International Journal of Research in Engineering and Science (IJRES)
ISSN (Online): 2320-9364, ISSN (Print): 2320-9356
www.ijres.org Volume 11 Issue 4 ǁ April 2023 ǁ PP. 474-478

Review on Durability of Alkaline-Activated Cement (AAC) and Its Microstructural Analysis Techniques

Imtiaz Ahmed. C.N[1]
[1] Student, Civil Engineering Department, M A College of Engineering, m21secm004@mace.ac.in

Abstract
As we all know that cement is one of the important construction materials which is used all over the world. But the large consumption of Ordinary Portland cement causes lots of environmental issues. The amount of CO_2 emission during the Production of Ordinary Portland cement results in the pollution of the atmosphere. Alkaline-activated cement (AAC) is one of the most suitable substitutes for Ordinary Portland cement. In Alkali-Activated Slag (AAS), various alumino-silicate materials such as ground granulated blast furnace slag (GGBS), fly ash (FA), etc are the main source materials for alkali activation. Various studies in AASC have proven that it have a superior durability in acidic environments as compared to OPC. Also the permeability is also very low as compared to OPC because it contain Ca. In this paper we evaluate the performance of AASC in sulphuric acid. The changes made by the acid exposure in the micro-structural level of the AASC is also evaluated here.

Keywords: *Alkaline-activated cement (AAC), ground granulated blast furnace slag (GGBS), fly ash (FA), Durability, Thermogravimetric analysis (TGA), X-Ray Diffraction (XRD)*

Date of Submission: 08-04-2023 Date of acceptance: 22-04-2023

I. INTRODUCTION

The Ordinary Portland cement produce large amount Of CO2 gas during its production process. The use of alkali-activated (AA) binder has gradually attracted attention since the late 1990s as another active effort to reduce CO2 emissions in concrete production [5, 6, 8].

1) The CO2 emission of concrete increases with its compressive strength, indicating that the contribution of the binder to the total CO2 footprint is more significant in OPC-based concrete [12] than in AA concrete. As AAC is a alternative to Ordinary Portland cement it should able to meet the mechanical strength, workability and durability requirements. But in case of AAC the lack of long-term durability data which helps to predict the structural integrity of the material is really a problem during the modern scenario. So, it is really essential to evaluate the durability of AAC so thet we can predict the long-term performance. This is a critical aspect, as quantification of AAC durability relates to the prediction of service life, and this is one of the main challenges facing the commercial adoption of AAC [11].

Now a days Ground granulated blast furnace slag (GGBFS) and fly ash (FA) are mainly used in the large-scale industrial production of AACs [6], the use of GGBFS and FA helps to develop high mechanical strength and other important properties [18]. As in the case of OPC acid attack is one of the main problem faced by the AAC so it is important to understanding the degradation kinetics and the mechanism of degradation of cement-based materials exposed to various acidic solutions. The terms ‘degradation kinetics’ and ‘alteration kinetics’ are commonly used in the acid attack/leaching studies to describe the variation of degradation related parameters (mass change, thickness change, altered depth etc.) with the age of acid exposure [20]. Concrete structures are vulnerable to the attack by aggressive aqueous environments in many situations, resulting in deterioration. The understanding of the alteration kinetics and mechanism of degradation of concrete by these aggressive aqueous media is a fundamental step towards the development of a durable concrete which will increase the service life or the safety of the structures.

The durability of an AAC based on GGBFS/FA blends is directly linked to the microstructure of the reaction products forming in these binders, as a function of the nature and concentration of the activator, and also the maturity of the material. So microstructural analysis is also a important step in this context.

Figure: 1 Acid attacked specimen [14]

1.1 Alkaline-activated cement (AAC)

Ordinary Portland Cement Concrete has been widely used since the 1800's. Research estimates that each ton of Cement produced, releases into the environment about 0.84 ton of Carbon Dioxide; directly during decomposition and indirectly because of the energy consumed during production. The total contribution of Cement to global Carbon Dioxide emissions is estimated to be between 5 and 8 percent. Today around 4.2 billion tons of Cement are produced annually with an expected growth rate of 2.5% per annum. Simultaneously, latently hydraulic cementitious matrices such as Fly Ash, Silica Fumes and Slag which are byproducts from various industries have been used as admixtures to replace some of the cement content. This leads to an improvement in concrete properties such as strength and permeability. The presence of cement, however, is required for the cementitious matrices' reaction to occur and produce the binding material in concrete. The idea behind alkali-activation is inhibiting the need for the cement's reaction and 'activating' the unaccompanied cementitious matrices [2]

The main benefits of using AAFA concrete or AAFA concrete blended with slag are at least two-fold: reduction in the environmental impacts resulting from cement production and reduction in stockpiling of common wastes. Compared to PC, this kind of binder material may be economical because the source material is an industrial by-product. Although the workability of fresh AAFA concrete decreases with incorporation of slag, the mechanical strengths significantly increase. The higher the slag content, the higher the increase in mechanical strengths [1].

Alkaline-activated cement (AAC) can be prepared by mixing the Alumino silicate Source, Alkali activator and aggregates in a particular ratio.

Figure: 2 Prepration of AAC

1.1.1 Constituents of AAC

Alkali activated system composed of one or combined alumino-silicate sources and one or combined alkaline activators. The activator solutions increase the pH value (e.g. hydroxides, silicates, carbonates or sulfates). Alumino-silicate source and alkaline activator can be pre-mixed as dry materials, then this dry binder be mixed with water, aggregates to produce a mortar or concrete. In another way, the alkaline activator solution can be added to the alumino-silicate source separately, then this wet binder be mixed with additional water (in case of need to dilute the concentration of alkaline solution), aggregates to produce a mortar or concrete. Alternatively, it is possible to produce AAC by mixing alumino-silicate source, alkaline activator, water, aggregates and admixtures without pre-producing the alkali activated binder separately [13, 15, 16, 19, 22, 25] presents the components of the alkali-activated system [7].

II. MATERIALS

The different materials are:

Sodium hydroxide (NaOH)

NaOH is obtained by brine electrolysis. Four types are available commercially: solids, flakes and beads. Solids are obtained by cooling molten caustic soda. Anhydrous NaOH has a specific gravity of 2.13 [3].

Sodium silicate (Na_2SiO_3)

Sodium silicate is an inorganic sodium salt which has silicate as the counterion. It is also called Sodium metasilicate or Waterglass. Na_2SiO_3 is generally produced from silica and carbonate salts by calcination and dissolving in water according to the required ratios.

Cementitious components

Commonly used cementitious materials for AAC are GGBFS, FA, Silica fume and Metakaolin (burnt clay) [10]

III. DURABILITY STUDY

The resistance of concrete to acid solutions can be tested in laboratory experiments in various ways: (1) realistic concentrations of the aggressive acid solutions can be used in combination with a sensitive method to detect the deterioration; (2) accelerated tests can be performed, in which the degradation rate can be increased in different ways (e.g. by means of higher concentrations of the acid solution, higher ratio of volume of acids to volume of specimens, higher temperatures, increasing the surface area to volume ratio of specimen, smaller sized specimen or alternate wetting and drying cycles etc.). since the extend of deterioration cannot be predictable because it depend upon the nature of the environment so extremely sensitive methods are needed to dectect the extend od deterioration in the conctrete [20].

The smaller specimen size was selected so as to have a higher surface area to volume ratio to enable accelerated testing. After preparing the specimen it is cured for in water for required period of time so that it helps to avoid calcium leaching and to attain the required strength before the acid exposure. After curing the specimen initial mass and thickness of the specimen is measured. Then prepare the sulphuric acid of required concentration generally 0.3M and put the cured specimen in the acid for required period of time. Tests on this specimen should be conducted at particular interval of time. Also, acid solution should be renewed frequently to maintain the aggressiveness of the solution.

After the required acid exposure time, the specimen should be taken out from the acid solution and uniform brushing should be done it helps to remove the loosely helded corroded particles on the surface of the specimen. After that different parameter that helps in the understanding of degradation kinetics such as mass change, thickness change and degradation depth that made by the acid solution is to be studied.

3.1. Mass change

Mass change can be measured by comparing the new mass after the acid exposure with the mass of the specimen before acid exposure. The mass of the specimen after initial curing can be taken with the help of a weighing balance before putting it into the acid. After the acid exposure the new mass is taken. The difference in mass can be prepresented in percentage with respect to the initial mass

3.2 Dimensional changes

as the specien is immerced in the acid solution, the chances for the change in thickness and surface area is more. When the specimen react with acid solution new products will be formed so that the chance for deterioration is also very high. Dimensional changes in the specimens were measured using a digital calliper. The change in dimensions is measured from different locations, at least six locations and the average value should be taken for calculating the dimensional change.

3.3 Compressive strength

The chances for decreasing the strength of the specimen is generally high since the specimen is immersed in the acid solution. So the change in the compressive strength of specimen can be determined. The compressive strength was determined by dividing the failure load by the altered cross-sectional area.

3.4 Deteriorated depth

The degradation depth measurements are important as they give an idea about the time taken for the degradation front to reach the reinforcement to cause corrosion in reinforced concrete structures. Hence, degradation depth could be considered as a primary indicator of attack and any technique which can capture the altered depth needs to be used for investigating the degradation kinetics. Degradation depth can be measured roughly using digital calliper based on visual changes by cutting the specimen perpendicular to its axis, but this is subject to errors and limitations in measurement accuracy. Alternatively, phenolphthalein solution can be sprayed to a fresh cut surface of the specimen and altered depth can be measured based on the colour changes (Pink colour). However, the complete degradation depth due to decalcification and mineralogical zonation may not be picked correctly using this method and the measurement accuracy will be compromised on smaller sized specimens. The image that obtained can be analysed using ImageJ software[23].

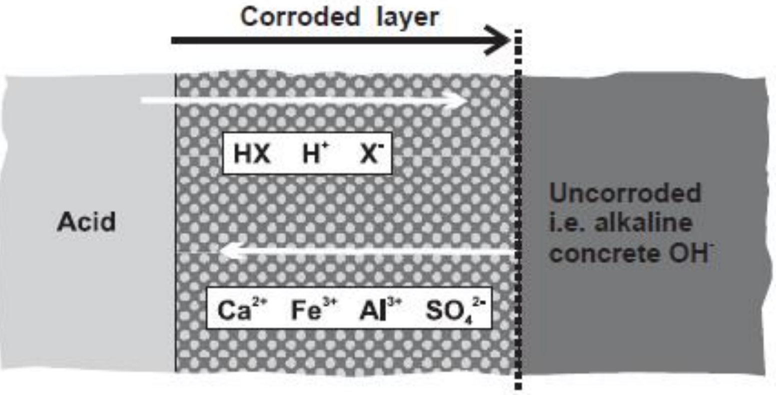

Figure : 3 Corrosion due to acid attack [21]

IV. MICROSTRUCTURE ANALYSIS

To understand the chemical transformation in materials before and after acidic exposure Thermogravimetric analysis (TGA) is done. The primary objective of conducting TGA is to estimate the portlandite and calcite content. The samples for TGA are prepared from prismatic paste. TG curve plot is made with Y axis representing the mass of the sample and X axis representing the temperature. The differentiation of the TG data gives the differential thermogravimetry plot which allows a better resolution of the alteration and in the identification of consecutive mass losses.

XRD has been used extensively for the examination of materials and thin films. Its effective use depends upon having a crystalline material. The technique is a bulk-sensitive analytical method, but can be used to provide information relevant to surface changes in suitable circumstances. For example, XRD can provide useful information on the extent to which surface treatment of a carbon system has affected the bulk of the material. It is possible to use XRD in a thin-film mode, employing very small take-off angles, to derive some surface information, but generally speaking it must be regarded as a bulk structural technique. Care should be taken while preparing the sample for XRD and TGA.

4.1 Sample Prepration of XRD and TGA

- The samples for TGA are prepared from prismatic paste of AASC
- Small sized specimen pastes samples
- slices were immersed in isopropanol solution for 2 days
- Take sample from isopropanol and dry the sample
- The dried samples were kept in vacuum desiccator until testing.
- The samples were powdered and the powder passing 75 Micrometre were taken for testing.

4.2 Benefits of XRD AND TGA

- Thermogravimetric analysis (TGA) can simulate roasting conditions and produce a small sample for chemical analysis or materials characterization

- A simultaneous thermogravimetric and differential scanning calorimetry (TG–DSC) can identify endothermic and exothermic reaction temperatures [24].
- It is a rapid and powerful technique for identifying unknown minerals and materials
- It only requires preparation of a minimal sample for analysis
- Interpreting the resulting data is relatively straightforward
- XRD measurement instruments are widely available [9].

REFERENCES

[1]. Alaa M. Rashad. (2013), "Properties of alkali-activated fly ash concrete blended with slag." Iranian Journal of Materials Science and Engineering, Vol. 10(1), pp. 57-64

[2]. Amer, Salma, Bakhoum, Michel, Yassa, Andrew, El Mahallaoui, Amr, Fahem, George, El Nahas, Eman, Hussam, Ahmed, and Abou-Zeid, Mohamed N. (2018), "Alkali-Activated Concrete," CSCE

[3]. C. Shi, D. Roy, P. Krivenko. (2003). "Alkali-activated cements and concretes" CRC Press ismail-18

[4]. Caijun Shi, Della Roy, Pavel Krivenko. (2014), "Alkali-Activated Cements and Concretes." https://doi.org/10.1201/9781482266900

[5]. Davidovits, J (2008). "Geopolymer Chemistry and Applications." Geopolymer Institute.

[6]. Duxson, P. and J.L. Provis, "Designing precursors for geopolymer cements. Journal of the American Ceramic Society", 2008. Vol. 91(12), pp. 3864-3869.

[7]. F. Pacheco-Torgal, J. Labrincha, C. Leonelli, A. Palomo, P. Chindaprasit. (2014), "Handbook of alkali-activated cements, mortars and concretes." Elsevier

[8]. F. Pacheco-Torgal, Joao P. Castro-Gomes and Said Jalali. (2008), "Alkali-Activated Binders: A Review: Part 1. Historical Background, Terminology, Reaction Mechanisms and Hydration Products." Construction and Building Materials, V0l. 22(7), pp. 1305-1314

[9]. https://www.scimed.co.uk/education/what-is-x-ray-diffraction-xrd/

[10]. Ismail Amer, Mohamed Kohail, M.S. El-Feky, Ahmed Rashad and Mohamed A. Khalaf. (2021), "A review on alkali-activated slag concrete." Ain Shams Engineering Journal, Vol. 12(2), pp. 1475-1499

[11]. Jannie S.J van Deventer, Rackel San Nicolas, Idawati Ismail, Susan A. Bernal, David G. Brice and John L. Provis. (2014), "Microstructure And Durability Of Alkali-Activated Materials As Key Parameters For Standardisation." Journal of Sustainable Cement-Based Materials, Vol. 4 (2). pp. 116-128

[12]. Keun–Hyeok Yang, Jin-Kyu Song and Keum-Il Song. (2013), "Assessment of CO2 reduction of alkali-activated concrete." Journal of Cleaner Production, Vol. 39, pp. 265-272.

[13]. M. Aly. (2019), "Performance of geopolymer concrete containing recycled rubber." Construction and Building Materials, 10.1016/j.conbuildmat.2019.02.121

[14]. M. Kumar, V. Revathi. (2023), "Durability studies in alkaline activated systems (metakaolin – bottom ash): A prospective study." Journal of the Spanish Ceramic and Glass Society, Vol. 62(1), pp. 40-55

[15]. M.S. El-Feky. (2020), "Effect of microwave curing as compared with conventional regimes on the performance of alkali activated slag pastes." Construction and Building Materials, 10.1016/j.conbuildmat.2019.117268

[16]. Ola Mayhoub.(2020), "Properties of slag based geopolymer reactive powder concrete." Ain Shams Engineering Journal, 10.1016/j.asej.2020.08.013

[17]. P. Duxson, A. Fernandez-Jimnez, J. L. Provis, G. C. Lukey, A. Palomo and J. S. J. van Deventer. (2007), "Geopolymer technology: the current state of the art." Journal of Materials Science, Vol. 42, pp. 2917–2933

[18]. Provis, J.L. and S.A. Bernal, Geopolymers and related alkali-activated materials. Annual Review of Materials Research, 2014. 44: p. in press, DOI 10.1146/annurev-matsci-070813- 113515.

[19]. Provis JL, Van Deventer JSJ. (2013), 'Alkali activated materials: state-of-the-art report." Springer Science & Business Media,RILEM TC 224-AAM, vol. 13.

[20]. Ramaswamy Koodalloor Parasuraman and Manu Santhanam. (2017), "Durability of Cementitious Materials in Acidic Environments." Conference: 15th NCB International Seminar on Cement, Concrete and Building Materials.

[21]. Robin E. Beddoe and Horst W Dorner. (2005), "Modelling acid attack on concrete: Part I The essential mechanisms." Cement and Concrete Composites, Vol. 35(12), pp. 2333-2339

[22]. T. Miyamoto, K. Torii, K. Akahane, S. Hayashiguchi. (2015) "Production and use of blast furnace slag aggregate for concrete." Nippon Steel Sumitomo Met Tech Rep, Vol. 109, pp. 102-108

[23]. Tom Damion, Rolands Cepuritis and Piyush Chaunsali. (2022), "Sulphuric acid and citric acid attack of calcium sulphoaluminate-based binders." Cement and Concrete Composites, Vol. 130

[24]. Tyler Salisbury and Jesse White. (2015), "The Advantages of Thermal Analysis Prior to Bench-Scale Roasting." Drying, Roasting, and Calcining of Minerals, DOI:10.1007/978-3-319-48245-3_4, pp.27-34

[25]. Y. Alharbi. (2021), "Engineering properties of alkali activated materials reactive powder concrete." Construction and Building Materials, 10.1016/j.conbuildmat.2020.121550