Abadel et al. [29] studied the strength, microstructure, and embodied energy of geopolymer mixtures by varying the ratio of the alkali solids to metakaolin from 0.1 to 0.5, as well as varying the ratio of sodium silicate to NaOH from 0.2 to 1.0. They found that when manufacturing a metakaolin geopolymer with a strength of 90 MPa, the molar ratio of silica to alumina should be greater than 2.3. Moreover, the ratio of sodium oxide to alumina should be between 0.6 and 1.2, and the ratio of water to sodium oxide should not exceed 12. Palumbo et al. [30] used fiber Bragg grating sensors to characterize the early-age curing and shrinkage of metakaolin geopolymer. It was found that their method could accurately control the early-age phases of the metakaolin geopolymer. Apart from these studies, metakaolin geopolymer can further be applied to civil engineering works, such as a low-cost and self-bearing thermal insulating core for thermostructural sandwich panels [31], cultural heritage restoration [32], and as a potential binder for granulation zeolites [33,34]. From these applications, it can be observed that various research topics in metakaolin geopolymer are still ongoing.

According to the aforementioned review, it can be observed that bender element testing has been applied to various research areas, in particular, to the setting time of materials. However, there is no previous research that has applied the bender element test to determine the setting times of metakaolin geopolymer pastes. In order to bridge this gap, this study proposes to use the piezoelectric bender element to investigate the initial and final setting times of metakaolin geopolymer pastes. For the experiment, metakaolin powder was prepared by treating kaolin at various temperatures of between 0 and 1200 °C. The metakaolin powder was then mixed with geopolymer solution at various mixing ratios. In addition, the geopolymer solution was prepared by adding 10 normal sodium hydroxide (10 N NaOH) concentrations to sodium silicate (Na_2SiO_3) at various solution ratios. Finally, the results from the bender element test, in terms of the setting times of metakaolin geopolymer pastes, were compared to those from the Vicat needle test, whereby the optimum mixing and solution ratios could be established. Moreover, the obtained optimum ratio is used to produce metakaolin geopolymer pastes for mixing with laterite soil samples. After this, the California bearing ratio (CBR) test was used for comparing laterite soils mixed with metakaolin geopolymer to control laterite soils.

2. Materials and Methods

2.1. Metakaolin Geopolymer

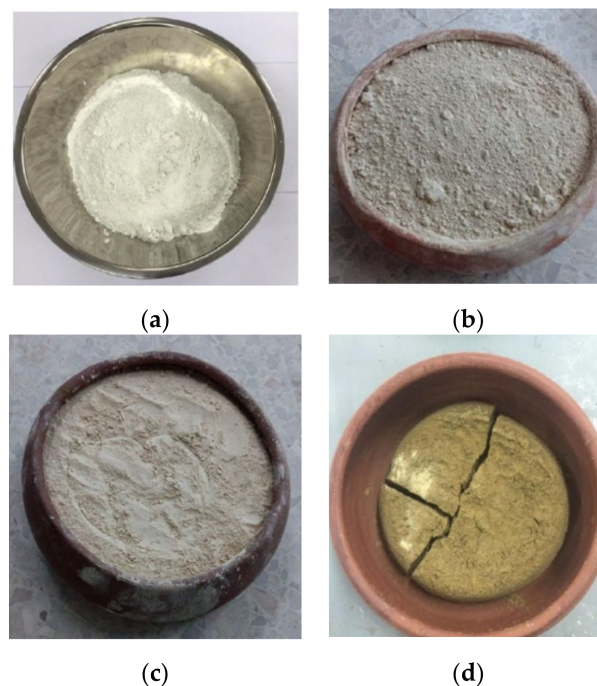
This study used kaolin with the chemical composition and basic engineering properties shown in Tables 1 and 2, respectively. Here, the particles of kaolin appeared to be very fine and passed easily through a number 400 ASTM mesh (38 μm). For control samples, the kaolin was not thermally treated and was designated as 0 °C. For the metakaolin geopolymer paste samples, the kaolin was treated at different temperatures from 200 to 1200 °C to obtain metakaolin powder, as shown in Figure 1a–d, respectively. It was observed that the metakaolin that was treated at 1200 °C, as shown in Figure 1d, appears to be ceramic and cannot be mixed with geopolymer solution. Therefore, it was not considered throughout the study. After thermal treatment, the metakaolin powder that was treated at 200 to 1000 °C was then mixed with a geopolymer solution. The mixing ratios between the metakaolin powder and the geopolymer solution were selected as 0.8:1.0, 1.0:1.0, 1.2:1.0, and 1.5:1.0. The geopolymer solution was prepared by adding 10 normal concentrations of sodium hydroxide (10 N NaOH) to the sodium silicate (Na_2SiO_3) at various solution ratios of 1.0:1.0, 1.0:1.2, 1.0:1.5, 1.0:2.0, 1.2:1.0, 1.5:1.0, and 2.0:1.0.

Table 1. Chemical compositions of kaolin.

Chemical Composition	Content (%)
SiO ₂	65–70
Al ₂ O ₃	17–20
K ₂ O	4.20
Na ₂ O	3.30
MgO	1.66
Fe ₂ O ₃	<1.60
TiO ₂	0.10
CaO	0.07

Table 2. Basic engineering properties of kaolin.

Properties	Unit
Moisture	1.0% max.
Particle sizes that pass through mesh No. 400 (38 µm)	100%
Finer than 2 microns	30–40%
pH	6.5 ± 1
Oil absorption	37 ± 2 gm/100 gm
Brightness	80 ± 2%
Liquid limit	46.84%
Plastic limit	34.19%
Plastic index	12.65%
Total unit weight	2.36 T/m ³

**Figure 1.** Kaolin after thermal treatment at various temperatures: (a) 200 °C; (b) 800 °C; (c) 1000 °C; (d) 1200 °C.

2.2. Piezoelectric Transducers

The piezoelectric ceramic transducer used here is a bimorph-type vibrator, fabricated by the Fuji ceramics corporation. Basically, this transducer is connected with two pieces of piezoelectric ceramic. When one piece is extended, another piece is compressed. Using this mechanism, the transducer can vibrate and send shear waves. For this transducer, there are two different connection methods, series and parallel types, as shown in Figure 2a,b,

respectively. The series type can be used for the highly sensitive sensing of a faint signal, while the parallel type can be used as an actuator due to its large displacement at a low voltage. Regarding these two types, further explanations will follow later.

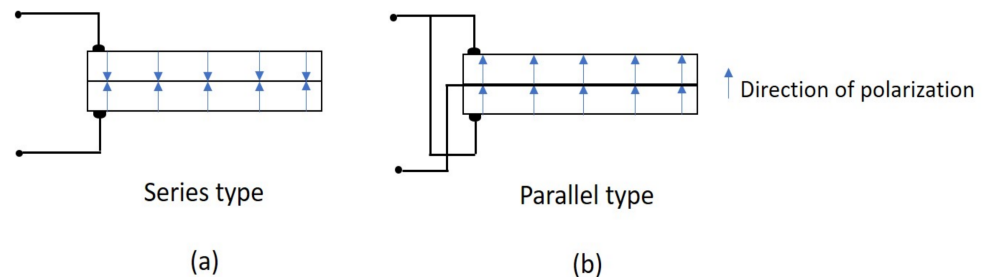


Figure 2. Piezoelectric ceramic transducers: (a) series type; (b) parallel type.

2.3. Shear Wave Velocity Measurement

Working according to the ASTM D8295–19 (the standard test method for the determination of shear wave velocity and initial shear modulus in soil specimens using bender elements) [35], the shear wave is generated here by a function generator with a sinusoidal waveform, with a frequency of 4–10 kHz and an amplitude of 20 V_{p-p}. After generation, the shear wave is sent to the transmitter of the bender element, in combination with measuring the sending shear wave via channel 1 of the oscilloscope, as shown in Figure 3. For this sending transmitter, a parallel-type connection is used due to its large deformation and strong sending signal. After leaving the sending transmitter, the shear wave travels through the metakaolin geopolymer paste within the reactor (in a crystal-clear plastic container) as shown in Figure 3. At the receiving bender element on the opposite side, a series-type connection is used due to its high sensitivity. Moreover, the receiving shear wave is detected via channel 2 of the oscilloscope, as shown.

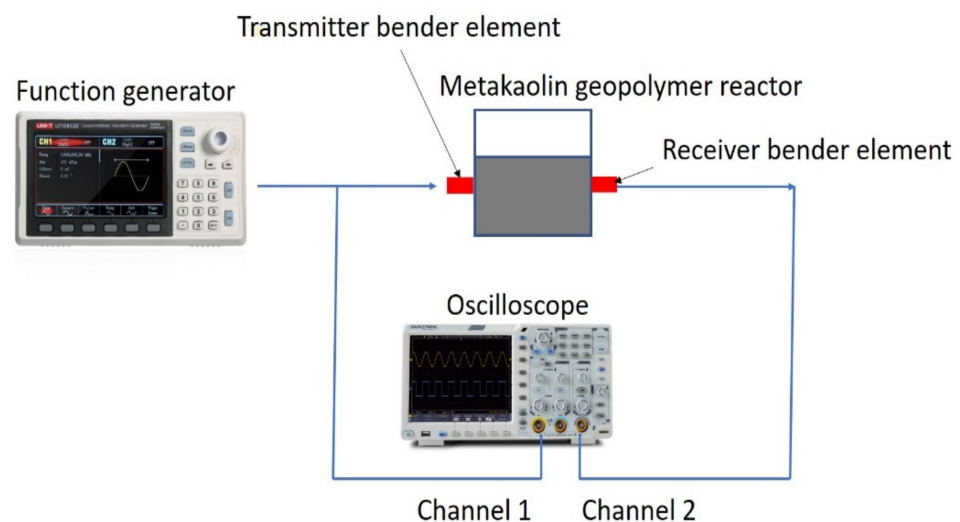


Figure 3. Bender element test setup.

Figure 4 shows an example of sending and receiving shear wave signals, and its offset time is known as shear wave travel time, T_s (s). This offset time is used to calculate the shear wave velocity v_s (m/s), as follows:

$$v_s = \frac{L}{(T_s - T_c)} \quad (1)$$

where L is defined as the length of the metakaolin geopolymer specimen measured between two bender element transducers (m), whereas T_c is defined as the time delay correction (s).

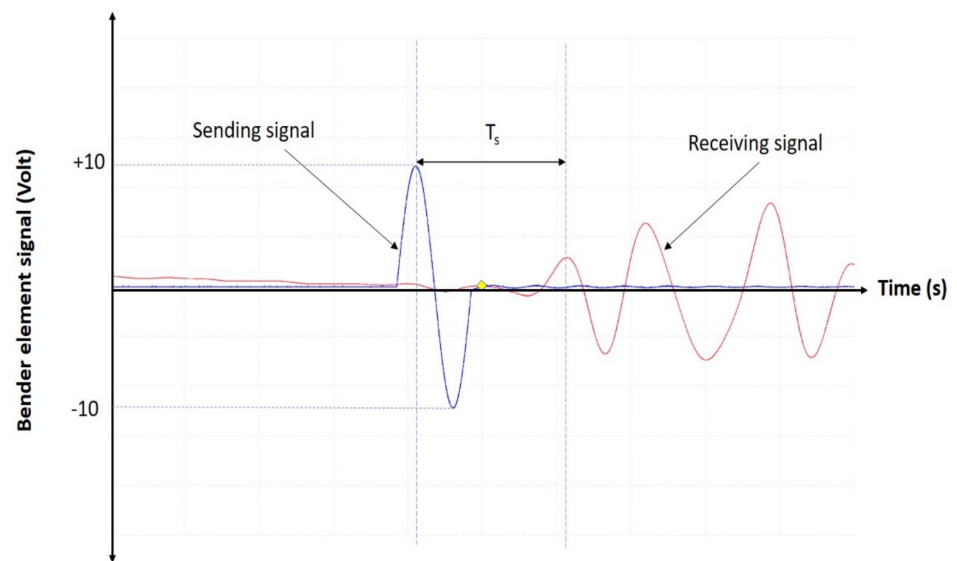


Figure 4. Example of sending and receiving shear waves.

2.4. Vicat Needle

According to ASTM C191-08 (standard test methods for measuring the setting time of hydraulic cement with a Vicat needle), the Vicat needle test apparatus [36] in Figure 5 can be used to determine both the initial and the final setting times of metakaolin geopolymer pastes. In this study, the initial setting time is judged as a Vicat penetration of 25 mm, while the final setting time is judged to be whenever there is no occurrence of Vicat penetration.



Figure 5. Vicat needle test apparatus.

2.5. Laterite Soil

For this study, a set of laterite soil samples were taken from the Bo Phloi District, Kanchanaburi, Thailand. These were classified as sandy clay (CL) according to the unified system, or A-4 based on the AASHTO system. Table 3 shows the soil engineering properties of the laterite soil samples. These laterite soil samples were used in the CBR tests in accordance with ASTM D1883-99. For these tests, 3 different mixing methods were carried out, i.e., A, B, and C. For mixing method A, the laterite soil was blended together 5 separate times with a mixture of metakaolin and geopolymer solution, then each of these 5 mixtures was assembled layer by layer. For mixing method B, the laterite soil and the metakaolin were first mixed together, then the geopolymer solution was subsequently added. For mixing method C, both the metakaolin and the geopolymer solution were first mixed together, then the laterite soil was mixed in later. It is noteworthy that in these tests, the solution ratio between NaOH and Na₂SiO₃, as well as the mixing ratio between the metakaolin and the geopolymer solution, were selected based on the results of the bender element tests.

Table 3. Basic engineering properties of the laterite soil samples.

Properties	Unit
Liquid Limit, LL	20%
Plastic Limit, PL	13.53%
Plastic Index, PI	6.47%
Specific gravity, G _s	2.76
Maximum dry unit weight	2.069 T/m ³
Optimum moisture content	10.17%

3. Results and Discussions

3.1. Effect of Thermal Treatment on Metakaolin

Figure 6 shows the relationship between the shear wave velocity, V_s , through metakaolin geopolymer pastes measured for 180 min and the solution ratio of NaOH and Na₂SiO₃, measured at various mixing ratios of metakaolin powder and geopolymer solution. Figure 6a shows the scenario of the control sample with no thermal treatment, i.e., 0 °C. It was found that the maximum V_s is about 147 m/s when the solution ratio between NaOH and Na₂SiO₃ is equal to 2.0:1.0, as well as a mixing ratio between the metakaolin powder and the geopolymer solution equal to 1.0:1.0, 1.2:1.0, and 1.5:1.0. With the same solution ratio and the same mixing ratio, Figure 6b,c show that the maximum V_s is about 147–175 m/s for metakaolin treated at 200 °C, and about 183–235 m/s for metakaolin treated at 800 °C, respectively. Figure 6d shows the scenario of metakaolin when it is treated at 1000 °C. It can be seen that V_s reaches a maximum at 360 m/s, when the solution ratio is equal to 2.0:1.0 and the mixing ratio is equal to 1.0:1.0. However, it is clear that V_s at a solution ratio of 1.2:1.0 and 1.5:1.0 is not available when the temperature treatment is at 1000 °C, because geopolymerization was found to be so rapid that the setting time could not be measured. This is one of the significant limitations in utilizing the metakaolin geopolymer in real-life civil engineering work. Again, the metakaolin sample treated at 1200 °C was not considered as a variable throughout this study because the sample turned into a ceramic after thermal treatment, meaning that it was impossible to mix it with the geopolymer solution, as mentioned in Section 2.1.

Figure 7 shows the relationship between the shear wave velocity, V_s , and the elapsed time of bender element testing on metakaolin geopolymer pastes at different treatment temperatures. For comparison purposes, the solution ratio between NaOH and Na₂SiO₃ was measured at 2.0:1.0, while the mixing ratio between the metakaolin powder and the geopolymer solution was measured at 1.0:1.0. It was observed that V_s increased with the passage of time. When using metakaolin treated at 0 and 200 °C, the V_s of both is identical and increases from about 115 m/s to 146 m/s after 180 min of testing, showing no significant geopolymerization in terms of V_s . For metakaolin treatment at 800 °C, V_s increases from 122 to 183 m/s, or by 50%. However, for metakaolin treatment at 1000 °C,

the V_s drastically increases from 138 to 361 m/s, or by 162%. Therefore, the metakaolin treatment temperature at 1000 °C is considered to be optimum in this study.

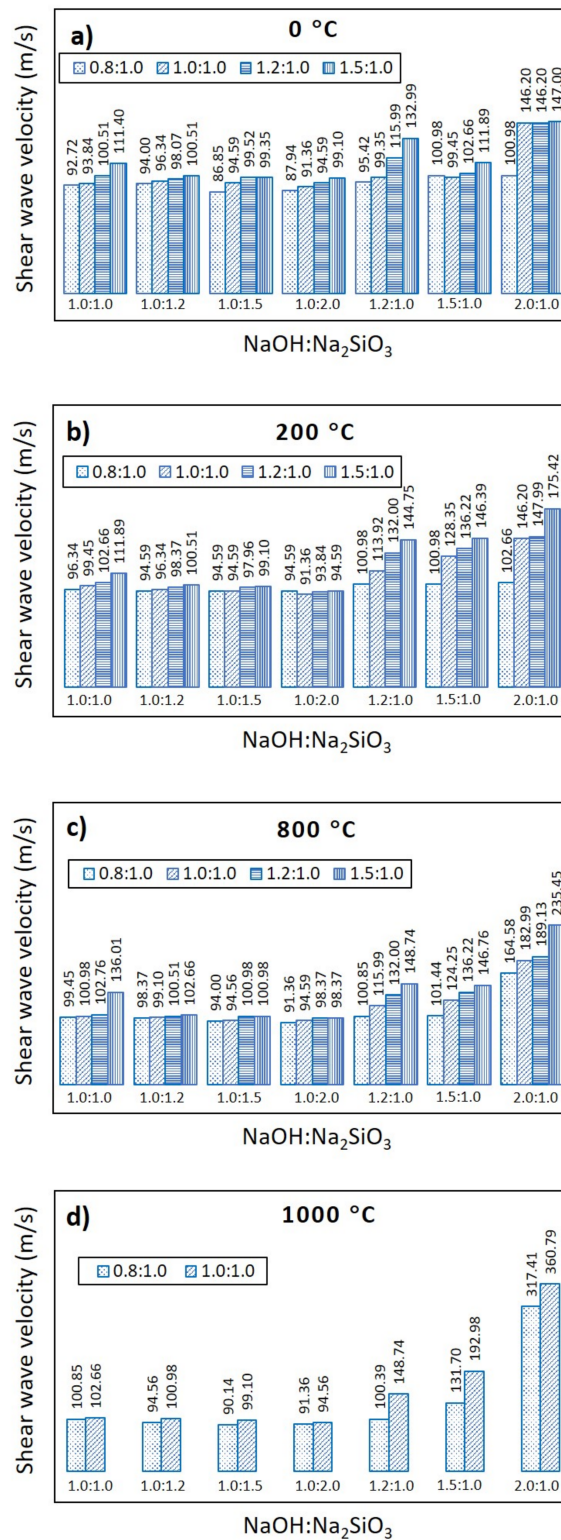


Figure 6. Shear wave velocity vs. NaOH:Na₂SiO₃, with various ratios of metakaolin powder and geopolymer solution and various metakaolin treatment temperatures: (a) 0 °C; (b) 200 °C; (c) 800 °C; (d) 1000 °C.

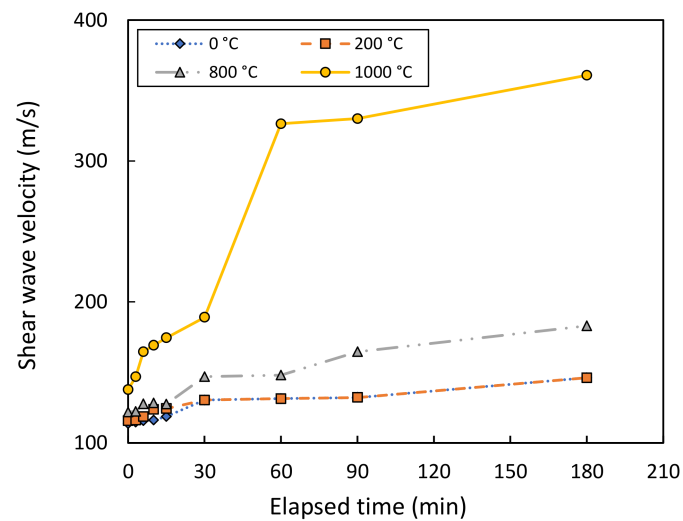


Figure 7. Shear wave velocity vs. the elapsed time of bender element testing, measured at different treatment temperatures.

Figure 8 shows plots between Vicat penetration through metakaolin geopolymer pastes and the elapsed time, as the solution ratio and the mixing ratio were assumed to be 2.0:1.0 and 1.0:1.0, respectively. From the plot, it can be seen that the initial setting time for a 25-mm penetration of the Vicat apparatus was faster with a higher metakaolin treatment temperature. For the control sample designated as 0 °C, the initial setting time of metakaolin geopolymer pastes was equal to 123 min. Using the metakaolin treated at 200, 800, and 1000 °C, the initial setting time became faster at 71, 48, and 12 min, respectively. As measured until there was no penetration of the Vicat needle, the final setting time for metakaolin treated at 1000 °C was about 30 min, whereas that at other temperatures was about 180 min or more. This seems to agree with the test results of V_s , as shown in Figure 7, where a temperature of 1000 °C was considered to be the optimum metakaolin treatment temperature.

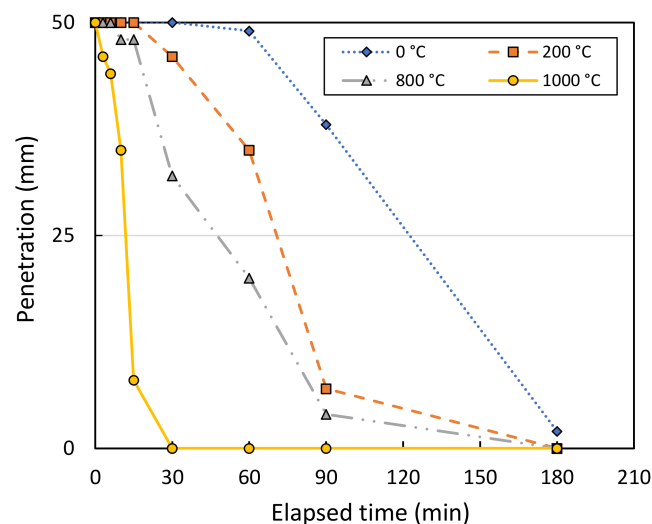


Figure 8. Penetration vs. the elapsed time of Vicat needle testing, measured at different treatment temperatures.

3.2. Effect of the Ratio of NaOH: Na_2SiO_3 for Preparing the Geopolymer Solution

The effect of varying the solution ratio between NaOH and Na_2SiO_3 on shear wave velocity, V_s , and the Vicat penetration can be investigated. For comparison purposes, this study assumes the mixing ratio between metakaolin treated at 1000 °C and the geopolymer

solution to be 1.0:1.0. Figure 9 plots the relationship between V_s and the elapsed time of bender element testing on metakaolin geopolymer pastes. It can be seen that V_s remains constant when the solution ratio is equal to 1.0:1.0, 1.0:1.2, 1.0:1.5 and 1.0:2.0. This means that there is no occurrence of geopolymerization. However, the geopolymerization occurs when the solution ratio is equal to 1.2:1.0, 1.5:1.0, and 2.0:1.0. Moreover, the V_s increases from 131 to 149 m/s and 136 to 193 m/s when the solution ratio is equal to 1.2:1.0 and 1.5:1.0, respectively. At a solution ratio of 2.0:1.0, V_s increases from 138 m/s to its maximum at 361 m/s. Hence, the optimum solution ratio between NaOH and Na_2SiO_3 is recommended here as being 2.0:1.0. Furthermore, Figure 10 presents the Vicat needle test results for metakaolin geopolymer pastes mixed at various solution ratios. It is clear that Vicat penetration is equal to 50 mm after 180-min testing for a solution ratio of 1.0:1.0, 1.0:1.2, 1.0:1.5, and 1.0:2.0. This shows that there is no setting of the metakaolin geopolymer pastes, meaning that there is no occurrence of geopolymerization. However, geopolymerization occurs only in the case of a solution ratio at 1.2:1.0, 1.5:1.0, and 2.0:1.0, since the initial setting time is equal to 8, 8, and 12 min, respectively. Moreover, the corresponding final setting time is equal to 15, 15, and 30 min, respectively.

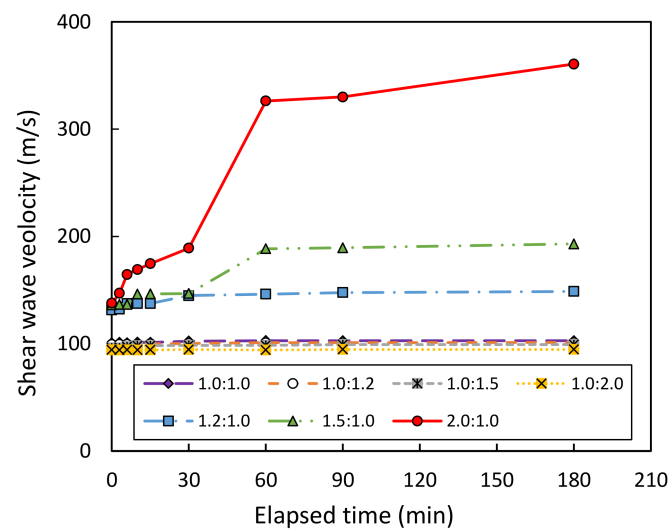


Figure 9. Shear wave velocity vs. elapsed time of bender element testing for various ratios of NaOH to Na_2SiO_3 .

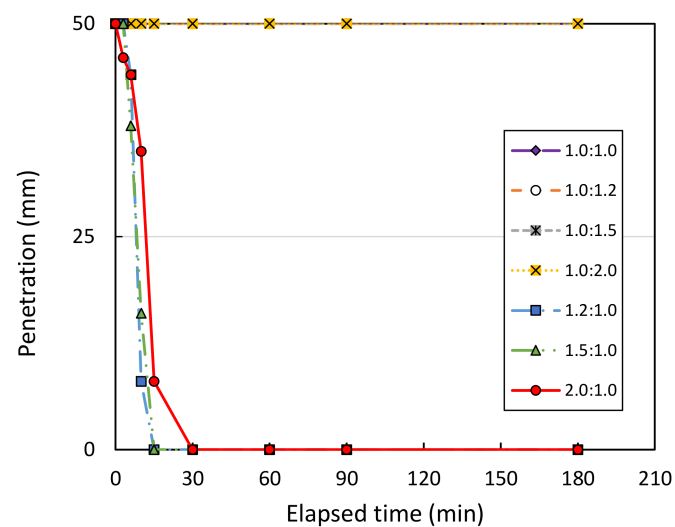


Figure 10. Penetration vs. elapsed time of Vicat needle testing for various ratios of NaOH to Na_2SiO_3 .

3.3. Effect of Mixed Proportions between the Metakaolin and the Geopolymer Solution

The relationship between the shear wave velocity, V_s , and the elapsed time of bender element testing, as well as between the penetration and the elapsed time of Vicat testing, can be used to determine the effect of varying the mixing ratio between the metakaolin powder and the geopolymer solution. For comparison purposes, this study assumes the solution ratio to be between NaOH and Na_2SiO_3 at 2.0:1.0 in combination with the metakaolin treated at 800 °C and 1000 °C. Figure 11 shows that V_s tends to increase with elapsed time in the case of the metakaolin treated at 800 °C. Moreover, the V_s at 180 min of bender element testing was equal to 165, 183, 189, and 235 m/s, when the mixing ratio is equal to 0.8:1.0, 1.0:1.0, 1.2:1.0, and 1.5:1.0, respectively. Hence, the mixing ratio of 1.5:1.0 is considered to be optimum in this study due to the maximum V_s . In the same way, Figure 12 reports the relationship between V_s and the elapsed time of bender element testing on metakaolin treated at 1000 °C. Only the mixing ratio at 0.8:1.0 and 1.0:1.0 is measured, because the metakaolin mixed using the other ratios could not be tested due to rapid drying, as mentioned in Section 2.1. It can be seen that the V_s for a mixing ratio of 1.0:1.0 is higher than that of 0.8:1.0. For example, the V_s for a mixing ratio of 1.0:1.0 at 180 min of bender element testing is equal to 361 m/s, while that of 0.8:1.0 is equal to 317 m/s. Hence, the mixing ratio of 1.0:1.0 is considered to be optimum, according to its maximum V_s .

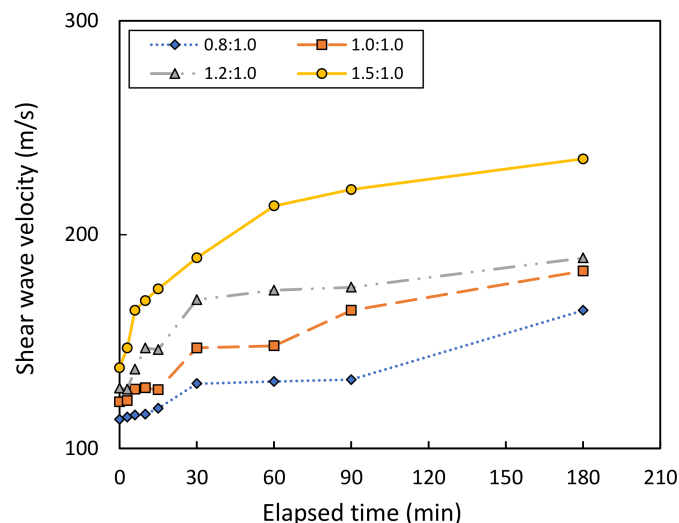


Figure 11. Shear wave velocity vs. the elapsed time of bender element testing for metakaolin treated at 800 °C, using different mixing ratios.

Figure 13 shows the Vicat needle test results on metakaolin geopolymer pastes with various mixing ratios, between metakaolin treated at 800 °C and the geopolymer solution. Note that the solution ratio between NaOH and Na_2SiO_3 is assumed to be 2.0:1.0. The initial setting time of metakaolin geopolymer pastes at 25-mm penetration is equal to 123, 48, 23, and 12 min for mixing ratios of 0.8:1.0, 1.0:1.0, 1.2:1.0, and 1.5:1.0, respectively. However, the final setting time at which there is no penetration is equal to 185, 180, 90, and 30 min for the mixing ratios of 0.8:1.0, 1.0:1.0, 1.2:1.0, and 1.5:1.0, respectively. Figure 14 shows the Vicat needle test results on metakaolin geopolymer pastes with various mixing ratios, between metakaolin treated at 1000 °C and a geopolymer solution. The initial setting time is equal to 12 min for the mixing ratio of 0.8:1.0, and 1.0:1.0. However, the final setting time is equal to 60 and 30 min for mixing ratios of 0.8:1.0 and 1.0:1.0, respectively. Hence, this finding confirms that a mixing ratio between the metakaolin powder and the geopolymer solution of 1.0:1.0 can be considered optimum for metakaolin treated at 1000 °C.

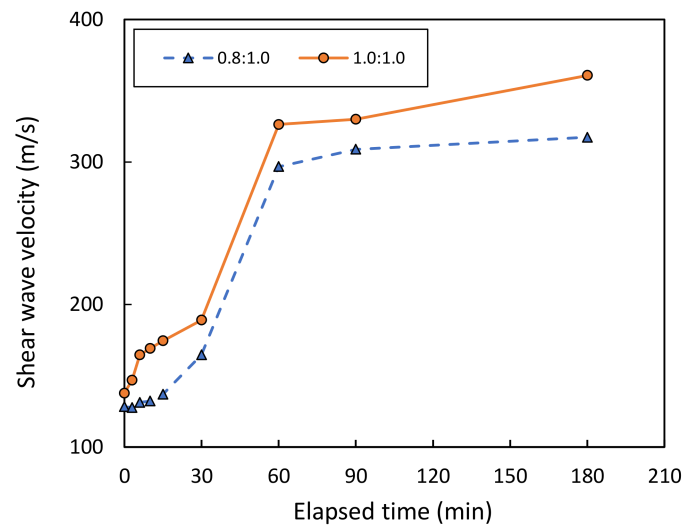


Figure 12. Shear wave velocity vs. the elapsed time of bender element testing for metakaolin treated at 1000 °C, using different mixing ratios.

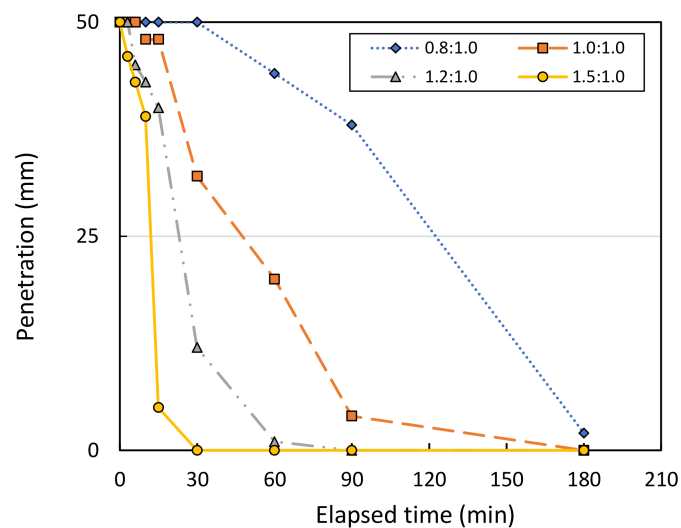


Figure 13. Penetration vs. the elapsed time of Vicat needle testing for metakaolin treated at 800 °C, using different mixing ratios.

3.4. Relationship between Shear Wave Velocity and Setting Times

Figure 15 shows the relationship between the shear wave velocity, V_s , established via the bender element tests and the penetration through metakaolin geopolymer pastes established via the Vicat tests. For comparison purposes, this study assumes the metakaolin treated at 1000 °C, and the mixing ratio between the metakaolin powder and the geopolymer solution, to be 1.0:1.0. By varying the solution ratio between NaOH and Na_2SiO_3 from 1.2:1.0 to 2.0:1.0, the relationship between V_s and penetration appears to be linear. The initial setting time, set at 25-mm penetration, can be calculated as equal to 136, 142, and 168 m/s for solution ratios of 1.2:1.0, 1.5:1.0, and 2.0:1.0, respectively. Moreover, the final setting time when there is no penetration can also be calculated as equal to 138, 148, and 187 m/s for solution ratios of 1.2:1.0, 1.5:1.0, and 2.0:1.0, respectively. From these results, it can be established that the V_s of metakaolin geopolymer pastes reaches the maximum value when the solution ratio between NaOH and Na_2SiO_3 is equal to 2.0:1.0. Hence, a solution ratio of 2.0:1.0 is considered to be optimum in this study.

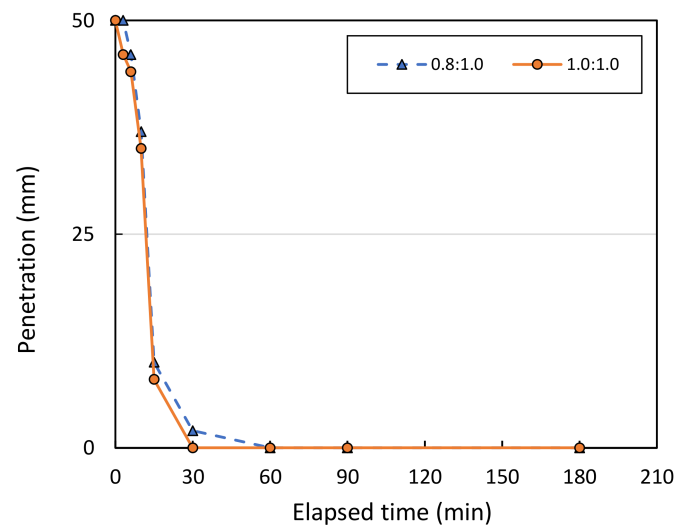


Figure 14. Penetration vs. the elapsed time of Vicat needle testing for metakaolin treated at 1000 °C, using different mixing ratios.

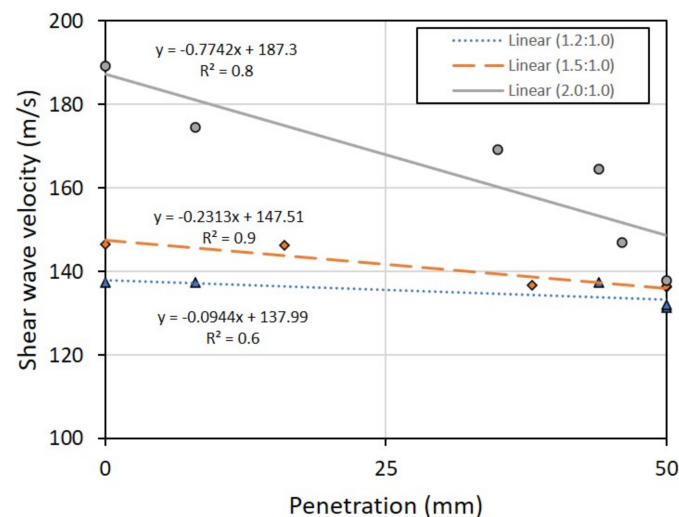


Figure 15. Shear wave velocity from bender element tests vs. penetration from Vicat tests.

3.5. Effect of CBR on Mixing Methods for Laterite

Figure 16 shows the CBR test results on laterite soils mixed with metakaolin geopolymer. For comparison, this study assumes the solution ratio between NaOH and Na₂SiO₃ to be 2.0:1.0, and the mixing ratio between the metakaolin treated at 1000 °C and the geopolymer solution to be 1.0:1.0. Figure 16a shows the unsoaked CBR test results. The control laterite soil sample, which was compacted at an optimum moisture content of 10.17%, showed unsoaked CBR test results of 20.07% at 0.1 inches of penetration, as well as 21.02% at 0.2 inches of penetration. Mixing method A showed unsoaked CBR test results of 34.04% at 0.1 inches, as well as 45.38% at 0.2 inches of penetration. Moreover, mixing method B yielded the maximum unsoaked CBR test results of 59.01% at 0.1 inches, as well as 65.05% at 0.2 inches of penetration. Finally, mixing method C presented unsoaked CBR test results of 49.50% at 0.1 inches, as well as 61.86% at 0.2 inches of penetration. From these results, mixing method B is considered to show the best performance in the unsoaked CBR tests, and its CBR values were about 3 times higher than those of the control sample. Figure 16b shows the soaked CBR test results. The control laterite soil sample, which was compacted at an optimum moisture content of 10.17%, showed soaked CBR test results of 8.16% at 0.1 inches, as well as 9.24% at 0.2 inches of penetration. It is clear that mixing

method A gave the maximum soaked CBR test results of 18.50% at 0.1 inches, as well as 24.11% at 0.2 inches of penetration. Moreover, its CBR values were about 3 times higher than those of the control sample. Figure 16c shows the swelling CBR test results. The control laterite sample, which was compacted at the optimum moisture content of 10.17%, showed swelling CBR test results of 0.0036%. It can be observed that method B provides the best swelling CBR test results of 0.41%.

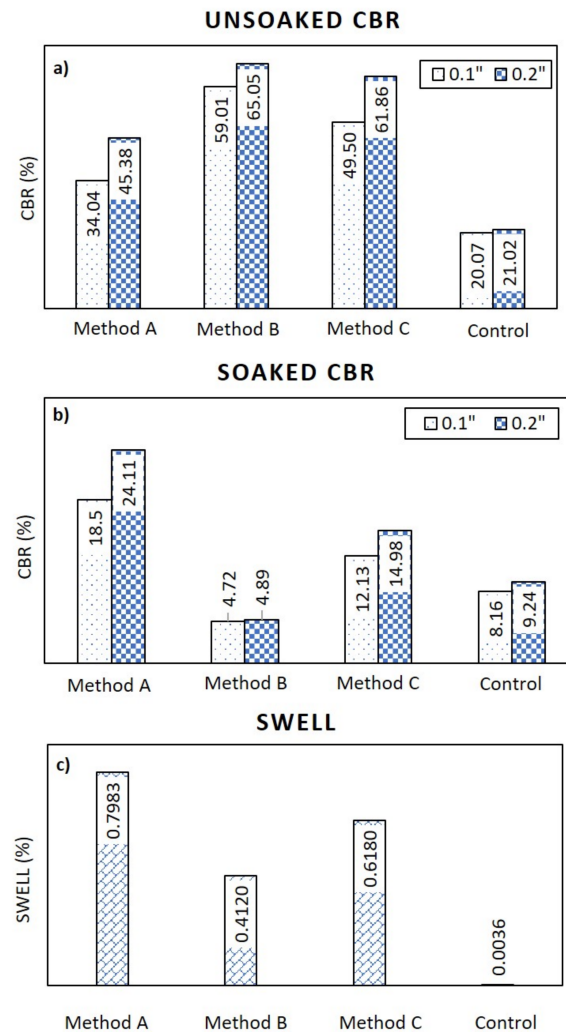


Figure 16. CBR test results for different mixing methods and conditions: (a) unsoaked (b) soaked (c) swelling.

4. Conclusions

This study uses piezoelectric ceramic sensors in combination with bender element tests to determine the initial and final setting times of metakaolin geopolymer pastes. These setting times are investigated via the shear wave velocity from the bender element and are compared to the penetration tests using the Vicat needle apparatus. Metakaolin was prepared from kaolin, with thermal treatment at 0 (control sample), 200, 800, 1000, and 1200 °C. These metakaolin samples were then mixed with the geopolymer solution in various ratios. Moreover, the geopolymer solution was prepared by adding 10 normal concentrations of sodium hydroxide (10 N NaOH) to sodium silicate (Na_2SiO_3) at various ratios. From the findings of this study, it can be summarized that:

- By varying the treatment temperature, the solution ratio between sodium hydroxide (NaOH) and sodium silicate (Na_2SiO_3), as well as the mixing ratio between the metakaolin powder and the geopolymer solution, their optimum value can be investi-

gated. In other words, the optimum treatment temperature in producing metakaolin is herein found to be 1000 °C; the optimum solution ratio and the optimum mixing ratio were found to be 2.0:1.0 and 1.0:1.0, respectively.

- Assuming solution ratios between NaOH and Na₂SiO₃ to be 1.2:1.0, 1.5:1.0, and 2.0:1.0, the relationships between shear wave velocity and Vicat penetration appear to be linear, for a mixing ratio between the metakaolin treated at 1000 °C and the geopolymer solution of 1.0:1.0. According to these three solution ratios, the initial setting time was found to be 136, 142, and 168 m/s, respectively, but the final setting time was found to be 138, 148, and 187 m/s, respectively. Moreover, the optimum solution ratio was found to be 2.0:1.0, according to the maximum shear wave velocity.
- Assuming the solution ratio between NaOH and Na₂SiO₃ to be 2.0:1.0, as well as assuming the mixing ratio between metakaolin treated at 1000 °C and the geopolymer solution to be 1.0:1.0, CBR tests can be conducted on laterite soils mixed with metakaolin geopolymer and on control laterite soil samples. From the tests, mixing method B is found to be the best for the unsoaked CBR test; its CBR values were about 3 times higher than the control sample. For the soaked CBR test, mixing method A was found to be the best because its CBR values were about 3 times higher than the control sample. However, mixing method B was found to be the best for the swelling CBR tests as it demonstrated the best CBR test results.
- For further study, there are two recommendations. First, a study that extensively focuses on the strength development of metakaolin geopolymer pastes should be conducted, although the current study focuses on the shear wave velocity, which might imply their strength. Second, the use of scanning electron microscopy (SEM) testing was also recommended, in order to further observe the microstructure of metakaolin geopolymer pastes.

Author Contributions: Conceptualization, K.P.; methodology, K.P.; validation, K.P. and J.I.; investigation, K.P. and J.I.; resources, K.P. and J.I.; data curation, K.P.; writing—original draft preparation, K.P. and A.P.; writing—review and editing, K.P. and A.P.; visualization, K.P. and A.P.; supervision, K.P. and A.P.; project administration, K.P. and A.P.; funding acquisition, A.P. All authors have read and agreed to the published version of the manuscript.

Funding: This research was funded by the National Science, Research and Innovation Fund (NSRF), and King Mongkut's University of Technology North Bangkok, with grant number KMUTNB-FF-65-30.

Institutional Review Board Statement: Not applicable.

Informed Consent Statement: Not applicable.

Conflicts of Interest: The authors declare no conflict of interest. Moreover, the funders had no role in the design of the study; in the collection, analyses, or interpretation of data; in the writing of the manuscript, or in the decision to publish the results.

References

1. Iamchaturapatr, J.; Piriyaikul, K.; Ketklin, T.; Di Emidio, G.; Petcherdchoo, A. Sandy soil improvement using MICP-based urease enzymatic acceleration method monitored by real-time system. In *Advances in Materials Science and Engineering*; Elsevier: Amsterdam, The Netherlands, 2021. [\[CrossRef\]](#)
2. Shirley, D.J.; Hampton, L.D. Shear-wave measurement in laboratory sediments. *J. Acoust. Soc. Am.* **1978**, *63*, 607–613. [\[CrossRef\]](#)
3. Brignoli, E.G.M.; Gotti, M.; Stokoe, K.H. Measurement of shear waves in laboratory specimens by means of piezoelectric transducers. *Geotech. Test. J.* **1996**, *19*, 384–397.
4. Piriyaikul, K. A development of a bender element apparatus. *J. KMUTNB.* **2010**, *20*, 363–369.
5. Kim, D.; Yu, J.; Byun, Y. Piezoelectric ring bender for characterization of shear waves in compacted sandy soils. *Sensors* **2021**, *21*, 1226. [\[CrossRef\]](#) [\[PubMed\]](#)
6. Lee, J.; Park, G.; Byun, Y.; Lee, C. Modified fixed wall oedometer when considering stress dependence of elastic wave velocities. *Sensors* **2020**, *20*, 6291. [\[CrossRef\]](#)
7. Kim, J.; Lee, J.; Arnold, C.; Kim, S.Y. Evaluation of thawing and stress restoration method for artificial frozen sandy soils using sensors. *Sensors* **2021**, *21*, 1916. [\[CrossRef\]](#)

8. Park, J.; Lee, J.; Won, J.; Kim, J. Evaluation of small strain soil stiffness during freeze-thaw cycle: Transition from capillarity to cementation examined using magnetic and piezo crystal sensors. *Sensors* **2021**, *21*, 2992. [[CrossRef](#)]
9. Ahn, C.; Kim, D.; Byun, Y. Expansion-induced crack propagation in rocks monitored by using piezoelectric transducer. *Sensors* **2020**, *20*, 6054. [[CrossRef](#)]
10. Kim, S.Y.; Lee, J.; Park, J. Response of transitional mixtures retaining memory of in-situ overburden pressure monitored using electromagnetic and piezo crystal sensors. *Sensors* **2021**, *21*, 2570. [[CrossRef](#)]
11. Gabryś, K.; Sas, W.; Soból, E.; Gluchowski, A. Application of bender elements technique in testing of anthropogenic soil-recycled concrete aggregate and its mixture with rubber chips. *J. Appl. Sci.* **2017**, *7*, 741. [[CrossRef](#)]
12. Ju, T.; Achenbach, J.D.; Jacobs, L.J.; Guimaraes, M.; Qu, J. Ultrasonic nondestructive evaluation of alkali-silica reaction damage in concrete prism samples. *Mater. Struct.* **2016**, *50*, 60. [[CrossRef](#)]
13. Zhu, J.; Cao, J.N.; Bate, B.; Khayat, K.H. Determination of mortar setting times using shear wave velocity evaluation curves measured by the bender element technique. *Cem. Concr. Res.* **2018**, *106*, 1–11. [[CrossRef](#)]
14. Liu, S.; Zhu, J.; Seraj, S.; Cano, R.; Juenger, M. Monitoring setting and hardening process of mortar and concrete using ultrasonic shear waves. *Constr. Build. Mater.* **2014**, *72*, 248–255. [[CrossRef](#)]
15. Reinhardt, H.W.; Grosse, C.U. Continuous monitoring of setting and hardening of mortar and concrete. *J. Constr. Build. Mater.* **2004**, *18*, 145–154. [[CrossRef](#)]
16. Petcherdchoo, A. Sensitivity of service life extension and CO₂ emission due to repairs by silane treatment applied on concrete structures under time-dependent chloride attack. *Adv. Mater. Sci. Eng.* **2018**, *2018*, 2793481. [[CrossRef](#)]
17. Petcherdchoo, A. Probability-based sensitivity of service life of chloride-attacked concrete structures with multiple cover concrete repairs. *Adv. Civ. Eng.* **2018**, *2018*, 4525646. [[CrossRef](#)]
18. Piotr Rożek, O.B.; Florek, P.; Mozgawa, W.; Król, M. Alkaline activation of kaolin group minerals. *Crystals* **2020**, *10*, 268.
19. Petcherdchoo, A. Service life and environmental impact due to repairs by metakaolin concrete after chloride attack. *RILEM Bookser.* **2015**, *10*, 35–41.
20. Sullivan, M.S.; Chorzepa, M.G.; Hamid, H.; Durham, S.A.; Kim, S.S. Sustainable materials for transportation infrastructures: Comparison of three commercially-available metakaolin products in binary cementitious systems. *J. Infrastruct.* **2018**, *3*, 17. [[CrossRef](#)]
21. Selmani, S.; Sdiri, A.; Bouaziz, S.; Joussein, E.; Rossignol, S. Effects of metakaolin addition on geopolymer prepared from natural kaolinitic clay. *Appl. Clay Sci.* **2017**, *146*, 457–467. [[CrossRef](#)]
22. Vali, K.S.; Murugan, S.B. Effect of different binders on cold-bonded artificial lightweight aggregate properties. *J. Adv. Concr. Constr.* **2020**, *9*, 183–193.
23. Wang, X.; Lee, H. Microstructure modeling of carbonation of metakaolin blended concrete. *Adv. Concr. Constr.* **2019**, *7*, 167–174.
24. Attanasio, A.; Pascali, L.; Tarantino, V.; Arena, W.; Largo, A. Alkali-activated mortars for sustainable building solutions: Effect of binder composition on technical performance. *J. Environ.* **2018**, *5*, 35. [[CrossRef](#)]
25. Nas, M.; Kurbetci, Ş. Durability properties of concrete containing metakaolin. *J. Adv. Concr. Constr.* **2018**, *6*, 159–175.
26. Petcherdchoo, A.; Hongubon, T.; Thanasisathit, N.; Punthutaecha, K.; Jang, S.-H. Effect of curing time on bond strength between reinforcement and fly-ash geopolymer concrete. *Appl. Sci. Eng. Prog.* **2020**, *13*, 127–135. [[CrossRef](#)]
27. Elimbi, A.; Tchakoute, H.K.; Njopwouo, D. Effects of calcination temperature of kaolinite clays on the properties of geopolymer cements. *Constr. Build. Mater.* **2011**, *25*, 2805–2812. [[CrossRef](#)]
28. Alaskar, A.; Shah, S.N.R.; Keerio, M.A.; Phulpoto, J.A.; Baharom, S.; Assilzadeh, H.; Alyousef, R.; Alabduljabbar, H.; Mohamed, M. Development of pozzolanic material from clay. *J. Adv. Concr. Constr.* **2020**, *10*, 301–310.
29. Abadel, A.; Albidah, S.A.; Attheeb, A.; Alrshoudi, F.A.; Abbas, H.; Al-Salloum, Y.A. Effect of mortar ratios on strength, microstructure & embodied energy of metakaolin geopolymer. *J. Adv. Concr. Constr.* **2021**, *11*, 127–140.
30. Palumbo, G.; Iadicicco, A.; Messina, F.; Ferone, C.; Campopiano, S.; Cioffi, R.; Colangelo, F. Characterization of early age curing and shrinkage of metakaolin-based inorganic binders with different rheological behavior by fiber Bragg grating sensors. *Materials* **2018**, *11*, 10. [[CrossRef](#)]
31. Murri, A.N.; Medri, V.; Papa, E.; Laghi, L.; Mingazzini, C.; Landi, E. Porous geopolymer insulating core from a metakaolin/biomass ash composite. *Environments* **2017**, *4*, 86. [[CrossRef](#)]
32. Ricciotti, L.; Molino, A.J.; Roviello, V.; Chianese, E.; Cennamo, P.; Roviello, G. Geopolymer composite for potential applications in cultural heritage. *Environments* **2017**, *4*, 91. [[CrossRef](#)]
33. Vegere, K.; Vitola, L.; Argalis, P.P.; Bajare, D.; Krauklis, A.E. Alkali-activated metakaolin as a zeolite-like binder for the production of adsorbents. *Inorganics* **2019**, *7*, 141. [[CrossRef](#)]
34. Pereira, P.M.; Ferreira, B.F.; Oliveira, N.P.; Nassar, E.J.; Ciuffi, K.J.; Vicente, M.A.; Trujillano, R.; Rives, V.; Gil, A.; Korili, S.; et al. Synthesis of zeolite A from metakaolin and its application in the adsorption of cationic dyes. *Appl. Sci.* **2018**, *8*, 608. [[CrossRef](#)]
35. Piriyaikul, K.; Iamchaturapatr, J. Horizontally mounted bender elements for measuring shear modulus in soaked sand specimen. *Adv. Mater. Res.* **2014**, *931–932*, 496–500. [[CrossRef](#)]
36. Ylmén, R.; Jäglid, U.; Steenari, B.M.; Panas, I. Early hydration and setting of Portland cement monitored by IR, SEM and Vicat techniques. *Cem. Concr. Res.* **2008**, *38*, 1336–1342. [[CrossRef](#)]