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Fly ash morphology and surface modification via mechanical activation: A review

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Abstract

Fly ash (FA) F low reactivity, slow hydration reaction and low early strength, restricted its industrial usage to less than 25 wt %. Ash properties were modified by mechanical activation to achieve higher added value product. The activation depends on the equipment type and their particle size range of milling. This paper reviewed the milling equipment effect on particle size, surface properties, and chemical compositions of activated ash. Increasing in the surface area, pozzolana activity and the reduction of crystalline dense layers of fly ash F, leading to microstructure and structural variations which raised the ash industrial applications.

Key Words: *Fly Ash, F Surface Properties, Mechanical activation, Milling equipment, Pozzolanic activity*

Introduction

Sustainable ash management requires the converting of waste materials as new raw materials for other industries. Silicate or alumino-silicate based raw materials found in fly ash released from thermal power plant are produced in enormously large amounts worldwide, and its disposal landfill implies environmental and social problems. However, the requirements of these kinds of materials are extraordinarily heterogeneous. Therefore, it is imperative to control and adjust their characteristics, to enhance the overall performance. The amendment can be reinforced by usage of few approaches such as mechanical, thermal or chemical activation. Coal fly ash (CFA) dense glassy surface layer, chemically stable, protects the active inner constituents, which generates low reactivity particle surface. Ash comprises of porous, spongy and amorphous particles. The reliability of the FA can crumble to encourage its chemical activity if the glassy chain, which incorporates Si, AL and low carbon, is exposed to activation methods. Activation methods are utilised to control and alters the ash characteristic for specific persistence in some fields. FA applications are often expanding if the ash has larger specific area and finer particle (Babel, 2003). One of these methods is the grinding mechanical activation (fig. 5), which enhances the reactivity of fly ash through three approaches: a) Mechanical dispersion (size reduction), Figure 1, where the advanced reactivity is the end result of the higher surface area exposure, b) Surface activation, the place that mechano-chemical reactions appear on the particle surface, and, c) Mechano-chemical activation, internal components of the particle radically change to different structures (Gabor, 2016).

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Activation of Fly Ash by Milling

The main milling equipment accustomed to grind ash into smaller particle size are; rod milling, ball milling, vibration milling, and electromagnetic mill as mentioned in Table 1. Ball milling consists of a rotating hollow cylinder, partially filled with balls. The material (to be ground), is fed in via a hole trunnion at one point and an equal trunnion at the opposite end, to enable the product to go through it. The outlet is typically sheltered with a rough shade to rest the break of the balls. It is frequently loaded with particulates 30%–40% of its size (Coulson and Richardson, 2002). Rod mills are comparable to ball mills, with log rods for grinding media, it received feed up to about 50 mm (Brook, 1991) Figure 2. Vibration mill is occupied to 80% of its capacity with porcelain or stainless-steel balls. During milling, the whole physique of the mill undergoes a very low and frequent vibration, which is generated through an eccentric motor and dimension discount takes

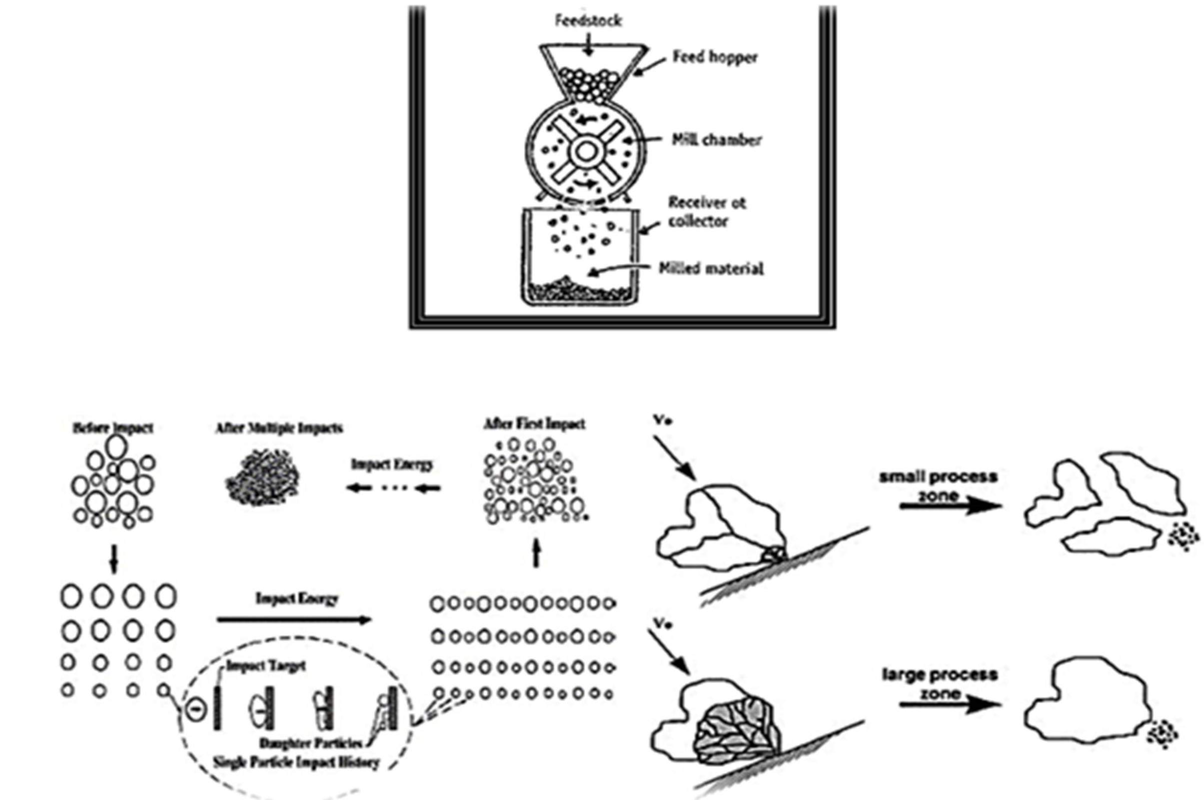

Figure 1. Milling Size Reduction Mechanism - (Sushant and Archana, 2013).

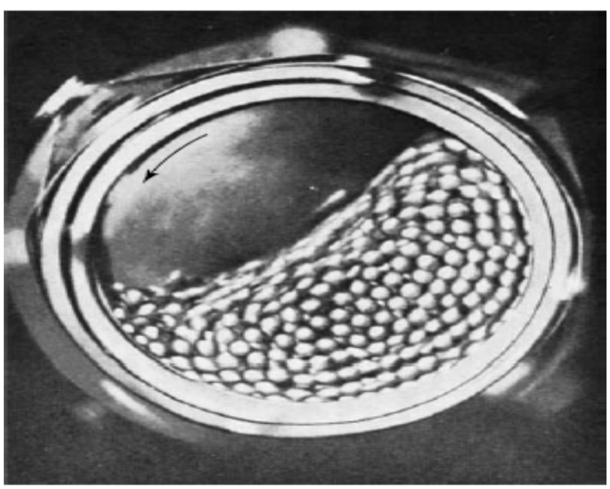

Figure 2. Ball Mill (Coulson and Richardson, 2002).

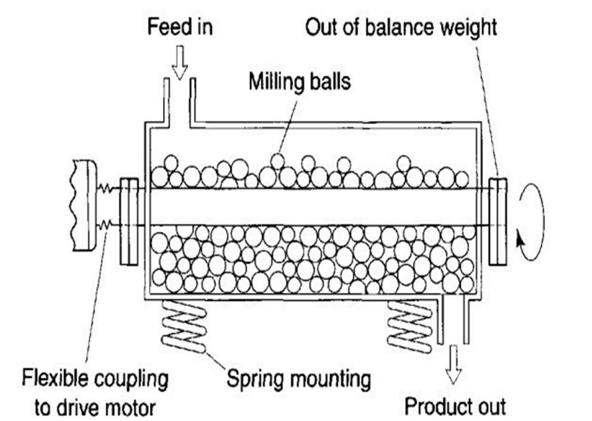

Figure 3. Eccentric Vibratory Mill (Brook, 1991).

place via repeated impact. Vibration mills (Figure, 3) are like ball mills, the particles are crumpled between porcelain or metallic balls in the mill body (Brook, 1991; Wołosiewicz *et al.,* 2015). Grinding technique (Figure, 4) in the electromagnetic mill is carried out by the way of the ferromagnetic grinding media (small rods), which cross in rotating electromagnetic field (Xue *et al.,* 2004). Milling or grinding reduces the size of the ash by transforming large, spherical particles into smaller, irregularly shaped particles, which subsequently negatively affects rheology. Grinding consumes more energy to obtain fine particle size distribution, and there are size limitations. The advantage of grinding is that the entire grinding volume only contains the defined particle size distribution, not the fine and coarse particle size distributions. For particles of 50µm, the energy required to grind the fabric to the specified size follows the Von Riddinger grinding law (Equation 1), while the particle size greater than 50µm follows the Bond grinds law (Equation 2):

For the particle size< 50 µm,

$$W_R=C_R/d_E-1/d_A \quad (1)$$

For the particle size > 50 µm,

$$W_B=C_B/d_E-1/d_A \quad (2)$$

Where,

W_R, W_B: Grinding work (kJ/kg).

C_R, C_B: Grinding coefficient

d_E, d_A : Sizes of the ground material

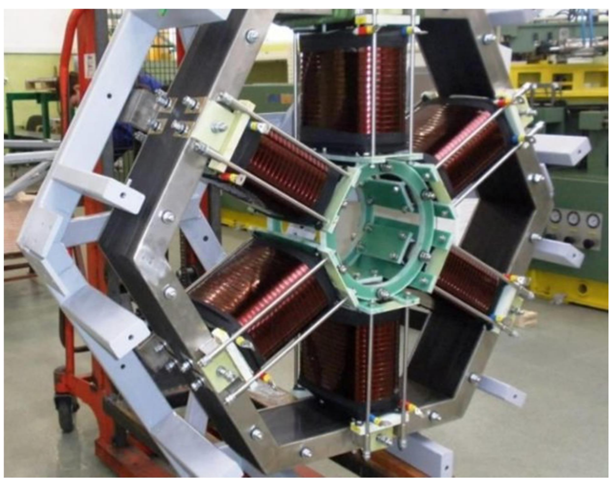

Figure 4. Electromagnetic Mill – (Wolosiewicz-Glab *et al.,* 2016).

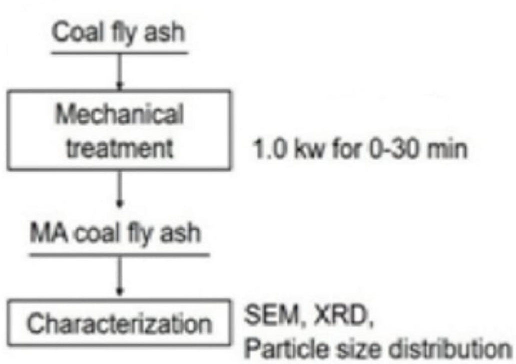

Figure 5. Flow Chart of the Mechanical Activation Methodology (Matsuoka *et al.,* 2019)

Milling changes many physicochemical properties of fly ash. The structure of the material becomes disordered. That creates surface defects. In other words, metastable forms can be found (Palomo *et al.,* 1999). This lead to the change in the structure and surface properties, depending on the activation procedure and application needed. It will affect the dispersibility of the particle size distribution (PSD), specific gravity (SG) and specific surface area (SSA), morphology and mineralogical composition (Gabor, 2016). Milling mechanical activation is designed for the destruction of the long-range order intermetallic phase and creation of either a disordered intermetallic or an amorphous phase. In addition, the mechanical activation improves the pozzolanic activity of fly ash (Bouzoubaa *et al.,* 1997; Varma *et al.,* 2013; Palomo *et al.,* 1999; Paya *et al.,* 1995).

This paper reviews the surface changes of Class F fly ash, when mechanically activated, in particular the specific surface, specific density, mineralogical phases and reactivity properties. Different mechanical activation ways are used to control fly ash characteristics and its application.

Materials and Methods

Class F Chemical Composition

Fly ash (FA) particles are generally spherical, with a size ranging from 0.5 µm to 100 µm. Class F ash contains more than 70% of SiO_2, Al_2O_3 and Fe_2O_3 and less than 10 % CaO. FA shows an existence of amorphous glass, mullite, quartz (crystalline silica), hematite and small amounts of lime. The intensity of quartz with mullite forms a chemically stable and dense layer (Gabor, 2016).

Fly F silicate, or silicate -aluminate (Pozzolana Material) bosses a small or absent self-hardening, which reduces its utilization to remarkable limit (Rosenberg, 2020). The hydrated silicate in fly ash develops the strength while the lime fills the voids, by forming CSH, give the mixture the same consolidation properties as Portland cement. The reaction of fly ash and lime in concrete increases the strength .But by adding the original F-class flight ash to 15% to 25% in concrete (Varma *et al.,* 2013), decreasing the cements activity was observed (Varma *et al.,* 2013; Sanytsky *et al.,* 2018) due to the low quantity of CaO. The pozzolanic activity of fly ash increases the compressive strength in future stages (Patil and Anandhan, 2015), but low strength was observed at the early curing time. The microstructure of fly ash F before one year on curing exhibits a copious amount of un-hydrated spherical fly ash particles (Nalbantoglu, 2004). To solve the Class F drawback in order to achieve sufficient strength throughout the total stages of curing, it is appropriate to mend the ash surface and reactivity by applying activated methods (Akinrotimi *et al*., 2015).

Experimental Methodology

Mechanical activation changes the physical and chemical properties (particle size distribution, specific surface area (SSA), crystal phase composition). The particle size (PS) distribution can be determined by dry or wet sieving, sedimentation or laser scattering methods (Gabor, 2016). Specific surface area (SSA) of solids is determined by the BET (Brunauer, Emmett and Teller), which is commonly used to evaluate the gas adsorption data and generate a specific surface area result expressed in units of area per mass, (Gabor, 2016). Chemical composition of FA can be determined by the Energy Dispersed X-ray analysis (EDX) (Akinrotimi *et al*., 2015, 2015; Saha, 2018; Paul *et al.,* 2007). The structure of the material can be predicted by Fourier Transform Infrared Spectroscopy (FTIR) in transmission mode. X-ray diffraction (XRD) technology is used for mineral phase analysis. The morphology and particle shape of the material can be studied by optical or scanning electron microscope (SEM) or transmission electron microscope (TEM) (Gabor, 2016).

Results and Discussion

The paper reviewed the effect of mechanical activation applying types of equipment. Many parameters within a lot of industrial applications had been reviewed in Table 1. The necessary per treatments and the studied factors helps the researcher in this field to have summarized view about the mechanical activation research and their changes in fly F properties.

Changes in particle size and Specific density

The milling process can be divided into three phases. The first phase is the initial grinding phase, where the increase in fineness is very obvious, and the time is 0 to 30 minutes. The second phase is the development and grinding phase, in which the increase in fineness slows down, and the time is about 30 minutes to 50 minutes. The third phase is the delay time, which is about 50 minutes to 70 minutes, in which the fineness no longer increases or sometimes decreases a little. This is because the particles are so fine that they cluster together under the action of electrostatic forces (Nalbantoglu, 2004). These results are compatible with the remarks in Table 2 (Helaand Orsakova*.,* 2013; Patil and Anandhan, 2012; Kumar *et al.,* 2007; Hela and Bodnarova, 2013). It should be considered that the product of the mechanical activation (SSA, PS) depends on the type of activation device used. Akinrotimi *et al*., (2015) point out that simple grinding equipment (Rod Mill) can only effectively grind rough materials, but not for fine materials less than 40μm. Sanytsky *et al.* (2013) used the electromagnetic mill to obtain ultrafine fly ash contents ≤ 20 μm (more than 90%). Large amount of particles smaller than 1 μm , consisting of spherical silica-alumina grains which contain large amounts of alkali metals (Patil and Anandhan, 2012).

During the grinding process, the increase in specific gravity was recorded. Obviously, 50 minutes of grinding is a critical point, which means that the specific gravity will increase rapidly before this point, and the specific gravity will increase more moderately after this point. However, the specific gravity eased after 50 minutes of grinding. That is because the fineness of FA is reduced at this stage (Palomo *et al.,* 1999). Fineness enhanced the specific gravity rise and this elaborated the increase in cement mortar density when 25 percentages of cement were replaced by milled fly ash, Shows that the upgrade of the particle filler of the mortar combines a finer particle size (Hela and Bodnarova, 2013). For concrete application, the slump increased when milled fly ash added to the concrete admixture .The higher the slump, the higher workability of concrete, (Kumar *et al.,* 2007). Researchers have found that 50-60 wt% of activated fly ash can be used to replace activated fly ash with higher strength or equivalent to commercially available cement (Subhash *et al.,* 2014). For environmental protection considerations, activated F fly ash can be used substitute for cement in concrete because it improves workability,

Table 1. Parameters affected the Activated Fly Applications.

No	References	Milling Machine	Pre Treatment	Parameters studied	Affected Application
1	Akinrotimi *et al.*, (2015)	Rod Mill	--------	Milling time- SSA ,SG and comprehensive strength	Mortars
2	Hela and Bodnarova (2013)	Ball Mill	0.9wt% acrylic plasticizer to reach require conistency	Milling time- surface area- Workability- Comprehensive strength	Concrete
3	Sharma *et al.* (2015)	Ball Mill	Washed with distilled water then dryed at 100 °C for 24 hr.	Milling time - chemical compositions –crystalline phase	solid acid catalyst and catalytic support materials
4	Patil and Anandhan (2012)	Ball Mill	Washed with distilled water then dryed at 100 °C for 24 hr, then a magnetic separation carried out.	Milling Time-Crystal size- Morphology	Nanoparticles
5	Kumar and Kumar (2011)	Eccentric Vibratory Mill	(6 M)sodium hydroxide ((98% purity) used as alkaline activator	Milling time -surface area, Geopolymeration reaction	Geopolymer
6	Sanytsky *et al.* (2013)	Electromagnetic mill	Polycarboxylate type superplasticizer was included in cementitious systems as modifier	Milling time-Pozzolana reactivity- Workability- Comprehensive strength	Ultrafine fly ash in concrete

pumpability, cohesion, surface finish, ultimate strength and durability. For geopolymers, the fineness of fly ash plays an important role in improving its performance. The higher workability, the higher strength in early duration of geopolymerization reaction (Patankar *et al.,* 2012; Hela and Bodnarova, 2013).

Change in Specific Area and Reactivity

Rudolf Hela and Denisa remarked that the grinding in Ball mill for Class F fly ash increased the specific surface area with increasing milling time (Sharma *et al.,* 2015) Other authors represent the similar outcomes, using BET model, where the variation in BET of fly ash with different milling time increased marginally (Sharma *et al.,* 2015; Masuda, 2007; Kumar and Kumar, 2011). As mentioned above, the effect of milling device in SSA is shown in Table 2. SSA increase using ball mill, eccentric vibratory mill and electromagnetic mill in succession. Attrition mill had an effect on the increase the fly reactivity by 11% greater than using a vibratory mill in the geoplimeraization application (Hela and Bodnarova, 2013; Kumar *et al.,* 2007). Gabor, (2016) considering the particle shape of F fly ash. Compared with fresh fly ash with mostly spherical particles, the surface of the particles is more uneven and rough, with irregular shapes. This leads to an increasing of dislocations, the disclosure of the active surface and subsequently the surface reactivity (Patil and Anandhan, 2012; Kumar and Kumar, 2011). Patil and Anandhan (2012) found that milling of fly ash affects the surface properties and geopolymer reactivity. Heat evaluation peak as an indicator for an attribution to geopolymerisation shifted to low intensity, which indicates an increase of fly F reactivity and that the geopolymerisation reaction can be applied at ambient temperature using mechanically activated fly ash.

Changes in Mineralogical phases

The general characteristic mineralogical phases in fly ash are quartz, mullite, lime and hematite, (Gabor, 2016). During the process of mechanical activation, the energy influences directly

Table 2. Main results and Improvements of Mechanical Activated Fly Ash F.

No	References	Parameters / Time	Noted Results	General Observations /Remarks
1	Akinrotimi *et al*., (2015)	0 and 8 hr	● 31% reduction in PS for coarse particles. ● 20% increase in strength	● Specific gravity increases, with reduction of PS ● HVFA gained higher Strength
2	Hela and Bodnarova (2013)	0, 20, 45 and 75min 40, 60 min	● SSA increased by 25%,60%,88% 113%,140%. ● 40,60min milling gave the highest workability.	● 25% fly ash replaced cement ● Higher concrete slump . ● Higher comprehensive strength initiated in 40min for ninity days
3	Sharma *et al.* (2015)	5 hrs 10 hrs 15 hrs	● 6 % increase in silica oxide ● 5% decrease in Alumina oxide ● 26.8 % increase in SSA	● Decreases crystal size and the crystallinity of the fly ash. ● increase in, amorphous nature, specific surface area and surface roughness
4	Patil and Anandhan (2012)	0,10,20,30,40, 50 and 60 min	● 75% reduction in crystal size.	● Peak intensity increased with as an evidence of breaking down in quartz structure
5	Kumar and Kumar (2011)	0,5, 10, 20, 30, 45, 60 and 90 min	Increase in SSA :48.5%,55%,75.6%,139%,141%,165% Geopolymerisation reaction started after 32 hrs, for 45 min milling samples	● 40 to 60 wt% of the total cementitious materials. ● The heat evaluation peak shifted towards lower time and its intensity increased indicating increased reactivity of fly ash .
6	Sanytsky *et al.* (2013)	0, 5, 10 and 15 min	Interaction with $Ca(OH)_2$ increased by 31.8% . ● 45.5 % increase in strength for 90 days. ● 6% increase in workability.	-20% is the optimum cement replacement The interaction of ultrafine fly ash with $Ca(OH)_2$ Increases. -Increasing the active surface in 2-3 times.

the crystal and molecular substructure. Reduction in crystalline phases was remarked by an increase in silica percentage of crushed fly ash, which enriched the present of amorphous phase (Gabor, 2016).

Paul *et al.* (2007) found that, the crystalline of ash F ground for 60 hours is significantly reduced (from 35% to 16%) (Gabor, 2016).Paul outcomes showed a destruction in quartz and hematite crystals phase. These remarks also agreed with Patil and Anandhan (2012) which showed a reduction in crystalline phases with milling time increase. Sharma *et al.* (2015) represented the coexistence of amorphous components after mechanical activation. Crystallite size and crystalline content decreased through the drop in quartz, mullite and iron oxide amounts .and an increase in amorphous phase occurred. The higher the amorphous phases, the higher fly ash reactivity was seen (Sharma *et al.,* 2015). The important IR bands mentioned in Table 3, in the normal range of silicon, oxygen and aluminium band .Which changed due to the milling of Fly F ash.

Table 3. Important IR Bands, (Patil and Anandhan, 2012)

Band (cm^{-1})	Band Assignment
3700-3400	OH stretching of Si-OH group
1643	OH stretching
750-800	Si-O-Si symmetric stretching
550-600	Si-O-Al stretching
485	Si-O-Fe stretching

Patil and Anandhan (2012) showed that the peak at 1092 cm^{-1} has broadened in min using milled FA compared to fresh FA, which is Si-O-Si stretching (Saha, 2018). The peak intensity used to be increased with rising milling time as a proof for the breaking down of the quartz shape and formation of Si–OH group (Antoni and Hardjito, 2015). XRD patterns indicated no alternate in the mineralogy and peak intensities in the samples milled up to 60 min, however, a mild minimize in peak intensity and broadening of quartz and mullite was once observed in ninety min milled fly ash (Kumar and Kumar, 2011; Patil and Anandhan, 2012). The milling consequences confirmed adjustments in the peak of IR intensity corresponding to Si–O–Si bending (460 cm-1), T–O–Si (T = Si, Al) and asymmetric stretching (913, 1090and 1160 cm-1). The Si–O–Si symmetric stretching band found at 798 cm^{-1}, which was once almost absent in the untreated ash, seemed after the forty five and 60 min. Their XRD outcomes and IR spectra indicated that the impact of milling on fly ash extends beyond new surface creation due to particle breakage and it undergoes structural adjustments via mechanical activation (Sharma *et al.,* 2015). Patil and Anandhan (2015) determined that Si–O–Si symmetric stretching band located at 798 cm^{-1} of IR intensity in the milled fly ash samples. This band was once absent in unavtivated fly ash and commenced to show up after the milling from 5 to 60 min, which verifies the amorphous segment existence. Kumar and Kumar (2011) indicated that the milling of fly ash harms the crystalline shape which regarded in the minimization of quartz and mullite after milling to ninety min. Patil and Anandhan (2012) investigated the pozzolan activity of fly ash as a characteristic of the extent of bounded $Ca(OH)_2$ with fly admixture after 30 days. There was once a formation of calcium silicates and aluminosilicates (C-S-H and C-S-A-H phases) with a low CaO/SiO_2 ratio in the course of the response process. The interaction between fly ash and $Ca(OH)_2$, used to be grown as a characterization of higher reactivity. Mechanical activation results in slight increase the silica percentage, indicating the amorphous nature. The proportion of alumina reduces and the share of silica improved.Transformation of quartz phases into a glassy phase is faster in MA (Sharma *et al.,* 2015). This means that the mechanical activation extended to create new surfaces due to particle breakage. So the utilization of fly F in cement geotechnical and Nano solid application will improve due to the amorphous nature, pozzolanic phases that approves to be appeared after milling (Gabor, 2016; Hela and Orsakova, 2013).

Conclusion

In this paper, mechanical activation of fly F ash through milling was reviewed. Four milling equipment, Rod, Ball, Eccentric Vibratory and electromagnetic mills can be used to reduce the particle size from coarse to ultra-fine size. The milling time was shorten when higher energy mill was used/applied. Milling or grinding process increases the fly ash reactivity and pozzolanic action. The importance of higher amount of fly F to be utilized in cement replacement, geopolymer and Nano particles applications. The drawback of fly ash F, particularly low strength in early concrete admixture, and low reaction rate in geopolymer had been resolved. In spite that the effect of milling alone did not have sufficient research studies in the adsorption process, since its applied with other activation methods like acidification and alkalization .Modification of fly ash surface properties and morphology, can enhance the application of fly ash to be a valuable commercial products.

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