Peat-wood fly ash as cold-region supplementary cementitious material: Air content and freeze-thaw resistance of air-entrained mortars

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Abstract

Fluidized bed combustion fly ash (FBCFA) is a promising industrial side stream to be used as a partial cement replacement material. Untreated and milled FBCFAs from co-combustion of peat and wood were used to replace 20% of Portland cement in air-entrained and non-air-entrained mortars. Additionally, equivalent mortars containing fly ash from pulverized coal combustion (CFA) were prepared to compare FBCFAs to more conventional, standardized cement replacement material. The study found that both FBCFAs produced mortars with similar compressive strengths compared to a reference, indicating that milling did not affect reactivity of ashes. Air-entrained FBCFA-containing mortars had about the same amount of entrained air compared to the reference mortar. FBCFAs outperformed

CFA as a cement replacement material, which produced lower compressive strengths and reduced the amount of entrained air. Non-air-entrained mortar containing CFA suffered severe damage during the freeze-thaw (F-T) experiment, unlike non-air-entrained mortars containing untreated or milled FBCFA. The addition of an air-entrainment agent improved F-T resistance of all mortars, except those that contained milled FBCFA, which nevertheless had good F-T resistance. This first-of-its-kind investigation of the suitability of peat-wood FBCFAs as a supplementary cementitious material in air-entrained mortars suggests a potential use of FBCFAs in cold region concreting.

Keywords: sustainable concrete, frost resistance, frost damage, biomass ash, grinding, air content

Introduction

During recent decades, fluidized bed combustion (FBC) has gained popularity around the world due to its suitability for various fuels that may have fluctuations in quality, such as biomass, peat, municipal waste, and low rank coal. Compared to pulverized combustion, FBC can produce less NOx due to lower combustion temperature, and SOx emissions can be mitigated by injecting limestone into a boiler, which adsorbs sulfur compounds. The current challenge of FBC is that it produces fly ashes with variable quality, no standardization, and unestablished utilization. One potential way to utilize high volumes of FBC fly ash is to use it as supplementary cementitious material (SCM), which has already shown promising results (Rajamma et al. 2015; Rissanen et al. 2017; Sata et al. 2007; Sheng et al. 2007; Wang and Song 2016; Zhao et al. 2015).

In cold climates, concrete is often exposed to recurring freezing. This damages the concrete because during the freezing process, water expands and causes internal stress to the material. This stress eventually leads to deterioration of the concrete if internal stress exceeds the strength of the material. Frost damage of concrete can be avoided if concrete can be kept dry, but in practice, this is often impossible. Concrete's resistance against frost damage can be improved by using air-entrainment agents (AEAs), which are surface-active chemicals. AEAs induce small and well-dispersed bubbles into fresh concrete, and these bubbles remain air-filled during the curing. Typical air content for freeze-resistant concrete is 5–6% (Hewlett 2003) while air content for air-entrained mortar mortar is around 8–21% ("ASTM C91-05, Standard Specification for Masonry Cement" 2005; Dransfield 2003; Hewlett 2003). In hardened concrete, pores formed from bubbles protect concrete by reducing internal stress caused by freezing water. The basis for this phenomenon is that air in these pores contracts as temperature decreases, thereby relieving the stress caused by freezing water. Additionally, part of the freezing water can escape from capillary pores into these air voids where it cannot cause damage.

It is well known that conventional fly ashes originating from pulverized coal combustion (PCC) can interfere with the performance of AEAs, because they often contain unburned carbon. This carbon can absorb molecules in AEA, reducing the amount of effective AEA molecules (Gao et al. 1997; Hill et al. 1997). Similar behavior has also been observed with granulated ground blast furnace slag and silica fume (Cyr 2013). PCC fly ash can also increase surface scaling of concrete (Cyr 2013).

In air-entrained concretes, cement replacement using fly ash from co-combustion of biomass and coal have been reported to cause similar problems as conventional fly ash from PCC. Fly ash from co-combustion biomass and coal has been reported to increase the requirement for AEA (Shearer et al. 2010; Wang et al. 2008), decrease the effectiveness of AEA (Kosior-Kazberuk and Józwiak-Niedzwiedzka 2010), decrease the quality of air entrainment, (Kosior-Kazberuk and Józwiak-Niedzwiedzka 2010) and decrease the surface scaling resistance of concrete (Kosior-Kazberuk and Józwiak-Niedzwiedzka 2010; Kosior-Kazberuk 2013). Contrary to this, Johnson et al. (2010) reported that fly ashes with low loss on ignition (LOI) (0.4–0.9%) did not interfere with the performance of the AEA. It is possible that properties of co-combustion fly ash are closer to the properties of conventional coal fly ash because biomass can have a negligible effect on ash quality due to its lower ash content compared to coal (Johnson et al. 2010).

In air-entrained concrete, cement replacement by fluidized bed combustion fly ash (FBCFA) from combustion of coal has been reported to increase AEA dosage of concrete (Glinicki and Zielinski 2008) and to decrease surface scaling resistance (Glinicki and Zielinski 2009). Both positive (Józwiak-Niedźwiedzka 2012) and negative (Glinicki and Zielinski 2008) effects on the quality of the air void system have been reported. In addition, one study reported that FBCFA from coal combustion decreased freeze-thaw (F-T) resistance of non-air-entrained concrete (Naik et al. 2005). Omran et al. (2018) reported that concretes in which 15–25% of the cement was replaced by FBCFA from biomass combustion had good F-T durability. On the other hand, FBCFA had a negative effect on surface scaling resistance and spacing factor.

In addition, there are studies that used fly ash from biomass combustion, but the combustion method has not been stated. Wang et al. (2008) reported that pure wood fly ash did not increase AEA dosage in a similar way as ashes from coal combustion and co-combustion of coal and biomass. Nagrockienė and Daugėla (2018) used fly ash from biomass combustion to replace 5–30% of cement. At a replacement rate of 15–20%, properties related to F-T resistance, such as compressive strength and open and closed properties, were at the same or better level than in the reference mix. Researchers noted that up to the 15% replacement level, concrete had the same or better predicted durability than the reference mix. Ipatti (1988) examined the effect of peat fly ash to the freeze-resistance of concrete. That study reported that cement replacement using peat fly ash in air-entrained concrete resulted in increased compressive strength and good freeze resistance. Used fly ash had a high SiO_2 content (62%) and low LOI (0.46%).

Overall, research focusing on other than pulverized coal fly ashes is quite limited, and most of these studies have been done for fly ashes originating from FBC of coal or from co-combustion of coal and biomass. However, it is known that both combustion method and fuel significantly affect the physical and chemical properties of fly ash, which in turn affects fresh and hardened state properties of concrete and mortar.

The aim of this study was to examine how partial cement replacement using un-treated and milled FBCFA from co-combustion of peat and wood affects AEA performance and F-T resistance of conventional and air-entrained mortars. These are important properties, especially in cold regions where peat and biomass are available for energy production. Additionally, fly ash from pulverized coal combustion was used to compare the performance of FBCFA to a more conventional, standardized SCM.

Materials

FBCFA used in this study originated from circular FBC of peat and wood. The burning temperature in the boiler was around 790°C. In order to study the effect of milling, FBCFA was milled using a laboratory size tumbling ball mill. A small amount of isopropanol was used as a grinding aid to prevent the agglomeration of fly ash during the milling. Milling was continued to the point where median particle size of ash remained constant. Milled FBCFA is referred to as M_FBCFA. Coal fly ash (CFA), originating from pulverized combustion of coal, was used to compare the performance of FBCFA to a more conventional SMC. Cement used in this study was sulfate resistant Portland cement type CEM I 42,5 N -3R (SR-sementti, Finnsementti). Sand used in mortars was CEN Standard sand (CEN-Standard Sand, Normensand GmbH). The AEA used was in liquid form and it was based on synthetic tensides (Airmix, Finnsementti). The super plasticizer (SP) used in the mortars was polycarboxylate based (SemFlow ELE 20, Semtu).

Methods

Characterization of materials

Chemical composition of materials was determined using the X-ray fluorescence method (XRF). Analysis was done for melt-fused tablets using a wavelength dispersive XRF spectrometer (AxiosmAX, PANalytical). LOI was measured by the thermogravimetric method using an automatic drying and ashing system (prepASH, Precisa Gravimetrics AG). Carbon content of the fly ashes was measured using CHNS/O elemental analyzer (2400 Series II CHNS/O Analyzer, PerkinElmer). Particle size distribution (PSD) of cement replacement materials was analyzed using a laser diffraction particle size analyzer (LS 13 320, Beckman Coulter). Analysis was done in wet mode using isopropanol as a carrier medium, and the data were analyzed using the Fraunhofer optical model. Density of materials was measured using a helium pycnometer (AccuPyc II 1340, Micromeritics).

Mix design

Mortar mix design was based on the EN 196-1 testing standard (SFS 2016). However, some modifications were made. To study the effect of cement replacement using FBCFA, M-FBCFA, and CFA, a 20% mass based replacement rate was selected. SP was used in every sample, and dosage of SP was based on pre-experiments so that the mixtures without AEA would have approximately the same workability. Five different levels of AEA were used to produce mortars with different air contents. A water-to-powder ratio of 0.45, instead of the original 0.5, was selected to prevent mortars from having too high flowability. The mix designs of the various mortars are presented in Table 1.

Mortar mixing

The mixing of mortars was done according to cement testing standard SFS-EN 196-1 (SFS 2016). Immediately after mixing, the flowability of mortars was evaluated using the flow table method described in standard SFS-EN 1015-3 (SFS 1999). Next, mortar was mixed in the mixer for one minute using a fast mixing speed. Immediately after the mixing, the density of the mortar was measured using two identical cylinder-shaped plastic containers. First, half of the cylinders were filled with mortar, and then the mortar was compacted using a tamper. After this, the cylinders were set on a jolting apparatus described in SFS-EN 196-1 (SFS 2016) and jolted 60 times to remove excess air from the mortar. Finally, the rest of the containers were filled with mortar and the same compaction procedure was used. After filling the containers, the surface of the mortar was leveled and all the excess material was removed from the sides of the containers. The weights of the empty and full containers were recorded. After the weighing of the cylinders, mortar was loaded back into the mixer and mixing was continued for 30 seconds at a fast mixing speed. Finally the casting of the mortar was done according to SFS-EN 196-1 (SFS 2016). After the casting, the mortars were wrapped in plastic and cured under laboratory conditions. The next day, the mortars were removed from molds and cured in plastic containers filled with water.

Air content

Air content of the mortars was calculated by comparing the real density of fresh mortar, ρ_R, to theoretical density similar to the ASTM C138 standard (ASTM 2017). However, measurement devices and protocols of standards were modified to be more suitable for lab scale experiments done with mortar.

The density of fresh mortar, ρ_{Fresh}, was calculated using equation (1).

$$\rho_{Fresh} = \frac{M_{Mortar}}{V_{Mortar}}, \quad (1)$$

where M_{Mortar} is mass of mortar in the container and V_{Mortar} is the volume of the container.

The theoretical density of mortar, $\rho_{Theoretical}$, was calculated using equation (2).

$$\rho_{Theoretical} = \frac{M_{Total}}{\frac{M_C}{\rho_C}+\frac{M_W}{\rho_W}+\frac{M_S}{\rho_S}+\frac{M_R}{\rho_R}+\frac{M_{AEA}}{\rho_{AEA}}+\frac{M_{SP}}{\rho_{SP}}}, \quad (2)$$

where M_{Total} is total mass of mortar mixture, M_C is mass of cement, ρ_C is density of cement, M_W is mass of water, ρ_W is density of water, M_S is mass of sand, ρ_S is density of sand, M_R is mass of used replacement material, ρ_R is density of corresponding replacement material, M_{AEA} is mass of AEA, ρ_{AEA} is density of AEA, M_{SP} is mass of SP, and ρ_{SP} is density of SP.

Finally, the air content of fresh mortar was calculated using equation (3).

$$Air\ content\ (\%) = \left(\frac{\rho_{Theoretical}-\rho_{Fresh}}{\rho_{Theoretical}}\right) \times 100 \quad (3)$$

Freeze-thaw resistance

To study the mortar's resistance against damage caused by repetitious freezing and thawing, mortars were exposed to 90 F-T cycles. The experiment was modified from ASTM standard C-666 (ASTM 2015). After the mortars were cured 28 days, they were put in small plastic boxes (three prisms per box) and water was added to the box so that mortars were half immersed in water during the experiment. F-T cycles were produced in a climatic test chamber (WK3-180/40, Weiss Technik). At the beginning of the F-T cycle, temperature was first kept at 15°C for two hours. During the next two hours, the temperature was dropped to -20°C where it stayed another two hours. Finally, during the last two hours, the temperature was raised back to 15°C. Specimen Ref.0.05 was not subjected to F-T-experiment due to an error in sample handling.

Evaluation of mortars' F-T resistance was based on compressive strength and relative dynamic modulus of elasticity, determined before and after the F-T experiments. The relative dynamic modulus of elasticity was obtained using an ultrasonic pulse velocity tester (Ultrasonic pulse velocity tester, Matest) which measured the time of an ultrasonic pulse going through mortar samples. These measurements were made before and after the samples were exposed to F-T cycles and during the experiment at intervals of 18 cycles. Specimens were removed from the climatic test chamber approximately 24 hours before measurement and kept fully immersed in water at room temperature. After measurement, the specimens were returned to the F-T cabinet and the experiment was continued. The relative dynamic modulus of elasticity was calculated using equation (4).

$$\text{Relative dynamic modulus of elasticity (\%)} = 100 \times \frac{V_n^2}{V_0^2}, \quad (4)$$

where V_n is the velocity of ultrasonic pulse after n F-T cycles and V_0 is the velocity of pulse before F-T experiments.

After 90 F-T cycles, the compressive strengths of the mortars were determined and compared to the compressive strengths of corresponding mixtures (on the 28th day), which were not exposed to F-T cycles. Finally, F-T resistance was calculated using equation (5).

$$\text{Freeze-thaw resistance (\%)} = 100 \times \frac{f_{cft}}{f_{c28}}, \quad (5)$$

where f_{cft} is compressive strength after the F-T experiment and f_{c28} is compressive strength after 28 days curing.

Results and discussion

Characterization of materials

FBCFA consisted mainly of SiO_2, Fe_2O_3, Al_2O_3, and CaO (see Table 2). The sum of SiO_2, Al_2O_3, and Fe_2O_3 was 72.6% which fulfills the requirement of fly ash standard EN 450-1 (SFS 2013). FBCFA had 1.5% LOI value and 0.3% carbon content. FBCFA contained 3.5% SO_3, which is slightly higher than the limit of fly ash standard EN 450-1. Otherwise, FBCFA fulfilled the chemical requirements of EN-450-1. CFA was mainly composed of SiO_2, Al_2O_3, and Fe_2O_3. The sum of SiO_2, Al_2O_3, and Fe_2O_3 was 82.9%. LOI and carbon content for CFA were 1.3% and 1.1%, respectively. Chemical composition of cement was typical for sulfate resistant cement. Sand was almost pure SiO_2.

Median particle sizes of cement, FBCFA, M-FBCFA, and CFA were 9.4, 15.9, 3.2, and 11.7 µm, respectively (see Fig. 1). PSD of FBCFA was a little bit narrower compared to cement. Milling of FBCFA clearly decreased the particle size and increased the span of particle size distribution. The PSD of CFA was similar to cement, but it had a higher share of slightly larger particles.

Effect of AEA on fresh state properties of mortars

Flowability of mortars clearly increased with increasing dosage of AEA (Fig. 2). Small air bubbles probably act as “ball bearings” in mortars, which allows particles to bypass each other more easily, leading to decreased viscosity and lower yield stress. It is well known that FBCFA can decrease the flowability of mortar or concrete (Fu et al. 2008; Li et al. 2012; Rissanen et al. 2018; Sata et al. 2007; Sheng et al. 2007). Despite the preliminary trials performed for mortars, flowability of M-FBCFA was somewhat higher compared to other mixtures. This indicates that SP dosage for M-FBCFA could be even lower than suggested in Table 1, when targeting similar flowability with other mixtures. This is in line with other studies reporting that milling of FBCFAs can decrease the water requirement of concrete or mortar (Fu et al. 2008; Li et al. 2012; Rissanen et al. 2018). Air entrainment had the lowest impact on flowability of mortars containing FBCFA. Apparently, the irregularly shaped ash particles of FBCFA have an opposite effect on workability. This could be a positive effect in air-entrained concrete as it could stabilize air bubbles and increased viscosity could prevent unwanted loss of entrained air from fresh mortar.

Neither un-milled nor milled FBCFA had a significant effect on performance of AEA, unlike CFA, which clearly decreased the effectiveness of AEA (Fig. 3). FBCFA had the same or slightly higher air content compared to the reference (Ref.) when AEA dosage was low. At 0.05% AEA dosage, Ref. had slightly higher air content than FBCFA or M-FBCFA. When AEA dosage was increased to 0.2%, FBCFA had the highest air content (43%), while Ref. had slightly lower air content (40%). Air contents of M-FBCFA were slightly lower than those of Ref. and FBCFA at every AEA dosage. However, the difference compared to Ref. increased as the amount of AEA increased. It is possible that slightly lower viscosity of mortars containing M-FBCFA caused entrained air to escape from fresh mortar. Similarly, high viscosity of mortars containing FBCFA could help to prevent loss of entrained air.

Air contents of mortars containing CFA clearly were lower compared to other mortar mixtures. The only exception to this trend was the mortar specimen containing 0.2% AEA. This specimen had the same air content (40%) as Ref. It is possible that when AEA dosage is high enough, air content is affected also by the rheology of the mortar, rather than just by AEA concentration. At lower AEA dosages, CFA required approximately two to three times higher AEA dosage to achieve similar air content as other ashes. CFA probably contains a small amount of unburned carbon, which was enough to absorb a significant amount of AEA. Apparently, the content of unburned carbon is much lower in FBCFA, as low carbon content suggests. Possible variations in the properties, such as accessible surface area and surface chemistry of carbon particles, can also explain why fly ashes had different effects on AEA (Gao et al. 1997; Hachmann et al. 1998; Hill et al. 1997).

Air contents of fresh mortars without AEA were 5.6, 6.8, 4.4, and 3.7% for Ref., FBCFA, M-FBCFA, and CFA, respectively. This result could indicate that FBCFA having highly irregular particle shape could entrap some air in the fresh mixture, while M-FBCFA and CFA had opposite effects. Similar observations were done in the study by Johnson et al. (Johnson et al. 2010) who reported that 20% cement replacement using conventional coal fly ash slightly reduced the air content of non-air-entrained concrete.

Compressive strength

Air content of fresh mortar correlated well with 28-day compressive strength (Fig. 4). Both FBCFA and M-FBCFA had similar compressive strengths compared to Ref. This result suggests that FBCFA and M-FBCFA produced hydration products that had a positive impact on compressive strength. In the case of CFA, pozzolanic reactions were probably slower, which explains why compressive strength of CFA at the age of 28 days was slightly lower.

Similarly, compressive strengths measured after F-T experiments correlated well with air content of the fresh mortars (Fig. 5). FBCFA had slightly better compressive strength compared to Ref. at fresh mortar air contents below 20%, which are more relevant for practical use. With higher air contents, however, performance clearly decreased. M-FBCFA had similar compressive strength as Ref.

when air content was low, however, with higher air contents, performance of M-FBCFA seemed to decrease. Compressive strengths of mortars containing CFA were clearly the weakest after the F-T experiment. At 10% air content, compressive strength was 40% lower compared to Ref. and even 48% lower compared to FBCFA. CFA mortar without AEA suffered severe damage during the experiment, and compressive strength could not been determined.

It should be noted that all mortars where 0.2% AEA dosage was used suffered from severe damage during the F-T experiment. For this reason, several compressive strength specimens from these mixes had to be discarded, which naturally decreased the reliability of the data. Fresh mortar air contents exceeding 35% are clearly excessive for practical use. FBCFA mortar containing 0.2% AEA dosage was destroyed during the F-T experiment, and compressive strength could not been determined.

Few studies have reported that milling of coal fly ash from FBC (Li et al. 2012; Zhao et al. 2015) and pulverized combustion (Hamzaoui et al. 2016) is beneficial for mechanical properties of mortars when FBCFA is used for partial cement replacement. In this study, such behavior was not observed. The reason for this could be different physical properties of FBCFA originating from biomass combustion as well as different milling parameters. In a study by Zhao et al. (2015), milling increased specific surface area (SSA) from approximately 0.5 to 0.8 m^2/g, and in a study by Hamzaoui et al. (Hamzaoui et al. 2016), from 0.8 to 2 m^2/g. Ohenoja et al. (2016) milled FBCFA from combustion of biomass and peat using pin mill and ball mill. Only pin mill at the highest milling speed was able to increase SSA from 3.1 to 6.7 m^2/g, while ball mill and pin mill at lower speeds had little effect on SSA. Additionally, previous studies (Rissanen et al. 2018) showed that milling of FBCFAs was able to increase SSA only 15% and 16%. It seems that milling has very limited effect on SSA of fly ashes, which already have a high surface area. Similarly, some studies reported that milling of fly ash could increase the share of amorphous phases leading to increased reactivity (Fu et al. 2008; Hamzaoui et al. 2016; Zhao et al. 2015). On the other hand, Ohenoja et al. (2016) showed that milling did not increase the amount of reactive CaO, SiO_2, Al_2O_3, or Fe_2O_3.

Freeze-thaw resistance

The best F-T resistance was achieved with air content of 14% (see Fig. 6). When air content of fresh mortar was over 30%, F-T resistance decreased rapidly due to weak mechanical properties of mortars. Air entrainment had a positive effect on F-T resistance of mortars with FBCFA, when air content of fresh mortar was around 12%. In this mixture, compressive strength was even slightly higher after the F-T experiment, compared to specimen, which was not subjected to the experiment. Apparently, AEA dosage provided good protection against F-T cycles. When air content increased, the F-T resistance decreased and the specimen that had an air content of 43% was destroyed during the experiment. In the relevant air content range (10–20% for mortars), F-T resistance of M-FBCFA mortars were better or on par with Ref. F-T resistance of all mortars decreased as expected at non-realistically high air contents of 30% or higher.

In the case of CFA, AEA was essential for F-T resistance. Mixture without AEA suffered from severe damage (Fig. 6b), and F-T resistance could not been determined. The reason for this could lie in the combined effect of low strength, high water content, and low air content. Slower reactivity of CFA leads to lower compressive strength compared to other specimens without AEA. Due to slower reactivity, mortars with CFA probably had a higher amount of capillary pores, which contained a higher amount of water, which created higher stress during freezing. This caused severe damage in this mortar, which had the lowest amount of entrained air. The addition of AEA clearly increased air content and F-T resistance of mortars to similar levels compared to Ref. and other fly ashes. The best result for CFA (97%) was obtained with AEA dosage of 0.015%, which produced 23% air content (Fig. 6).

AEA also had a positive effect on F-T resistance of the no-ash Ref. F-T resistance of the mixture without AEA was 83%, and the best result (93%) was obtained when air content of mortar was 18%.

Relative dynamic modulus (RDM) as a function of fresh mortar air content (Fig. 7) showed a similar trend with F-T resistance (Fig. 6). FBCFA and M-FBCFA demonstrated the same or better performance than Ref. within the whole data range. RMD clearly decreased in specimens that showed signs of damage during the F-T experiment. However, RDM was clearly a less sensitive measurement of F-T damage compared to compressive strength. In most samples, RDM decreased only slightly from the original 100%, and in some cases, RDM even slightly increased. Even the most deteriorated samples, CFA0 and FBCFA0.2, reached RDM of 72% and 85%, relatively. This could suggest that F-T experiment damages occurred mainly on the surface of the mortars and did not cause internal cracking of the matrix. It is also possible that during the experiment, mortars still absorbed water that could increase the speed of the ultrasonic pulse in the sample.

Conclusions

Milled as well as un-milled wood-peat combustion ashes led to improvement in mortar F-T performance in relevant air contents (10–20%). This was more evident with the non-milled ash with 10% air content, which improved F-T performance by 20%, as measured by UCS after 90 F-T cycles. In addition, the presence of these ashes did not affect the total amount of entrained air, and therefore did not seem to affect the functioning of AEAs.

Ashes from pulverized coal combustion led to decreased F-T performance of the mortars. It increased AEA requirement two to three times, and at 10% air content, lowered F-T performance by 40% and led to a fully destroyed sample at 4% air content.

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Data Availability Statement

All data, models, and code generated or used during the study appear in the submitted article.

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