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Preparation and characterization of vitrified slag/geopolymers for construction and fire-resistance applications

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Abstract Incinerator ash transforms into vitrified slag through thermal plasma treatment and quenching processes. Thus, the heavy metals contained in the ash can be solidified into the framework structure of the vitrified slag, thus reducing the threat to the environment. This study used vitrified slag as a raw material in preparing geopolymers by applying alkali solutions of predetermined molar ratios at ambient temperature. The influences of alkali solutions at various SiO₂/ Al₂O₃ and SiO₂/Na₂O molar ratios on the properties of the geopolymers were evaluated. A foaming agent was also added during geopolymerization process to increase the porosity of the geopolymers; this could improve the fire resistance characteristics of the geopolymers. The test results indicated that the alkali solution at 57 SiO₂/Al₂O₃ and 0.75 SiO₂/Na₂O molar ratios can produce geopolymers with optimal mechanical properties and fire resistance characteristics. Adding foaming agent can greatly improve the fire resistance of the geopolymers because of the increased porosity. According to the test results, the geopolymers fabricated using vitrified slag and alkali solutions can be potential materials for construction and fire resistance applications.

Y.-C. Ding (⊠) · Y.-S. Fang · T.-W. Cheng Institute of Mineral Resources Engineering, National Taipei University of Technology, 1, Sec. 3, Zhongxiao E. Rd., Taipei 10608, Taiwan, ROC e-mail: ycding2014@gmail.com **Keywords** Incinerator ashes · Quenching slag · Geopolymer materials

1 Introduction

Taiwan produces approximately one million tons of incinerator ash annually. The incinerator ash is commonly treated using the cement-based solidification technique. However, this technique increases the volume of waste and exhibits heavy metal dissolution problems after long periods of acid rain wash. In recent years, the Institute of Nuclear Energy Research has used thermal plasma to treat the incinerator ash at a temperature higher than its melting point. At such high temperature, the toxic organic matters can be decomposed and the toxic heavy metals can be bonded into the SiO₂ framework structure to create stable vitrified slag. This vitrified slag can then be recycled as a useful resource for various applications [1].

Currently, the vitrified slag can be used for only low-value applications, such as graded material for road construction and bituminous concrete overlay. To increase the application value of the vitrified slag, this study used the vitrified slag for increasing its application value as a raw material in preparing geopolymers because of its high aluminum and silica content. The Si and Al in high alkali solutions can be dissolved from vitrified slag and create an aluminum silicate colloid. The aluminum silicate colloid transforms into a zeolite-like polymeric framework structure after dehydration. The geopolymers prepared using the vitrified slag exhibit excellent mechanical and fireresistance characteristics; they can be used for construction and heat insulation applications. The following tasks were conducted in this study.

- The vitrified slag was used as a raw material and mixed with alkali solutions to prepare geopolymers. The influence of alkali solutions at various SiO₂/Na₂O molar ratios and curing time on the physical and mechanical properties as well as fire resistance capability of geopolymers were evaluated.
- 2. In addition to the sodium silicate solution, sodium aluminate solution was also added for preparing geopolymers. The effect of SiO₂/Na₂O and SiO₂/Al₂O₃ molar ratios and curing time on the physical and mechanical properties as well as fire resistance capability of the geopolymers were also evaluated in detail to determine the optimal geopolymeric reaction parameters.

1.1 Geopolymer

Joseph Davidovits introduced the term "geopolymer" materials, and he has also applied numerous patents regarding geopolymerization processes [2-5]. Davidovits used a hydrothermal synthesis method to prepare geopolymers by mixing aluminum silicate minerals with an alkali solution to create a zeolite-like structure at a temperature below 150 °C; this synthesized material is either an amorphous or hypocrystalline 3-dimensional aluminum silicate mineral [6]. According to previous studies, the geopolymer cement solidifies in 1.5-2 h and its compressive strength can reach 20 Mpa in 4 h [7, 8]. Davidovits also discovered that a geopolymer prepared using (Na,Ca)-Poly(sialate) and (K,Ca)-Poly(sialate-siloxo) exhibits highly favorable fire resistance and heat insulation characteristics even when exposed directly to 1200 °C.

A geopolymer is a 3-dimensional framework aluminosilicate material, and its structure is composed of SiO₄ and AlO₄ tetrahedral structures interlinked by a shared O atom [9, 10]. Geopolymers can be fabricated by mixing aluminosilicate materials (metakaolin) or glass power with sodium hydroxide/sodium silicate solutions [11]. Geopolymers exhibit the advantages of high compressive strength, fire resistance, low



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shrinkage, acid resistance, and low thermal conductivity [12].

Numerous minerals and related wastes containing Si and Al can be used to synthesize geopolymers. The Si and Al ions in the minerals and wastes can be dissolved by an alkali metal solution to create a silicate and Al hydroxide colloid mixture; the alkali metal solution acts as an activating agent. With this geopolymeric reaction, a geopolymer with a framework structure can then be solidified through a drying and dewatering process.

1.2 Alkali solutions

Xu and Van Deventer [13] pointed out that adding sodium silicate solution provides extra Si ions to activate the cementing characteristic of the precursor for geopolymeric reaction and to increase the mechanical strength of the geopolymer. Davidovits [2] also suggested that the hexahedral-coordinated Al in the sodium aluminate solution may affect the tetrahedral-coordinated Al reaction and improve the mechanical strength of the geopolymer. Verdolotti et al. [14] prepared a geopolymer by using Pozzolana and sodium aluminate and soldium hydroxide solution. This resulted in the increase of the geopolymer's compressive strength. and Van Deventer ***[15] suggested that alkali solution controls the dissolution of aluminum silicate materials (minerals) and rate of geopolymeric reaction.

2 Experimental

In this study, vitrified slag was used as a raw material in preparing geopolymers. The alkali solutions were prepared by mixing predetermined quantities of sodium silicate, sodium aluminate, and sodium hydroxide solutions to create various SiO₂/Al₂O₃ and SiO₂/Na₂O molar-ratio alkali solutions. The vitrified slag was then mixed with alkali solution at a 3:1 solid/liquid ratio to create geopolymers. This was followed by physical, mechanical, and fire resistance tests. Potassium permanganate and hydrogen peroxide were also added as foaming agents during the geopolymerization process to improve the fire resistance characteristics of the geopolymers. Figure 1 shows the experimental flowchart.





2.1 Plasma-vitrified slag

Plasma-vitrified slag was provided by the Institute of Nuclear Energy Research. This slag was first pulverized to particle sizes ranging between 2.75 and 704 μ m with D_{50} at 130.9 μ m. According to X-ray diffraction analysis, the vitrified slag is an amorphous material which is favorable for preparing geopolymers. Acme Analytical Laboratories Ltd. analyzed the chemical composition of the vitrified slag. The vitrified slag is mainly composed of 36.12 % CaO, 28.59 SiO₂, 18.86 Al₂O₃, and 8.68 % Fe₂O₃. The other minerals found in vitrified slags include Cr₂O₃, MgO, P₂O₅, TiO₂, ZnO, CuO, MnO₂, Na₂O and K₂O, but all less than 2.0 %. Because the original raw material of incinerator fly ashes contains large amount of calcium, therefore the vitrified slag had high CaO content. According to the TCLP test results, all of the measured heavy metal dissolutions comply with EPA requirements.

2.2 Alkali solutions

The sodium aluminate solution $(Na_2Al_2O_4)$ (purchased from Taiwan Organic Co, Ltd.) used in this study contains 26 % of Al_2O_3 and 19 % of Na_2O and creates a 1.4 Al_2O_3/Na_2O molar-ratio solution.

Sodium silicate solution (purchased from Taiwan First Chemical Co., Ltd.) contains 29 % of SiO₂ and 9.1 % of N₂O and creates a $3.2 \text{ SiO}_2/\text{Na}_2\text{O}$ molar-ratio solution.

As shown in Tables 1 and 2, the alkali solutions at various SiO_2/Na_2O and Al_2O_3/Na_2O molar ratios were prepared by mixing with a predetermined amount of NaOH solution.

2.3 Foaming agent

The foaming agents were prepared by mixing 0.5 wt% of 0.1 N potassium permanganate (KMnO₄) with 0.4–1.6 wt% of H₂O₂. Adding H₂O₂ into KMnO₄ leads to a chemical reaction that produces oxygen and creates a porous structure in the geopolymers.

2.4 Physical and mechanical properties tests

The mechanical properties tests of geopolymer conducted in this study include setting time tests, compressive strength tests and flexural strength tests. For setting time tests, CNS786 "Method of Test for Time of Setting of Hydraulic Cement (by Vicat Needle)" was adopted to measure the setting time of geopolymers. The compressive strength tests of geopolymers were conducted in accordance with CNS2889/R3053

 Table 1
 Sodium silicate solutions of various SiO₂/Na₂O molar ratios

Sample no.	Na-0.75	Na-1.00	Na-1.25	Na-1.50	Na-1.75
SiO ₂ /Na ₂ O molar ratios	0.75	1.00	1.25	1.50	1.75

Table 2 Alkali solutions with various Al₂O₃/Na₂O and SiO₂/Na₂O molar ratios

Sample no.	Al-19/Na-0.75	Al-19/Na-1.00	Al-19/Na-1.25	Al-19/Na-1.50	Al-19/Na-1.75
SiO ₂ /Na ₂ O molar ratios	0.75	1.00	1.25	1.50	1.75
SiO ₂ /Al ₂ O ₃ molar ratios	19	19	19	19	19
Sample no.	Al-57/Na-0.75	Al-57/Na-1.00	Al-57/Na-1.25	Al-57/Na-1.50	Al-57/Na-1.75
SiO ₂ /Na ₂ O molar ratios	0.75	1.00	1.25	1.50	1.75
SiO ₂ /Al ₂ O ₃ molar ratios	57	57	57	57	57

"Method of Test for Compressive (Crushing) Strength of Fired Whiteware Clay Materials". The flexural strength tests of geopolymers were conducted in accordance with CNS2888/R3052 "Method of Test, Modulus of Rupture Unfired Clays" to measure the 4-point flexural strength of geopolymers. For physical properties of geopolymers, CNS619/R3013 "Method of Test for Apparent Porosity, Water Absorption and Specific Gravity of Refractory Bricks" was adopted to measure the bulk density, porosity, and water adsorption of geopolymers. For fire resistance tests, $15 \times 15 \times 1$ cm geopolymer plates (7 days of curing) were prepared for fire-resistance tests. A flame temperature of 1100 ± 5 °C controlled by a thermal couple was directed to the surface of the tested plate for 30 min. Another thermal couple was placed at the back of the tested plate to record the temperature at a 1-min interval.

3 Results and discussion

3.1 Setting time of geopolymers

For geopolymers fabricated at various SiO_2/Na_2O molar ratios, the setting time of vitrified slag geopolymers increased as the SiO_2/Na_2O molar ratios increased from 0.75 to 1.75. As illustrated in Fig. 2, the final setting time for Na-1.75 is 145 min, which is approximately 285 min earlier than that of Na-0.75 (430 min). This is probably because a high concentration of Si ions in the





Fig. 2 Setting time of geopolymers prepared with various SiO₂/Na₂O molar ratios

alkali solution can promote the formation of a geopolymer precursor and accelerate the hardening of geopolymer. More time is required for geopolymeric reaction at a low SiO₂/Na₂O molar ratio because of the high Na ions concentration.

Alkali solutions prepared at various SiO_2/Na_2O (0.75–1.75) and Al_2O_3/Na_2O (19 and 57) molar ratios were also used to fabricate geopolymers. Figures 3 and 4 show the setting times of these geopolymers, indicating similar results as those observed in Fig. 2 (i.e., the higher the SiO_2/Na_2O molar ratio is, the shorter the setting time). However, the samples prepared by applying a high molar ratio (Al-57/Na-1.75) demonstrated a slightly shorter setting time than



Fig. 3 Setting time of geopolymers prepared with 19 Al_2O_3/Na_2O molar ratio solutions



Fig. 4 Setting time of geopolymers prepared with 19 Al_2O_3/Na_2O molar ratio

that of those prepared by applying a low molar ratio (Al-19/Na-1.75). These test results indicated that the concentration of Al ions can affect the setting time of the geopolymers.

3.2 Physical properties of geopolymers

The specific gravity of the geopolymers prepared using the alkali solutions specified in Tables 1 and 2 are in the range of 1.99-2.07. The porosity (26.5–27.1 %) and water adsorption (12.9–13.3 %) of the geopolymers fabricated at various SiO₂/Na₂O molar ratios were also highly similar. By adding Na₂Al₂O₄ (19 and 57 Al₂O₃/Na₂O molar ratios), we observed that the porosity and water adsorption of the geopolymers increased with the SiO₂/Na₂O molar ratio of the alkali solution (Fig. 5). This is presumably because the low alkalinity solution could not extract adequate Si and Al ions to facilitate the geopolymeric reaction. Even though the reaction may well be completed, the geopolymerization was not pronounced and not all starting glass were dissolved. Since the higher the Si/Na ratio of alkali solution, the shorter the setting time (higher viscosity) of geopolymer, it may reduce the integrity of the geopolymer structure, and thus increase its porosity and water adsorption rate. Based on the physical properties of the vitrified-slag geopolymers, the alkali solutions at 19/57 Al₂O₃/Na₂O and 0.75/1.0 SiO₂/Na₂O molar ratios were used to prepare geopolymers for the following mechanical properties and fire resistance tests.

3.3 Compressive strength of vitrified slag geopolymers

Figure 6 shows the compressive strength of geopolymers prepared at various SiO₂/Na₂O molar ratios, indicating that the compressive strength increases as the SiO₂/Na₂O molar ratio decreases. The compressive strength of the Na-0.75 sample can reach 37 and 76 MPa after 3 and 14 days of curing, respectively. Figure 6 also indicates that the compressive strength of all of the prepared geopolymers decreased after 28 days of curing. The geopolymeric reaction was completed after 28 days of curing and the compressive strength remained constant even after 60 days of curing. Panias et al. [16] pointed out that the dissolution of Si and Al ions from aluminum silicate materials can be accelerated by increasing the alkalinity of alkali solution. A complete geopolymeric



Fig. 5 Porosity and water adsorption of geopolymers prepared with various SiO_2/Na_2O and Al_2O_3/Na_2O molar ratios



Fig. 6 Compressive strength of geopolymers made with various SiO₂/Na₂O molar ratios

reaction can be achieved by dissolving high amounts of Si and Al ions from vitrified slag, and this increases the compressive strength of the geopolymer.

Figure 7 indicates that geopolymers fabricated using the mixture of sodium silicate and sodium aluminate solutions exhibit a much higher compressive strength than do those fabricated using only sodium silicate solutions. Phair and Van Deventer [15], Brew and Mackenzie [17] and Verdolotti et al. [14] indicated that the 6-coordinated Al from sodium aluminate can provide sufficient Al ions to facilitate the geopolymeric reaction. The compressive strength of the geopolymers fabricated at 0.75 SiO₂/Na₂O and 19/57 Al₂O₃/Na₂O molar ratios was higher than those fabricated at a 1.0 Al₂O₃/Na₂O molar ratio. This is because increasing the alkalinity of the alkali solution can accelerate the dissolution of the Si and Al ions



Fig. 7 Compressive strength of geopolymers prepared with $19/57 \text{ Al}_2\text{O}_3/\text{Na}_2\text{O}$ molar ratios and $0.75/1.0 \text{ SiO}_2/\text{Na}_2\text{O}$ molar ratiossolutions

from vitrified slag, thus promoting the geopolymeric reaction. The compressive strength of Al-19/Na-0.75 and Al-57/Na-0.75 can reach 94.2 and 74.4 Mpa, respectively after 60 days of curing. The compressive strength of Al-19/Na-1.0 and Al-57/Na-0.75 decreased from 64.2 and 69.4 Mpa to 58.3 and 45.4 Mpa, respectively, after 28 days of curing. This can be attributed to the low concentration of Na ions in the alkali solution.

3.4 Flexural strength of geopolymers

The flexural strength test results of the geopolymers indicated nearly the same trend as that of the compressive strength test results. The geopolymers fabricated by adding sodium aluminate in the alkali solution exhibited higher flexural strength than did those fabricated without using sodium aluminate. Figure 8 indicates that Al-19/Na-0.75 (10 Mpa) and Al-57/Na-0.75 (8.5 Mpa) generally exhibit higher flexural strength than do Al-19/Na-1.0 (5.5 Mpa) and Al-19/Na-1.0(6.3 Mpa) after 60 days of curing. The low concentration of Na ions in the alkali solution presumably caused the reduced dissolution of Al and Si ions, thus resulting in an incomplete geopolymeric reaction.

3.5 FT-IR spectrum analysis

The FT-IR was used to verify the functional group of the geopolymers prepared using alkali solutions of various SiO₂/Na₂O and Al₂O₃/Na₂O molar ratios. Figure 9 indicates that no Si, Al bonding, and functional groups can be observed in the vitrified slag. Peaks for Si and Al bonding can be observed between



Fig. 8 Flexural strength of geopolymers prepared with 19/57 Al₂O₃/Na₂O molar ratios and 0.75/1.0 SiO₂/Na₂O molar ratios solutions



Fig. 9 FT-IR spectrum of geopolymers

450 and 1200 cm^{-1} for the synthesized geopolymers. Through further verification [18, 19], the peaks appearing between 450 and 470 cm^{-1} can be attributed to the Si-O-Si or Si-O-Al bending vibration. The AlO₂ functional group exists between 660 and 680 cm^{-1} . The symmetric stretching vibration of Si-O-Si or Si-O-Al appears at approximately 710 cm^{-1} and asymmetric stretching vibration of Si-O-Si or Si-O-Al can be observed between 950 and 1200 cm^{-1} . The structural characteristics of geopolymers observed in the FT-IR spectrum can prove the Si and Al bonding of the geopolymers prepared in this study. The Na₂CO₃, which serves as the reactant of Na ions and CO_2 , was also observed near 1460 cm⁻¹ [20], especially for geopolymers prepared using a 0.75 SiO₂/Na₂O molar ratio solution. The peaks that appear at 1420, 1650, and 3450 cm^{-1} represent the preserved stretching and O-H bending vibration of H₂O after geopolymerization [21, 22].

3.6 NMR analysis

²⁷Al was used to analyze the molecular structure of the geopolymers. Al tetrahedral and octahedral structures were discovered for vitrified slag at 52 and 9 ppm, respectively [23]. For geopolymers fabricated at various SiO₂/Na₂O molar ratios, a sharp peak appears

between 50 and 60 ppm which presumably represents the structure of $AlQ_4(4Si)$. It also means the framework structure with tetrahedral Al bonding was created in the geopolymer. The small peak appearing between 4 and 10 ppm in Fig. 10 represents small fractions of octahedral Al in the geopolymer. This octahedral Al can complement Al ions that are required for complete geopolymeric reaction [14].

3.7 Influence of foaming agent on the properties of geopolymers

The foaming agent was used in geopolymers prepared using 57 SiO₂/Al₂O₃ and 0.75 SiO₂/Na₂O molar-ratio alkali solution. After the addition of KMnO₄ and H₂O₂ into the vitrified slag and alkali solution mixture, the produced oxygen can create a porous structure in the geopolymer after solidification. It was found that the porosity of the geopolymers greatly increase as the amount of H₂O₂ addition increase from 0.4 to 1.6 %.

Table 3 shows the physical properties of the porous geopolymers. As the H_2O_2 addition increases from 0.0 to 1.6 %, the porosity and water adsorption of the geopolymers also increase from 25.18 to 63.90 and 11.95 to 60.72 %, respectively. The specific gravity also decreased from 2.10 to 0.69. According to the test results, the increase of porosity and water adsorption is assumed to be results from the open pores were formed.

The compressive and flexural strength of the porous geopolymers greatly decreased from 3.9 and 1.4 to 0.5 and 0.3 MPa, respectively, as the H_2O_2 increased from 0.4 to 1.6 %. Obviously, the highly porous structure of



Fig. 10²⁷Al NMR analysis of geopolymers

Table 3 Physicalproperties of geopolymersmade with various amountof H_2O_2 additon	H ₂ O ₂ (wt%)	Specific gravity	Porosity (%)	Water adsorption (%)
	0	2.10 ± 0.01	25.18 ± 0.43	11.95 ± 0.26
	0.4	1.65 ± 0.07	42.87 ± 2.28	27.75 ± 2.40
	0.8	1.58 ± 0.13	49.24 ± 2.32	39.52 ± 5.88
	1.2	1.06 ± 0.02	62.76 ± 0.89	59.48 ± 2.09
	1.6	0.69 ± 0.02	63.90 ± 0.84	60.72 ± 1.83

the geopolymers renders them an efficient fire-resistant material ideal for construction purposes.

3.8 Influence of porosity on the thermal conductivity of porous geopolymers

According test results, the the thermal conductivity of geopolymers decreases from 0.70 to 0.31 W/mK after adding 0.4 % H₂O₂ into the vitrified slag and alkali solution mixture. The thermal conductivity of geopolymers fabricated using higher H₂O₂ additions slightly decreased from 0.26 W/mK (0.8 % H₂O₂) to 0.23 W/mK (1.6 % H₂O₂). However, these test results also indicate that the thermal conductivity of porous geopolymers were slightly decreased by increasing the dosage of H_2O_2 from 0.4 to 1.6 %.

3.9 Fire resistance test of geopolymers

It should be noted here that this fire resistance test only consider the temperature changes on the back of geopolymer panel and the panel remains intact without rupture at the end of test.

As shown in Fig. 11, the temperatures measured at the back of geopolymers fabricated at 0.75 and 1.0 SiO₂/Na₂O molar ratios were nearly identical. The temperature measured at the back of the geopolymer plates were approximately 528 °C after burning by flame at 1100 ± 5 °C for 30 min. This indicates that the SiO₂/Na₂O molar ratio exerts no obvious influence on the fire resistance of the geopolymer. However, the geopolymers fabricated using 0.75/1.0 SiO₂/Na₂O and 57 SiO₂/Al₂O₃ molar-ratio solutions generally exhibit higher fire resistance than do those fabricated using 0.75 and 1.0 SiO₂/Na₂O molar-ratio solutions. The temperatures measured at the back of the geopolymers for Al-57/Na-0.75 and Al-57/Na-1.0 were 462 and 491 °C, respectively after burning for 30 min.

Figure 12 illustrates the measured temperatures of the geopolymers fabricated by adding foaming agent. As expected, the geopolymer fabricated using 1.6 % H₂O₂ foaming agent exhibits the lowest temperature (369 °C) measured at the back of the geopolymer plate because of its high porosity. The temperatures measured at the back of the plate remain nearly the same for all geopolymers tested after 10 min of burning. These test results demonstrated that the geopolymers fabricated by adding foaming agent exhibit excellent heat insulation characteristics and can be used as fire resistant materials.



Fig. 11 Influence of SiO₂/Na₂O and SiO₂/Al₂O₃ molar ratio on the fire resistance of geopolymers



Fig. 12 Influence of foaming agent on the fire resistance of geopolymers

4 Conclusions

- In this study, the geopolymers fabricated using vitrified slag (product of thermal plasma treated incinerator ash) and alkali solutions were found to be potential materials for construction and fire resistance applications.
- The higher the SiO₂/Na₂O molar ratio is, the shorter the observed setting time of geopolymers. The geopolymers fabricated by adding sodium aluminate solution generally exhibit longer setting times than do those fabricated using only sodium silicate solution.
- The functional groups of the geopolymers were verified using FT-IR. A Si–O–Si or Si–O–Al bending vibration was observed at 450–470 cm⁻¹. The AlO₂ functional group was observed between 660 and 680 cm⁻¹. The symmetric stretching vibration of Si–O–Si or Si–O–Al appeared at approximately 710 cm⁻¹ and the asymmetric stretching vibration of Si–O–Si or Si–O–Al was observed between 950 and 1200 cm⁻¹.
- 4. For geopolymers fabricated at a low SiO₂/Na₂O molar ratio (0.75), more Na ions in the solution can dissolve more Si and Al ions from vitrified slag to facilitate geopolymeric reaction and retain its long-term strength.
- 5. The geopolymers fabricated by adding sodium aluminate in the alkali solution exhibited higher mechanical strength, better physical properties, and greater fire resistance than did those fabricated using only sodium silicate solution. The test results obtained in this study indicated that a vitrified slag geopolymer can be an efficient construction material in the future.
- 6. The porous geopolymers fabricated by adding foaming agent demonstrated highly favorable fire resistance. Temperatures between 369 and 406 °C were measured at the back of porous geopolymers after burning at 1100 °C for 30 min. This excellent heat insulation property can render it as an efficient fire resistance material.

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