



Influence of nano-SiO₂ on the strength and microstructure of natural pozzolan based alkali activated concrete

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HIGHLIGHTS

- AAC was developed using natural pozzolan (NP) from Red Sea coast of Saudi Arabia.
- A SS/SH ratio of 2.5 for the selected NP was suitable to achieve superior strength.
- Replacement of NP with nano-SiO₂ enhanced the strength and microstructure of concrete.
- 5% replacement of NP with nano-SiO₂ resulted in higher compressive strength.
- Use of nano-SiO₂ enhanced the polymerization and formation of C-A-S-H and N-A-S-H.

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ABSTRACT

Development of alkali-activated binder (AAB) is fast becoming a viable alternative to ordinary portland cement (OPC). However, key engineering properties of such a binder depend strongly on the chemical composition and fineness of the source materials in addition to the concentration of alkaline activators. In order to improve the reaction kinetics and subsequently, the mechanical strength of alkali activated concrete (AAC), nano-SiO₂ was proposed as a partial replacement to natural pozzolan (NP) in this study. As the composition of alkaline activators also plays a vital role on the properties of AAC, trial mixtures were prepared with varying sodium silicate to sodium hydroxide (SS/SH) ratio. Subsequently, NP was partially replaced with nano-SiO₂ in the selected mix to enhance the properties further. Fresh properties, such as, setting time of alkali-activated pastes (AAP) and the workability in terms of flow of mortar were determined. Evolution of compressive strength of concrete cured at 60 °C was monitored. Scanning electron microscopy (SEM) and X-ray diffraction (XRD) were utilized to determine the morphology and mineralogy of the AAP, respectively. In addition, FTIR was used to identify functional groups formed in the AAP. The study reveals that a SS/SH ratio of 2.5 was suitable in achieving superior strength. AAC prepared using NP and nano-SiO₂ exhibited improved strength and microstructural characteristics. However, 5% nano-SiO₂ showed significant enhancement in the compressive strength and microstructural characteristics as compared to those prepared with other replacement levels. Consequently, the results of this study provides important information on developing NP-based sustainable building material with remarkable enhancement in the properties by adding nano-SiO₂.

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1. Introduction

Alkali activated binder (AAB) is emerging as a viable alternative to ordinary portland cement (OPC) developed with a view to

decrease greenhouse gas emissions associated with the production of OPC. These binders are synthesized utilizing source materials that are mainly composed of silica and alumina [1–7]. Fly ash, metakaolin and palm oil fuel ash (POFA) have been used as precursors in developing AAB [8–10]. When these source materials are activated with alkaline solution, unlike the C-S-H gel which is formed in case of OPC, C-A-S-H or N-A-S-H type gel or both are the main reaction products within a three-dimensional network depending on the availability of calcium in the system [11,12]. The structure of this binder resembles with synthetic zeolites,

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however, it is predominantly amorphous in nature. These new class of AAB have multiple benefits, including; lower environmental impact [13], improved strength [14] and durability [15].

Although there are several advantages of using AAB, its production relies on the activation of source materials with alkaline activators and development in strength is rather slow when cured at ambient temperature [16] to that of elevated temperature ranging from 40 to 80 °C [17,18]. Further, the strength development of AAB depends on the chemical composition of the source material and its fineness [19,20]. As the fineness increases so does the reactivity with the alkaline activators resulting in improved mechanical and microstructural properties. Most of the cementitious materials used in the synthesis of AAB have particle size ranging from 1 to 20 µm [21]. The reactivity of the materials having particle size of more than 20 µm is low, whereas, those having smaller than 2 µm react rapidly within 24 h [21].

In order to improve reaction kinetics and subsequently the strength development of AAB, several methods have been tried including, increasing the fineness of the source material, incorporation of mineral additives and industrial by-products such as silica fume and GGBFS [22–24]. Fly ash was used as a main binder in the bulk of these studies. However, other alumino-silicate materials were also utilized. For instance, Moruf et al. [25] studied the effect of adding Al(OH)₃ on the strength and microstructural properties of alkali activated ground blast furnace slag-palm oil fuel ash based binder. Addition of 4% of Al(OH)₃ by weight of total binder content in the mortar mixture, marginally increased the strength by about 4% in relation to the mortar mix without Al(OH)₃. In another study, kaolinite and slaked lime were added to the Iranian Taftan pozzolan, having SiO₂, Al₂O₃ and CaO content of about 62%, 16% and 8%, respectively, and its impact on the strength and microstructural characteristics along with the nature of the binder were investigated even though the silica content was high in the starting material. There was strength reduction at some replacement levels when the specimens were cured at ambient temperature as well as elevated temperature under 2 MPa pressure for 3 h. It was reported that the addition of 20% kaolinite resulted in marginal improvement in strength by 3.5% as compared to the control mix [23].

Based on the results of the earlier studies mentioned above, it is noticeable that the strength and microstructural properties of AAB could not be substantially improved even with the addition of mineral admixtures, particularly kaolinite and Al(OH)₃. Despite these efforts, there is a scope of improving strength and microstructural properties of AAB by identifying deficiencies in the source material and incorporating them in addition to enhancing the reactivity by adding more finer materials. In this regard, the use of nanoparticles in the development of AAB is growing as they are highly reactive and improve the microstructural properties of the binders [26]. Particularly, nano-SiO₂ is a highly reactive amorphous pozzolanic material that predominantly consists of SiO₂ with enormous specific surface area exhibiting enhanced pozzolanic reactivity [27]. Additionally, its physical influence on the microstructure through compact particle packing makes it a viable solution to improve the properties of AAB. Further, the mechanical properties, such as compressive strength and tensile strength of AAB can be improved by using these nanomaterials [28]. However, as the nanomaterials in general can cause health risk to the human body when inhaled or absorbed, extra precautions need to be put in place while handling these materials [29]. Also, they should be stored well and avoid accidental release into the atmosphere which can cause ecological imbalance.

Several studies were previously undertaken to evaluate the effect of replacing fly ash with nanomaterials. In one such study, high calcium fly ash was partially replaced with 1–3% of nano-SiO₂ and nano-Al₂O₃ for synthesizing AAB for a particular alkaline activator concentration and their ratio [30]. It has been reported

that strength increased by more than 30% compared to the control mix at 2% replacement level despite the fact that the starting material contained only 29.32% of SiO₂ along with 25.79% of CaO [30]. In another study, fly ash comprising 64.97% and 26.64% of SiO₂ and Al₂O₃, respectively, was replaced by 6% nano-SiO₂. After 28 days of room curing, there was about 15% enhancement in strength recorded for a SS/SH ratio of 1.75 [31]. Along with the fineness of materials, alkaline activator content and their ratio plays a vital role on the strength development and microstructural properties of AAB, and these were not taken into consideration while the nanomaterials were incorporated in the fly ash-based AAB in the earlier studies [30,31].

Natural pozzolan (NP), whose chemical composition varies widely from source to source, is a supplementary cementitious material (SCM) which possesses a great potential for being used as precursor material in developing AAB. In the past, NP has not been explored considerably as AAB. Further, the effects of incorporating nano-SiO₂ on the properties of NP-based AAB have not been studied earlier. Therefore, in this research, natural volcanic pozzolan having moderate 40.24% of SiO₂, and sizable amounts of Al₂O₃ and CaO of 11.90% and 11.83%, respectively, has been used as the primary precursor material. As this NP is not rich in silica, compared to the natural pozzolan from other parts of the world [23], the properties of the binder developed utilizing this volcanic pozzolan could only be enhanced when this deficiency is compensated. Therefore, the objective of this research work was firstly to determine suitable SS/SH ratio for the development of AAC utilizing natural volcanic pozzolan in order to obtain optimum strength. Secondly, to improve its properties further by incorporating nano-SiO₂. Also, the study aims to understand the effect of different replacement levels of nano-SiO₂ on the mechanism of strength development, enhancement in microstructural characteristics as well as nature of the binder so formed. It is envisaged that the results obtained would be beneficial in understanding the behaviour and practical application of the end product.

2. Methodology of research

2.1. Materials

2.1.1. Natural pozzolan and nano silica

Natural pozzolan (NP) used in the reported study was a powdered form of volcanic rock from Red Sea coast in the western region of Saudi Arabia and nano-SiO₂, aqueous dispersion of colloidal silica approximately 50% solids by mass. The chemical composition of NP determined by X-ray fluorescence (XRF) technique is shown in Table 1. The specific surface area and average particle size of NP used are 442 m²/kg and 30 µm, respectively. Table 2 shows the properties of the nano-SiO₂.

2.1.2. Alkaline activators

The alkaline activators used were a combination of aqueous sodium silicate (SS), and 14 M sodium hydroxide (SH) solution. The silica modulus of sodium silicate was 3.3 and its composition includes; H₂O: 62.50%, SiO₂: 28.75% and Na₂O: 8.75%.

2.1.3. Aggregates

The fine aggregate (FA) used was dune sand with a specific gravity of 2.62 in saturated surface dry condition. Crushed limestone, having specific gravity of 2.56 was used as coarse aggregate.

2.2. Experimental program

2.2.1. Mix design

In order to determine the suitable composition of alkaline activators, trial mixtures were prepared with SS/SH, ratios of 2.0,

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