



# Milling of peat-wood fly ash: Effect on water demand of mortar and rheology of cement paste

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## HIGHLIGHTS

- Milling of fluidized bed combustion fly ashes was studied.
- Effect on water requirement of mortar and rheology of paste was examined.
- Fly ash particle morphology is the main reason for high water requirement.
- Tapped density correlates with water requirement and rheology.

## ARTICLE INFO

### Article history:

Received 7 December 2017

Received in revised form 28 April 2018

Accepted 3 May 2018

### Keywords:

Fluidized bed combustion

Rheology

Morphology

Viscosity

Correlation

Packing

Biomass ash

Water requirement

## ABSTRACT

The milling of fluidized bed combustion fly ashes is a promising method to improve ashes' properties as a cement replacement material. Two fly ashes from the co-combustion of peat and wood, as well as inert sand were milled at varying times. The physical properties of materials, water demand of mortar and rheology of cement paste were studied. At 25% cement replacement rate, the milling decreased the water demand of mortar by 10% and the yield stress of cement paste by 33%. It was found that milling disintegrated irregularly shaped particles, which were the main reason for high water demand of ashes, and tapped density could be used as a simple parameter to estimate the water demand for all studied materials.

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## 1. Introduction

To implement the principles of circular economy, industrial residues should be used as secondary resources instead of landfilling them as waste. Fluidized bed combustion fly ash (FBCFA) is one of those residues that not have established practices for utilization. One way to utilize FBCFA is to use it as a partial cement replacement material. The utilization of FBCFA has been demonstrated in several studies [1–7], and the results are promising. One challenge related to the utilization of FBCFA is that partial cement replacement is reported to increase the water demand [1,2] of the materials in their fresh state, unlike fly ash (FA) from pulverized coal combustion, which has the opposite effect [1,8]. An

increased water-to-binder ratio decreases the durability and compressive strength of concrete, but it may also be problematic in other applications where FBCFAs could be used, for example, in alkali-activated cements and materials whose strength rely solely on the hydration of the ash.

The increased water demand, which can be related to different supplementary cementitious materials, has been associated with the following material characteristics: irregular particle shape [9,10], high porosity [1], high specific surface area [2,3,9], high volume [2], high carbon content [1,4,11], high free CaO [2], high content of soluble salts [12] and other chemical factors [11]. Several of these characteristics are typically found in FBCFA. In addition, the packing density of the mixture is known to have an effect on water demand [10,13,14].

Milling of FBCFA can be done to modify the physical and mineralogical properties of FBCFA, and it has been the subject of several

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previous studies, e.g. [5,6,9,15–19]. However, most of these studies are done for fly ashes originating from combustion of coal and only one study [15] focuses on fly ashes which are from fluidized bed combustion of peat and biomass. The effect of milling on water demand of fly ash from fluidized bed combustion is studied only in few articles [6,9,15,16] which are mostly focused on self-hardening applications. Effect of milling of fluidized bed combustion on water demand of mortar samples, where part of cement is replaced by fly ash, is examined in two studies [6,9] which both indicated that milling of fly ash lowered the water demand. On the other hand, study of [15] showed that milling of FBCFA from combustion of peat and wood can have different effects on water demand of self-hardening samples depending on milling method and parameters. There have been also conflicting observation how milling affects to specific surface area [5,15] and bulk densities [5,16], which are both associated to water demand.

It is still unclear how well results from previous studies apply to fly ashes originating from fluidized bed combustion of peat and wood, since almost all previous studies regarding the milling of fly ash from fluidized bed combustion are done for ashes originating from coal combustion plants. These studied coal fly ashes have been substantially higher loss on ignition (LOI), which indicates to high content of unburned carbon, as well as higher content of SO<sub>3</sub> and CaO due to de-sulfurization with limestone injection [6,9]. According to Deschamps [17] the amount of added limestone can be 30–50 % from the mass of fuel. On the contrary, typically in the fluidized bed combustion of peat and wood there is no need to limestone injection. Therefore FBC of peat and wood can produce fly ashes, which have low contents of SO<sub>3</sub> and free-CaO [18]. In addition, LOI values of peat and wood FBCFA is typically low. It is likely that fluidized bed combustion of coal, together with massive amount of injected limestone, produces fly ash which chemical and physical characteristics, like morphology, differs from fly ashes of fluidized bed combustion of peat and wood. Thus, these ashes may behave differently when milled and used as a cement replacement materials. For these reasons, there is still a need for comprehensive study of the effect of milling on the water demand of mortar containing peat-wood fly ash from FBC and, to clarify how much and in what extend the physical properties effect on water demand. Therefore, for this study two FBCFA having negligible chemical effect to the water demand was chosen. These fly ashes were originated from co-combustion of wood and peat and they were milled at different times using a tumbling ball mill and then characterized using various techniques, i.e. particle size distribution, specific surface area, packing density and particle shape. Milled sand was used as a reference material being inert in nature and having angular particle shape. The water demand of mortars containing 25% of the original and milled cement replacement materials was measured using the flow table method and was then compared with a rheological analysis of the paste samples.

## 2. Materials

Two FAs from wood and peat combustion were retrieved from different CFB boilers; they were not exposed to humid conditions before the experiments. The burning temperature of FA1 is around 790 °C and FA2 around 890 °C. The original untreated FAs are referred to as FA1 and FA2. The sand used as a cement replacement material was milled CEN-Standard sand (CEN-Standard, Normensand GmbH) according to standard SFS-196-1:2016 [19]. The cement used in the current study was type CEM I 52, 5 R- SR5 (Valkosementti, Finnsementti) according to standard SFS-EN 197-1 [20]. Aggregate sand used in this study was CEN-Standard sand (CEN-Standard, Normensand GmbH).

## 3. Methods

### 3.1. Milling of materials

A laboratory-size tumbling ball mill (Germatec) was used to mill FAs and sand into different particle sizes. The volume of the milling chamber was 10 L. Milling media consisted of 180 steel balls with a diameter of 30 mm. The batch size of the milled material was constant: 1.7 kg. The rotation speed of the milling chamber was 84 rpm. FA1 was milled for 30, 60, and 90 min, and the milled samples are referred to as FA1-30, FA1-60, and FA1-90, respectively. FA2 was milled using the same milling times, and the samples are referred to as FA2-30, FA2-60, and FA2-90. The sand was milled for 60, 120, and 180 min, and these samples are referred to as sand-60, sand-120, and sand-180, respectively. Longer milling times, compared to FAs, were chosen for sand, since it had larger original particle size than FAs.

### 3.2. Characterization of the materials

Particle morphology was investigated using a field emission scanning electron microscope (FESEM) (ULTRA PLUS, Zeiss). Samples were sputter coated with platinum, and imaging was done using 5 kV voltage. The particle size distribution (PSD) of different replacement materials were determined using a laser diffraction particle size analyzer (LS 13 320, Beckman Coulter). The particle size distribution of unmilled sand was analysed by dry sieving. The measurements were carried out on wet ashes with 2-propanol to prevent the reactions of materials during the measurement. Data were analyzed using the Fraunhofer optical model. The span of the PSD was calculated from volumetric particle size distributions using the following equation:

$$\text{Span of particle size distribution} = \frac{D_{90} - D_{10}}{D_{50}} \quad (1)$$

where  $D_{90}$  is the diameter at which 90% of the sample's volume is composed of particles that have a smaller diameter than this value.  $D_{50}$  is the diameter at which 50% of the sample's volume is composed of particles that have a diameter less than this value, and  $D_{10}$  is the diameter at which 10% of the sample's volume is composed of particles that have a diameter less than this value. The specific surface area of materials was measured using an accelerated surface area and porosimetry system (ASAP 2020 Plus physisorption, Micrometrics). Measurements were based on the physical adsorption of N<sub>2</sub> gas on a solid surface, and results were reported as Brunauer-Emmett-Teller (BET) isotherm. The tapped density of the materials was determined by measuring 100 ml of powder in a graduated cylinder, which was followed by tapping using a mechanical tapping machine. The volume of the sample was recorded, and the tapped density was calculated based on the sample weight and volume after 2500 tapping cycles. Tapping density instead of loose bulk density was chosen since it provided more reproducible data. The chemical composition of the cement replacement materials was analyzed using an X-ray fluorescence method (XRF). The analysis was done for the melt-fused tablets using a wavelength dispersive XRF spectrometer (AxiosmAX, PANanalytical). The free CaO content was determined using a method described in standard SFS-EN 451-1:2017 [21]. Loss on ignition (LOI) at 950 °C was measured by thermogravimetric analysis using automatic drying and ashing system (prepASH, Precisa Gravimetrics AG). A diffractometer (SmartLab, Rigaku) was used to identify crystalline phases in FAs and cement. In order to analyze the composition of crystalline phases, samples (2.7 g) were ground 4 min together with TiO (rutile) (0.3 g), which served as internal standard for Rietveld refinement. The Rietveld refinement was done using diffractometer's software. The step width, scan speed, and angle

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