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Review article

Critical methods of geopolymer feedstocks activation for suitable industrial applications

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ABSTRACT

As health and safety issues emanating from human activities on terrestrial environment is becoming ever challenging, the production of Ordinary Portland Cement is identified as a key contributor. This technology threatens environmental quality by emitting significant quantity of carbon dioxide (CO_2) that threatens Net Zero delivery. Consequently, the development of cement alternatives with substantial CO_2 reduction/sequestration during production has become imperative. Geopolymers obtained from industrial residues are poised as promising alternatives in managing environmental systems but selection of appropriate method of activation has limited their wider industrial applications. This article discusses four key activation methods and their combinations used in four main feedstocks to advise on their energy requirements, product compressive strength and environmental/industrial applications. Reviewing and characterising 302 published literatures with focus on most relevant and recent advances in the field, this review found that hybrid techniques combining mechanical activation method produces geopolymers with the highest compressive strength and thus the best method. Geopolymer made by mechano-chemical activation method of slag achieved the highest compressive strength while geopolymer produced by microwave assisted activation of clay and ultrasonic activation of fly ash cum slag are most economical in curing energy demand. Hybrid activation is the current development in the field and integration of this method with mechanical activation is poised as the future geopolymer activation technology as it demonstrates greatest efficiency potential.

1. Introduction

Production of Ordinary Portland Cement (OPC) used in Civil Engineering construction generates significant carbon dioxide (CO_2) emissions during the process of calcination of limestone and other raw materials. The effect of CO_2 on environment is reported in Refs. [1–13]. To reduce the quantity of CO_2 emitted during production of construction materials globally, research on environmentally friendly construction material has evolved and is advancing. A common cementitious material identified as a potential OPC replacement is geopolymers [14–19]. Geopolymers were first discovered in the 1970's by Davidovits who created the amorphous three-dimensional alumina-silicate binder materials [20–26] and named them geopolymers [27]. They are ceramic and thus inorganic in nature. They form alumino-silicate compound that is covalently bonded in non-crystalline (amorphous) structure [28]. Its raw materials are basically waste byproducts of various industrial operations such as fly ash and blast furnace slag. As an alternative to

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Table 1
Vital review papers in the field of geopolymer, feedstock and activation method.

S/N	Author	Paper title	Focus	Scope
1	Mackenzie, K. J. D., & Welter, M. (2014).	Geopolymer (aluminosilicate) composites: Synthesis, properties, and applications.	* Synthesis, properties, and application of geopolymer matrix * Reviews the characteristics of fibre-reinforced geopolymer composites.	Discussed only one activation method
2	Singh, B., Ishwarya, G., Gupta, M., & Bhattacharyya, S. K. (2015).	Geopolymer concrete: A review of some recent developments. Construction and Building Materials	* Alkaline activation method * Mechanical/chemical behaviour of material	Limited to fly ash Limited to one activation method
3	Duxson, P., Fernández-Jiménez, A., Provis, J. L., Lukey, G. C., Palomo, A., & van Deventer, J. S. J. (2007)	Geopolymer technology: the current state of the art	showed that the raw materials and processing conditions are critical in determining the setting behavior, workability and chemical/physical properties of geopolymer production	* Limited to one activation method
4	Ramesh, G. (2021).	Geopolymer Concrete: A Review.	This paper help understand Geopolymer Concrete. It gave a quick review of the Geopolymer Concrete.	Focused on one activation method.
5	Zhang, M., Guo, H., El-Korchi, T., Zhang, G., & Tao, M. (2013)	Experimental feasibility study of geopolymer as the next-generation soil stabilizer	illustrated that metakaolin based geopolymer can be an effective soil stabilizer for clayey soils	* Focused on one activation method * Limited to one feedstock material
6	Palomo, A., Grutzeck, M. W., & Blanco, M. T. (1999)	Alkali-activated fly ashes: A cement for the future	mechanism of activation of a fly ash was highly alkaline solutions	* Focused on one activation method * Limited to one feedstock material
7	Davidovits, J. (2013).	Geopolymer Cement a review.	Reviewed various feedstock material	* Focused on one activation method
8	Habert, G., D'Espinose De Lacaillerie, J. B., & Roussel, N. (2011).	An environmental evaluation of geopolymer based concrete production: Reviewing current research trends	* Life Cycle Assessment methodology. * Suggested the use of industrial waste that is not recyclable within other industries and secondly on the production of geopolymer concrete using a mix of blast furnace slag and activated clays.	* Discussed two only two feedstock materials * Limited to determining only the environmental impact of geopolymer production
9	Mehta, A., & Siddique, R. (2016).	An overview of geopolymers derived from industrial by-products.	* Pointed out that most works had been carried out on fly ash based geopolymers whereas very few works have been reported on the potential of other SCM's * Stated that each feedstock material required different method of processing, different curing regimes as well as different mixture design	* Discussed mostly Alkaline fusion activation method. * Focused on only curing time and compressive strength.
10	M. I. Abdul Aleem, P. D. A. (2012)	Geopolymer Concrete: A Review.	reviewed the constituents of geopolymer concrete, its strength and potential applications.	* Focused on one activation method * Limited to one feedstock material
11	Djobo, J. N. Y., Elimbi, A., Tchakouté, H. K., & Kumar, S. (2016).	Mechanical activation of volcanic ash for geopolymer synthesis: effect on reaction kinetics, gel characteristics, physical and mechanical properties.	Mechanical Activation	* Focused on one activation method * Limited to one feedstock material
12	Bao, S., Qin, L., Zhang, Y., Luo, Y., & Huang, X. (2021)	A combined calcination method for activating mixed shale residue and red mud for preparation of geopolymer	Higher reactivity and compress strength were obtained for combined calcination.	Limited to one feedstock material
13	Antunes Boca Santa, R. A., Bernardin, A. M., Riella, H. G., & Kuhnen, N. C. (2013)	Geopolymer synthetized from bottom coal ash and calcined paper sludge	The compressive strength results were about 10–25 MPa	* Limited to paper sludge and bottom ash * Compressive strength were limited to one activation method
14	Allahverdi, A., & Mahinroosta, M. (2013).	Mechanical activation of chemically activated high phosphorous slag content cement.	* Feedstock were first chemically activated before the mechanically activated. * Mechanical activation greatly affects the compressive strength development	Limited to slags.
15	Balczár, I., Korim, T., Kovács, A., & Makó, É. (2016	Mechanochemical and thermal activation of kaolin for manufacturing geopolymer mortars – Comparative study.	* Mechanochemical activation of kaolinite can be a new and valuable method to manufacture geopolymer binders * Optimisation of the grinding process is a key issue to produce geopolymer mortars with adequate.	* Limited to one feed stock material. * Focused one activation method

(*continued on next page*)

Table 1 (*continued*)

S/N	Author	Paper title	Focus	Scope
16	Sun, Y., Zhang, P., Hu, J., Liu, B., Yang, J., Liang, S., Xiao, K., & Hou, H. (2021)	A review on microwave irradiation to the properties of geopolymers: Mechanisms and challenges	* Promising method of activation.	* Focused on one activation method * Energy consumption cannot be accurately measured. * Poor compressive strength

OPC, geopolymers deployment supports environmentally friendly construction practices in which it

enables more than 25 % cost reduction and 79 % decrease in CO_2 emission [29]. Research on geopolymers based on metakaolin, fly ash, and various minerals has delivered product which has its compressive strength increased to about 65 MPa [30,31]. Research in this field may have dated back to the 1950s when Victor Glukhovsky discovered alkali-activated materials by mixing materials from volcanic ash and rock with alkali-activating solutions. However, in the late 1970s, Davidovits created an alkali-activated material using naturally occurring materials rich in silicon and aluminum which include kaolin and which is bounded in a solution of alkaline liquid [32].

Alkali activated materials (AAMs) and geopolymers are binding systems created when an alkali source reacts with aluminosilicates rich feedstocks [25,27,33–36]. Alkali sources which are mostly utilized include sodium-hydroxides or potassium-hydroxides [37,38]. Akali-activation impacts the product's final characteristics and compressive strength based on PH and available alkali ions [39]. Life Cycle Assessment (LCA) is the best technique to analyze environmental impact of geopolymer cements [40,41], whilst the worst part of geopolymer carbon footprint is the alkali activators [16,42–50]. Pozzolanic materials such as ground granulated blast-furnace slag (GGBS), metakaolin (MK), pulverized fly ash (PFA), and their combinations are the basic feedstock geopolymeric binders [51]. Their properties are found significantly influenced by the Al–Si source and activator [20,52–54]. These aluminosilicate-rich materials are extensively used in evolving research in the field [55,56]. However, their limited availability limits their wider acceptability and deployment – demonstrated by the report [56] on challenges of fly ash global supply chain. About 40 % of coal-fired power companies in the United States have shut down in the past five years, while all power plants will be retired in the United Kingdom and the Netherlands by 2026–2030 [57]. Nonetheless, the global demand for GGBS as a supplementary cementitious material for OPC and concrete producers is substantial [56]. Therefore, it is crucial for researchers to find effective novel geopolymeric binders.

Critical 16 review papers in the field of geopolymer, feedstock and activation method are identified. These are presented in Table 1. The Table outlines paper title, focus and scope. Analysis of the focus and scope reveals that feedstock activation methods in geopolymer synthetization has not been extensively covered in literature. Findings show that activation method is specific on geopolymer feedstock because effective material activation significantly depends on the activation method and the binder used. This is due to the material chemistry, chemical structure, and microstructure as well as the binder properties [58–60]. As geopolymers which are activated optimally have demonstrated better shear strength and durability in comparison with conventional binder and concrete whilst leaving trivial environmental imprint [61–68], research into critical methods of geopolymer feedstock activation has become attractive. Literature to date focuses on either feedstock or activation mechanism. No publication to date comprehensively compared and contrasted feedstock activation mechanisms for geopolymer cements. Current research is carried out to augment this knowledge gap. This study reviews methods of activation of geopolymer feedstocks to advise on their suitability for industrial applications. The review discusses the physical and mechanical properties of each feedstock and matches them with the appropriate activation methods. It outlines activation process energy requirement for each feedstock and curing temperature and duration to report on the improvement achieved in the compressive strength of feedstock product. This research demonstrates commonality of feedstocks among the activation methods to advise on technology penetration in the industry whilst presenting the limitations of the methods.

2. Method and structure of review process

For replicability and reproducibility, a systematic review method is implemented. This involves deployment of organized search strategies. Six academic paper search engines comprising ScienceDirect, Google scholar, Research gate, Springer, Google and Scopus are used for paper exploration. Key papers searched include journal article, conference paper, book, PhD thesis and dissertation. Keywords which informed the search include geopolymers, feedstocks activation, cement alternative, Ordinary Portland Cement (OPC), industrial residue, environmental issues from OPC, industry decarbonization methods, environmental impact of OPC. Selection criteria are: (i) Paper publication date must be from 1979, (ii) Paper has at least one keyword of current manuscript in abstract, (iii) Paper focus has strong alignment with current review paper focus. Search results produced 280+ journal articles, 11 conference papers, 7 textbooks, 2 PhD theses and dissertation. Analysis techniques employed include Venn diagram, Bar chart, and Quantitative analysis. Fig. 1 presented the schematic of the literature review process. The distribution of paper types over the publication range is presented in Fig. 2. The figure shows that 84.26 % of the papers are within 14 years of publication.

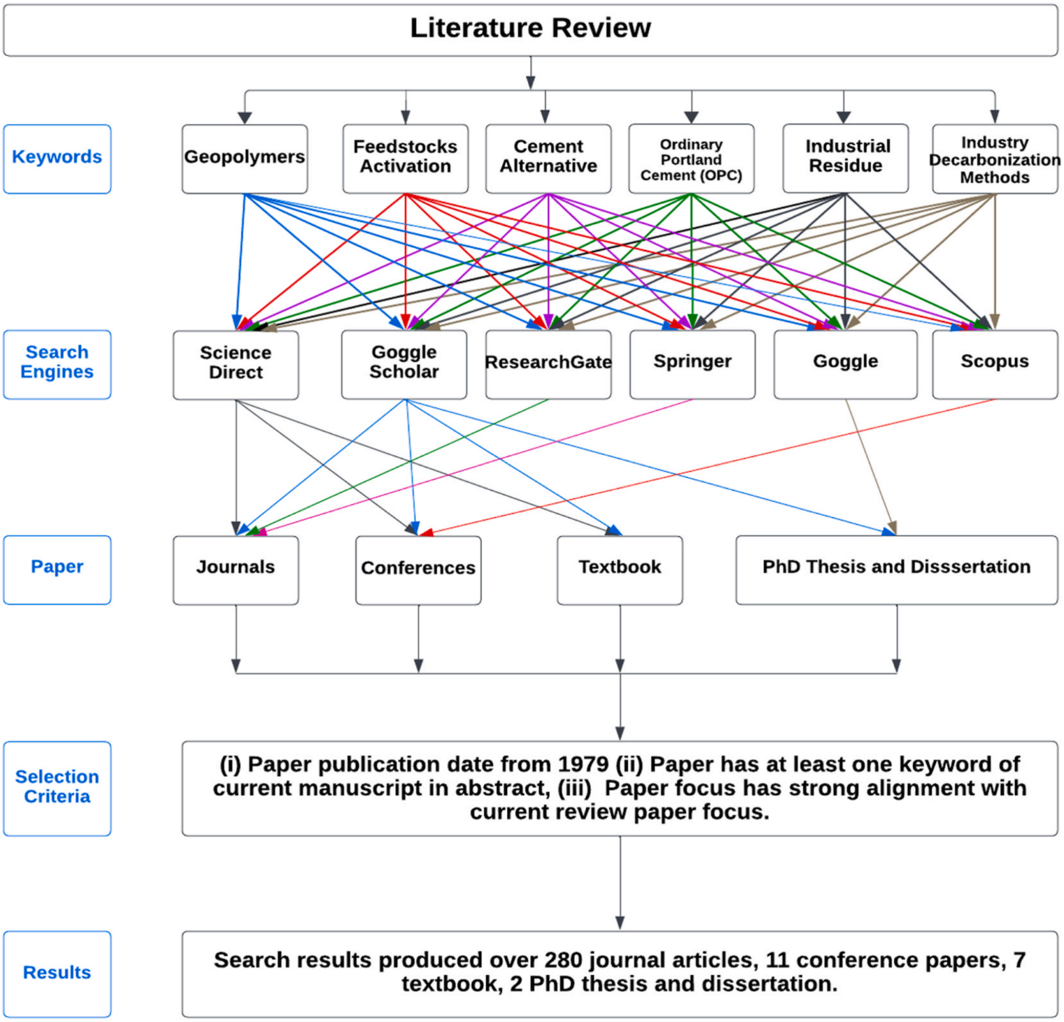


Fig. 1. Schematic of the literature review process.

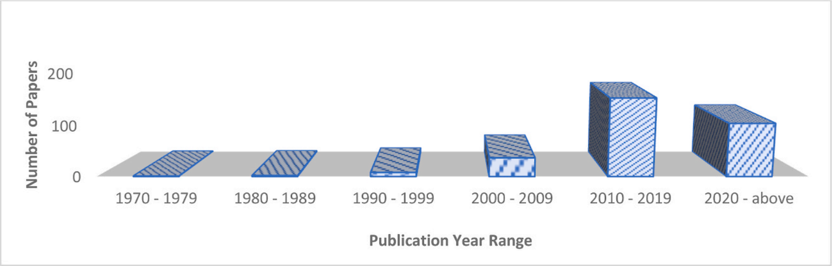


Fig. 2. Number of papers against publication year range.

3. Geopolymers feedstock materials

Several geopolymer feedstock materials exist but four main materials are discussed in this research. These are Kaolin, Fly ash, Slag, and Mine tailings. The materials comprise silica and alumina phases [69,70] and have been demonstrated as good materials for making geopolymers [71,72]. A detailed discussion on them is presented thus.

3.1. Kaolin

Kaolin, shown in Fig. 3(a)–is a whitish material [73]. It is a member of group of kaolinitic clays which comprises an even ratio of

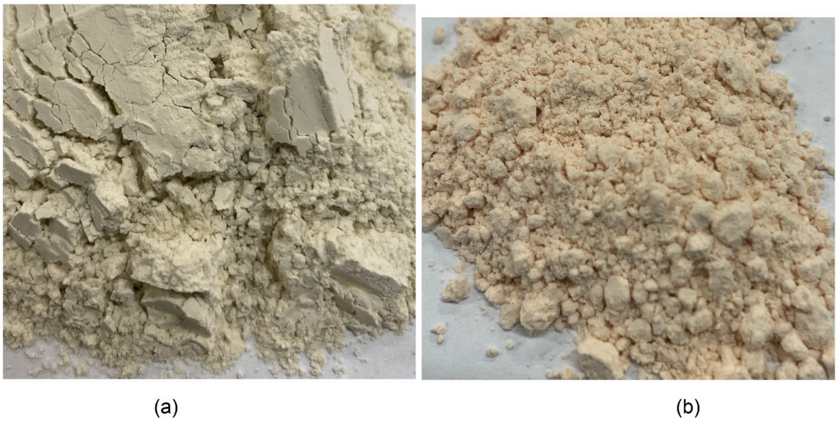

(a) (b)

Fig. 3. (a) Kaolin (b) metakaolin.

clay minerals and have both octahedral and tetrahedral crystal structure [74–76]. Kaolinite, a typical clay mineral and the predominant mineral in kaolin deposits, has low permeability [77–79]. It is relatively pure and useable for commercial purposes [80]. Owing to its unique physical and chemical characteristics, kaolinite can be used in a wide range of applications [80]. Metakaolin (MK), shown in Fig. 3(b)–is an anhydrous alumino silicate formed by the calcination of kaolin. When activated in alkali solution, it demonstrates high reactivity owing to its amorphous state [81–87]. The use of kaolin and MK (calcined kaolin) to produce geopolymer has piqued the interest of researchers, globally [75]· In 2012, concrete mix was created through activation of calcined kaolin with an alkali (a mixture of 6–10 mol of sodium hydroxide, NaOH and sodium silicate (Na_2SiO_3). The mixture was baked in an oven at 80 °C to generate a solidified product which was then pulverized to a fixed particle size powder. Increase in compressive strength resulting from stronger bond was observed in the geopolymer properties [88]. Kaolin geopolymers has demonstrated good volume stability in water. It does not crack or disintegrate - when solids-to-liquid ratio of the mix is in right proportion in concentrations ranging from 0.16 to.

0.36. Key properties of geopolymers are resistant to chemical attack and good stability in adverse weather condition - demonstrated in their high durability [89].

The researchers [90] demonstrated how compressive strength of geopolymers can be regulated through mechanical activation [28]. They mechanically activated kaolin by dry ball milling at 250 rpm. The activated kaolin shows possession of crystalline hydrated structure. Synthesizing alkaline aluminosilicate gel by mixing alkaline solutions ($Na_2SiO_3 + NaOH$), they blended the gel with mechanically activated kaolin to produce a higher strength geopolymer. They reported that the strength regulation is occasioned by the presences of crystalline hydrated phase induced by the process. Presence of carbonated species in the mixture accounts for deterioration of mechanical properties of geopolymers.

The authors [91–93] prepared and characterized kaolin. They obtained geopolymer which is more stable and better mechanical properties. Geopolymer composites are produced when NaOH and Na_2SiO_3 are used as activators, water-cooled slag is used as start material and kaolin is fired at.

800 °C for 2 h, heated at 5 °C per min and resulting substance cured for ninety days. Geopolymers produced in this process show improvement in the mechanical properties [94]. Generally, geopolymers produced from kaolin and alkaline activated binder have demonstrated improved mechanical properties [95–98] but higher cost of basic ingredients impacts on its cost [99].

3.2. Fly ash

Since 1950s fly ash has been available globally and has been utilized in the construction of highways, worldwide [60,100,101]. Fly ash, shown in Fig. 4, is a waste material, a fine glass powder and a residue extracted from fumes produced during combustion of coal in electricity generation [100,102–105]. The substance consists of earth elements which are mostly silica, alumina, and iron. In the manufacture of Portland cement concrete, fly ash is utilized as a supplementry cementitious material (SCM) [106] because they are more eco-friendly than OPC [107]. Moreover, research has demonstrated that the water-reducing ability of fly ash can increase the fluidity of freshly laid concrete [108]. Two main categories of fly ash exist. These are low-calcium fly ash (created when anthracite or bituminous coal is burned), and high-calcium fly ash (formed when lignite or sub-bituminous coal is burned) [109–111]. However, as a source material, low-calcium fly ash is deemed preferrable over high-calcium fly ash. This is because calcium, in excessive concentrations, may influence the polymerization reaction and therefore affect the microstructure of the resulting composition [112–115]. The concrete industry and the Engineering Plan Approval advised that the use of fly ash as OPC has the potential to reduce CO_2 emission considerably [116]. One most significant advantage of utilizing fly ash for geopolymer synthesis is its abundance in comparison with alternate source materials [117,118]. In 2004 and 2005, a high-alkaline solution was used to chemically activate low-calcium (ASTM C 618 Class F) fly ash to form a paste which bind loose coarse and fine aggregates, as well as other unreacted materials in the mixture to form geopolymer concrete. The produced geopolymer demonstrated better properties which include greater compressive strength, more resistant to sulphate attack, low creep properties, and exhibited minimal drying shrinkage - making it ideal for construction purposes [119,120]. Low-calcium fly ash from a coal-burning power plant was utilized by Ref. [121] to create

Fig. 4. Fly ash (FA).

the binder for manufacturing concrete. Geopolymer produced this way showes an outstanding compressive strength, trivial drying shrinkage, improved creep properties, greater resistance to sulphate and acid attack.

Researchers [122] mixed fly ash with NaOH and Na_2SiO_3 as activators to produce geopolymers. They reported on the relationship among concrete strength, fly ash content and water absorption rate. When compared to normal concrete, geopolymer concretes demonstrate superior resistance to 3 % H_2SO_4 acid attack. In the studies [123,124], the effect of aggregate content, alkaline solution, fly ash ratio, Na_2SiO_3 to NaOH ratio, and curing method on mixtures are investigated. The study reported on the competitiveness of the mechanical properties of geopolymers in relation to the OPC concrete. The advantages of geopolymers over OPC concrete, especially in seawater environment, are outlined. Low calcium fly ash-based geopolymer concrete was cured at room temperature of 23 °C with no extra heat. A tiny quantity of additives was mixed with fly ash to speed up the early-stage reaction. The results demonstrate that appropriate geopolymer mixes for curing period could be created utilizing low calcium fly ash and additives as a cement substitution [125]. The researchers [126] investigated the effect of heat-curing on the transport properties of geopolymer concrete based on low-calcium fly ash. They reported on both electrical resistivity and compressive strength of the geopolymer. To improve fly ash bonding and thus mechanical properties of geopolymer made from it, a fifty-percentage mix content is recommended [127]. Similarly, to prevent premature deterioration of low calcium fly ash based geopolymer concrete creek bed in a high salinity, appropriate consideration of binder chemical reactions and prelim durability property testing are considered necessary as reported by Ref. [128]. In 2018, researchers [129] explored the mechanical properties and short-term durability characteristics of geopolymer concretes based on fly ash and slag. They reported that 100 % fly ash shows greater durability resistance when compared to OPC specimens. Many researchers including [130–134] proposed that fly ash activated by alkaline activator based on geopolymer technology has the potential to significantly reduce carbon footprint of OPC concrete. The technology is poised to have huge potential for applications in the concrete industry as an alternative binder to Portland cement.

3.3. Slag (GGBS)

Ground Granulated Blast-furnace Slag (GGBS) is a byproduct of iron production process from the blast furnaces [135]. It is shown in Fig. 5 as a brown substance but can also be whitish in colour. Naturally, it is a cementitious material with good workability used in concrete production. Concrete and cementitious production were the first industrial and commercial applications that began

Fig. 5. Slags.

around 1859 [136,137]. For many years, GGBS has been used as a geopolymeric component of concrete and in composite cements production. GGBS other properties include good pumpability, high compaction qualities, reduced permeability, increased resistant to chloride penetration, good protection from sulphate strike, high resistance against alkali silica reaction (ASR), a really low heat of hydration and an improved chemical stability [138–148]. Demonstration of these great properties has increased the attractiveness of research into the material. The researchers [149] studied the characteristics of concrete made using heated GGBS slag (GGBFS). Their results appear to indicate that applications involving high temperatures would be suitable for GGBFS- containing concrete. In an effort to lessen environmental issues associated with the production of cement and waste management [150], examined the potential for using limestone powder and blast furnace slag (BFS) as cement substitutes in concrete. Their findings demonstrate that LP and GGBS replacement levels of 5–10 % yield the highest compressive strength. The investigators [136,151,152], investigated the use of GGBS as a pozzolanic partial replacement for cement in concrete. These findings presented indicate that GGBS could be used successfully as feedstock geopolymer material. Many researchers [153,154] have reported on the relationship between utilization of GGBS and conservation of conventional construction materials that are rapidly depleting. The mechanical performance of concrete, including compressive strength, split tensile strength, and flexure, is enhanced with GGBS due to the pozzolanic reaction and micro fill of voids, however, an increased dosage decreases the physio-mechanical properties of the concrete [135,155]. As an alternative construction material, GGBS can minimize environmental pollution by reducing CO_2 emissions during production as well as the rate of consumption of natural resources.

3.4. *Mine tailings (MT)*

Mine tailing (MT), shown in Fig. 6, is a feedstock material obtained from mine processing processes. MT is a type of tailing. The term "tailings" refers to a byproduct among many mineral processing plants. Tailings are combinations of squashed rock and processing fluids from mills, washeries, or extractors that remains after economic metals, minerals, mineral fuels, or coal have been extracted from a mine resource [156]. These facilities create two distinct product categories

that can be classified as either economic or non-economic. Generally referred to as tailings, the non-economic product is made up of waste (by-product), trace amounts of valuable minerals or metals, chemicals, organic materials, and process water [157,158]. Mine tailings are the residual waste products generated during extraction of mineral ore's economic fraction [159–161]. Since the early ages, billion tons of heavy metals (copper, lead, cobalt, zinc, cadmium, and chromium) have been extracted, and the production of mine tailings today is estimated to be between 5.5 and 8 billion tons annually globally [162]. Presence of this amount of tailing makes research on its management economically attractive [163,164] and the realization of the potential of utilizing it in the production of geopolymeric materials has accelerated the research in the area [165–169]. Researchers [170] investigated the durability and leaching behavior of MT-based geopolymer brick specimens. The experimental findings suggest that a key element influencing the leaching behavior is its solubility/reaction rate. Other forms of tailing can also be utilized [171–173]. Authors.

[174] investigated the visibility of utilizing iron ore tailing (IOT) as a substitute to fine aggregate in the production of ultra-high-performance concrete. The presence of many inert phases in the tailings and the fact that their average particle size was substantially bigger than that of cement suggest that they could be used as fine aggregates. Sulphidic tailings from gold mine and phosphate mine tailings were used for the production of geopolymers by Refs. [175,176], the findings showed that GGBFS and sulphidic mine tailings from gold mining locations can be utilized as a raw material in alkali-activation [177–179]. Phosphate tailings could also be recycled to create sustainable geopolymers with good mechanical qualities. The researcher [180] used copper mine tailings in concrete production as a partial replacement for cement. The findings indicated that the compressive strength of concrete specimens produced with varying ratios of cement replacing mine tailings decreased with increasing replacement percentage, and the loss of compressive strength was higher at young ages, which was caused by delayed hydration process. The compressive strength of geopolymers made from mine tailings alone is typically lower. However, the addition of other ingredients appeared to have a more pronounced impact on the strength development [181].

Fig. 6. Mine tailings.

4. Feedstock activation methods

Feedstock activation methods (AMs) are presented in six sub-headings. Sub-section 3.1 is the introduction. The critical methods of activation are discussed in sub-sections 3.2 to 3.6.

4.1. Introduction

There are two distinct stages of activation in the formation of geopolymer. These are binder and feedstock activations. Binder activation is widely described in literature. It is the mixing of feedstock material with alkali activator solution prior to curing to form geopolymer. Feedstock activation is a pre-process to the binder activation. The process increases the reactivity of bulk feedstock material prior to binding. Thus, feedstock activation is a critical pre-processing step which ensure high strengths and durability of geopolymer. As limited extensive and systematic structured reviews exist on feedstock activation methods, this review focuses on the method to provide an evaluation of the methods for suitable industrial applications. It also aims to provide information on the relationship between the methods and the properties of the geopolymers formed - especially their compressive strengths.

A schematic representation of the development process of geopolymer cement is shown in Fig. 7. The process in simplified into four stages which are input material, activation, processing and output. The stage 1 is the input – which involves the collection of the feedstock material. The feedstock is passed into the stage 2 – which is the activation stage. Five critical activation methods are identified. These are the mechanical, thermal/calcination, Hybrid, micro-wave assisted treatment (MAT) and ultrasonic. The methods are discussed in detail in sub-section 3.2 to 3.6. After activation, the activated feedstock is processed further in stage 3 – which is the processing stage. This stage involves mixing of feedstock aggregates with alkali solution and allowing the matrix to cure under suitable conditions. At the stage 4 - which is the output stage - the geopolymer cement are produced after the matrix have cured.

About 64 published peer reviewed papers are examined and used for this investigation. Venn diagram representation of the composition of the papers are presented in Fig. 8(a). The figure shows that 48 of the 64 papers are on AMs while the other 16 papers are on hybrid methods, (HM). The papers on AMs, shown in Fig. 8 (ai), comprises 24 papers on mechanical activation, 19 papers on thermal/calcination, 4 papers on ultrasonic activation, and 1 paper on microwave-assisted acid activation (MAT). Similarly, the papers on HM, shown in Fig. 8(aii) consist of 8 papers on mechanical and thermal activation, 6 papers on mechanical and chemical activation, 1

paper on chemical and thermal/calcination, and 1 paper on mechanical, thermal/calcination and chemical activation.

Further details on the papers in terms of the AMs – including HM - and the corresponding feedstock materials used are presented in Fig. 8(b). Fig. 8(bi) shows the feedstock materials commonly used in each of the AM. This include feedstock that can be used in more than 1 a.m. Kaolin, Tailings, and Fly Ash demonstrate increased usage in more than 1 a.m. This is an added advantage in their processability. Similarly, Fig. 8(bii) presents the feedstocks used in HM. Kaolin is found to be used by the HM consisting of mechanical, chemical and thermal/calcination. This demonstrated its popularity potentially due to its high processability within the HM.

A detailed systematic structured review of the papers to characterize the AMs and HMs in terms of feedstock used, process energy requirement, product compressive strength, product curing temperature, and product curing time are presented in Table 2. Critical analyses of the information in the table is presented in Figs. 7–9. Fig. 7 depicts a plot of the compressive strength of the various geopolymers against the activation method and feedstock. It is observed that geopolymer made by mechano-chemical activation method of slag achieved the highest compressive strength of 55.6 MPa. Furthermore, geopolymer made by mech-thermal activation of

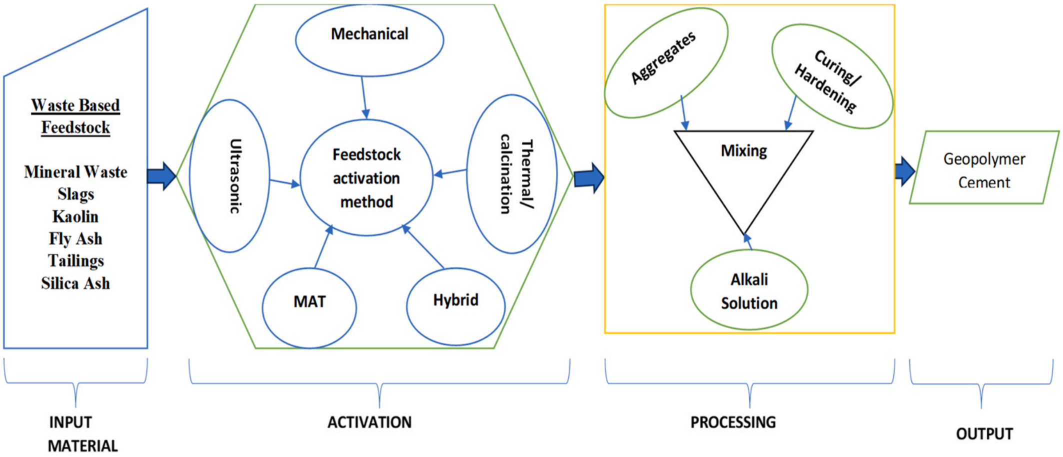
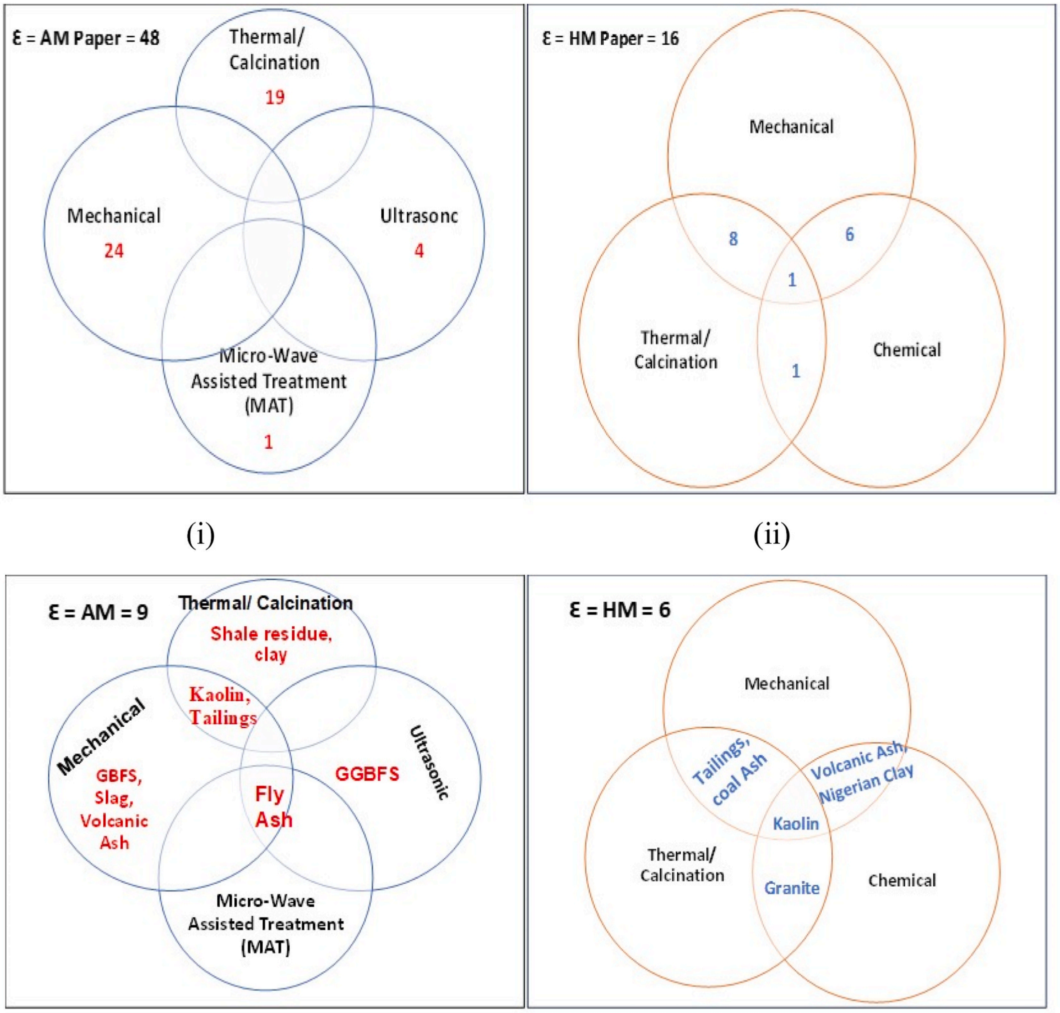


Fig. 7. The development process of geopolymer cement.



(i) (ii)

(i) (ii)

Fig. 8. (a)Venn diagram of papers reviewed on geopolymer feedstock activation methods. (i) Activation method (AM); (ii) Hybrid Method (HM) (b): Venn diagram of feedstock in various activation methods:(i) Activation method (AM); (ii) Hybrid Method (HM).

Table 2

Detailed analysis of feedstock activation methods.

S/ No	Activation Method	Feedstock	Process Average Energy	Improved Compressive Strength (MPa)	Curing Temperature (^{0}C)	Curing Time (Days)	Ref.
1	Mechanical	GBFS	200–500 rpm for 30–120 min	19 to 45		1 to 28	[182]
		Fly Ash (FA)		16 to 45	40 to 100	2 to 28	[183]
		Slag		23 to 25	20 to 80	28	[184,185]
		Kaolin		20.25 to 26.4	70 to 100	3	[186]
		Tailings		19 to 25	20 to 80	28	[187]
		Volcanic Ash		20.5 to 37.9	27 to 80	28 to 90	[188]
2	Thermal/Calcination	Kaolin	650 °C to 750 °C	20.08 to 28.55	20 to 60	28	[189–194]
		Shale Residue		29.53 to 32.54	20 to 60	28	[195]
3	Micro-Wave Assisted Treatment (MAT)	Clay	800W and 2.45 GHz	–	–	1–2min	[196]
4	Ultrasonic	FA, GGBFS	SiO2/K2O molar ratio of 1.25–1.86	little to no effect	5 to 30	2	[197–200]
5	Mech-Thermal	Kaolin	600–800 °C	10 to 55	105	1 to 28	[201–205]
		Tailings		10 to 25	100	2	[206]
6	Mechano-Chemical	Slag	600–800 °C	20 to 55.6	20 to 60	28	[207]
		Kaolin		20 to 35	20 to 60	14	[208]
7	Thermochemical/Alkali fusion	FA, Bentonite, Tailings	550 °C for 60–120 min at a rate of 1–5 °C/min	28.2 to 40.6	20 to 80	2 to 28	[209–225]
		Volcanic Ash		13.89 to 19.6	27	2 to 28	[226–228]

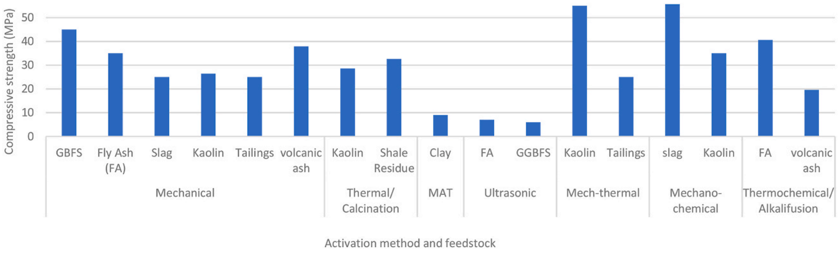


Fig. 9. Plot of compressive strength against activation method and feedstock.

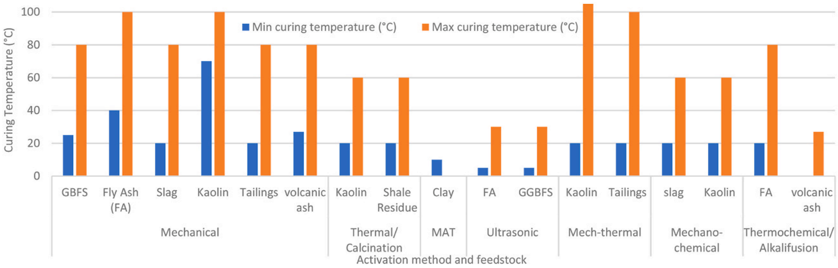


Fig. 10. Plot of curing temperature against activation method and feedstock.

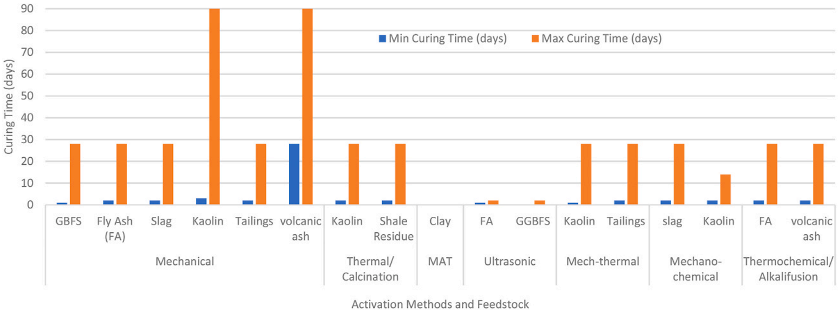


Fig. 11. Plot of curing time against activation method and feedstock.

Kaolin is in the second at 55 MPa while geopolymer made by mechanical activation of GBFS feedstock ranked third at 45 MPa. Similarly, the plot of curing temperature against activation method and feedstock presented in Fig. 8 reveals that mech-thermal activation of Kaolin requires the highest curing temperature among the activation methods. The temperature is circa 105 °C. Polymers produced by activation of fly ash and Kaolin as well as those produced by mech-thermal activation of Tailings have equal curing temperature requirements of about 100 °C. These have demonstrated to be high energy processes. The figure shows that geopolymers made by ultrasonic activation of fly ash and GGBFS demonstrate lowest curing temperature requirement and thus are the best process in this regard. Moreover, a plot of curing time against activation method and feedstock is presented in Fig. 9. It is observed that geopolymer made from mechanical activation of Kaolin and Volcanic ash requires the highest curing time and thus are not ideal in this regard. Geopolymer production by MAT activation of Clay is found the most economical in terms of curing time. Geopolymer production by ultrasonic activation of fly ash is identified the second-best process. A more specific discussion of each activation method is presented in the following sub-sections.

4.2. Curing thermal energy

The curing thermal energy (E), measured in degrees centigrade second (°C hr) is defined as the quantity of heat over time required to cure the produced geopolymer. It is mathematically represented as:

$$E = Qt = c\Delta Tt$$

$$\frac{E}{C} = \Delta Tt \qquad (1)$$

Where: Q is the heat required (joules), ΔT is the change in temperature over the curing process, t is the time duration of the curing process, c is the specific heat capacity of the geopolymer, and $\frac{E}{C}$ is the energy per unite specific heat capacity of the geopolymer. Implementing Eq. (1) and rationalizing the values of $\frac{E}{C}$, Fig. 10 presents a plot of the rationalize values against the activation method and feedstock. This is a plot of the consumed over time in curing the respective geopolymers. The plot shows that polymers produced by MAT activation of clay and ultrasonic activation of fly ash and GGBFS are the best economic process with minimal energy requirement. (see Fig. 11).

4.3. Mechanical activation

It is one of the most effective activation methods for industrial solid waste because grinding has demonstrated increase in reactivity of geopolymeric materials [60,181] (see Fig. 12). This activation method is performed by using a mill grinder consisting of mainly a ball mill, vibratory and stir media. A vibratory disc mill and a ball grinning machine used in mechanical activation are shown in Fig. 13.

(a) and Fig. 13 (b), respectively. The researchers [229–231] has reported on the effectiveness of ball mill grinding. Other researchers including [232–238] also reported on potential increase in reactivity when the technique is employed. Other investigators [239] reported on increase compaction of geopolymeric when the technique is used. Mechanical activation technique is the key step to creating geopolymers [240] due to its significant impact on the specimen strength [241]. With reference to Table 2 [234,242–245], show that the geopolymers can achieve an increased compressive strength in the range of 60 %–80 %. Authors [246] demonstrated achievement of better compressive strength of 30 %–80 % when the curing time is increased from 70 °C for three days to 100 °C for one day. Most recently [247], stated that more than one activation technique for recycled waste could produce better results. Combining

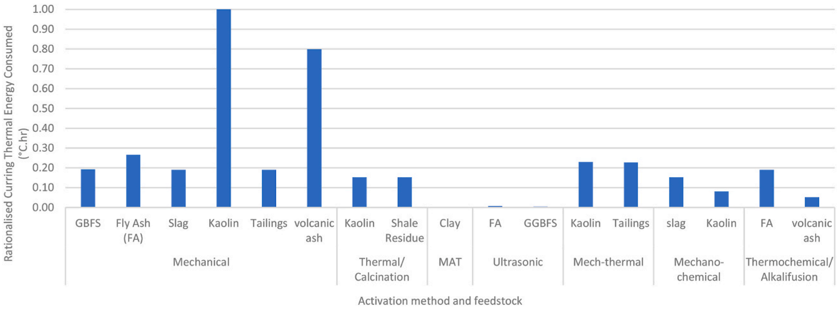


Fig. 12. Plot of curing thermal energy consumed against activation method and feedstock.

(a) (b)

Fig. 13. (a) SIEBTECHNIK TEMA T1000 vibratory disc mill (b) An 8000 M Mixer/MILL ball grinning machine.

mechanical, thermal and chemical activation is advised more so as [248] demonstrated quicker kinetic reaction of the geopolymer formation when performed with kaolin.

4.4. Calcination activation

This process involves the heating of waste material in a furnace/oven to an elevated temperature within a specific time range to remove moisture, volatile substance, oxidize a portion of the material, or render the solids friable/smaller before chemically activating the material. The furnace/oven could be electric or gas or other fuel fired. Fig. 14 shows a Carbolite GERO ELF 11/23 electric fired furnace. The temperature used during burning or calcining has an impact on the final product's pozzolanic reactivity. The calcining temperatures causes the release of hydroxyls that increases the reactivity of the substance [249]. Table 2 shows the materials used for calcination activation technique are kaolin, coal ash, paper sludge and tailing (red mud). The geopolymer made from the feedstock demonstrated increase in compressive strength. This result is achieved owing to production of geopolymeric gel and denser matrices, when the calcination temperature is between 700^{o}C to 950^{o}C for one to 2 h and a curing temperature of 80 °C with increased time. The table also showed that coal ash and paper sludge were mixed in various proportions and the samples achieved good results [250]. It is observed that increase in the ratio of SiO_2/Na_2O increases the compressive strength of the geopolymer while increase in NaOH reduces the setting time [251]. However, the effect of the latter can be controlled with increase in curing time because [245,250] utilized 10–15 ml of NaOH and 7 days curing time to achieve 10–27 MPa compressive strength polymer. This result demonstrates a 5.8–7.8 % increase from the 7 days curing compressive strength when cured for 28 days. Calcination activation technique has demonstrated potential as a good activation method which increases the reactivity of feedstocks [252],but the process produces 6–8 % CO_2 in industrial scale [253] which makes it a contributor to global warming. This finding necessitated the need to develop a hybrid method [254].

4.5. Micro-wave assisted treatment (MAT) activation

Micro-wave assisted treatment has been used for several applications - either to reduce treatment time duration, increase efficiency, improve solidification or stabilization [255–258]. MAT has been shown to be an effective technique [259,260] as it has an increased efficiency rate, a reduced energy consumption and trivial release of secondary pollutant during treatment [261]. In this technique, the grain boundary area is expanded and reconfigured, which promotes both plastic and viscos flow of particles. It supports mass transfer, particles compaction and forms the boundary which lowers substance pore holes and volume to generate polycrystalline sintered body [262]. It also significantly improves the geopolymerization process with a reduced polymerization time of 15 min as opposed to 28 days and an optimal compressive strength of 18.8 MPa [258].

Microwave-assisted geopolymer preparation is a potential approach for preparing building material and immobilizing heavy metals. The incorporation of microwave technology into the geopolymer manufacturing process has significantly increased mechanical properties, optimized the microstructure, and enhanced heavy metal embedment [260]. Although the use of microwave technology in geopolymers has numerous benefits, it is still in its development phases and faces a number of obstacles before being used in manufacturing environments. Notably, only 40 %–70 % of the energy from electricity is converted into microwave energy [263,264]. Components of geopolymers have varied dielectric characteristics. Frequent interaction of microwave with molecules may result in a local temperature that is substantially greater than the bulk structure.

Fig. 14. Carbolite GERO ELF 11/23 electric fired furnace Furnace

[265]. The phenomenon causes local overheating and erroneous temperature readings, which challenges analysis on how microwaves affect geopolymers [260].

4.6. Ultrasonic activation

Ultrasonic activation technique uses a frequency of over 20 kHz to 1 GHz to cause a rapid change in pressure through cavitation. The cavitation process forms radicals that support the formation of composite [266]. This condition aids geopolymerization [267]. In 2022 [268], stated that ultrasonic activation technique may not be required to produces geopolymers because the specimen produced were not far different from the conventional ones. However, referring to Table 2, this statement may be limited to fly/bottom ash materials.

4.7. Hybrid method

There have been numerous challenges with the activation process of geopolymers [34,269–274]. Geopolymer production, as have been discussed, can be challenging because the process needs special materials handlings at high temperature [275]. To create the best activation method, a hybrid approach is proposed by Ref. [247]. Hybrid method combines two or more activation techniques. These include mechanical and thermal, alkali fusion, mechanical and chemical (mechanochemical as often referred to as [276–278]), or a combination of mechanical, calcination, and alkali fusion. The hybrid method has been performed by many researchers that include [131,279–284]. Further discussion will focus on the alkali fusion method.

5. Emerging feedstock activation techniques base on hybrid method

Four key emerging technologies of feedstock activation based on hybrid methods are presented. The hybrid method basically comprising a combination of two of more activation methods. These are discussed in the sub-headings 4.1 to 4.4.

5.1. Mechanical and thermal activation

Mechanical plus thermal activation is a common method of activating feedstock materials. The method affects the mineral composition, chemical bond or functional group, microstructure, and thermal characteristics of the feedstock material [285]. This method is done by grinding and vibration the feedstock material to reduce it particle size followed by the calcination of the reduced particle size. The process makes it possible to improve the physio-mechanical properties of the cured geopolymer by up to 40 % [286] which substantially improve and impact the cementitious $_{\text{activity}}$ [247,250,287–290].

5.2. Mechano-chemical activation

Mechanochemical activation is achieved by grinding the feedstock material in the presence of an additional chemical additive. Alkali-mechanical activation is the most reported in the literature. The process involves grinding feedstock with an alkali in a grinder prior to geopolymerisation. The performance of SCMs is greatly enhanced in excess of 44 MPa [281] by the use of mechano-chemical activation. In some instances, admixtures are used to alter grinding of feedstock materials and cause changes in the structure of cementous minerals [291]. This practice is demonstrated by Refs. [248,281,292]. Common chemical additives include phosphoric acid and aluminum silicates.

5.3. Thermo-chemical activation

Thermochemical activation is the thermal activation of a material in the presence of a chemical additive. This is commonly demonstrated in the literature and the process utilizes NaOH as the

chemical additive. NaOH usage causes dehydration and/or a decarbonation processes that forms Na-rich crystalline phases [293]. The resulting new phases are a form of alkali fusion. Alkali fusion, a result of thermochemical activation, is used to digest materials containing silicates and replace sulfonic acid group with a hydroxyl group in a substitute aromatic ring [294,295]. The low reactive raw material is an alkali which is thermally activated by mixing the material with an alkali source such as sodium hydroxide - followed by calcination at a greater temperature than the melting point of the alkali source [296,297]. The modification of the crystalline phase is referred to as an alkali fusion. This method simply changes the aluminosilicate source mineral structure by producing more amorphous phases or some Na-rich crystalline phases [293]. Due to its microstructural and mineralogical modifications, alkali fusion has a significant impact on the reactivity of geopolymers [181].

Alkali fusion uses feedstocks which include fly ash [298,299], volcanic ash [300,301], volcanic scoria [294], granite [302], kaolin [303], and phosphate sludge [304]. Table 2 shown a couple of these. The authors [300] produced geopolymer which is synthesized from alkali fusion method by adding metakaolin to balance the Na/Al ratio. The resulting product exhibited low setting time of 6.5–15 min, low shrinkage and increased compressive strength. Researcher [302] reported on the potential of granite waste as geopolymer. They employed alkali fusion method, increasing the Na_2O content and achieved good compressive strengths ranging from 6 MPa to 40 MPa. Optimal condition for alkali fusion for mine tailing is found to be 10 wt percent of NaOH at 550^{o}C [293]. Furthermore, the degree of geopolymerization reaction for fly ash is 71 % of Al_2O_3 at a curing temperature of 37^{o}C [298]. However, this technique is mostly

Table 3
Advances in geopolymer activation methods.

S/No	Activation Methods		
	Previous	Current	
	Previous method	Single method	Hybrid method
1	Mechanical activation (MA) e.g., as grinding and crushing	Mechanical activation e.g., planetary milling, ball milling.	MA + CA, MA + Alkali fussion, MA + Calcination, MA + TA
2	Chemical Activation (CA) e.g.,	Alkali Fussion	MA + Alkali fussion, MA + TA
3	Thermal Activation e.g., Heating,	Thermal Activation e.g., Calcination.	MA + TA
4	–	Microwave assisted Treatment	MA + MAT
5	–	Ultrasonic Activation method (UAM)	MA + UAM
6	–	–	Thermochemical

suited for recycling and utilization of low reactive aluminosilicate wastes. Although an efficient activation process as the addition of alkali more than 20 % into the mix increases the reactivity, it reduces the physio-mechanical properties of the produced geopolymer and a 10 % is proposed [293,302,305]. When compared to mechanical activation method using volcanic ash, samples from mechanical activation are found superior with compressive strength in the domain of 25, 39 and 33 MPa to alkali fusion in the range 15, 17 and 10 MPa. Results are found dependent on the inclusion of water structure caused by muscovite which altered the mineralogical composition of the geopolymer formation [294].

5.4. Mechanical and calcination and chemical activation

This is the type of activation technique that combined three methods. These are mechanical, thermal and chemical activations. This technique involves mechanically activating the feedstock first. An alkali source in then added in the mix which is calcinated at an elevated temperature greater than the melting point of the alkali source. The investigators [301] recorded a rise in strength between 15 MPa and 41 MPa at 28 days curing – although the geopolymer strength decreased after 60 days of curing. Authors [303] recorded a maximum compressive strength of 28.2 MPa with 8 mol of NaOH silicate geopolymer at 27 °C within 28 days of curing.

5.5. Current and future development in the field of geopolymer activation

The methods of geopolymer activation (previous and current) both revealed in the review papers and projected based on the findings from the review are presented in Table 3. In analyzing the current development, it is found that the most relevant activation methods are thermochemical, mechanical and thermal activations. Furthermore, recent advances are identified as alkaline fusion, planetary & ball milling, and mechanochemistry.

6. Conclusions

As environmental issues arising from pollution are becoming uncontrollable, development of methods to reduce them has become urgent. Identifying the development of alternatives for OPC is promising but acquisition of knowledge on adequate characterization of feedstock activation methods is crucial to increase the technology attractiveness for wide penetration. Several conclusions are drawn from the findings of this extensive review. It outlines the critical role of geopolymer materials in the development of alternative cement for improved environmental management.

This review finds that hybrid activation method which integrates mechanical activation is poised as the future geopolymer activation technology because it demonstrates greatest efficiency potential. This is a key finding of this review that is not reported in the literature. Kaolin, an environmental residue, is identified as the most widely used feedstock followed by an industrial residue, fly ash. Metakaolin and metal mine tailings are found to be good sources of geopolymer cement with great compressive strength. Penetration of kaolin as feedstocks is heightened by the

realization that it is used by the hybrid method comprising mechanical, chemical and thermal/calcination. This demonstrates its popularity owing to its high processability. Fly ash is observed as the second most widely used feedstock after kaolin. The feedstock has exhibited properties which position it as eco-friendly supplemental cementitious waste materials rich in silica, alumina and iron. Its deployment demonstrates the potential of reducing global warming by about 48 %.

Geopolymer made by mechano-chemical activation method of slag (GGBS) achieved the highest compressive strength of 55.6 MPa. This demonstrates slag's strength as a good geopolymeric component of concrete, composite cements, and attractive alternative construction material - especially when improved mechanical properties are required. Geopolymer made by mech-thermal activation of kaolin has an average compressive strength of 55 MPa and sits in the second position. In terms of curing temperature, geopolymers made by ultrasonic activation of fly ash and GGBFS used the lowest curing temperature. The process is proposed as the best in terms of curing temperature demand. Furthermore, geopolymer production by MAT activation of clay is found the most reasonable in terms of curing time. This is followed by geopolymer produced by ultrasonic activation of fly ash. With the finding that geopolymers produced by MAT activation of clay and ultrasonic activation of fly ash and GGBFS require the least curing energy, the activation technique is proposed the best economic process in term of minimal energy requirement.

Feedstock activation techniques integrating mechanical activation method are found the most effective techniques because the inclusion of the method produces geopolymers with high compressive strength. The impact of the method draws from its ability to decrease waste particle size to increase its reactivity.

This review demonstrates the significant potential feedstock activation poses in expanding current geopolymer feedstock availability. However, further research is needed to refine and optimize these techniques.

CRediT authorship contribution statement

Oluyemi Kehinde: Writing – review & editing, Writing – original draft. **David J. Hughes:** Writing – review & editing, Supervision. **Emeka H. Amalu:** Writing – review & editing, Supervision.

Declaration of competing interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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