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State of the Art: Process of Pozzolan Formation from Ash and its Applications

Estado del arte: proceso de formación de puzolanas a partir de cenizas y sus aplicaciones

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Abstract

A brief presentation is made on the problem of fly ash from sugar production, which can be transformed into pozzolan through physical, thermal, chemical or mixed activation, and then used as a chemical corrector and additive for the production of Portland cement. The conversion of the ash into pozzolans is carried out with hydrothermal processes that include repetitive actions of incubation, filtration and drying, for which an alkali solution is used at high temperatures, where compounds such as zeolites are formed from the amorphous aluminosilicates present, being necessary in some cases to adjust the Si/Al ratio to produce the desired type of pozzolan. It is concluded that the use of fly ash, as a chemical corrector in the raw clinker mix, depends on the purity of the limestone and the evaluation of the chemical balance of the mix.

Keywords: *Pozzolan; fly ash; industrial byproducts; sustainability*

Resumen

Se hace una breve presentación sobre el problema de las cenizas volantes provenientes de la producción de azúcar, las cuales se pueden transformar en puzolana por medio de activación física, térmica, química o mixta, para luego ser utilizadas como corrector químico y adición para la producción de cemento Portland. La conversión de las cenizas en puzolanas se lleva a cabo con procesos hidrotérmicos que incluyen acciones repetitivas de incubación, filtración y secado, para lo cual se utiliza una solución álcali a altas temperaturas, en donde se forman compuestos como zeolitas a partir de los aluminosilicatos amorfos presentes, siendo necesario en algunos casos, ajustar la proporción de Si/Al para producir el tipo de puzolana deseada. Se concluye que el uso de cenizas volantes, como corrector químico en la mezcla cruda del clínker, depende de la pureza de la piedra caliza y de la valoración del balance químico de la mezcla.

Palabras clave: Puzolana; ceniza volante; subproductos industriales; sostenibilidad

1. Introduction

In energy cogeneration, different types of organic materials can be used, alone or mixed together, such as municipal solid waste, sewage plant sludge, biomass, bagasse, and coal, and it is necessary to know and adapt the incineration (combustion) technologies to the energy requirements of the fuel (Gallego Ocampo, 2015). In the Colombian sugar industry, it is very common to use the coal-bagasse mixture as a fuel in power boilers, due to the thermodynamic capacity generated (high calorific value) for the production of electrical, mechanical and thermal power (Margallo et al., 2015); (Mohee et al., 2015).

During combustion, large quantities of ash are generated, which are considered to be waste. Therefore, in most cases, the sugar industry disposes of the ash on roads and empty lots, thus wasting the potential of the byproduct as a cementing agent, a substitute for clinker and cement in mortars and concrete, since the ash contains in its structure pozzolanic minerals such as Al, Si, K, Ca, Br, Fe, Mg, Na, Mn with their corresponding oxides (Al_2O_3, SiO_2, SiO_4, K_2O, CaO, Br, Fe_2O_3, MgO, Na_2O, MnO). These react chemically during combustion resulting in the formation of quartz (SiO_2) and corundum (Al_2O_3) at 560°C due to the presence of Ca and S and the fusion of quartz, silica and alumino-silicates at temperatures between 600°C and 800°C. However, anorthite ($CaAl_2Si_2O_8$) is formed at 700°C when CaO reacts with Al_2O_{32} and SiO_2 (Jagadesh and Ramachandramurthy, 2015); (Melissari, 2012); (Van Dyk, 2006); (Zhang et al., 2013a).

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2. Ash

Ash is the final product of the combustion reaction at temperatures between 1200°C and 1700°C, where the fuel can be coal, bagasse, sludge, municipal solid waste and the oxidizing agent can be air and/or gas. During combustion, processes of vaporization, precipitation, nucleation and coalescence take place, as shown in (Figure 1). In this case, the organic and inorganic parts of the fuel undergo chemical and physical transformations at high temperatures (Mejía et al., 2014); (Melissari, 2012).

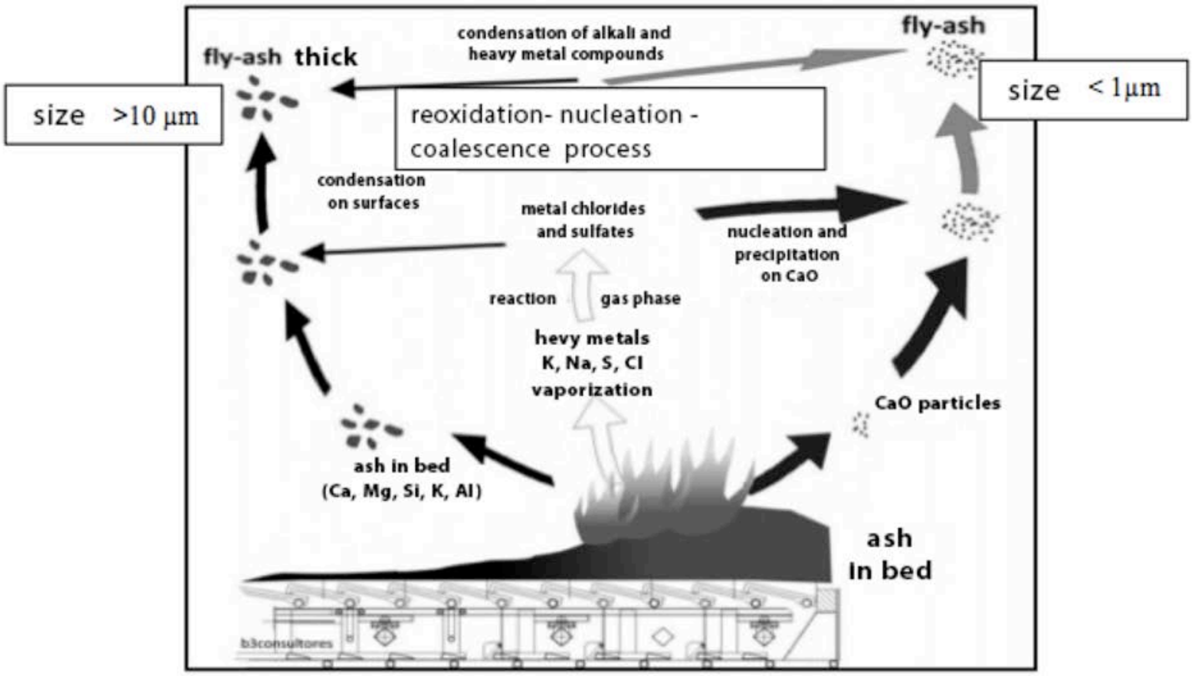

Figure 1. *Ash formation mechanism. Source: (Melissari, 2012)*

The ash may contain alumina and silica compounds including mullite, quartz, hematite and calcite and some inorganic compounds such as lime and sulfates, where, at low calcium contents, crystalline compounds such as quartz and mullite predominate, while at high calcium contents aluminosilicates such as tricalcium aluminate, calcium silicate and tetracalcium aluminosilicate predominate (Fotovat et al., 2009); (Jankowski et al., 2006). On the other hand, if the sugar cane bagasse is calcined at 600°C for 6 hours, ash can be obtained with a weight percentage of 84% SiO_2 in both crystalline and amorphous phases (Paula et al., 2009).

3. Pozzolanic Materials

Pozzolanic materials are characterized by mineral particle packing, where the amorphous silica when in contact with water solubilizes and reacts with the Ca+2 ions present in $Ca(OH)_2$, forming hydrated hydroxide silicates similar to those produced in cement hydration reactions as shown in (Equation 1), where the high silica, alumina and iron oxide content favors the pozzolanic activity of the ash (Abiodun and Jimoh, 2018); (Ribeiro and Morelli, 2014).

$$Ca(OH)_2 + H_4SiO_4 \rightarrow CaH_2SiO_4 \cdot 2H_2O \qquad (1)$$

The pozzolanicity of a siliceous and aluminous compound is a function of the chemical composition, the amorphousness of the phases, and the fineness. These variables depend on the activation temperature (between 600°C and 800°C), the capture method (wet or dry) and the storage method since if the material is captured wet, the treatment process becomes complicated (Ribeiro and Morelli, 2014).

The ash as it leaves the incineration process would have a low pozzolanic activity due to its large particle size. In this case, the ash would act as an inert material or without activity (filler), so it is necessary to submit it to grinding and sieving processes (Kumar and Kumar, 2011); (Margallo et al., 2015); (Park et al., 2016); (Sahoo, 2016;). The average pozzolan particle size can vary between 6 and 13 μm depending on the gradation, distribution and fineness of the cement (Bentz et al., 2011); (Erdoğdu and Türker, 1998); (Givi et al., 2010); (Seraj et al., 2017). Coincidentally, in Colombia, cement is made with a particle size between 8 and 14 μm (Mendoza et al., 2014); (Tobón and Restrepo, 2010).

4. Physicochemical processing for the activation of ash as pozzolan

The physicochemical changes of volume and surface occurring in the ash during size reduction favor the reversible desorption of water, chemical reactivity for ion exchange during mineral transformations, the formation of alkali metal silicates (K, Na) by the fusion and chemical interaction of quartz and silica with other components of the ash. They occur by the fusion of alumino-silicates; the decomposition of carbonates, oxalates, chlorides, and other inorganic salts; the volatilization of alkaline and other heavy metals; the fragmentation of particles by thermal shock and rapid emission of gases from them; the coalescence and agglomeration of mineral particles (Blissett, 2012); (Hela et al., 2013); (Sahoo, 2016).

When the ash is subjected to hydrothermal processes in an alkali solution such as NaOH, Na_2CO_3, $NaNO_3$, $CaCl_2 \cdot 2H_2O$, $Ca(NO_3)_2$ and KOH at high temperatures, it tends to form new compounds such as zeolites from the amorphous aluminosilicates present, which dissolve in the reaction mixture and then group, nucleate and grow as crystals on the surface of the coal ash (because of the presence of trace elements such as TiO_2, MnO, MgO, Na_2O and K_2O, carbonates and sulfates). The degree of dissolution of aluminosilicates determines the conversion percentage and the speed of the pozzolanic formation, which can be affected by chemical factors, such as the amount of calcium hydroxide available for the reaction, nature of the active phase, SiO_2 content in the ash, $Ca(OH)_2$/aluminosilicate ratio, crystallographic composition, alkalinity of the aqueous phase, Si/Al ratio, reaction time and physical factors such as surface area, solid/water ratio, temperature, pressure, heating sources (conventional ovens, autoclaves, microwaves (MW), ultrasound (US), among other sources). In some cases, it is necessary to adjust the Si/Al ratio by adding aluminates or silicates to produce the desired type of pozzolan. The evaluation of the effectiveness of the chemical treatment is determined by activation tests or pozzolanicity indicator tests such as the Frattini test or the mechanical activity index performed for Portland cement (Hollman et al., 1999); (Muraza et al., 2013); (Ribeiro and Morelli, 2014); (Zhang et al., 2013a).

Direct hydrothermal synthesis of pozzolan (incubation-induction period) is performed by mixing 500 g of fly ash in 1.25 dm^3 of a 2 M NaOH solution, followed by heat treatment at 90°C for 96 h. During this time, the Si concentration is stabilized at 6800 mg/dm^3. During the process, a maximum concentration of Si is reached (start of crystallization), while the aluminum reaches a maximum concentration at the beginning of the reaction and then it decreases to zero, one hour after the start of the Si crystallization process. At 400°C and 600°C, the carbon is lost during calcination and at 800°C crystalline silica compounds are formed, causing a decrease in the pozzolanic activity. Therefore, the optimum temperature to generate amorphous silica with a pozzolanic activity index of 77% and an ignition loss of 5.7% is 600°C (Cordeiro et al., 2009); (Feng et al., 2018).

Other hydrothermal treatments include repetitive actions of incubation, filtration and drying (solid residue - crystal formation) at the same mixing and incubation conditions but at different times to adjust the Si/Al molar ratio between 0.8 and 2. The first incubation process is carried out for 6 h, and then it is submitted to the filtration process to separate the solid residue (formed crystals) and adjust the filtration. The solid residue is dried, while the Si/Al molar ratio is adjusted to the filtration. In the second process, the adjusted filtration is incubated for 48 h, while the filtration and drying are carried out again. The new filtration is mixed with the solid residue obtained during the first incubation process and incubated again for 24 h (Behin et al., 2016).

Conventional hydrothermal methods for ash conversion are industrially inefficient due to excessive time and associated costs and low efficiency in the production of zeolites, as well as the amount of water used to neutralize the NaOH solution since, after the activation treatment, it becomes a problem when disposing the liquid into the sewage system. However, the same treatments have been carried out but with zero emissions to the sewage system, reducing water and NaOH consumption between 50% and 90%, by recirculating the NaOH solution along the process (Behin et al., 2016).

The pozzolan formation process (hydrothermal synthesis) with microwaves allows to shorten the crystallization time by the homogeneous heating of the synthesis mixture at atmospheric pressure and to accelerate the nucleation process (fast reaction speed), thus producing pure zeolite crystals with different morphologies, thanks to inductive heating, the specific dissipation of energy in the water molecules present in the surface boundary layer and the immobilization of heavy metal ions and cations such as Ba, Cu, Cr, Mn, Ni, Pb and V *present in the ash, while the same amount of Si and Al is dissolved (Bukhari et al., 2014); (Kim and Lee, 2009); (Muraza et al., 2013); (Tanaka et al., 2008); (Zhang et al., 2013b).*

Also, the ultrasonic treatment carried out at 300 W with alkaline solutions with NaOH concentrations between 1 M and 5 M (at a solid-liquid ratio of 0.5 g/cm^3) at different time intervals between 30 minutes and 2 h improves mass transfer from acoustic cavitation and the collapse of microbubbles generated in the liquid phase, producing a phase change corresponding to the formation of hydroxysodalite. This accelerates chemical processes such as the dissolution of Al/Si, strengthens the bonds of particles at the solid/gel interface (increased crystallization), and accelerates the speed of the secondary nucleation (Andaç et al., 2005); (Belviso et al., 2011); (Hums et al., 2015); (Wang and Zhu, 2005).

5. Uses of Pozzolan

The use of pozzolans in cement factories as a chemical corrector and additive for the production of Portland cement provides significant advantages at both an economic and environmental level because by replacing a portion of the Portland cement with pozzolans, the generation of greenhouse gases produced during the calcination of the limestone (CaO) when producing cement is reduced while operating costs are also reduced (Gorai and Ash, 2018).

5.1 Chemical corrector for the obtaining of Clinker

A chemical corrector is a raw material that exposes a high composition of silica oxide (SiO_2), iron (Fe_2O_3), and aluminum (Al_2O_3). It is added to a mixture of crushed limestone and clay slurry to obtain the clinker, where the chemical composition is a conclusive element in the development of the cementitious properties so that the quality of the clinker depends on the chemical control of the slurry (Enríquez, 2019).

The chemical correctors are added to balance the composition of the clinker components (calcium silicates, tricalcium aluminate, and calcium aluminoferrite), which are generated in the sintering of the clays and limestone. The balance is made when the quantity of the compounds present in the raw mixture is not enough to produce the clinker. Materials called "second raw materials" are used such as clay, shale, sand, iron ore, bauxite, fly ash, and slag (Benzaazoua et al., 2010).

The proportion of fly ash used as a chemical corrector in the raw clinker mixture ranges from 5% to 25%, although it depends on the purity of the limestone and the assessment of chemical balance (Ferrari et al., 2012). The advantages of using industrial byproducts as a chemical corrector are reduced operating costs associated with the primary raw material and improved physicochemical properties of Portland cement (Enríquez, 2019). On the other hand, the requirements for a chemical corrector are low levels of impurities to avoid problems in the manufacture of the clinker, as in the case of magnesium oxide (MgO), which compromises the durability of the cement if its raw material content is greater than 5% (Ahmaruzzaman, 2010); (Blissett, 2012); (Mayor, 2014); (Toro et al., 2014).

5.2 Portland Cement Additive

The industrial byproduct is considered pozzolan if the concentration of calcium hydroxide in solution is less than the saturation concentration. The pozzolanic effect is established by a decrease in the concentration of CaO in the liquid phase since the calcium hydroxide generated through the hydration of the cement is sequestered and combined by the pozzolan (Ribeiro and Morelli, 2014).

Due to their physicochemical characteristics, fly ash is used as an additive for prefabricated and cement clinker because of its high pozzolanic potential (Rougeau, 2004). The addition of bagasse fly ash with pozzolanic reactivity can form composites with cementitious properties that meet the standards given in Colombian NTC 3493 and US ASTM C618 standards (ASTM, 2005); (Icontec, 2017). Although care must be taken when using bagasse ash as an additive, since an excess of organic material produces unburned products that contain alkaline sulfates, which react with tricalcium aluminate, forming ethtringite. This is an expansive substance that affects the mechanical properties and durability of the composite (Valencia et al., 2018). Therefore, there are limitations in the use of pozzolans from bagasse ash, in the case of the percentage of coal. This mineral is the remainder of the result of the mixture of mineral coal and bagasse, which is known in the industry as "unburned products". For this reason, it is recommended that the mineral be less than 5% since it forms components that negatively influence the durability and properties of hydraulic concrete (Camilo and Gutiérrez, 2006); (Ribeiro and Morelli, 2014).

The substitution of CaO and OH- ions by ash from bagasse generates a pozzolanic reactivity independent of the temperature of ash calcination (Onésippe et al., 2010); (Ribeiro and Morelli, 2014). In a concrete mix, it is feasible to replace between 10% and 50% of the ordinary Portland cement with pozzolanic bagasse ash, which can increase the durability of the concrete, especially the resistance to chloride penetration (Nunes, 2009); (Paula *et al., 2009); (Rerkpiboon et al., 2015); (Xu et al., 2018). However, the substitution of bagasse ash in the 15% - 20% range by Portland cement produces the best performance in compressive strength tests (Pereira et al., 2018).*

6. Conclusions

The high content of silica, alumina and iron oxide favors the pozzolan activity of the ash, so its reactivity depends on the chemical composition, phase amorphousness and fineness, as well as the activation temperature. It is necessary to establish the hydrothermal synthesis method that best suits the chemical requirements for the activation of the ash as pozzolans.

To obtain ash with a SiO_2 content of 84% (weight percentage), both in the crystalline and the amorphous phases, a thermal activation process must be carried out at calcination temperatures around 600°C for 6 hours (hydrothermal synthesis). The degree of dissolution of the aluminosilicates in the ash determines the conversion rate and the speed of the formation of pozzolan.

Conventional hydrothermal methods for the transformation of ash into pozzolans are industrially ineffective. On the contrary, modern pozzolan-forming processes, such as hydrothermal synthesis with microwaves and ultrasound, allow the efficient transformation of the byproduct into pozzolans.

The use of fly ash from sugar cane bagasse as a chemical corrector in the raw clinker mix will depend on the purity of limestone and the assessment of the chemical balance, so up to 20% of the Portland cement can be replaced in a concrete mix and the compressive strength can be improved.

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