

Effects of delay time and curing temperature on compressive strength and porosity of ground bottom ash geopolymer mortar

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Abstract

This research investigated development of ground bottom ash (GBA) for a new geopolymer material. The compressive strength and porosity of the geopolymer mortar containing GBA were tested. The GBA-based geopolymer mortar mixtures were designed by control liquid to GBA ratio for all tests. Extra water was also used to maintain the workability of geopolymer mortar. The sodium hydroxide (NaOH) concentrations of 10 M and 15 M were used. Furthermore, sodium silicate (Na2SiO3) to sodium hydroxide (NaOH) ratios by mass such as 0.50, 1.50 and 2.50 were also used as alkali activator solutions. Effects of delay time and no-delay time, heat curing as well as the concentrations of NaOH and Na2SiO3 to NaOH ratios on compressive strength and porosity of the GBA geopolymer mortar were investigated. It was found that the increasing of NaOH concentration and Na₂SiO₃ to NaOH ratio reduce the workability of GBA geopolymer mortar. The high concentration of NaOH at 15 Molar gave the compressive strength at 1 day higher than that of GBA geopolymer mortar with low NaOH of 10 M. Furthermore, the GBA geopolymer mortar with high Na₂SiO₃ to NaOH ratio at 2.5 gave the highest compressive strength. 15 M-2.5 Si/Na mortar with delay time before heat curing at 75°C gave the highest compressive strength of 23.0 MPa at 1 day. Moreover, the porosity of GBA geopolymer mortar was related to its compressive strength. The GBA binder is a new geopolymer material with good properties for concrete work.

1. Introduction

Many investigations have been directed toward the utilization of waste materials in mortar and concrete works. The waste materials from industrial and agricultural by-product such as fly ash, rice husk ash, bagasse ash, rice husk-bark ash, and palm oil fuel ash have used as supplementary cementitious materials for replacement of Portland cement, [1-6]. These ashes are pozzolanic materials that can be incorporated in mortar and concrete to enhance a good concrete property. The pozzolanic materials are the materials with silica or silica and alumina as the major chemical compositions [7,8]. The silica or silica and alumina can additionally react with the calcium hydroxide from the hydration reaction, resulting in improve strength as well as mortar and concrete properties. It is not only the improvement of a mechanical property but also durability especially porosity within the mortar and concrete [9,10]. Furthermore, it reduces the environmental problem, and reduces the landfill for disposed of the waste materials. In 2019, Thailand produced the Portland cement as much as 30.09 million tons. It is well known that the clinker manufacturing process in the cement industries are known to contribute to the greenhouse effect, through the release of CO_2 gas into the atmosphere: about 0.9 tons of CO_2 is released during the production of every ton of clinker [11]. To address these issues, this research aimed to use the bottom ash, which was waste material from power plant, as geopolymer material represent using Portland cement in mortar.

Bottom ash, disposed fly ash, is a waste product from coal combustion power plant. The bottom ash has been disposed of as waste to landfill. Many countries found that the bottom ash, waste material causes environmental problems such as air pollution and contamination of metals from the ashes to groundwater. Although many researches [12-14] studied the pozzolanic property of fly ash in concrete, it is little research for the bottom ash especially bottom ash-based geopolymer material. It was because large particles and low pozzolanic reactivity of the bottom ash resulted in low compressive strength in mortar and concrete. This was also confirmed by Jaturapitakkul and Cheerarot [15] who reported that ground disposed fly ash or ground bottom ash as small particles had very important factor to enhance the compressive strength of mortar. Several researches [16-18] developed the geopolymer materials by using silica and alumina activated in high alkali solution. Fly ash is a main pozzolan

source to produce the geopolymer material. In order to create the geopolymer material, the pozzolanic materials are activated by the alkali–silicate dissolution at high pH with the presence of soluble alkali metal silicate. The mortar and concrete made from the geopolymer material gave strength and durability similar to those from normal concrete that using Portland cement [19,20].

Therefore, this research aimed to study and develop the bottom ash for using a new material for geopolymer mortar without using Portland cement. However, the bottom ash has to be improved in its physical property by grinding as fine particles in order to increase its reactivity. The ground bottom ash (GBA) based geopolymer was activated with alkaline liquids, and then was cured under the several temperatures. To address the environmental problem from the disposed of bottom fly ash to landfill, the bottom ash-based geopolymer mortar is one of the options for concrete work without using Portland cement. This is not only reducing the environmental problem from the waste of industry but also increases the potential of using the bottom ash as industrialization in concrete work.

2. Materials and experimental program

2.1 Materials

The materials used in this study are mentioned as follows:

2.1.1 Bottom ash

Bottom ash from Mae Moh electricity power plant at Lampang province was used as a pozzolanic material. Ground bottom ash (GBA) was prepared by using ball mill grinding machine. The GBA was ground to fine particle until percentage of the particles retained on a sieve No. 325 (45 μ m) was 3% to 5% by weight. The fineness, specific gravity and median particle size (μ m) of GBA materials are given in Table 1. The Blaine fineness of GBA was 3,800 cm²·g⁻¹. The specific gravity of the GBA was 2.24. The particle size distribution of GBA is given in Figure 1. It was found that the median particle size of GBA was 5.5 μ m. The particle size distribution test used in this research consisted of Mercury intrusion porosimetry (MIP) from Thailand.



Figure 1. Particle size distribution of GBA.

Table 1. Physical properties of ground bottom ash (GBA).

Physical properties	Ground bottom ash (GBA)		
Median particle size (µm), d ₅₀	5.5		
Retained on a sieve no. 325 (%)	3.0-5.0		
Specific gravity	2.24		
Blaine fineness (cm ² ·gm ⁻¹)	3,800		

2.1.2 Natural sand (Fine aggregate)

A river sand with specific gravity of 2.45 and a fineness modulus of 2.52 in saturated surface dry condition was used in this research.

2.1.3 Alkali solutions

2.1.3.1 Sodium hydroxide (NaOH)

The concentrations at 10 M and 15 M of sodium hydroxide (NaOH) were prepared by mixing sodium hydroxide pellet with reverse osmosis water. Next, it was kept at room temperature for 24 h before use it as liquid for alkali activators solution. Sodium hydroxide used in this work consisted of commercial grade from Thailand.

2.1.3.2 Sodium silicate (Na₂SiO₃)

Sodium silicate (Na₂SiO₃) was also used as liquid for alkali activator solutions (Na₂O = 15.32%, SiO₂ = 32.87% and H₂O = 51.81% by mass). Sodium silicate used in this research also consisted of commercial grade from Thailand.

2.1.3.3 Sodium silicate (Na₂SiO₃) to sodium hydroxide (NaOH) ratios

In this work, sodium silicate (Na₂SiO₃) to sodium hydroxide (NaOH) ratios by mass at 0.50, 1.50, and 2.50 were used as alkali activator solutions.

2.2 Method of testing

2.2.1 Mix design

Geopolymer mortars were made from ground bottom ash (GBA) by mixing the GBA with the reiver sand at a ratio of 2.75. Sodium hydroxide (NaOH) of two concentrations, 10 M and 15 M were used, and the sodium silicate (Na₂SiO₃) to sodium hydroxide (NaOH) ratios by mass of 0.50, 1.50, and 2.50 were also used as alkali activator solutions. A liquid solution to GBA ratio ((Na2SiO3+NaOH)/ GBA) of 0.68 was used for this experiment. The percentage of extra water by weight of GBA was added into the geopolymer mortar mixtures in order to maintain flowability. The GBA mortar mixtures were cast in room temperature at 25±3°C. The standard mortar flow of 110±5% was maintained as control workability, and easily placed GBA mortar into $50 \times 50 \times 50$ mm of cube mould. The mortar flow test was performed in accordance with ASTM C230 [21]. The fresh GBA geopolymer mortar was vibrated during cast for 10 s by using a vibrating table. Next, the specimens were wrapped with plastic sheet to prevent the losing of moisture. The mix proportions of GBA geopolymer mortars are given in Table 2.

Samples	Na ₂ SiO ₃ to NaOH ratio	Concentration of NaOH	Extra water	Flow	
		(M)	(%) by mass	(%)	
10 M-0.5 Si/Na	0.50	10	2.4	111	
10 M-1.5 Si/Na	1.50	10	2.8	109	
10 M-2.5 Si/Na	2.50	10	3.5	108	
15 M-0.5 Si/Na	0.50	15	3.6	109	
15 M-1.5 Si/Na	1.50	15	3.8	107	
15 M-2 5 Si/Na	2 50	15	39	106	

Table 2. Mix proportions of geopolymer mortars.

Notes: X M-Y Si/Na; X is the concentration of NaOH. Y is Na2SiO3/NaOH ratio.

2.2.2 Method of curing

The methods of curing for the GBA geopolymer specimens were evaluated as description below:

2.2.2.1 Delay time (Delay time for 1 hour before heat curing the sample)

In this case, the mortar specimens were predetermined delay time for 1 h at room temperature before heat curing. Next, the specimens were kept at 45°C, 75°C, and 90°C to curing in heat chamber for 24 h. After that, the specimens were cooled down, and they were demoulded in testing room at temperature of 25 ± 3 °C until the testing date (at 1 day).

2.2.2.2 No-delay time

After wrapping the specimens with plastic sheet, the specimens were put in the heat chamber to curing at 45°C, 75°C, and 90°C for 24 h. After heat curing, the specimens were cooled down, and they were demoulded in testing room at temperature of 25 ± 3 °C until the testing date (at 1 day).

2.2.3 Compressive strength test

The compressive strengths of $50 \times 50 \times 50$ mm cube specimens were tested by investigating on parameters as described below:

1) Delay time (Delay time for 1 h before heat curing the specimens)

2) No-delay time (Suddenly heat curing after casting the specimens)

Both delay and no-delay times, the compressive strength tests were performed at the age of 1 day in accordance with ASTM C109 [22]. The reported strengths are the average of six mortar specimens that were tested for compressive strength. Effects of the curing temperature, the delay and no-delay times before curing, the concentrations of sodium hydroxide, and the sodium silicate to sodium hydroxide ratio on compressive strength were investigated. The compressive strength test used in this research consisted of 100 tons universal machine from Thailand.

2.2.4 Porosity test

GBA mortar specimens were dried by heating at temperature of $100\pm5^{\circ}$ C until they were constant weight. Next, they were placed in desiccators under vacuum condition for 3 h. The set–up was finally

filled with de-aired, and distilled water to measure the effective porosity of the mortar [5,23] at the age of 1 day. The porosity was calculated by using Equation (1).

$$p = \frac{(W_a - W_d)}{(W_a - W_w)} \times 100 \tag{1}$$

where, *p* is effective porosity (%), *Wa* is saturated specimen weight (g) in air, *Wd* is dried specimen weight (g) after 24 h in oven at 100 ± 5 °C, and *Ww* is specimen weight (g) in water. The reported results of porosity are the average of three samples.

2.2.5 Abbreviations of specimens

The abbreviations of the GBA mortar specimens are given as follows. For the example, 10 M-0.5 Si/Na means the mortar contained 10 M of NaOH and a 0.5 ratio of Na₂SiO₃ to NaOH while 15 M-2.5 Si/Na means the mortar contained 15 Molar of NaOH and a 0.5 ratio of Na₂SiO₃ to NaOH.

3. Results and discussions

3.1 Chemical compositions and morphology of GBA

The physical properties and chemical compositions of the GBA were determined using X-ray diffraction (XRD) and X-ray fluorescence (XRF), respectively. The chemical constituents of GBA are given in Table 3. The main chemical compositions of GBA were 40% of SiO₂, 21% of Al₂O₃, 14% of Fe₂O₃, and 16% of CaO with the loss on ignition (LOI) of 4.4%. The sum of SiO₂, Al₂O₃, and Fe₂O₃ of 75% was slightly higher than 70% that required for natural pozzolan according to ASTM C618 [7]. The XRD pattern of GBA is shown in Figure 2. The XRD pattern confirmed that GBA contains mainly of

Table 3. Chemical components of ground bottom ash (GBA).

Compositions	Ground bottom ash (GBA)
SiO ₂	40
Al ₂ O ₃	21
Fe ₂ O ₃	14
CaO	16
Na ₂ O	3
MgO	6
K ₂ O	2
SO ₃	-
LOI	4.4
SiO ₂ + Al ₂ O ₃ + Fe ₂ O ₃	75



Figure 2. X-ray analysis (XRD) pattern of GBA.



Figure 3. SEM photograph of GBA.

SiO₂, Al₂O₃, and Fe₂O₃. SEM photograph (Figure 3) showed the morphology of GBA. The GBA had angular and irregular shapes with different size in the range of 2.0 μ m to 10.0 μ m. This investigation was also similar to finding of Sathonsaowaphak *et al.* [24].

3.2 Workability of geopolymer mortar

The workability of GBA geopolymer mortar depended on the concentrations of NaOH at 10 and 15 Molar as also shown in Tables 2. At 10 M NaOH, the flowability of GBA geopolymer mortar decreased with increasing in the ratio of Na2SiO3 to NaOH. The flows of GBA geopolymer mortar with 0.50, 1.50, and 2.50 of Na₂SiO₃ to NaOH ratios were 111%, 109%, and 108%, respectively. In addition, it was found that the 111%, 109%, and 108% of flows were obtained from additional waters, 2.4%, 2.8%, and 3.5% by weight of GBA, respectively. These results showed that an increase in Na₂SiO₃ to NaOH ratio resulted in reduced the flowability of GBA geopolymer mortar. The result was confirmed from those researchers [17,19,20,24] who reported that in order to maintain the flowability of geopolymer mortar, the additional water in mixture was needed with increasing of Na2SiO3 to NaOH ratios. Furthermore, the sodium silicate solution, Na₂SiO₃ is a very high viscosity solution. As a result, the increasing of Na₂SiO₃ also reduced the flow of GBA geopolymer mortar.

Similar result was found in 15 M of NaOH concentration, the results showed that the flowability of GBA geopolymer mortar also decreased with increasing in the ratio of Na₂SiO₃ to NaOH. GBA geopolymer mortar with 0.50, 1.50, and 2.50 of Na₂SiO₃ to NaOH ratios had flow values of 109%, 107%, and 106%, respectively. In order to maintain the flowability of GBA geopolymer mortar, the extra waters of 3.6%, 3.8%, and 3.9% by weight of GBA were added in the mortar mixtures. Moreover, the results indicated that the increasing of NaOH concentration could be reduced the flowability of GBA geopolymer mortar. The results were observed from the extra water values from 15 M NaOH geopolymer mortars, 3.6% to 3.9% by weight of GBA, were higher than those of 10 M NaOH geopolymer mortars, 2.4% to 3.5% by weight of GBA. It has been shown from those results of other researchers [17,20,24]. This was because the increasing in the NaOH concentration increased viscosity the geopolymer mortar, and resulted in reduced the flowability of the geopolymer mortar.

3.3 Effect of NaOH concentration on compressive strength at 1 day

The compressive strength results of GBA geopolymer mortar with different concentrations of NaOH solutions and curing at 45°C, 75°C, and 90°C are shown in Figures 4, 5, and 6, respectively. It was found that the concentration of NaOH was significantly enhanced compressive strength at 1 day. At 15 M of NaOH, the compressive strengths development of GBA geopolymer mortar were higher than those of 10 M of NaOH. In addition, the compressive strength development was also found in the same trend at all temperature cures as well as the delay time before curing. For example, at temperature cure of 75°C, the compressive strengths of 15 M NaOH mortars ranged from 14.5 MPa to 23.0 MPa, while compressive strengths of 10 M NaOH mortars ranged from 10.5 MPa to 17.8 MPa. The result is consistent with previous researches [17,19,20,24]. It was because the increasing of NaOH concentration resulted in increased Na ions as high alkaline liquids concentration within silicate solution. It was not only enhanced high reactive from silica ratio within the alkaline liquid purity but also enhanced the geopolymerization.



Figure 4. Compressive strength of GBA geopolymer mortar with different concentrations of NaOH solutions at a curing temperature of 45°C.



Figure 5. Compressive strength of GBA geopolymer mortar with different concentrations of NaOH solutions at a curing temperature of 75°C.



Figure 6. Compressive strength of GBA geopolymer mortar with different concentrations of NaOH solutions at a curing temperature of 90°C.

3.4 Effect of Na₂SiO₃ to NaOH ratios on compressive strength at 1 day

The compressive strength results of GBA geopolymer mortar with various Na₂SiO₃ to NaOH ratios at curing temperatures of 45°C, 75°C, and 90°C are shown in Figures 7, 8 and 9, respectively. The results indicated that the GBA geopolymer mortar with Na2SiO3 to NaOH ratio of 2.5 gave highest compressive strength at all curing temperatures when compared to Na2SiO3 to NaOH ratios at 0.5 and 1.5. For example, at a curing temperature of 75°C, the compressive strength of 15 M-2.5 Si/Na mortar (no-delay time) was as high as 18.0 MPa. The compressive strength of 10 M-2.5 Si/Na mortar (nodelay time) was lower at 16.5 MPa. In case of the delay time, the compressive strength of 15 M-2.5 Si/Na and 10 M-2.5 Si/Na mortars were 23.0 MPa and 17.8 MPa, respectively. It was found that the increasing in Na2SiO3 to NaOH ratio resulted in enhanced the compressive strength of GBA geopolymer mortar. This was due to the increasing in Na2SiO3 to NaOH ratio increased Na ions content of geopolymer mixture. The high reactive from silica ratio within the alkaline liquid purity were used to balance the charges to form the alumino-silicate networks from geopolymerization [17,20]. These results indicated that the effect of Na₂SiO₃ to NaOH ratio on the compressive strength of GBA geopolymer mortars was therefore very significantly.



Figure 7. Compressive strength of GBA geopolymer mortar with various Na₂SiO₃ to NaOH ratio at a curing temperature of 45°C.



Figure 8. Compressive strength of GBA geopolymer mortar with various Na_2SiO_3 to NaOH ratio at a curing temperature of $75^{\circ}C$.



Figure 9. Compressive strength of GBA geopolymer mortar with various Na₂SiO₃ to NaOH ratio at a curing temperature of 90°C.

3.5 Effects of curing temperature on compressive strength at 1 day

Figures 10 and 11 show the effect of curing temperature on the compressive strength of GBA geopolymer mortar for no-delay time and delay time, respectively. It was found that GBA geopolymer mortars gave compressive strength at curing temperature of 45°C lower than that of 75°C for curing temperature. At 45°C of curing temperature, no-delay time and delay time of curing, the compressive strength of the GBA geopolymer mortars ranged from 6.0 MPa to 17.5 MPa and 7.8 MPa to 21.0 MPa, respectively. In addition, the compressive strength of geopolymer mortars GBA had trend to increase when they were cured at higher temperature of 75°C. At 75°C of curing temperature, no-delay time and 12.0 MPa to 23.0 MPa, respectively. The highest compressive strength was obtained from 15 M of NaOH and Na₂SiO₃ to NaOH ratio of 2.5 with curing temperature of 75°C.



Figure 10. Effect of curing temperatures on compressive strength of GBA geopolymer mortar with no-delay time.



Figure 11. Effect of curing temperatures on compressive strength of GBA geopolymer mortar with delay time.

However, it seems that the compressive strengths of geopolymer mortars GBA were decreased when the curing temperature was increased to be 90°C. It was observed from at 90°C of curing temperature, no-delay time and delay time of curing, the compressive strengths of GBA geopolymer mortars ranged from 9.5 MPa to 18.0 MPa and 8.5 MPa to 22.0 MPa, respectively. The result of reduction in compressive strength was probably because the small cube specimen ($50 \times 50 \times 50$ mm), which had high surface to volume ratio, resulted in more weakness in GBA geopolymer mortars when the high temperature was cured at 90°C. Furthermore, at high temperature curing of 90°C, the GBA geopolymer mortar was high loss of moisture as well as the alkali activators. Consequently, the substantial moisture loss resulted in the reduction in compressive strength [17,20]. These results showed that the initial curing at high temperature was significantly process to improve the geopolymerization for compressive strength development of GBA geopolymer mortar. However, it was noted that the high temperature curing at 90°C should be considered in reduction on compressive strength of the GBA geopolymer mortar.

3.6 Effects of delay time and no-delay time before heat curing on compressive strength at 1 day

The results of the compressive strength of GBA geopolymer mortar for no-delay time and delay time at curing temperatures of 45°C, 75°C, and 90°C are shown in Figures 12, 13 and 14, respectively. In the case of delay time, the specimens were put in the oven at room temperature for 1 h before start heat curing. For no-delay time, the specimens were put in the oven, and then immediately heat curing. The curing temperatures were controlled at 45°C, 75°C, and 90°C for 24 h. It was found that at all curing temperatures, the delay time method gave compressive strengths higher than those of GBA geopolymer mortars with no-delay time of curing. For no-delay time, the compressive strengths of GBA geopolymer mortars ranged from 10.5 MPa to 18.0 MPa at curing temperature of 75°C. At 75°C of



Figure 12. Effects of delay time and no-delay time on compressive strength at 1 day at a curing temperature of 45°C.



Figure 13. Effects of delay time and no-delay time on compressive strength at 1 day at a curing temperature of 75°C.



Figure 14. Effects of delay time and no-delay time on compressive strength at 1 day at a curing temperature of 90°C.

curing temperature with delay time 1 h, the GBA geopolymer mortar gave the highest compressive strength with range from 12.0 MPa to 23.0 MPa. As a result, it was concluded that the delay time for 1 h before heat curing was significantly increased compressive strength of the GBA geopolymer mortar. This was due to the delay time allowed the dissolving of silica and alumina which were the main ingredients for the alumino–silicate of geopolymerization [17,19].

3.7 Porosity of GBA geopolymer mortar

The porosity results of GBA geopolymer mortar with various Na₂SiO₃ to NaOH ratios, concentration of NaOH solutions for delay time and no-delay time at a curing temperature of 75°C is shown in Figure 15. It was found that the porosities of 15 M NaOH of GBA geopolymer mortar ranged from 10% to 21% while 10 M NaOH of GBA geopolymer mortar ranged from 16% to 24%. The result showed that the porosities of 15 M NaOH of GBA geopolymer mortar were lower than those of 10 M NaOH of GBA geopolymer mortar.

Furthermore, the porosity of GBA geopolymer mortar with Na2SiO3 to NaOH ratio of 2.5 was lower than those with Na2SiO3 to NaOH ratios of 1.5 and 0.5. Moreover, the test results also showed that the delay time for 1 h affected to reduce the porosity of the GBA geopolymer mortar especially 15 M NaOH of the GBA geopolymer mortar. The relationship between the compressive strength and percentage porosity of all GBA geopolymer mortars at a curing temperature of 75°C is shown in Figure 16. The results indicated that the porosity of GBA geopolymer mortar was related to its compressive strength. The porosity of GBA geopolymer mortar was decreased with the increasing of its compressive strength. It was the same trend of those results of normal mortars that using ordinary Portland cement. The use of GBA binder to produce GBA geopolymer mortar reduced the porosity because its fine particle size and high surface area enhanced the geopolymerization. The result was confirmed by those results of the researchers [17,19,20].



Figure 15. Porosity of GBA geopolymer mortar at a curing temperature of 75°C in oven for 24 h.



Figure 16. Relationship between compressive strength and porosity of GBA geopolymer mortar at a curing temperature of 75°C in oven for 24 h.

4. Conclusions

Based on the experimental results, the following conclusions can be drawn:

1) The increasing of NaOH concentration and Na₂SiO₃ to NaOH ratio reduce the workability of GBA geopolymer mortar. In addition, the higher extra water was required when NaOH concentration and Na₂SiO₃ to NaOH ratio was increased in order to maintain the workability of the GBA geopolymer mortar.

2) The high concentration of NaOH at 15 M gave the compressive strength at 1 day higher than that of GBA geopolymer mortar with lower NaOH concentration at 10 M. Furthermore, GBA geopolymer mortar with high Na₂SiO₃ to NaOH ratio at 2.5 gave the highest compressive strength when compared to lower Na₂SiO₃ to NaOH ratios of 0.5 and 1.5.

3) The highest compressive strength was obtained from 15 M of NaOH and Na₂SiO₃ to NaOH ratio of 2.5 with delay time before heat curing at 75°C. It was 15 M-2.5 Si/Na mortar that gave the highest compressive strength of 23.0 MPa at 1 day. It was noted that the high temperature curing at 90°C should be considered in reduction on compressive strength of the GBA geopolymer mortar.

4) The porosity of GBA geopolymer mortar was related to its compressive strength. The compressive strength of GBA mortar increased with the decreasing of the porosity. It was the same trend of those results of normal mortars that using ordinary Portland cement.

5) The GBA binder could not only be used as a new geopolymer material in mortar, but could also help to reduce the environmental problems such as the disposed of the bottom ash as waste to landfill. The GBA binder is one of the options for geopolymer material from industrial waste in concrete work.

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