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Use of alkali-silica reactive sedimentary rock powder as a resource to produce high strength geopolymer binder

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HIGHLIGHTS

- Use ASR sedimentary rock powder to synthesis high strength geopolymer.
- Geopolymer binder is densified by filling the voids in matrix with rock powders.
- Incorporation of sedimentary rock powder enhances the compressive strengths by 15–30%.
- Metakaolin geopolymer binder with 67 wt% rock powder can achieve a high strength of 80 MPa.

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ABSTRACT

This paper reports on an innovative way to utilize alkali-silica reactive (ASR) rocks as a natural resource to produce high strength geopolymer binder. Excavation of the Jurong rock caverns in Singapore has produced large quantities of sedimentary rocks. These rocks, however, cannot be used for ordinary Portland cement concrete production due to their ASR reactivity. An alternative way to beneficially utilize these rocks is to produce geopolymer binder. The excavated rocks were classified based on petrography into four types and then converted to powder form in a sequence of steps. The rock powders were used to synthesize geopolymers by replacing metakaolin in different replacement ratios. Results showed that geopolymer binder with 67 wt% rock powder and only 33 wt% metakaolin can achieve a high compressive strength of 80 MPa. Incorporation of sedimentary rock powder enhanced the compressive strengths by 15–30% as compared to pure metakaolin geopolymers. Microstructure analysis revealed that the enhancement in compressive strengths were primarily due to the densification of binder by filling the voids in matrix with rock powders.

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1. Introduction

Singapore is a city-state with a limited land area of 719.1 km^2 and a population density of 7697 per km^2 [1]. With increasing population and globalization, more efficient ways of infrastructure are currently being explored by the Singapore government. One among such efforts is to utilize the underground space and as a consequence large quantities of sedimentary rocks have been dug out. Due to shortage of locally available aggregates, the potential use of these excavated rocks for concrete production has been explored. Initial reports studying properties of the excavated rocks using mortar-bar test [2], however, revealed that at many excavation sites these rocks were alkali silica reaction (ASR) active and were not suitable for concrete production. ASR in concrete refers to the reaction of certain siliceous phases present in some aggregates with alkali hydroxide in cement paste, which forms a gel that can swell by absorbing water from surroundings and induce expansive pressure to damage the concrete. Concrete deterioration caused by ASR is one among the many factors affecting the service life of reinforced concrete structures [3]. As a result, these excavated rocks are currently piled up and occupied space which is a concern for Singapore.

Geopolymer is a potential alternative binder to the ordinary Portland cement (OPC). It is well known that OPC production is one of the major causes of greenhouse gas emission [4,5]. Converting a ton of raw material into cement releases nearly a ton of carbon di-oxide (CO_2) [6] and cement industry alone accounts for around 5–8% of global CO_2 emission [7]. Geopolymer is a greener construction material compared to OPC [8,9]. It is because many aluminosilicate rich industrial by-products such as fly ash can be used as precursors for geopolymer synthesis. In geopolymerization,

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aluminosilicate sources chemically react with alkaline silicates to yield polymeric Si–O–Al–O bonds [9–11]. Thus, geopolymer binder possesses excellent mechanical properties [12], acid resistance [13], fire and heat resistance [14–19], good thermal insulation [20–24] and refractory properties [25]. Its application for cement and concrete [26–29], high tech composites [30–32] and in medicine [33] has also been explored.

Furthermore, it has been shown that geopolymer binder does not experience deleterious ASR. Expansive sodium-calcium silicate gels are unlikely to form in a geopolymer binder due to low calcium content in geopolymer precursor [34]. Rather, it has been indicated that ASR may be beneficial to geopolymer [26] as the reaction medium consists of alkali, silicon and aluminum. In fact, the alkali-silica reaction is to some degree the foundation of geopolymer chemistry [35].

Current research considers the utilization of the excavated ASR prone sedimentary rocks as a natural resource to produce high strength geopolymer binder. The sedimentary rocks were firstly classified based on their grain size and color. After crushing the rocks into powder, they were characterized using scanning electron microscopy (SEM), energy-dispersive X-ray (EDX) spectroscopy and X-ray powder diffraction (XRD). The potential of the rock powders to dissolve in alkaline medium and participate in geopolymerization reaction was also investigated. Further, the rock powder was used in conjunction with metakaolin to synthesize geopolymer binders. Properties of these binders were investigated by measuring the compressive strengths, by observing the microstructures using SEM and by characterizing the chemical structure using Fourier transform infrared (FT-IR) spectroscopy.

2. Experimental program

2.1. Characterization of rock samples

Sedimentary rocks from excavation of Jurong Rock Caverns (JRC), Singapore were collected from the site and used in the current study. JRC is located at a depth of 130 m beneath Banyan Basin in Jurong Island of Singapore. The 60-hectare underground space is utilized for storage of liquid hydrocarbons such as crude oil and condensate [36]. Jurong formation had developed by deposition of sediments from weathered material of older rocks in a shallow marine basin. JRC rocks are thus categorized as sedimentary [37]. The collected JRC rock samples from the site with size in meter scale were classified into four types based on their grain size and color (Table 1). Grain size here refers to the diameter of individual grains of sediment in bulk rocks.

After classifying the rocks, they were broken into smaller pieces using a hammer and one kilogram of each rock type was collected. The collected rock pieces, with size similar to that of large gravels, were crushed using a jaw crusher. The crushed rock pieces were then milled using a ball milling machine for about 30 min to obtain the original sedimentary rock in a powdered form. The rock powders were then sieved and their properties were studied.

Particle size distribution of rock powder was measured after ball milling using a laser particle size analyzer. 5 g of rock powder was added to 20 ml of distilled water to generate 10–20% obscuration. The instrument default optical parameters were used with a refractive index of 1.33 for water. SEM was conducted using a thermal field emission SEM (FE-SEM) at magnification levels of 25,000 and 50,000 for the powdered rock samples. Samples were mounted on a stub using a carbon tape and coated with a platinum layer using a sputter coater with 20 mA current for duration of 30 s prior to SEM imaging. EDX was carried out to identify the element composition of each rock sample. XRD powder diffractogram for each rock sample was obtained using powder XRD diffractometer with Cu Kα radiation. Samples were step scanned from 5° to 70° 2θ at 0.02° 2θ steps.

Table 1
Classification of JRC rock samples.

Type	Grain size (mm)	Macro color	Rock classification
I	0.005–0.02	Black	Fine siltstone
II	0.01–0.08	Dark grey-black	Coarse siltstone
III	0.04–0.2	Grey-dark grey	Coarse siltstone or fine sandstone
IV	0.1–0.4	Grey-light grey	Sandstone

The extent of dissolution of rock samples was also investigated. It is important to conduct such a study since the rocks are ASR prone and can provide reactive silicon (Si) and aluminum (Al) species and become a potential geopolymer precursor. It has been suggested that the process of geopolymerization begins with the dissolution of Si and Al species from the aluminosilicate material in an alkaline environment and continues with stages such as speciation equilibrium, gelation, reorganization, polymerization and hardening, with all these stages actually happening simultaneously [38,39]. Hence, it is significant to know the extent of dissolution of rock powders to know their potential to act as geopolymer precursor. Extent of dissolution of rock samples was determined by mixing 0.5 g of each rock sample with 20 ml of alkaline solution (2, 5, and 10 N of NaOH) at room temperature for 5 h using a magnetic stirrer. After centrifugation and filtration, the solution part was diluted to 0.2 N alkaline concentration and neutralized by 36% HCl. Inductively coupled plasma atomic emission spectroscopy (ICP-OES) was used to analyze the filtered solutions. A similar approach has been previously used [10] in order to estimate the extent of dissolution of natural aluminosilicate minerals.

2.2. Synthesis of geopolymer incorporating sedimentary rock powders

Type III rock powder, metakaolin, and sodium silicate solution were used for synthesis geopolymer. As will be discussed in the Section 3.1, all the four types of rock powder had similar composition, microstructure and extent of dissolution, and therefore type III rock powder was selected as a typical material to synthesize geopolymers. Metakaolin is produced by calcination of kaolinite clay at temperature ranging from 500 to 800 °C depending on the purity and crystallinity of precursor clay [40]. It is generally X-ray amorphous. The metakaolin used in this research was purchased from BASF. It has chemical composition as described in Table 2. The mean particle size of metakaolin used in this research was 1.3 μm.

Sodium silicate solution was prepared by dissolving varying amounts of amorphous silica (i.e. fumed silica) into freshly prepared sodium hydroxide solution. The amorphous silica or fumed silica was more than 99.8% pure SiO_2 and was a manufactured product from the Cabot Cooperation. Sodium hydroxide solution was prepared by dissolving a calculated quantity of NaOH pellets (99.8% pure, purchased from Schedelco) into ultrapure water collected from a Milli-Q water filtration station. The dissolution of fumed silica in sodium hydroxide solution was done to obtain a clear solution. Solutions were stored for about 24 h before use to allow them to cool down to room temperature and to achieve equilibrium.

Geopolymer samples were prepared using different proportions of rock powder (type III) and metakaolin and different quantities of sodium silicate solution were added to them. The corresponding mix design parameters (Si/Al molar ratios, Al/Na molar ratios, and w/solids mass ratios) were calculated based on calculations considering the moles of silicon, aluminum, sodium and water available from the source materials, namely metakaolin, rock powder, and sodium silicate solution and the values of these

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