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# Durability in Materials Obtained by Alkaline Activation, A Review on the State of the Arte Focused on Municipal Solid Waste Incineration Slags-MSWIS

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**ABSTRACT**

The present work on durability in materials obtained by alkaline activation (AAM), in state-of-the-art review format, is structured as follows. (i) The concept of alkali activated materials-composition-performance; (ii) The prediction of the useful life of AAM-methodologies and planning; (iii) The durability and properties of Engineering-mechanical tests-standards of specifications and tests (iv) Degradation processes of Chemical Matrix-resistance to sulfates ($SO4^{2-}$), acids (HCl), ($H4^2 2SO_4$), ($HNO_3$), ($H_3PO_4$), (HF), ($H_2CO_3$ ) and sea waters; (v) Degradation processes by mass transport-permeability, porosity, corrosion, carbonation, efflorescence and ice-thawed.

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## Introduction

We define the structure of this work in order to correspond with the global vision regarding the great concern about climate change, the concrete associated with world growth, the production and consumption of ordinary Portland cement referred to, previously responsible for 5 to 8% of total anthropogenic $CO_2$ emissions of which 95% of $CO_2$ is developed during cement production and most recently reported with a probability of reaching 10 to 15% of these emissions by 2020, this production that has been contributing for decades to the degradation of the terrestrial platform through the exploitation of calcareous materials ($CaCO_3$) and clayey ($Al_2O_3$-$2SiO_3$-$H_2O$), the increase in the production of carbon gel ($CO_2$) with the corresponding contamination of the atmosphere, thus increasing the degree of concern of the scientific and environmental community in the study and research of new alternative materials to Portland cement, ordinary considered as supplementary cementitious materials, whose application in the field of engineering and civil construction has been studied with great approaches in the concepts of construction durability, knowledge of useful life planning methodologies, analysis of applicable standardization, approach and determination of estimated useful life in case studies as well as the implementation of measures to promote durability, and also the studies of technical, environmental, socio-economic and financial impact on the partial or total use of slag from the incineration of municipal solid waste as a complementary cementitious material [1-3]. These alternative technological improvements with new supplemental cementitious materials cane be reliably expected to reduce $CO_2$ emissions by a factor of two, which will be far from the quadruple reduction targets of the Intergovernmental Panel on Climate Change (IPCC), as these new materials are known to account for a lower level of carbon dioxide emissions than Portland cement [4,5]. Worldwide, Portland cement production in the 1990s reached 1,200 million tons per year (Figure 1), having exceeded 2,600 million tons per year at the end of the 2000s and, with projections pointing to production above 5,200 million tons per year in 2050 [6].

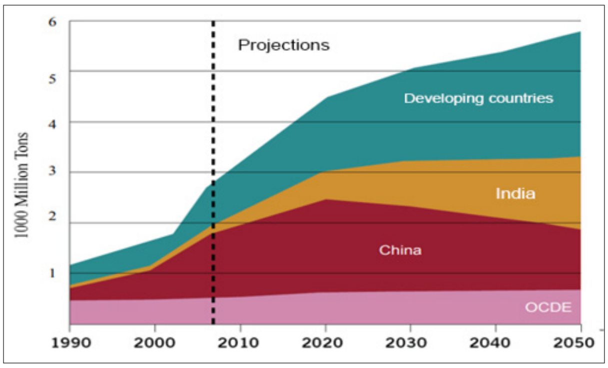


**Figure 1:** Forecast of Global Consumption of Portland Cement: Adapted from [6,7]

The need to achieve carbon neutrality by 2050 has increased interest in ligands obtained by alkalin activation as a more environmentally friendly alternative in traditional Portland cement-based processes in the construction sector.

## Durability of Construction

As for durability, according to Van Deventer, the question of whether geopolymer concrete or AAMs are durable remains another important obstacle to broader commercial adoption [8]. That means it's the question, not the answer, that remains the obstacle. Some would say that alkali-activated materials have undergone detailed research only recently and may not have decades of durability data to prove long-term stability, but this is not the case [9,10]. It can also be asked what evidence, in addition to structures that exist in service for long periods of time, existing binding systems have to prove their fundamental durability? The answer is that there is very little - but the durability of Portland cement is not questioned, although recent analytical studies show significant changes in the nature of hydrated Portland cement binder and cement-slag mixtures over a 20-year period [11].

Also according to Van Deventer in this context, alkali-activated systems have the unenviable task of showing themselves to be durable when the final measure, the presence of decades-old structures, is in the end the only accepted verification tool [8]. Most standard methods for testing the durability of cement and concrete involve exposing small samples to very extreme conditions – such as highly concentrated acid or saline solutions – for short periods of time. These results are used to predict how the material will work under normal environmental conditions for a period of decades or more [8]. Predictive models depend on concepts that include mass transport through porous means, reaction kinetics, and particle packaging. A deficiency of this approach to try to "prove" durability is that it can only provide indications of expected performance under actual environmental conditions rather than any kind of definitive proof. In addition, the structure of the research community is fundamentally based around the time scale of graduate research projects, 3-6 years. This inherently leads to the problem that what is considered a "long-term" test is at least a smaller order of magnitude than the expected service life of most concrete structures. Where projects are conducted for longer periods of time, there needs to be strong management and focus on the project, which is rarely seen. So the reality is that durability tests mean little in the absence of real-world validation [8].

For the same author, the adoption of new scale materials is intrinsically very slow, as periods of 20 to 30 years are required to verify test results, and this is clearly not a time scale on which marketing efforts can be based [8]. Therefore, there are two possible approaches, which are now being carried out in parallel: linking the durability of alkali-activated materials to the chemical structure using cutting-edge scientific techniques and putting into use as many real-world structures as possible with very well documented mixing projects as soon as possible. possible, so that they can reach the required ages of decades or more, as soon as possible [8].

Today society is thinking about "Sustainable Development", in order to avoid quality problems in civil construction, to take into account social, environmental and economic aspects, encouraging the development of methods and tools to assess the durability of construction products, both traditional and innovative products [12]. In this context, two complementary tools were developed by Lair, being 1-Data fusion procedure (left part of Figure 2) and 2-Analysis of Failure Modes and Effects (right part of Figure 2) [13,14].

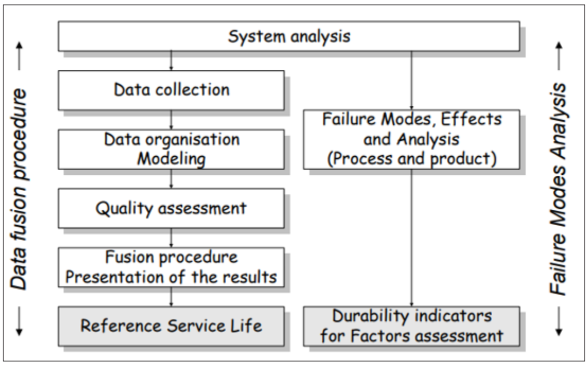


**Figure 2:** Durability Evaluation Methods and Tools [14]

**Alkali Activated Materials**

As the main connecting material used in concrete production, ordinary Portland cement accounts for about 5% of the world's $CO_2$ generation, whereas for economic and environmentally friendly reasons, it has been necessary to study other alternative building materials such as alkali activated binders [15-17]. Alkaline activated materials, also called "geopolymers", have been studied in recent decades as alternative binders to traditional Portland cement [18]. The study of alkali-activated systems has been studied since 1940, when Purdon observed the good development of mechanical resistance in the activation of slags with alkaline solutions and lime [19,20].

Glukhovsky Viktor, also devoted himself to the study of alkaline materials, having noticed in 1959 that these materials had a type of composition similar to that of many minerals and rocks that are constituents of the earth crust, having classified two groups according to the composition of the initial material: a)-$Me_2O$-$Al_2O_2$-$H_2O$ interpolymer or geopolymer system, being Me=Na, K... and M=Ca, Mg. Since the a)-($Me_2O$-$Al_2O_3$-$SiO_2$-$H_2O$) system refers to alkaline materials rich in $SiO_2$ and $Al_2O_3$, that when activated by alkaline solutions form an amorphous aluminosilicate material, responsible for the high mechanical resistance, while the system b)-($Me_2O$-MO-$Al_2O_3$-$SiO_2$-$H_2O$), produces a C-S-H gel forming calcium-rich compounds [21-22]. In recent years, several writers have reported research related to a large number of aspects on alkaline activation-based binders, as, among others: Dependence on the nature of the origin of the materials, since binders activated by alkalis synthesized from calcined sources have a higher compressive strength than common raw materials [23]. immobilization of toxic metals, reaction mechanisms and hydration products, the role of calcium in alkaline activation manufacturing operations and also, the development of lightweight materials in the construction [23-39].

**Composition**

In general, Davidovits expanded the concepts of alkaline activated materials by formulating a specific composition of silica ($SiO_2$) and alumina ($Al_2O_3$) reactive constituents of aluminosilicates that reacting among themselves in a strongly alkaline environment, derive three types of bonds: Si-O-Al-O; Si-O-Al-O-Si-O and Si-O-Al-O-Si-O-Si-O [40-41]. In (Figure 3), alkali activated slag exemplifies alkali activated cements with high calcium content, with $SiO_2$ + CaO representing more than 70% of the total weight of the ligant constituents and $Al_2O_3$ with less than 20%. The main reaction product is formed in the earlier states of hydration and is called C-S-H (hydrated calcium silicate). Alkaline products N-A-S-H and C-(N)-A-S-H (resulting from the replacement of $Ca^{2+}$ by $Na^+$) are formed more slowly due to the fact that the crystallization process is more time-consuming [42].

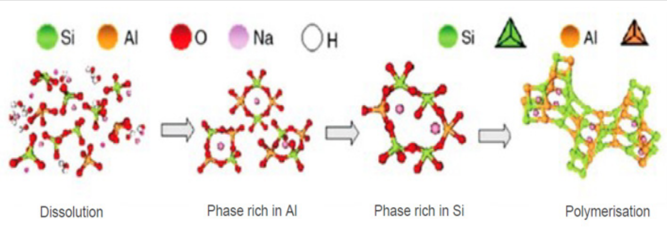


**Figure 3:** Alkali Activation Mechanism Proposed by Garcia-Lodeiro [43,44]

Alkaline activation involves a chemical reaction between various aluminosilicate oxides with silicates under various highly alkaline conditions, producing Si-O-Al-O bonds of polymer matrix, thus indicating that any Si-Al materials can be activated by alkali [23].

**Performance**

Regarding durability, materials with alkaline activation are highlighted in the reduction of both permeability and porosity, because an accessible porosity allows the entry of liquid and gaseous agents capable of promoting chemical changes inside the concrete [40]. Alkaline cements and alkaline hybrid cements in terms of durability show, in most cases, pc-like behavior, although it is true that they stand out for their excellent behavior against acid attack and their extraordinary fire resistance [45-49].

These indicators contribute to a large extent to the performance of this type of materials associated with the results, although a limited number of life cycle analyses of alkaline activation technology [16]. A reasonably extensive research program carried out in Germany provided much information on the selection of precursors and mixing designs for a variety of materials generally based on geopolymers [50-52]. However, geographical specificity plays a significant role in a thorough life cycle analysis and therefore there is a need for further studies in different locations, in addition to a wide range of mixing projects covering the broader spectrum of AAMs [16].

In the mid-1950s, and in the context of a demand for Portland cement alternatives in the former Soviet Union, Glukhovsky began investigating ligands used in ancient Roman and Egyptian structures [53]. Based on these observations, he developed binders called "soil-cement", combining alominosilicate residues, such as various types of slag, with alkaline solutions of industrial waste to form an alternative binder to cement. From the 1960s, the Glukhovsky Institute in Kiev, Ukraine, became involved in the construction of apartment buildings, railway dormants, stretches of roads, pipes, drainage and irrigation channels, floors for dairy farms, slabs and precast blocks, using alkali activated blast-oven slag [54,55]. Further studies of sections taken from these original structures showed that these materials have high durability and compact microstructure [56]. A large number of patents and standards have been produced in previous slag mixtures, but this documentation has been largely inaccessible in the West. The Kiev team continued under the supervision of Prof. Pavlo Kryvenko to develop mixing projects for different raw materials and applications [57-59].

In the late 1970s, Davidovits coined the term "geopolymer" for a variety of alkali-activated metacaulin binders that did not involve OPC-type phases in their chemistry, that soon gained interest from government and industrial organizations in the U.S [16,60]. During the 1980s and 1990s, an OPC/AAM hybrid concrete called Pyrament was developed and marketed. This concrete reached a resistance of 20 MPa in 4 h and was used in the 1991 Gulf War for rapid placement of runways. In 1993, Pyrament was applied to 50 industrial facilities in the U.S. 57 military installations, and seven other countries [61,62]. In 1994, the U.S. Army Corps of Engineers released a study showing that the Pyrament performed better than expected for other high-quality concrete [63].

However, in 1996, the marketing of Pyrament was terminated, not for technical reasons, but because of restructuring and corporate issues related to its manufacturer's parent company. Unfortunately, this event tarnished the image of concrete AAMs and much of the operational experience gained with Pyrament was lost. Pyrament was about twice as expensive as OPC concrete and still had a high OPC content, since its goal was technical performance rather than lower $CO_2$ emissions or cost reduction [64]. Thus, it is clear that geopolymer and alkaline activation technology has been known in the cement and concrete industry for approximately 50 years, but commercial acceptance of the technology so far has been limited until very recently. These obstacles that have hindered the growth of the AAMs industry are being quickly eliminated due to global concerns about $CO_2$ emissions and climate change, so the growing geopolymer industry in some parts of the world is in an enviable situation of being unable to cope with market demand due to complications in developing a high-volume supply chain in parallel (or possibly in competition with) the Portland cement concrete industry highly vertically integrated [16].

A commercial life cycle analysis was carried out in Australia and was coordinated by commercial producers of inorganic polymeric concrete. This life cycle analysis compared the AAMs concrete product with a standard Portland cement product available in Australia in 2007 based on the comparison of binder to binder and the comparison of concrete to concrete [16]. The linkage comparison showed an 80% reduction in $CO_2$ emissions, while the concrete comparison showed a slightly higher savings of 60%, as the energy cost of producing and transporting aggregates was identical for both materials. However, this study was again specific to a single site and a specific product, and it will certainly be necessary to conduct further analysis of new products as they reach the stages of development and marketing internationally [16].

**The Life Cycle of Materials**

The methodology (LCA) assesses the potential impact of materials, products and technology. Its procedure is standardised in international standards of the 2006 14040 series. The life cycle is defined as the consecutive and interconnected stages of a product system, from the acquisition of raw material or generation of natural resources to the final disposal (ISO 14040, 2006). Life cycle assessment is the compilation and evaluation of inputs, outputs and potential environmental impacts of a product system throughout its life cycle, structured in four main steps: Definition of objectives and scope, Inventory analysis, Impact Assessment and Interpretation [65,66]. The environmental impact of geopolymers remains a recent open debate, especially as they present themselves as an alternative to conventional concrete. Initially, Duxon stated that $CO_2$ emissions in the production of geopolymers were much lower than in OPC production [65,67]. These statements were based on two indicators such as: the reduction of water use and the non-need for superplasticizer additives. This first assessment did not take into account the impact of industrial waste production, such as ash and blast furnace slag, which are not accessible to all countries (e.g. Europe) [68]. The first ACL of geopolymers was published by Weil [52]. Having been made a comparison between 1m3 of ice-defrost resistant concrete of class XF2 and XF4 according to DIN EN 206-1, DIN 1045-4 (CEM I 32.5R) and 1m3 of geopolymer with a slag/fly ash with ash ratio of approximately 80/20 cured at room temperature [69-71]. The LCA considered three impact categories: Abiotic Depletion Potential (ADP), Global Warming Potential (GWP) and Cumulative Energy Demand (CED). The geopolymer surpassed CEM I concrete in global warming potential (GWP) by a factor 3, and with a similar impact, in abiotic depletion potential (ADP) and also in cumulative energy demand (CED). Habert show, however, that the production of geopolymer concrete has a greater environmental impact in other categories than global warming due to the production of sodium silicate [72]. There are environmental indicators to consider: Abiotic depletion; Global warming; Destruction of the ozone layer; Ecotoxity of marine water; Terrestrial ecotoxity; Human saxonity; Eutrophication; Acidification and Photochemical Oxidation [73]. Life cycle assessment is the compilation and evaluation of inputs, outputs, and potential environmental impacts of a product system throughout its life cycle. It has four main stages; 1-Definition of objective and scope, 2-Inventory analysis, 3-Evaluation, and 4-Impact interpretation, as (Figure 4) [74].

The relationships between the parts of ISO 15686 and the planning of the useful life of buildings can be interpreted in (Figure 5), which specify their general principles of planning the useful life of a building or other built asset and present an indicative structure for the execution of planning. These general principles can also be used for decision-making on maintenance and replacement requirements, also serving as a guide for other parties, including requirements and guidance on estimating the life of a building's components, which contribute to the life of buildings.

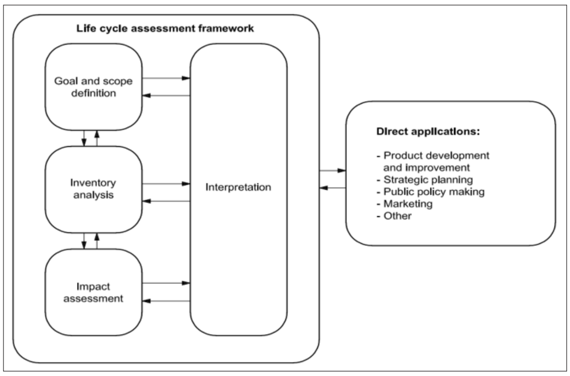


**Figure 4:** Key Steps on Life Cycle Assessment, Adapted from [75]

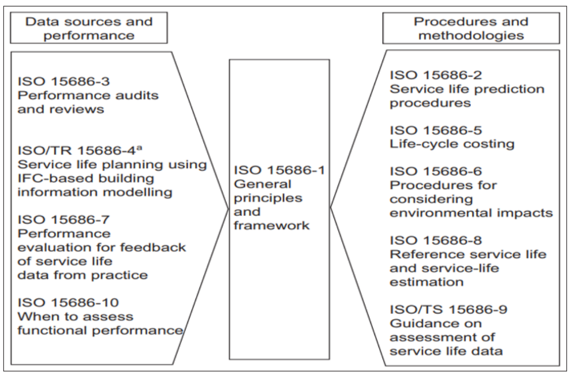


**Figure 5**: Relations between the Parts of ISO 15686 and the Planning of the Useful Life of Buildings, Adapted by [76]

**The Forecast**

The prediction or estimation of the useful life according to ISO 15686 can be addressed with the reading of three north-south vertical axes, according to Figure-6, being the first axis with test data and degradation model (part-2), the second axis with origin in the reference useful life on certain conditions (part-8), combining with the indications on data and production documentation (part-9) and finally the third axis originated in the documentation with information on life (part-7).

These three vertical axes finally converge, directly predicting the useful life, when under the same conditions of use and or for the same performances of the components, or, in the absence of these latter obligations, the Factor method for the proper provision of the useful life may be used.

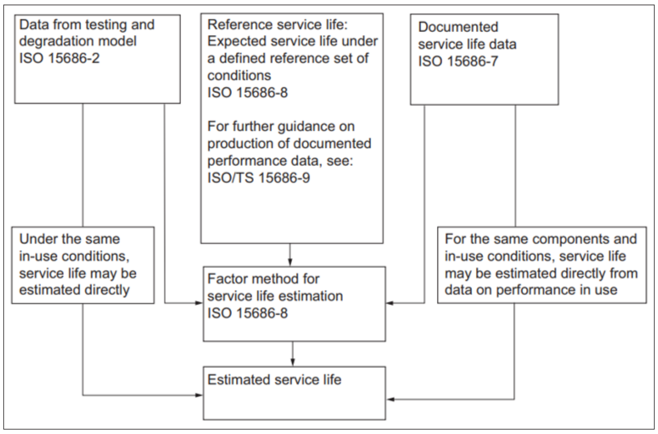


**Figure 6:** Methods and Tools for Defining the Prediction of Life, Adapted from [76]

**Methodologies and Planning**

In the field of methodology and planning, it is common for there to be complex problems from the collection of data to the decision, such as meteorology, toxicology, traffic management and others, to which experts take different approaches, starting, in most cases with the collection of product data, its definition, its environment and others, so that they can understand and model all the phenomena involved [77]. Finally, from this modeling, it extracts decision elements taken in the recommendations, alternative comparison elements, evaluation parameters to be used and others. In this approach, and especially in the problem of life-expectancy assessment, one of the biggest obstacles to decision making is to be able to deal with both uncertain and heterogeneous information, often with management of uncertainty and ignorance, where the solution is co-exploration of data, i.e. "Simultaneous exploration of multiple points of view in a data or in a method to process it" [77]. This approach enriches the analysis (complementary information, analysis and exploitation of the conflict) and conducts synthesized and consensual information. In addition, managing uncertainty and ignorance increases the credibility of the results. To bring these procedures together, it was suggested the model in Figure 7, with the following four main steps: (1)-Data collection (2)- Data organization and modeling (3)-Fusion procedure and (4)-Reportage [77].

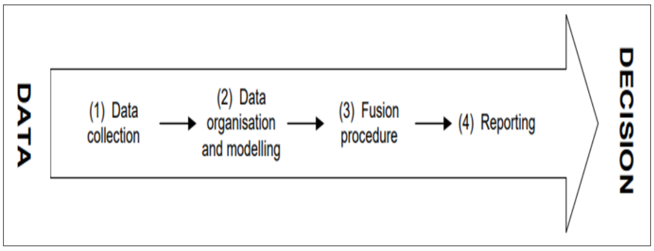


**Figure 7:** Proposed Approach to Methodology and Planning, Adapted from [77]

Where, the steps (1) and (2) several models with different points of view are analyzed, including the evaluation of construction products, in step (3) the data are uniond by extracting the concensual information in the viable format for the stage (4) [77].

Currently there are several tools and methods for durability assessment (field tracking studies, expert opinion, accelerated tests, natural weathering, modeling (reliability models...), materials science... But its use implies some problems: non-reproducibility and traceability of field tracking studies, subjectivity of expert opinion, duration of accelerated tests and natural weathering, relevance of the torture test, quality and amount of knowledge required for modeling (these studies are available only for simple and known materials or products, for one or two degradation phenomena) [77]. Data collection consists of collecting all available durability data from the product or one of its components, in its intended environment or in one of its parts [77]. In fact, two types of lifetime data can be collected: - Data that fully represents the system in your predicted environment. - Data representing only a part of the system (component), and/or a part of the predicted environment (a degradation phenomenon) [77]. All this information is dispersed (variety of sources and studies), in a dissimilar way (scale and formalism of uncertainty) and with different quality (force of hypothesis...) [77].

## The Durability and Properties of Engineering

For Duxson, Figure 8, the durability of these AAM materials is precisely the determining factor that differentiates them from Portland cement [67,78]. To these advantages should be added the fact that these binders allow the reuse of some materials such as waste from mines and quarries, and still have a high capacity for the immobilization of toxic and radioactive waste, which gives them an indisputable environmental value [79]. For there is a direct relationship between durability and different comprehensive engineering properties, including geopolymeric structure, surfaces and interfaces, chemistry, physics, aquatic environment, additives, binders, alkaline metals, the electric charge balance of cations, the electrical load of the surface, composition, execution, synergy and competition, the role of the ligating phase, diffusion transport, nucleation and crystallization, precipitation of impurities, alkaline-ground metals, particles that do not react, the Incapsulates and others, all referenced in this Figure-8, in a broad relationship between durability and engineering properties.

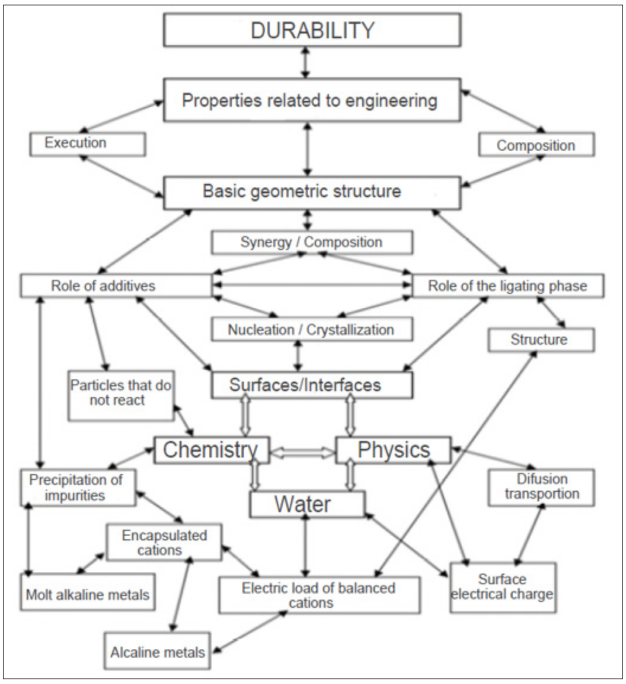


**Figure 8:** Schematic Diagram Showing Some of the Relationships between the Various Areas that Constitute the Technology of Geopolymeric Ligands, Adapted from and Quoted in [7,67]

## Chemical Matrix Degradation Processes

Alkaline cements and alkaline hybrid cements in terms of durability show, in most cases, pc-like behavior, although it is true that they stand out for their excellent behavior against acid attack and their extraordinary fire resistance [45,46,47,48,80,81].

Although most concrete structures are not subjected to highly acidic conditions, there are some situations where this becomes a problem and, in these circumstances, the life of concrete structures can be severely reduced. Acid rain, acid sulfate soils, animal use and industrial processes can produce acids that can potentially degrade concrete [82-87]. However, the most economically important industrial cause of acid-induced damage in infrastructure elements is corrosion by biogenic sulfuric acid, which usually occurs in sewage pipes, and is an important focus of research in several long-standing studies around the world, with various technical solutions (whether related to the manipulation of concrete by the pipe itself or by the use of coatings) developed and implemented [88-92]. Many of the procedures used in acid attack testing of concrete are similar, in a general sense, to leaching tests, several of which specifically involve exposure to acidic conditions. The most important mode of acid attack on a linker, whether based on OPC or AAMs, occurs through the degradation of concrete by ion exchange reactions. This leads to a breakdown of the nano and microstructure of the matrix and the weakening of the material. In some cases, this can be extremely fast and serious, and acidic conditions can be induced by industrial or biogenic processes. In a lab test, different parameters are adjusted to mimic the real-life situation as closely as possible, or to accelerate degradation and thus get results faster, and the extent and way this acceleration is applied will influence the test results. These parameters include the pH and concentration of the acid solution, the physical state of the sample (monolith or powder, mortar or concrete paste), temperature, acid replenishment rate, presence or absence of mechanical action/flow, wetting and alternating drying, heating, alternating cooling and pressure. These parameters should be carefully selected and should always be reported along with test results. In addition, the choice of the selected degradation measure (loss of strength, loss of mass, depth of penetration) can lead to different conclusions about the relative performance of concrete types, in particular when the binder chemistry is quite different between samples [93]. A combination of several relevant indicators will often be required. In addition, the preparation and conditioning procedures of the sample and maturity at the time of the test are extremely important.

AAMs have often been advertised as being highly acid resistant; this has proven to be, in fact, an important driver for academic and commercial developments in this area for many years [94,95]. However, many of the claims submitted have not been sufficiently tested to allow the use of AAMs in acid exposure applications where long-term performance is critical. In addition, the applied tests were generally designed for Portland cement classifiers and have not yet been validated for AAMs and can therefore provide the expected information on the performance of the "real world" [96,97]. Due to the use of different raw materials, curing durations, mixing designs, sample formats, acid exposure conditions and performance parameters in each published study on acid resistance of AAMs, it is very difficult to make an immediately meaningful comparison between the results obtained. Many of the test methods used vary drastically from the conditions foreseen in service; for example, 70% nitric acid at room temperature, or 70% $H_2SO_4$ at 100 °C [95,98]. Tests in conditions close to those foreseen in service are expected to provide more representative results, but with potentially longer test durations; this is more or less universal in the development of accelerated test methods.

**Degradation Processes by Mass Transport**

Currently, the chemistry understanding of steel corrosion within AAMs binders is still probably insufficient to allow the development of specific testing methods for the chemistry of these materials. This is particularly the case for AAMs materials based on GBFS or other metallurgical slag containing sulfates, which generates a reducing environment within the linker and causes complexities in electrochemistry that are not yet well understood. It will certainly be necessary to analyze and understand the mixtures in large-volume GBFS with OPC (which have reached a more advanced stage in the analysis of AAMs, due to the greater maturity of this research topic), in order to obtain a deeper understanding of the influence of sulfate chemistry in the corrosion rates of steel as to its complexity [99]. The effects of the presence of high concentrations of alkalis and, in particular, the interaction between carbonation, chlorides and alkalis, as well as the relationship between the transport properties and the chemistry of steel corrosion at the rebar-paste interface, provide a fruitful ground for researchers in the coming decades, and much remains to be understood in this area of research. Thus, it seems important to recommend that whatever the test methods selected for the analysis of AAMs, a complete report of the conditions and experimental details in each published study is essential to provide the reader with the ability to understand and use the results of the work [99]. This is universally important in implementing durability testing, but is particularly critical in areas such as corrosion testing, where there are so many misunderstood parameters that can potentially influence the results obtained in all tests performed. In most reinforced concrete applications, the predominant modes of structural material failure are more related to the degradation of the built-in steel reinforcement than the alloyer itself [99]. Thus, a key role played by any structural concrete is the supply of cover depth and sufficient alkalinity to keep steel in a passive state for a long period of time [99]. Loss of passivation usually occurs due to the entry of aggressive species such as chlorides, and or loss of alkalinity by processes such as carbonation [99]. This means that the mass transport properties of the hardened ligand are essential to determine the durability of concrete, and therefore the analysis and testing of the properties related to the transport of alkali-activated materials become fundamental [99]. Sections dedicated to the chemistry of steel corrosion in alkali-activated binders and efflorescence (which is a phenomenon observed in the case of excessive alkaline mobility) are also incorporated into the study and discussion due to their close connections with the transport properties [99].

**Permeability and Porosity**

There are numerous studies on the relationships between microstructures and permeability of Portland cement-based concrete, including some presented and revised in detail in, many different porosimetric techniques are available, some of which have been formally standardized in different jurisdictions, but most of which rely on commercially available or institute-built laboratory equipment for sample analysis [99-102]. Also, to obtain results and information taken in standardized and non-standardized analytical protocols, there are original publications with complete experimental details in each instance, namely the reports presented in RILEM TCs 116-PCD and 189-NEC, which contain descriptions and analysis of many of the available techniques on the permeability of concrete [99,103,104].

Gas sorption analysis (usually using nitrogen, argon, or helium) can also be used to calculate pore size distributions and surface areas through various algorithms such as the popular Brunauer-Emmett-Teller (BET) and Barrett-Joyner-Halenda (BJH), and mercury intrusion porosimetry (MIP) is also widely used in the study of hardened phases of the ligant [105,106]. Some controversy involves the applicability of MIP to complex pore geometries, such as those observed in building materials, but these complications may also provide opportunities for more advanced analytical procedures such as multicycle MIP and Wood metal intrusion to provide more understanding of the pore geometries present in the material [107-110]. Water permeability analysis also provides useful information when applied to hardened concrete – where water is forced into the sample under pressure or where capillary suction is used to attract water to the sample. Each of these test classes is able to provide essential information for understanding the structure and durability of AAM concrete and for predicting its performance in service. There is no universally applicable technique that can provide a complete multiscale characterization of a complex material, such as an AAMs or concrete linker; a more complete toolkit of techniques is required to get details on the length scales of interest. The BJH method for pore size distribution calculations has been standardized in several jurisdictions, but for porous materials in general and not with specific application for cements or building materials [106,111,112]. Although this method has been compared unfavorably in recent years to more advanced methods of converting gas sorption data into pore size distribution information, this remains the method that has been applied more widely for the extraction of gas pore size distribution information sorption data for alkali-activated binders [113,114]. Lloyd, e Zheng used the BJH technique to observe the refinement of pores in AAMs derived from fly ash with increasing activator concentration, which is consistent with the conceptual understanding of the formation of these materials [110,115].

**Capillarity**

Capillarity tests have shown that the BFS concrete pore networks activated with alkali are sufficiently refined and tortuous to lead to a fairly low extent of capillary sorvability city in these materials, although porosity was, in most cases, similar or higher to that of comparable Portland cements [45,116,117]. The use of a higher module activator, or a lower water content in alkali-activated BFS systems, reduces water absorption rate and sorption decreases with increased curing time in humid conditions [118,119]. Attempts were also made to model the flow rate through the BFS concrete pore networks activated with alkali, describing capillary pores as a pore shaped elliptical model, which was to some extent successful, although it is known that the actual geometry of the alkali-activated agglutinating pore network involves significant "cartridge" effects, where larger radius pore volumes are accessible only through narrow necks [110,120-122]. Very high capillary suction of highly porous alkali-activated metakaolin or natural pozzolan-based binders is potentially problematic in many applications and can lead to efflorescence if alkali movement is not properly controlled, but also provides possible applications in thermal control, providing a water source for evaporative cooling [123,124]. Another standardized test method is the Initial Surface Absorption Test (ISAT), as described in BS 1881-208, which uses a narrow capillary placed in contact with the surface of a sample of dry concrete (or in service) and calculate the sorbity of the concrete from the rate of movement of the water from the capillary to the material [125]. This test has the advantage of being relatively fast (approx. 1 h per sample), but does not seem to have been applied to alkali-activated concretes in the currently available literature. Similar methods include the EN 772-11 test, which is specified for mortars and also measures the water flow of an external pipe in the pore structure of the material [126].

## Carbonation

There is limited knowledge about carbonation in AAMs, Byfors identified higher carbonation rates in F-concretes (GBFS activated by sodium silicate), when compared, in accelerated tests, with common concretes [99,127]. These results are in accordance with the observations of Bakharev, who also reported greater susceptibility to carbonation in concrete prepared AAMs with sodium silicates and BFS than in reference concretes to the common Portland cement base, when evaluated under accelerated carbonation conditions [99,128]. On the other hand, Deja identified that GBFS mortars and concretes activated with alkali showed carbonation depths comparable to those obtained for Portland cement reference samples, along with increased compression strengths and longer $CO_2$ exposure time [99,129]. This was associated with a refinement of the pore structure, as carbonates precipitated during the carbonation reaction. This was more noticeable in silicate-based activated samples than in sodium carbonate-activated samples [99]. It is important to note that accelerated carbonation of the specimens in this study was induced using a carbonation chamber at a relative humidity temperature of 90% and fully saturated with $CO_2$ [99]. These results should be interpreted very carefully because, in such high relative humidity, pore saturation in these specimens is such that even when exposing AAMs to extremely severe $CO_2$ concentrations, the carbonation reaction does not develop in the same way as it would be at lower relative humidity values [99]. This is consistent with the trends identified by Byfors, which identified that the carbonation of AAMs is faster when materials are exposed to lower relative humidity [99,127]. The relative humidity conditions in which the carbonation of AAMs is evaluated are critical, as the shrinkage by drying and subsequent carbonation can be favored in low humidity conditions that can induce microcracks in the material, increasing the progress of carbonation [99]. Studies conducted by Bernal in GBFS/concretes with metakaolin mixture showed that the progress of carbonation and the consequent increase in total porosity are legitimately higher when the samples are carbonated at 65% relative humidity, compared to carbonated samples at values 50 or 80% relative humidity [99,130]. However, after longer periods of carbonation exposure, the effect of relative humidity becomes less relevant, and slightly increased carbonate depths are identified with increased relative humidity [99]. For carbonation tests of GBFS and GBFS/ metakaolin enabled by calcium silicate, where the samples were not submitted to the drying process before the test, a separate measurement of the water absorption of non-carbonated samples provided a good indication of how the drying effects during the test period would slow, probably the early stages of carbonation [131].Testing samples with low water absorption (i.e. when the pore network is initially saturated and refined) at high relative humidity provides a very low carbonation rate in the early stages of the test, as the carbonation rate of the saturated binder is relatively slow, and subsequently there is an acceleration of the carbonation process as the drying front enters the sample and allows the continuation of carbonation [99]. Very little attention has been paid to the evaluation of carbonation retraction in construction materials in general, a fact that is associated with the stresses induced in cement pastes as a consequence of the formation of carbonation products, initially in the pore network and then in the advanced carbonation conditions in the binder gel [132]. There is no specific standard method to measure carbonation retraction, but only in a few studies this behavior was evaluated, procedures similar to those described in ASTM C596-09 or ASTM C1090-10 are adopted [99,133-135]. In this case, shrinkage is measured at different times of $CO_2$ exposure to correlate the carbonation depth with any dimensional changes presented by the samples. The extension of carbonation shrinkage in AAMs is, in terms of the available literature, completely unknown. However, Shi identified that during 75 days of exposure of a GBFS paste activated by alkali at 15% $CO_2$ and 53% relative humidity, cracks were observed a few days after the start of the carbonation test, as a combined effect of shrinkage by drying and shrinkage by carbonation [99,136].

## Full-Scale Applications

While the recent and growing demand for alternatives, greener to traditional concrete, has led to more research on AAMs, this technology and its application in construction projects are not new. The development of AAMs was accentuated in the post-World War II period, with the first applications in the 1950s when concrete, based on explosion slag, activated only with calcium hydroxide ($Ca(OH)_2$) or in combination with sodium sulfate (Na2SO4), called "Purdocement" was first used in Belgium for the realization of several buildings [137,138]. Since then, numerous structures have been carried out, including hydraulic works, pavements, roads, conventional precast products, and more recently, large-scale molded-in-situ projects, as follows (Table-1).

**Table 1: Examples of Full-Scale Applications, Adapted from** [141]

| Year | Location | Work of Construction | Material | Ref. |
|---|---|---|---|---|
| 1952-1959 | Brussels, Belgium | Parking 58 | Purdocement (GGBFS + PC activated by $Ca(OH)_2$ or $Na_2SO_4$) | [137] |
| 1960-1980 | Mariupol, Ukraine | 2-storey and 15- storey residential buildings | Alkali-hydroxide activated GGBFS concrete | [137] |
| 1966 | Odessa, Ukraine | Drainage collector No. 5 | Alkali-carbonate activated GGBFS concrete | [137] |
| 1974 | Krakow, Poland | Storehouse | Precast steel-reinforced alkali-carbonate activated GGBFS concrete | [137] |
| 1986-1994 | Lipetsk, Russia | 24-storey residential building | Alkali-carbonate activated GGBFS concrete | [137] |
| 1988 | Yinshan County, Hubei Province P.R. China | 6-storey office and retail building | Sodium sulfate-activated Portland-slag cement concrete | [137] |
| 2009 | Melbourne, Australia | Salmon St Bridge | E-Crete precast footpath panel segments (180 precast footway units) | [139] |
| 2009 | Brisbane, Australia | Murrarie Plant site bridge | EFC precast bridge decks | [140] |
| 2010 | Melbourne, Australia | Thomastown Recreation and Aquatic Center | E-Crete footpaths and driveways | [139] |
| 2012 | Melbourne, Australia | Melton Library | E-Crete precast panels and in-situ works | [139] |
| 2013 | Queensland, Australia | Global Change Institute (GCI) Building, University of Queensland | EFC – 33 precast floor beam-slab elements | [140] |
| 2013 | Irvine, California, USA | Sustainable concrete solar-powered house | Precast alkali-activated fly ash concrete members | [141] |
| 2013 | Yuozhong District, Chongqing, P.R China | Chongqing Research Institute of Construction Science office building | Cast in-situ alkali-activated GGBFS concrete | [141] |
| 2014 | Toowoomba, | Toowoomba | EFC-cast in-situ heavy-duty pavements | [140] |
| 2017 | London, UK | Thames Tideway Central, Kirtling Street | Cenfree-cast in-situ | [142] |
| 2020 | Wageningen, Netherlands | Cycle bridge | RAMAC (prefab) | [141] |
| 2021 | Chatham,UK | Chatham railway station (step-free access foundation) | Cemfree-300 m3 cash in-situ | [142] |
| 2021 | Le Havre, FRA | Grand Port Maritime du Havre | Exegy-concrete barrete (17 m depth) | [141] |

## Conclusions

- Recent studies of climate change have been the case that new cementitious materials are known to be responsible for a lower $CO_2$ emission level than Portland cement;
- The question of whether geopolymer concrete or AAMs are durable remains open, and constitutes an obstacle to wider commercial adoption;
- Regarding durability, alkaline materials are highlighted in the reduction of both permeability and porosity and stand out for their excellent behavior against acid attack and extraordinary fire resistance;
- Currently, the understanding of the chemistry of steel corrosion within the AAMs binders is still likely insufficient to allow the development of specific test methods for the chemistry of these materials, since the effects of the presence of high concentrations of alcalis and, in particular, the interaction between carbonation, chlorides and alkalis, as well as the relationship between the transport properties and the chemistry of steel corrosion at the regal-paste interface, provide a fruitful ground for researchers in the coming decades, and there is still much to be understood in this area of research.
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