REVIEW



Alkali-Activated Materials and Geopolymer: a Review of Common Precursors and Activators Addressing Circular Economy

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Abstract

Introduction The vast increase in CO_2 and waste generation in recent decades has been a major obstacle to sustainable development and sustainability. In construction industry, the production of ordinary Portland cement is a major greenhouse gas emitter with almost 8% of total CO_2 production in the world. To address this, *Alkali-activated materials* and *geopolymer* have more recently been introduced as a green and sustainable alternative of ordinary Portland cement with significantly lowered environmental footprints. Their use to replace Portland cement products generally leads to vast energy and virgin materials savings resulting in a sustainable concrete production. In doing so, it reuses the solid waste generated in industrial and manufacturing sectors, which is aligned with circular economy. In turn, it reduces the need for ordinary Portland cement consumption and its subsequent CO_2 generation.

Objective To provide further insight and address the challenges facing the substitution of ordinary Portland cement, this article reviews different types, mechanisms, and result of mechanical and durability properties of *alkali-activated materials* and *geopolymer* reported in literature. Finally, it discusses future projections of waste materials that have cementitious properties and can replace ordinary Portland cement and be used in *alkali-activated materials* and *geopolymer*.

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Introduction

The search for sustainable means in engineering practices is one of the major quests in this century. From the advised *sustainable development goals* (*SDGs*) formed by the UN [1, 2], to the incorporation of circular economy in waste management, the vast urbanization and population growth of the metropolitan areas has created a conundrum of cleaner production and consumption to address the insurmountable urbanization issues such as waste production. Such vast increase in waste materials production, as well as the ever-increasing CO2 production in recent decades has been a major obstacle to sustainable development and sustainability. To address this, new concepts of waste management such as circular economy that adopt a lifecycle view of each material are being further incorporated into different fields such as construction area.

Construction industry has been consistently reported to count for use of almost 40% of total energy production [3], where only the cement production accounts for 8% of total global CO₂ production, annually [4, 5]. In this industry, the unbridled need for construction materials and binding agents has dedicated an ongoing body of research in understanding the most efficient means feasible to produce a more sustainable binding agent especially through entertaining new systems that further utilize waste materials. One of the most recent alternatives in this area can be viewed as the emergence of *Alkali-activated materials* (*AAMs*) and geopolymer whereby the ordinary *Portland cement* (*OPC*) is replaced by *supplementary cementitious materials* (*SCMs*) that have binding-ability and are generally recognized to be the most promising waste materials added to the mixture substituting cement and acting as binding agents.

Since their recognition, AAMs have found a variety of applications including eco-friendly concrete [6, 7], ceramic formation [8, 9], and refractories [10]. Yet, as a result of substitution of OPC, depending on the type and content of the SCMs added, a medium in liquid or solid form is required to increase the alkalinity of the mixture for binding purposes. This process is also called *activation* which is followed by the dissolution of the aluminosilicate bearing materials (aluminum and silicate bearing material) also known as *precursors* such as calcined clays (e.g., metakaolin), ground granulated blast furnace slag (GGBFS), coal fly ash, or other aluminosilicate-rich materials mixed with highly alkaline solutions (e.g., sodium hydroxide or sodium silicate) [11–13].

Understanding this process, the variation in systems and properties as a result of the type of primary *precursor* and *activator* is the first step toward entertaining this technology in construction industry. Yet, since aluminosilicate sources used in AAMs are mainly amorphous, the reaction mechanism of alkali-activated materials has been a source of academic debate that has resulted in terminology, function, and often mechanism contradictions. With such considerations, this review provides information based on the terminology and functionality provided by the cited references.

In that respect, this review aims to provide a compiled content based on both general and in-depth findings associated with AAMs and geopolymer technology as well as their prospective strength and durability characteristics. Such content would serve the literature by providing a basic understanding of this concept that supports total use of waste materials as binding agent.

Circular Economy

The incorporation of new concepts such as circular economy in waste management can be seen as a result of unsustainable material development and mismanagement of resources. In construction industry, the need for cheaper and vastly available materials as well as a detailed life-cycle design has always been a major driver toward sustainability and greener buildings. Such efforts, however, have been entertained through a static view of materials availability within a dynamic and hybrid world where emergence of a new material often equals scarcity of the other. In this environment, while the construction industry is experiencing a paradigm shift in the availability of certain supplementary cementitious materials, such as coal fly ash, it is imperative to search for newer waste materials in a hybrid and dynamic waste management economy of systems. In such systemic review, this article tends to highlight the use of alternative sources of waste materials that are projected to see higher available quantity, in the future, whose promising results are briefly shown in the literature.

Alkali-Activated Materials

Historically, cementitious materials were chosen and used in construction due to their availability and often favorable properties. During the 1940s and 1950s, Purdon [14] and Glukhovsky conducted major research on AAMs that was by then named "*soil cements*" whereby a high content of Slag-based aluminosilicate materials (an industrial waste material) was used as OPC substitution. Through the documented results, proved the possibility of using such, at the time vastly available, waste material to reduce the need for OPC and reduce the associated costs. Following such conceptual development, during the 1970s, Davidovits [15] investigated fire-resistant materials that led to the use of metakaolin as precursor in AAMs that further named "geopolymer" [16–18]. From there, a vast body of research interested in developing a sustainable binding agent started to entertain the use and study of the physicochemical properties of AAMs through the use of a variety of, mainly waste-based, cementitious materials and activators.

The basic function of AAMs can now be seen and divided into three groups of (1) low calcium system (also referred as geopolymer), in which low calcium SCMs, such as fly ash (class F), provide silicon and aluminum (Si + Al) as the main reactive binding agents; (2) high calcium system, in which silicon and calcium (Si + Ca) establish a product of calcium aluminum silicate hydrate (C-A-S-H) network which has a lower setting time and can harden in ambient temperature [19–21]; and (3) hybrid systems where OPC is often used with high volume SCMs that each partially contribute to the final and hardened materials. In all types of AAMs, however, equal to lesser degree of greenhouse gas emissions, being a perfect material for repair purposes [22] and having a reduced durability issues, compared to OPC system, are reported [23–25].

Mainly due to the use of high-volume waste materials and their respectively lower reactivity, another material (an activator) in solid or liquid form has to be added to the mixture to chemically increase the reaction rate. According to the type of activator used (e.g., whether liquid or solid), Further research by Torgal [26–28], Luukkonen [29, 30], and Provis and Bernal [18, 31] research groups have further divided the AAMs to two different activation techniques with one that tends to use liquid activator, referred to as "*two-part alkali-activated materials*," while the other uses cheaper and often more eco-friendly activator in solid form that is referred to as "*one-part or ready mix alkali-activated materials*".

With solid activator being much more recently introduced, the foundation on which the one-part AAMs is developed is dealing with the hazardous properties of the liquid activators that are highly corrosive and their transportation to the construction site requires following a specific set of protective measures [32]. In contrast, Through using solid activators (One-part AAMs), lower cost of the material, ease in transportation, lower environmental footprint [11], and most importantly, the ability to be utilized by only adding water to the mixture which mimics the current OPC applicability can be achieved. Yet, the chemical mechanism hierarchy, as discussed above, follows the same trajectory with tendency to dissolve the Si, Al, and Ca structure of the precursor and result in hardened material, irrespective of the type of activator (e.g., whether solid or liquid) [18, 33].

The various types of activators and the respective amount used, both in solid and liquid form, in that respect, increases the alkalinity (pH) of the medium and act as a catalyzer [34]. This reaction takes place according to the compositional elements available in the medium, and thus, the degree of this reactivity can partially be defined by it to the point that a mixture medium with an overall pH of 14 can potentially result in 50 times more strength development than the same medium with a pH of 12 [35]. The resulted mechanical property, nevertheless, is not only dependent on the type and content of the activator and precursor, but a variety of other factors including the *curing regime*, the *type and content of aggregate*, as well as presence of fillers.

Another major influential factors in the resulted AAMs properties include but are not limited to: the *total molar ratios* such as silicon to aluminum (Si/Al), *alkali concentration*, and *the ratio of used activators* (in case of using two different activators for optimum results). Unless such factors are addressed thoroughly, AAMs cannot potentially address the structural or durability purposes, defined or expected. Such shortcomings include shrinkage issues [10, 36, 37], alkali-silica reaction tendency [9, 38, 39], and efflorescence [40, 41]. Yet, the variation in properties due to mixture ratio and understanding the effect and multi-functionality of the chain of factors makes the very basis for this review. In that context, the following sections provide an insight on the three mentioned systems Fig. 1.

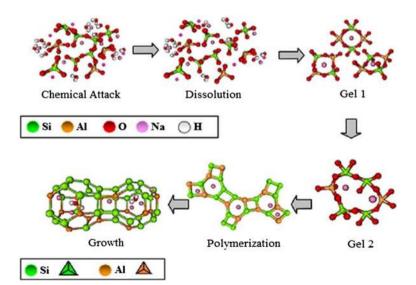


Fig. 1 Alkali-activation process in graphical illustration [42]

High Calcium-Based Systems

The development of alkali-activated systems based on calcium-rich precursors has been entertained for over a century [31]. In this system, precursors such as GGBFS, and class C fly ash are used in a relatively moderate alkaline condition [43]. The calcium participation in this system can be in form of (1) Ca $(OH)_2$, (2) substituting cations within the mixture and bond with it, or (3) react with dissolved silicate and aluminate species to initially form C-S-H gel [44]. This addition of high calcium materials mainly changes the setting time and the chemistry of the product [45] where, Al³⁺, Si⁴⁺, Ca²⁺, and Mg²⁺ are reported to be the main network modifiers in the reaction chain. These modifiers with alkalis result in formation of calcium aluminate silicate hydrate (C-A-S-H) [45-48]. Thus, the higher level of calcium leads to faster hardening of C-A-S-H gel phase, lower setting time [46, 48], and higher early strength [47, 49–51]. This gelation process has been reported to increase strength over longer periods of time if followed by curing in ambient conditions, similar to OPC concrete system which develops calcium silicate hydrate overtime [47]. On the opposite side, however, higher drying shrinkage and cracking [52], higher risk of steel corrosion through chloride ion exchange [53], and loss of durability especially at high temperatures (above 300°C) [54] have been reported for high calcium systems. These adverse effects on the characteristics of AAMs, are reported to be related to molar ratio of Ca/Si, Al/Si, and the type and amount of activator as well as the pH level of the medium, and as described by many, the formation of gypsum due to the presence of calcium [18, 31]. Although these factors are discussed at length in the following sections, for further information on the kinetics of high calcium systems, diverse range of Ca-aluminosilicate precursors, and precursor chemistry, reader is referred to [18, 31, 43, 55–58]

Low Calcium-Based Systems

Low calcium-based alkali-activated binders were initially developed as a fire resistance material replacing organic polymeric materials while receiving attention for low cost alternatives to fire resistant ceramics and OPC concrete [31, 59]. The content of low calcium system is primarily comprised of aluminum- and silicon-rich materials where the calcium content is kept relatively low. The fundamental binding structure in this system is reported to be highly disordered and include numerous cross-linked Si and Al in a tetrahedral coordination [31]. The low calcium systems have been reported for their lower permeability [60], better fire resistance [18, 61], longer setting time [18, 21], lower shrinkage and carbonation [45, 60], less porous microstructure [62], and higher chloride resistance [60] compared to high calcium systems. The reaction of this system can be defined in three stages that are distinctly different from the localized precipitation of C-A-S-H that takes place in high-calcium systems [45] which include (1) dissolution of Si-O-Si, Al-O-Al, and Al-O-Si bonds provided by the precursor (often referred as nucleation stage), (2) coagulation or polycondensation in which a coagulated structure between the disbanded/disaggregated composition from precursor takes place and (3) crystallization, in which crystals begin to develop and shape an inorganic hardened and 3dimensional polymer structure [18, 31, 43]. This process results in a high molecular or macromolecule polymer that is referred to as inorganic or geopolymer [18, 43]. Geopolymers are aluminosilicates that form through a hydrothermal condition. Such condition includes polycondensation of geopolymers in concentrated water-based cements or resins whose byproduct is an amorphous binding material with low crystallinity [59]. To start this reaction process, however, major thermal (60–90°C) or alkaline media is required [18, 23, 43, 63, 64]. This thermal curing, along with other factors including the SiO₂/Al₂O molar ratio, can adjust the degree of polymerization [62, 65] where if the proper mixture ratio is not met, the byproduct loses its applicability due to lack of strength development. In that sense, products containing Si-poor (Si/Al<1) and Si-rich (Si/Al>5) have been reported not to be suitable in constructional applications due to low strength and durability characteristics [31].

The final product, in this system, is N-A-S-H gel which is reported to have a higher setting time and often requires thermal curing to develop strength. In this process, the alkali alumina silicate with silicon and aluminum coherently binds and forms a group of 3-dimensional framework of silicate and aluminate that are water resistant where they show higher durability than OPC. In this system, the reaction usually requires specific thermal curing and does not react at room temperature; upon curing at temperature threshold of around $80-100^{\circ}C$ conditions, however, a conversion into crystalline ceramic phases can be observed. Different types of these crystalized byproducts are reported to withstand more than $1000^{\circ}C$ before being melted. Recent studies also showed geopolymers' good bonding with metals [66], refractory coating, and its application as a composite material adhesive [66], in which the degree of alkalinity can further tailor it, has created a great interest in concrete industry for further research [67].

Hybrid Systems

The production of hybrid systems consists of a combination of OPC or Portland cement clinker along with the use of an aluminosilicate and an alkali-binder where the byproduct has a CaO, SiO₂, and Al₂O₃ contents > 20% [43]. This blended system has been shown to provide promising results in terms of mechanical performance and durability and can be divided into two systems of (1) having *OPC or Portland cement clinker* and (2) having a combination of materials from *slag* family as well as another low calcium precursor such as class F, *fly ash* [31, 43, 67]. In this regard, the reaction byproducts are a combination of low and high calcium systems including C-A-S-H and N-A-S-H systems [43, 68]. In general, this system has been shown to have promising results that also do not require thermal curing as in low calcium system and can develop proper strength. [69], for instance, showed that hybrid system comprising of OPC and GGBFS activated with sodium hydroxide (NaOH) and sodium silicate (Na₂SiO₃) can reach 4.5 and 10.8 times higher compressive strength, respectively, with a dense microstructure as compared with the 100% OPC concrete system reference. The following sections further review the use of different activators and precursors.

Precursors and Aluminosilicate Sources

Precursors in AAMs are those aluminosilicate-bearing raw materials that were dissolve by the activators including fly ash [70, 71], GGBFS [29, 72], metakaolin [73], rice husk ash [74], red mud [75, 76], and certain other reactive materials rich in silica (SiO₂) and alumina (Al₂O₃) [77] that alter the binder's Si/Al ratio [78]. Since its discovery, a broad range of cementitious materials have been entertained as precursors in AAMs. Since their use is to provide a range of dissolved and reactive elemental materials that rearrange to harden, the variations in aluminum, calcium, and silicate contents of these precursors are the main measurable factors resulting in a variety of outcomes. Such outcomes are thus in direct relationship with the precursors' *Si/Al ratio, particle size, calcium content, as well as other elemental compositions* [79]. In that spirit, understanding the basic elements governing each precursor can provide the

proper understanding for the function each precursor can potentially play. To provide an insight on the elements constituting the fabrics of major precursors used, Fig. 2 shows the approximate difference in compositional variation in the common SCMs. Following this understanding, the following sections provide a brief overview of the most commonly used cementitious materials as precursors Table 1.

Fly ash

Fly ash can be divided into high-calcium (class C) and low calcium (class F) materials that are one of the most commonly used precursors in AAMs. The low calcium fly ash (class F) has been much more widely exercised as major precursor in AAMs due to its availability, and potentially better performance [18]. The use of low calcium fly ash allows a longer setting time and better workability than that of high calcium which is less available and often with more variable characteristics. Through the use of low calcium fly ash, due to reduce content of Ca, however, the reactivity of the mixture is drastically affected to the point that either another precursor with potentially higher content of calcium is used or thermal curing becomes the only measure to kickstart the chemical reaction [71, 72].

Ground Granulated Blast Furnace Slag (GGBFS)

GGBFS is one of the most commonly used precursors in AAMs mainly due to its rich sources of Ca^{2+} and Mg^{2+} . The production of GGBFS is estimated to be around

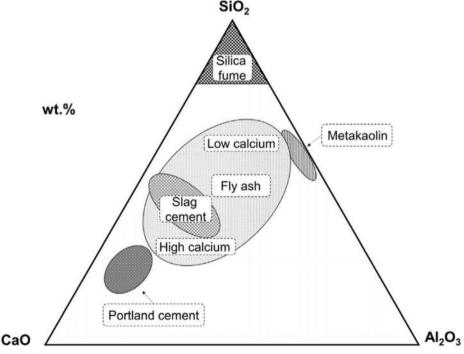


Fig 2 Ternary diagram of chemical composition of major cementitious materials [114]

Table 1 Characteris	stics of common waste mater	rials that can	be added to	Table 1 Characteristics of common waste materials that can be added to concrete and produce a more sustainable binding agents	able binding agents	
Name of additive	Usual Shape	Density (Kg/m ³)	Ave. particle size (µm)	Limitations	Benefits	References
OPC Silica Fume	Irregular and angular Spherical shapes	1440 130–600	0.1–250 0.15	Reduction in workability, and	Increase in compaction, mechanical strength, corrosion [81-86]	- [81–86]
Ground granulated blast slag (GGRS)	Angular with rough surface	1000-1300 125-250		increase in drying shrinkage Lower early age strength	resistance, density Increase in sulfate resistance, ITZ, and weathering resistance	[85, 87–90]
Fly Ash	Spherical shapes	540-860	0.5-300	Lower early age strength	Improve in workability, better long-term mechanical properties, lower shrinkage, and weathering	[85, 91–93]
Metakaolin	Porous, platy and angular 890	890	1-20	Reduction in workability	Enhances and octed autation resistance Enhances mechanical properties, Interfacial transition zone (ITZ) and microharchess	[85, 93–95]
Rice husk ash	Irregular shapes with high porosity	vith high 550–700	1–20	Variation in mechanical and durability properties according to the size and mulity	Reduces shrinkage and bulk density and enhances the microstructure packing	[66-96]
Glass powder	Angular shapes	1800	ı	Expansion, ASR, lower hydration	to ute size and quarity Expansion, ASR, lower hydration In fine scales can enhance the packing and increase and interaction	[100–105]
Red mud	Irregular and needle shaped particles	~ 2700–3- 200	0.8-50	High impurities	High alumina content can participate in geo-polymerization	[106–108]
Municipal solid waste incineration	Irregular	~ 1700	100 to above	·	Better leaching performance, increasing the denseness [109–112] and homogeneity	[109–112]
Paper sludge	irregular	660–1690	Below 100	1	Favorably adjusting Si/Al ratio	[113]

300 million tons annually [115] which promises a steadily available silica-rich material with low cost. GGBFS is generally consisted of SiO₂, CaO, Al₂O₃, and MgO, with almost the same composition as in metakaolin with different ratio and much more availability. GGBFS-based AAMs have been reported to have high early mechanical strength and durability against sulfate and acidic presence [116], lower greenhouse gas effect [117, 118], and higher fire resistance [119]. The reactions of slag are reported to be predominantly dominated by particle size. In that respect, the particles below 20 μ m with majority below 2 μ m are reported to react within the first 24 h of curing [18]. The use of GGBFS-based AAMs, however, is reported to have certain drawbacks including a high rate of shrinkage and subsequent formation of cracks as well as volumetric instability [120].

Recent studies, as in [121], have demonstrated that higher volumes of GGBFS within the mixture can increase the number and volume of microcracks, reducing the hardened state properties of the concrete. Yet, just as the content, the proper curing regime is also an effective influential factor in strength and quality development. As noted by [120], depending on the content, system, and type of activator, major variation in crack volume and strength development can be expected.

Metakaolin

Metakaolin results from the hydroxylation of kaolinite Si_2O_5 , $Al_2(OH)_4$ which occurs at around 750°C. Its main chemical components include silica (SiO₂, ~44.4–73%) and alumina (Al₂O₃, ~14.5–47.43%), with varying particle size of 1.20–38 µm and surface area of 2.16–22 m²/g [122, 123]. Metakaolin is a key component especially utilized with low calcium based AAMs that adjusts Si/Al of the binder. Metakaolin is reported to enhance the polycondensation rate and effectively increase reactivity of Class F fly ash to form denser nano- and micro-structures, reaching higher mechanical properties if cured at high temperature [61, 124].

Rice Husk Ash

Rice husk ash is a silica-rich aluminosilicate source after silica fume and nano-silica, having a content of more than 90% SiO₂, as compared to OPC with ~20%. Rice husk ash is reported to enhance the bonding by creating Si-O-Si bonds which promote better mechanical properties than Al-O-Al and Si-O-Al that is in the mixture [61, 125]. According to [75], the major varying factor in its reactivity can be traced to its particle size that produces better performance in smaller sizes.

Silica Fume

In general, silica fume represents a pure silica material; the use of which, in alkaliactivated mix, is generally as filler. In that regard, literature shows that the use of silica fume due to its small particles has an invariable positive effect on the mechanical and durability properties of the AAMs. This tendency is rooted in providing more available Si content for further reaction of aluminosilicate sources in the mixture [32] as well as acting as filler and reducing the permeability of the hardened AAMs [126].

Other Sources of Precursors

Due to vast generation of different waste materials, there are a number of attempts in finding a cheaper alternative for construction materials. In AAMs, red mud, paper sludge, glass powder, and mine tailings, municipal solid waste incineration ash (MSWI ash) has been reviewed in Table 2 for further reference.

Curing Techniques

In alkali-activated materials, since the chemical proportion of the mixture varies in accordance to the type of precursor used, often the low reactivity requires a specific technique to heighten the reactiveness of the mixture [18]. Depending on the system of alkali-activated materials, *heating (thermal curing or oven), sealing (wrapping), steaming,* and *water immersion* are usual techniques used to achieve optimum properties. In this context, in thermal curing, in order to increase the chemical reactivity at first hardening stages, most commonly, a temperature range of 60–80°C is used for the first 24 h [134, 135]. Such technique is advised to be used on low calcium system that has a lower reactivity rate. Using such technique, recent studies reported reduced porosity, and significant strength gain [18, 75, 136]. Elongated

Name	Ref.	description and comment
Red mud	[106, 107]	Red mud is the byproduct of alumina production and mainly consists of Fe, Al, and Si but has a lower Si/Al ratio, yet higher alkalinity, compared to major SCMs. Yet, it can be used in smaller quantities (>15%), in AAMs as filler of precursor replacement
Paper sludge	[127, 128]	Paper sludge is the byproduct of paper making process that is made of micro-fibers. Its use in AAMs has been reported to cause a reduction in drying shrinkage, flowability, as well as mechanical properties. Such results are partially associated with the major Fe and Si compositional ratio
Glass	[129–131]	1
Mine tailings	[132, 133]	Tailings as result of mining operations differ in their compositional contents. In AAMs, favorable Si/Al ratio, rapid mechanical strength development, and high acid resistance have been reported
Municipal solid waste incineration ash	[109–112]	MSW incineration ash is produced as the result of combustion plants where the resulted ash has an estimated 10–15% and 20–35% of its original MSW volume and weight, respectively. MSWI ash has been reported to have an intensely heterogeneous composition. In AAMs application, promising results such as better leaching performance, often better mechanical properties as compared to their SCM counterparts, have been documented

Table 2 other sources of waste-based aluminosilicate precursors

thermal curing, however, has been associated with drying shrinkage, higher porosity, and finally a loss of hardened state properties. Yet, water curing, depending on the system, has been reported to cause a dilution of reaction which is the result of reduced pH, resulting in lower strength gain, and in leaching of the activator [137]. As opposed to water immersion technique, wrapping and sealing can reduce the environment's effect on specimen, such as evaporation of water in one-part alkali-activated system, which has been reported to reduce porosity and overall shrinkage cracking [138–142]. Irrespective of the developed mechanical properties, proper curing technique can significantly affect physico-chemical and durability properties that can further be characterized through microscopic analyses such as Fourier transform infrared spectroscopy (FTIR) [143, 144], X-ray diffraction (XRD) [145], scanning electron microscopy (SEM) [30], thermogravimetric (TG/TGA), and differential thermal analysis (DTA). According to the results outlined by [145, 146], microwave curing appears to provide a harmonized effect on specimen that allows a full reaction aluminosilicate source, to react in the medium (Fig. 3).

Activators

Alkali activation is a complex and multi-chain system that takes place in alkalinity of the mixture where the aluminosilicate materials dissolve to form a new network structure. This process starts by ion exchange and hydrolysis of Si, Al, and their network breakdown. The alkaline solution mainly has two basic roles in the geopolymer mixture: (1) dissolving Si-O and Al-O bonding and their subsequent re-establishment in the geopolymer network and (2) charge-balancing of the mixture by alkali-metal cations [124]. In short, alkali activator acts as a catalyst in the reaction allowing the new and polymeric formation. [147], for instance, showed that the higher molar ratios of SiO₂/Al₂O₃ and Na₂O/Al₂O₃ lead to higher mechanical strength

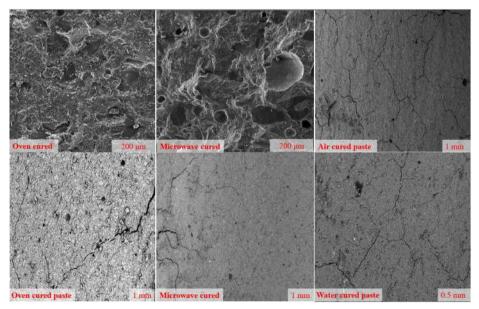


Fig. 3 Microstructural assessment of differently cured specimen, based on the result of [30, 145, 146]

and density, while in terms of activators, sodium hydroxide (NaOH) proved to have better performance than potassium hydroxide (KOH). Table 3 outlines the recommended molar ratio of geopolymer.

Depending on the state used, sodium hydroxide (NaOH), potassium hydroxide (KOH), sodium sulfate (Na₂SO₄), potassium carbonate (K_2CO_3) are major activators utilized in liquid form. In solid form, sodium metasilicate (Na₂SiO₃), sodium carbonate (Na₂CO₃), and potassium hydroxide (KOH) are the major activators used. In that spirit, the sodium-based alkaliactivators are generally more available at lower costs with high reactivity, while potassiumbased activators have been numerously entertained for high temperature applications [149]. Table 4 further reviews a basic description of the mentioned activators.

Liquid Activators and Mechanical Properties

To increase the pH of the medium to dissolve the precursor, liquid activators were first used in production of two-part alkali-activated materials. One of the major issues with such method was then found to be transporting and handling such corrosive liquid activator. Studies that compared the result of the two types of activators have shown that in case of utilizing liquid activators such as sodium hydroxide or sodium silicate, in higher thermal curing temperatures, a relatively higher degree of porosity can be expected [157]. Yet, in terms of CO2 production of the activators, it has been reported that hybrid use of activators (liquid and solid) can be the most optimized. [158], for instance, studied the use of composite solid activators by substituting solid sodium carbonate (Na₂CO₃ anhydrous) in half by liquid sodium carbonate (Na₂CO₃). In their research, it has been shown that Na₂CO₃ has far less greenhouse effect and cost almost half of Na₂CO₃ anhydrous Tables 5, 6.

Solid Activators

To avoid transportation of the hazardous liquid activator, more recently, much attention is paid to the development of solid activators that can potentially be used by just adding water to the medium. Sodium metasilicate (Na_2SiO_3) is the major type of activator used as solid activator. According to [166], activation through sodium metasilicate offers higher ultimate strength gain with lower porosity than sodium hydroxide. In that respect, [167] illustrated that the higher percentage of sodium metasilicate increases the heat of hydration, while [168] showed that sodium metasilicate-activated specimen develop a better early mechanical strength.

Other solid activators include paper sludge, red mud, and oyster shell that have recently been used. [29], for example, exercised the use of paper sludge as a dry-solid activator and a source of calcium carbonate along with liquid NaOH with different content ratios on slagbased geopolymer. It was reported that higher dosage of paper sludge, even in unreacted form, can increase compressive strength and act as filler in the geopolymerization process. In the

Range
3.50-4.50 0.80-1.20 15-17.50 0.20-0.28

 Table 3 Outlining the recommended molar ratios for geopolymer [15, 18, 148]

Name	Common state used	description and comment	Chemical structure
Sodium hydroxide	Liquid	Sodium hydroxide (<i>NaOH</i>), also known as <i>caustic soda</i> , is an inorganic compound that has a variety of uses in manufacturing processes including soaps, paper, dye, and petroleum byproducts. Since it is a <i>strong base</i> , it has a corrosive nature and can cause allergenic reactions and skin irritations [150]. It can be found in liquid and solid states that are both colorless and have no odor.	Na H
Sodium silicate	Liquid	Sodium silicate is a general name of any chemical compound that has sodium oxide, (Na ₂ O) _n , and silica ,(SiO ₂) _m , in it. It has a variety of applications in construction industry that includes sealing of concrete cracks, dissolving agent in AAMs, and setting accelerator [151]. The commercially available Sodium silicate has a pH of around 10 to 13, inversely relating to the silica content.	
Sodium carbonate	Solid	Sodium carbonate is another inorganic compound that is water- soluble. With formula Na ₂ CO ₃ , is has a high concentration of bicarbonate that increases pH or leads to dissolution of other matters within the medium [152]. This solid material can be produced from natural sources of trona and sodium carbonate brines as well as nahcolite mineral (naturally occurring sodium bicarbonate) sources [153] which commonly occurs as crystalline decahydrate that subsequently effloresces and forms an odorless and white powder [154].	
Sodium metasilicate	Solid	Sodium metasilicate is main component of sodium silicate with Na ₂ SiO ₃ formula. The production of Sodium metasilicate is an energy-intensive process that requires the fusion of Silica sand (SiO ₂) with sodium carbonate (soda ash) that occurs at around 1400°C [155].	
Potassium hydroxide	Solid	With formula KOH, potassium hydroxide is a strong base that is commercialized in pellets, flakes and powder that is known for its corrosiveness tendency to absorb moisture from the environment. The production of Potassium hydroxide is done through electrolysis of potassium chloride. Severe reactions, skin irritations and other hazardous side effects have been documented as a result of contact with it [156]	K H

Table 4 Major activators used in literature with their state (solid or liquid) as well as their description

same way, [11] exercised the use of red mud as a NaOH supplier and unburned fly ash as an aluminosilicate precursor in one-part Alkali-activated concrete. Comparing the resulted AAM, activated with NaOH versus that of red mud, both results have shown the same performance in terms of mechanical characteristics and Na/Si ratio. The authors then found the same linear increase between compressive strength and Na/Si ratio due to accelerated dissolution of aluminosilicate precursors. In that respect, almost the same results in mechanical properties between NaOH-activated and red mud-activated samples were found, concluding that the use

Binder	Alkali-activator	Mechanical pr (28 days)	operties	Aggregate	Curing	Reference
		Compressive (Mpa)	Split tensile			
SF-MK	Sodium hydroxide-sodium silicate	28	-	Max 0.6mm	Air	[146]
SF-MK	Sodium hydroxide-sodium silicate	38	-	Max 0.6mm	Heat 70°C-Air	[146]
SF-MK	Sodium hydroxide-sodium silicate	41.5	-	Max 0.6mm	Microwave	[146]
GGBFS	Sodium hydroxide-sodium silicate	52	-	Max 0.6mm	Air	[146]
GGBFS	Sodium hydroxide-sodium silicate	54	-	Max 0.6mm	Heat 70°C-Air	[146]
GGBFS	Sodium hydroxide-sodium silicate	65	-	Max 0.6mm	Microwave	[146]
MK	Sodium hydroxide-sodium silicate	35	-	Max 0.6mm	Air	[146]
MK	Sodium hydroxide-sodium silicate	39	-	Max 0.6mm	Heat 70°C-Air	[146]
MK	Sodium hydroxide-sodium silicate	47	-	Max 0.6mm	Microwave	[146]
FA	Sodium hydroxide-sodium silicate	37	-	Max 20mm	Air	[159]
GGBFS	Sodium hydroxide-sodium silicate	105	-	Max 20mm	Air	[159]
FA - GGBFS	Sodium hydroxide-sodium silicate	88	-	Max 20mm	Air	[159]
FA - RHA	Sodium hydroxide-sodium silicate	16	-	Max 20mm	Air	[159]
GGBFS - RHA	Sodium hydroxide-sodium silicate	57	-	Max 20mm	Air	[159]
GGBFS	Sodium hydroxide-sodium silicate	42.5	-	Max 19mm	Air	[160]
GGBFS	Sodium hydroxide-sodium silicate	32	3.1	Max 2mm	Steam	[161]
GGBFS	Sodium hydroxide-sodium silicate	39.5	3.3	Max 10mm	water	[162]
FA-GGBFS	Sodium hydroxide-sodium silicate	65	-	20mm	Heat 70°C—air	[163]
FA	Sodium hydroxide	1.5	-	N/A	Air	[164]
FA-GGBFS	Sodium hydroxide	18.3	-	N/A	Air	[164]
GGBFS	Sodium hydroxide	27.1	-	N/A	Air	[164]
FA	Sodium hydroxide-sodium silicate	42.8	-	N/A	Air	[164]
FA - GGBFS	Sodium hydroxide-sodium silicate	114.5	-	N/A	Air	[164]
GGBFS	Sodium hydroxide-sodium silicate	171.7	-	N/A	Air	[164]
FA-GGBFS	Sodium silicate	54.9	-	N/A	Air	[164]
GGBFS	Sodium silicate	173	-	N/A	Air	[164]
FA	Sodium hydroxide	45	-	Max 0.1mm	Heat	[165]
					(80°C)—air	[]

Table 5 The type of precursors and activators used in two-part AAMs

SF silica fume, GGBFS ground granulated blast furnace slag, FA fly ash, MK metakaolin

Binder	Alkali-activator	Mechanical p (28 days)	roperties		Aggregate	Curing	Reference
		compressive (Mpa)	split tensile	Flex			
GGBFS	Sodium metasilicate	66		6	Max 2 mm	Ambient	[169]
GGBFS	Sodium silicate	107	-	-	Max 2 mm	Plastic	[32]
Rice Husk	Sodium hydroxide	37	-	-	Max 2 mm	Plastic	[32]
SF	Sodium hydroxide	35	-	-	Max 2 mm	Plastic	[32]
GGBFS	Sodium silicate	54	-	5	Max 1.6 mm	Water	[30]
GGBFS	Sodium silicate	63.5	-	8.3	Max 1.6	Plastic	[30]
GGBFS	Sodium silicate	40.5	-	4.2	Max 1.6 mm	Air	[30]
GGBFS-FA	Sodium metasilicate-sodium hydroxide	75	-	-	N/A	N/A	[170]
GGBFS-FA	2	72	-	-	N/A	Steam 20°C	[170]
GGBFS	Sodium hydroxide-sodium oxide	21	-	-	N/A	Water 23°C	[171]
GGBFS	Sodium silicate	30	-	-	N/A	Steam 20°C	[172]
FA-C	Sodium metasilicate	49	3.5	4.7	Max 20 mm	Water	[173]
FA-C	Sodium metasilicate	45	3.1	6	Max 20 mm	Air	[173]
FA-C	Sodium metasilicate	52	4.75	5.2	Max 20 mm	Solar 23°C	[173]
FA-GGBFS	Sodium metasilicate	39	-	7.4	N/A	Air	[174]
FA-GGBFS	Sodium metasilicate	36	-	6.4	N/A	Heat 80°- C—air	[174]
FA-GGBFS	Sodium metasilicate - sodium carbonate - potassium hydroxide	44.9	-	-	Max 2.5 mm	Heat 40°- C—air	[175]
FA-GGBFS		40.9	-	-	Max 2.5 mm	Heat 60°- C—air	[175]
FA-GGBFS	Sodium metasilicate - sodium carbonate - potassium hydroxide	48	-	-	Max 2.5 mm	Air	[175]
FA-GGBFS	Sodium silicate-sodium carbonate-potassium hydroxide	27.9	-	3.4	Max 2.5 mm	Air	[176]

Table 6 The type of precursors and activators used in one-part AAMs

SF silica fume, GGBFS ground granulated blast furnace slag, FA fly ash

of red mud as a waste material can provide a desirable outcome. [21] exercised the use of oyster shell as solid activator in one-part alkali-activated binder using slag as the main precursor. In their study, 5% oyster shell was shown to be the most effective which improved

mechanical and microstructural properties. In higher dosage, however, it was shown to decrease the reaction rate and significantly increase capillary pores.

Alkali Concentration of Activators

Considering an aqueous solution to comprise of *solvent* and *solute*, alkali concentration is a measure of the dissolved moles in the solution. This, in other words, translates into the moles of the solute expressed by the volume liters as written in the equation below:

$$Molarity (M) \frac{The number of moles of solute (n)}{Volume of the solution (v)} = \frac{Mol}{Liter}$$

In alkali-activated materials, the molarity, or concentration of the activator, has been proven to be an accelerating factor in the hydrolysis of the aluminosilicate materials [177]. In lower concentrations, insufficient dissolution of the precursors as well as lower polymerization heat is reported [178]. This, however, should not be confused with optimum concertation. As discussed in length by [179, 180], unconditionally high concentration of alkaline activator can result in efflorescence, brittleness, higher porosity, and a reduction of overall mechanical and durability properties. This phenomenon can be traced to premature coagulation due to potentially faster dissolution of precursors in the mixture [177]. Outlines the variation of potassium hydroxide concentration versus the 28 days developed compressive strength, according to which, the optimum concentration for that specific set of variables is 10 M (Table 7).

Durability Factors

Given the generally higher porosity of AAMs as opposed to OPC system, the durability factors can potentially be more influential and variable. In general, the durability of concrete is significantly impacted by the presence of pores that allow aggressive substances including Cl⁻ and SO⁻²₄ to enter and transport within the

Concentration (KOH)	Si/Al	K/Al	K/Si	K ₂ SiO ₃ /KOH	Compressive strength (28 days)
6	2.4	0.15	0.36	1	19.5
8	2.4	0.18	0.44	1	20.5
10	2.4	0.21	0.51	1	24
20	2.4	0.38	0.91	1	21.5
30	2.4	0.54	1.1	1	21
40	2.4	0.7	1.69	1	23.5
6	2.46	0.13	0.33	1.5	14.5
8	2.46	0.16	0.39	1.5	17
10	2.46	0.18	0.45	1.5	29
20	2.46	0.31	0.77	1.5	26
30	2.46	0.44	1.08	1.5	16
40	2.46	0.57	1.39	1.5	21

Table 7 Different concentration of potassium hydroxide with its prospective Si/Al, K/Al, and K/Si ratios (data from [149])

concrete [124]. Yet, the presence of pores has been recognized to be dependent on factors including curing time, activator concentration, liquid to solid ratio (and water to binder), and the dosage and availability of silica and calcium to react within the mixture [60, 181]. In that respect, the content and type of aggregates, acid resistance, shrinkage, porosity, permeability, fire, and temperature resistance are major influential factors that directly affect durability and are reviewed in the following sections.

Alkali-Silica Reaction

The role of aggregate in AAMs is the same as in OPC concrete; however, high alkalinity and reactivity are reported to increase surface reaction with the binding agent. In this context, Alkali-aggregate (or alkali-silica) reaction is a chemical reaction caused between hydroxyl ions in alkaline medium and reactive silica in the aggregate that creates expansive sodium calcium silicate gel that increases cracking [18]. In this situation, the internal pressure generally cracks the concrete, reducing durability and mechanical properties of the concrete [182]. To avoid such phases, reduction in calcium content [183], as well as a more durable type of aggregate, can potentially increase acidic resistance of AAMs [124].

In general, given that three phases are met, alkali-silica reaction is reported to occur: (1) presence of excessive activator (high alkalinity), (2) availability of excessive moisture, and (3) the presence of reactive siliceous phases in the aggregates. The mentioned reactive silica in aggregates comprise of amorphous silica, unstable crystalline polymorphs of silica, poorly formed crystalline silica, and some type of defectively oriented quartz [184]. Apart from the mentioned factors, composition of the aggregate can also potentially affect the porosity, alkalisilica reaction, and total hardened state properties. [185], for instance, showed that the inclusion of ceramic aggregates reduces the drying shrinkage and due to their aluminum and silica content contributes to the alkalization and increases the surface bonding of aggregate and leads to higher compressive strength while absorbing more water from mixture and internal pores.

Acid Resistance

One of the major superiorities of AAMs is their acidic resistance. Acidic resistance is essential in repair applications and sewage structures that require high sulfate and acidic resistance. The deterioration mechanism of acid exposure starts by alkali cation exchange with hydronium ions [28] whereby the Si-O-Al bonds destabilize and forms Si-OH and Al-OH group of bonds [186, 187]. Through this process, soluble salts emerge that is the result of a significant loss of performance. Literature shows that the AAMs are capable of having 70–80% less acidic degradation compared to OPC [188, 189]. Such performance can be traced to the reduced amount of calcium present in the AAMs compared to OPC as well as higher resistance of SCMs to acid, as compared to cement clinker [31, 42] (Fig. 4).

Shrinkage

Shrinkage constitutes major causes of cracking that allows transportation of harmful substances. Shrinkage is generally categorized into (1) drying shrinkage, (2) plastic shrinkage, (3) autogenous shrinkage, and (4) carbonation shrinkage. Recent studies outlined the higher rate of drying and autogenous shrinkage in high calcium systems [37]. Yet the rate of reaction,

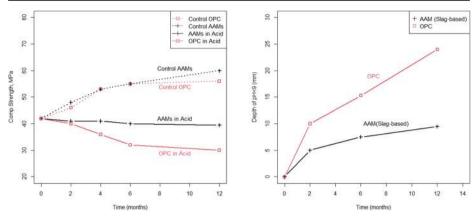


Fig. 4 Resketched from [189], showing the acid resistance of Slag-based AAMs and OPC samples

internal relative humidity, and surface tension have direct relationship with the size and amount of autogenous shrinkage. In that respect, internal curing, reduced or calculated Ca/Si ratio, as well as shrinkage reducing agents are advised to reduce shrinkage [190]. Such methods, however, have been shown to not only reduce the strength gain but also adversely affect setting time and modulus of elasticity [176, 191]. Yet, the influence of the type and content of activator used has been reported to be more pronounced [192]. As depicted in Fig. 5, higher content of activator generally results in higher overall autogenous shrinkage due to higher intensity of hydration. This phenomenon can be traced to the increased consumption of moisture of the micro-pores and available content of silica that results in volume change [192].

Porosity

Classification of pores within alkali-activated materials is the basis for understanding the multi-functionality of precursors, activators, and the type of adopted curing. The system of pores can be divided into *gel pores* that are within the C-S-H gel (in OPC system) or C-(N)-A-S-H gel (in AAMs), and *capillary pores* that refer to the space left by water not filled during

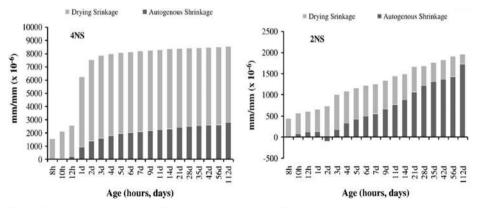


Fig. 5 Shrinkage variation through different contents of Sodium silicate activator (4NS: 4.5% Na₂O, 2NS: 2.5% Na₂O) [192]

hydration. Other pore classification is generally either based on their shapes, such as geometric pores [193], or their type, such as (1) open pores, (2) through pores (pores open from two sides), and (3) closed pores [194]. In size, however, they are generally categorized as micropores (below 20 nm), meso-pores (around 20 nm), macro-pore (20–50 nm), and fracture (above 50 nm) [195, 196]. Such pores are recognized to be unharmful, unfavorable, harmful, and detrimental, respectively, to the durability and strength of the concrete. To characterize the size of pores, mercury intrusion porosimetry (MIP) test is used as a general means to measure the volume and size of pores; the result of which is inversely related to bulk density.

In thermal curing, for instance, the water content within micro-pores is evaporated, leaving a more micro-pores that decrease the overall density. Thermal curing with reduced intensity (~60–80°C), however, has been reported to lower porosity that mainly increases the reactivity while allowing the formation of N-(C)-A-S-H gel [197, 198]. In the same way, higher CaO content and improper mixture ratio of activator can potentially affect the results of MIP test. In this context, [192] showed the influence of silica content and an increase in the amount of calcium silicate on total porosity. As shown in Fig. 6, by increasing the amount of silica and activator, possibly, due to an increase in polymerization, the total porosity decreases to almost half of its other counterparts.

Permeability

Unlike porosity, permeability characterizes the rate of flow of a substance (a fluid) through the porous concrete. In general, a variety of factors affect permeability and water absorption that include the content and type of precursors, activators' modulus and concentration, the materials' total surface area and size, materials' packing (cohesion), and water/binder ratio [60, 199, 200]. In terms of the type of precursors, [201] showed the volume of permeable voids with different ratios of GGBFS with fly ash, noting that C-A-S-H binding gels dominate the microstructure which is denser than their counterpart, resulted from fly ash. It was then shown that the OPC system has a relatively lower permeable voids compared to fly ash-based alkali-activated

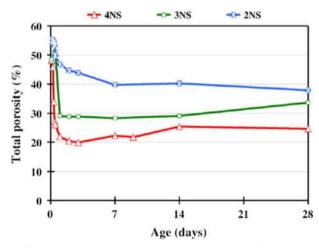


Fig. 6 Showing the influence of addition of activator on the total porosity of slag-based AAMs (4NS: 4.5% Na2O, 3NS: 3.5% Na2O, 2NS: 2.5% Na2O) [192]

materials. Such results are aligned with previous studies as in [202] where it was shown that each binding gel system promotes different pore structure and porosity. Yet, permeability has been shown by numerous studies [201, 203] to decrease through elongated hydration and polymerization in proper curing conditions which promotes chemical conversion of alumino-silicate materials. A consistent relationship, in that respect, has also been shown to exist between water permeability, total porosity, and pore diameters [204, 205] (Fig. 7).

Fire and Temperature Resistance

Alkali-activated materials have been reported to be significantly resistant to elevated temperatures, as compared to OPC system [206, 207]. [208], for instance, studied three main parameters including vitrification temperature (1200 and 1300 °C), heat treatment duration (2 h and 3 h), and the degree of alkalinity in specimen exposed to high temperatures. Using XRD and FT-IR spectrometry, it was found that albite, microcline, quartz, calcite, and lime to be the main components of their used precursor which, after being heated for 1300°C, in effect, resulted in the disappearance of calcite, guartz, and lime phases and the formation of new minerals with lower degree of crystallinity. Further research by [208, 209] showed that the disappearance of calcite, lime, and quartz minerals is due to the formation of calcium silicate phases which will increase by increasing the heat treatment. This phenomenon was then concluded to prove the role of heat treatment and higher temperature on the formation of new crystalline phases. In heat cured regime, it was reported that the specimen developed higher mechanical strength on heating up to 200–400°C, while a moderate strength decrease was noted from 700°C. After 1000°C, however, the strength and mass loss were noted to be significant, addressing that the effect of high temperature on phase transformations varies according to mix-design and Si/Al ratio (Fig. 8).

Materials Availability and Future Projections

Alkali-activated materials are generally viewed as major alternatives to ordinary Portland cement mainly consisting of waste materials. The increase in its use can be viewed as the excessive availability of SCMs such as fly ash and GGBFS during 1970s and 1980s. With the current trend in the reduction of fly ash production, as a result of environmental concerns (11–

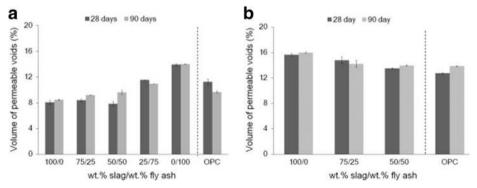


Fig. 7 Average volume of permeable voids of (a) mortar samples and (b) concrete samples based on alkaliactivated slag/fly ash blends [201]

Deringer

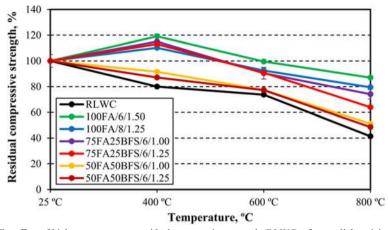


Fig. 8 The effect of high temperature on residual compressive strength (RLWC reference lightweight concrete, Fa fly ash, BFS GGBFS) [210]

20% until 2050 [208, 209]), it is safe to assume that the use, entertain, and practice of newer cementitious alternatives with often lower costs and lower greenhouse gas effects contribution are projected to be more attractive to cement scientists. The production of slag family, however, seems to remain unchallenged, and their use in different industries is uprising just as the search for newer aluminosilicate sources. Materials such as glass powder, paper sludge, MSWI ash, and red mud can directly replace cement because they have cementitious properties or can be used as fillers increasing mechanical and durability properties of final concrete.

In any case, however, the use of safe, clean, and rather cheap binders can open new opportunities for use in hybrid binder systems that resemble or exceed the properties of ordinary Portland cement with reduced environmental concerns. Current trend in use of solid activators and the documented results obtained from solid activators have provided a promising means to the future of alkali-activated materials with reduced environmental impact due to the type of activator and a cost reduction associated with the ease in transportation of liquid activators (Fig. 9: https://doi.org/10.1016/j.cemconres.2019.05.008, https://doi.org/10.1007/s40940-021-00155-9) (Table 8).

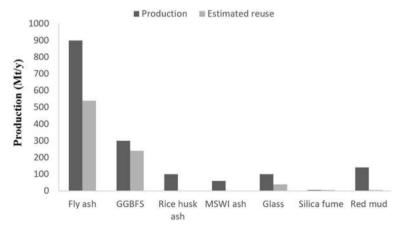


Fig. 9 Estimated production and reuse of different precursors (amounts in Mt/y)

Material	Major compositions	Production (Mt/y)	Estimated consumption (Mt/y)
Silica fume	Si	1-2.5	70–90%
Ground granulated blast furnace slag (GGBFS)	Ca-Si-Al	300-360	70–90%
Fly ash	Si-Al and Si-Ca-Al	900	58-64%
Metakaolin	Si-Al	2.2-2.6	N/A
Rice husk ash	Si-C-K ₂ O	100-200	Negligible
Glass powder	Si-Na-Ca	50-100	35-40%
MSWI ash	Si-Al-Ca	30-60	Negligible
Red mud	Fe-Al-Si	140	2–4

Table 8 Compiled from [213, 214], [215–223] (mt/y: million metric ton)

Conclusion

In this article, a review of precursors, activators, and their reported properties have been provided as well as the current state and projections of future material availability. The following further summarizes and highlights the points discussed within this review:

- Fly ash, ground granulated blast furnace slag, metakaolin, and rice husk ash are major *aluminosilicate sources* (precursors) used in literature.
- Three main systems in alkali-activated materials are *high calcium*, *low calcium* (*geopolymer*), *and hybrid* (*blended*) *systems* that are made of mixtures with different ratios of calcium and ordinary Portland cement contents.
- Sodium silicate and sodium hydroxide as opposed to sodium metasilicate are major activators used in two-part and one-part alkali-activated materials, respectively.
- Depending on the system, *heating*, *sealing* (*wrapping*), *steaming*, *and water immersion* is major curing systems used in alkali-activated materials.
- The use of proper *curing technique* can significantly affect the *porosity, mechanical, durability, and serviceability* of the alkali-activated materials.
- Low calcium-based alkali-activated materials, often requires thermal curing to increase their chemical reactivity.
- *High calcium-based* alkali-activated materials show a relatively *lower durability properties* such as higher shrinkage, as opposed to low calcium system.
- Both low calcium and high calcium systems have a *higher temperature resistance* as opposed to ordinary Portland cement systems.

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Code Availability The authors declare that no code is used for the purpose of this article.

Author's contribution Mehrab Nodehi: conceptualization; data curation; investigation; resources; writing original draft; Vahid Mohammad Taghvaee: validation.

Declarations

Consent to Publish The authors declare their consent to publish this article in the journal of Circular Economy and Sustainability.

Conflict of Interests The authors declare no competing interests.

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