

Long Term Effects on Stored Calcium Rich Fly Ash as Relevant to Cement Replacement

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Abstract

Storage of calcium rich fly ashes (CFA) is necessary when its production exceeds the demand for the product. In these cases, wetting of the CFA is needed in order to prevent dust emissions from the storage site. When CFA is wetted, reactions occur between the water and free lime, thus affecting the potential use of CFA as a partial cement replacement material. This paper deals with questions such as amount of added water, storage environment and time dependency and how this affects the CFA if it is to be used as a partial cement replacement material in the stabilization and solidification (s/s) of dredged materials. The paper concludes that stored CFA may be reused as a partial cement replacement material within the s/s application. Longer storage time as well as higher storage temperatures adversely affects its potential use as a partial cement replacement material.

Keywords: Calcium rich fly ash, stabilization & solidification, biomass, peat, lignite.

INTRODUCTION

Generation of electricity and heat by means of thermal power demands a product that can be ignited. This combustible material is usually coal, oil, gas, biomass and/or waste. If using solid fuels as coal, biomass and waste, a by product known as ash is formed in the process; fly ash (FA) and bottom ash (BA). Roughly 600Mt ashes are annually produced throughout the world and 70 weight % of these ashes are FA, 25 weight % BA and 5 weight % slag [1]. 41 weight % of all ashes are annually reused in some form [2].

Given the environmental impact of combustion i.e. CO₂ emissions, a widened use of biomass fuels and co-combustion is expected in the near future. This will however create new problems when reusing ashes and FA in particular. The reuse of FA within the concrete industry stand for a total of about 25 % but the reuse of non-coal derived FA within the concrete industry is prohibited [14]. Non-coal derived FA usually has higher amounts of calcium oxide, i.e. free lime, which acts expansively in concrete. Less than 20% of biomass or co-combustion FA is reused today in Europe [11]. A new standard, the EN 450, is however under development [9]. This standard will deal with the reuse of non-coal derived FA within the cement and concrete industry. If not, larger quantities of FA will be landfilled each year.

Reuse of FA within the construction sector has many advantages. Today, the most important incitement is probably based upon economical ground. FA is produced at a low cost and normally only logistical costs are associated with its reuse. At larger construction project types that require high amounts of cement, a partial substitute of cement with FA may save money. FA is of pozzolan nature, which means it will improve cement and concrete after its initial hardening e.g. higher compressive strength and lower hydraulic conductivity. An interesting method for the reuse of FA is the s/s-treatment method [4, 5, 6, 7, 8]. With the s/s-method, large quantities of soils may be treated in a more economical and environmental way. The polluted soil is mixed with a binder, commonly cement. By doing so, pollutants are stabilized within the soil structure and the soil is geotechnically solidified. s/s-treatment requires large amounts of binders and it accepts non-coal derived FA since no standards are associated with the type of FA used.

In Sweden, coal is rarely used as a fuel. More common is the use of biomass, peat and waste. It means that FA is normally of non-coal origin and thus derived from reuse within the cement and concrete industry. Beside their non-coal origin, the main part of the FA is produced during November-March. This means that it might be difficult to find available FA all year around, making the reuse of FA less attractive. Hence, FA must be stored prior to use. Little research have been made on the storage of CFA from biomass- and co-combustion with respect to presumptive usage of the FA as a cement replacement material. Storing FA in a cheap way requires an addition of water to prevent it from



dusting when stockpiled outdoors. Addition of water in CFA will however trigger self hardening properties of the material, thus causing detrimental effects within the CFA.

Storing FA with a water addition is the cheapest way which FA can be stored. If water is added in CFA, mineral transformations will occur thus affecting the FA potential of being able to partially replace cement. Knowing these reactions should be considered crucial if using stored CFA. Related research questions are:

- How does the initial water addition in the CFA affect its function as a part binder with cement?
- How does the storage environment (temperature, atmospheric exposure, precipitation) affect its function as a part binder with cement?
- How does an activation of stored CFA affects its function as a part binder with cement?

A laboratory series have been performed in two steps. The first part will deal with the development of newly formed minerals in the CFA alone and the development of pH, electric conductivity, water content and amount of free lime throughout an increased storage time. The second part will deal with the actual functionality of the CFA when used as a part binder, together with construction cement (CC) and granulated blast furnace slag (GGBS) in the s/s-treatment method.

This paper provides valuable guidelines to a producer of CFA if the CFA is meant to be reused within the construction industry, i.e. s/s-treatment.

METHODS

In order to answer the proposed research questions, CFA needed to be stored in a controlled environment and surveyed accordingly. Thus, creating a relevant storing environment for the CFA was crucial.

Two storage sites were constructed. The main storage site was placed outdoors at the Port of Gävle, roughly 200km north of Stockholm. Outdoor storing of the CFA allowed it to be exposed to natural variations such as rain, snow, cold and wind. The second storage site was located indoors at the Vattenfall laboratory in Älvkarleby, roughly 30km south of the Port of Gävle. The CFA were stored for a total time of 168d (February – July) in both environments. Throughout the storage time, extraction of CFA were made at seven (7) times, *table 1*

Table 1. Extraction of CFA from storage site expressed in days since start of storage, February 2010.

Extraction day	1	2	3	4	5	6	7
	(7d)	(14d)	(28d)	(56d)	(84d)	(112d)	(168d)

Three types of CFA were mixed with three (3) pre-decided water contents prior to storage; 10-12 %, 15-17 % and 20-22%. At each extraction day, outdoor stored CFA was extracted from the top and centre of each storage pile. At extraction day 4 (56d), 6 (112d) and 7 (168d), indoor stored CFA was extracted from the centre only. Each storage pile outdoors contained roughly 2 – 3 tons of wetted CFA and placed in 2x2m open boxes without a lid, *figure 1*.

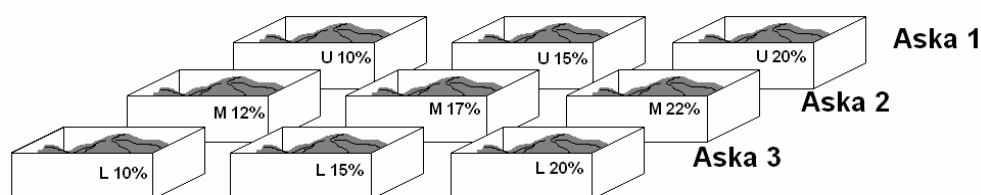


Figure 1. Outdoor storage site with boxes divided into type of CFA and amount of added water prior to storage. Aska 1 = Uppsala (peat), Aska 2 = Mälarenergi (biomass), Aska 3 = Lippendorf (lignite).

The corresponding piles indoors had a size of roughly 20 kg and placed in sealed buckets at a temperature of 20°C. Continuous temperature measurements were performed in each storage pile throughout the whole laboratory period. The exact same type of CFA was stored indoors as well as outdoors.

Besides survey of change in characteristics in the stored CFA alone, the extracted CFA was also used as a part binder in s/s-treatment of sediments. 40 % of the traditional binder consisting of CC and GGBS were exchanged with the stored CFA. The recipe for the s/s-treatment thus became 40 % CC,

20% GGBS and 40 % CFA and added with a ratio of 150kg binder/m³ of sediment. Mixing of sediment and binder took 5 minutes and was made with a Hobart mixer. After, mixing the s/s-treated sediment where put in test tubes, 4 from each batch. The test tubes where then left to harden under water in a cooling facility and tested, i.e. uniaxial strength after 28 and 91d respectively. No loading of the test tubes where applied during curing. The results from this would allow a widened understanding of the efficiency and the influence of time dependant changes within the added CFA when used in a practical sense, i.e. s/s-treatment.

Involved Materials

Two types of materials where used during this laboratory series; Sediment and binders (CC, GGBS and stored CFA). These two types of materials combined, form the basis of materials used within the s/s-treatment method.

Sediment

The sediments where excavated from the harbour basin at Gävle Port, Sweden. The sediments where kept in a cooling facility at 5°C in sealed containers under water. A geotechnical characterisation as well as a total content analysis where made.

Binders

The CC (CEM II/A-LL 42,5 R) and GGBS (Merit 5000) were only characterised in the beginning of the laboratory series since they where not stored.

Three types of CFA where used within the laboratory series. Their origin can be seen in *table II*.

Table II. FA used within the project.

Origin	Boiler	Fuel	Relation, energy %	Amount FA ton/year
Mälarenergi, SWE (Biomass)	CFB	Recycled wood, peat, stem wood	20/8/72	18 000
Uppsala, SWE (Peat)	Pulverized	Peat, wood chips	80/20	20 000
Lippendorf, GER (Lignite)	Pulverized	Lignite, sewage sludge	97/3	500 000

Throughout the storage time, characterization of the extracted CFA was made with respect to; pH, water content, amount free lime, particle distribution, SEM EDX-, FTIR- and XRD-analysis.

s/s Treated Material

s/s-treated soil samples with the extracted (top extracted-, centre extracted-, indoor stored- and activated-) CFA as a part binder were made. These soil samples were left to cure in a cooling facility at 5°C under water and evaluated, i.e. uniaxial testing after 28 and 91d curing.

RESULTS

Sediment

The sediments were very soft and in a liquid state. Geotechnical characteristics are found in *table III* and total contents are found in *table V*.

Table III. Characteristics of the sediment

Characteristics	Result
Total density, t/m ³	1,2
Water ratio W _n , %	235
Undrained shear strength, kPa	0,5
pH, -	6,9
TOC, weight %	4,8

CFA – Fresh State

The basic properties of the three (3) types of CFA are shown in *table IV* and the total content analysis is shown in *table V*. It is noticeable that the CFA from lignite has a much higher total density and compact density than the two other CFA. The amount of SiO₂ is higher in CFA from biomass. The amount of SiO₂ and CaO in peat and lignite CFA are very similar due to the used fuel. In peat CFA an elevated amount of MgO is found, which originate from the flue gas cleaning with dolomite lime stone (CaMg(CO₃)). The dolomite lime stone is ground and added in the boiler. The flue gas cleaning reduces sulphur and elevated levels of sulphur can be found in biomass CFA (2,68 %) and lignite CFA (2,87 %) since flue gas cleaning take place after the extraction of FA. Corresponding amount of

sulphur for peat CFA is 1,67 %. LOI is lower in CFA from lignite where the boiler works at a higher temperature.

XRD-analysis reveals the presence of minerals in the CFA, *table VI*. Given these results the mineral composition of the three (3) CFA are considered to be similar. The amount of each mineral is however shifting. Thus, varying results with respect to uniaxial strength of s/s-treated sediment with stored CFA as a part binder are to be expected.

Table IV. Characteristics of fresh CFA.

Characteristics	Biomass CFA	Peat CFA	Lignite CFA
Total density, t/m ³	0,78	0,99	1,50
Compact density, t/m ³	2,667	2,671	2,885
TOC, weight %	<1,0	<1,0	<1,0
pH, -	12,6	12,7	12,5
Electric conductivity, mS/m	700	1100	300
Free lime, weight %	2,3	20,1	7,1
Blaine Surface, cm ² /g	5710	6460	1950
LOI 1000°C	4,9	9,7	0,5

Table V. Total content in sediments and CFA.

Element, %TS	Sediment	Biomass CFA	Peat CFA	Lignite CFA
SiO ₂	60,3	47,4	23,4	27,7
Al ₂ O ₃	12,3	8,97	6,02	15,3
CaO	1,43	18,3	39,3	37,4
Fe ₂ O ₃	5,59	5,11	9,74	6,48
K ₂ O	3,15	4,55	1,03	0,354
MgO	1,8	2,63	7,21	4,54
MnO	0,065	0,45	0,117	0,194
Na ₂ O	2,18	1,53	0,471	0,174
P ₂ O ₅	0,232	1,64	1,01	1,13
TiO ₂	0,557	1,22	0,289	1,01
Summa	87,6	91,8	88,6	94,3
LOI 1000°C	12,1	4,9	9,7	0,5

Table VI. Mineral constitution of the CFA.

Biomass CFA	Peat CFA	Lignite CFA
Quartz, SiO ₂	Quartz, Lime, CaO	Quartz Lime
Anhydrite, CaSO ₄	Anhydrite	Anhydrite
Periclase, MgO	Periclase	Periclase
Calcite, CaCO ₃	Calcite	
Tricalcium aluminate, Ca ₃ Al ₂ O ₆	Tricalcium aluminate	
	Portlandite, Ca(OH) ₂	Srebrodolskite, Ca ₂ Fe ₂ O ₅
	Gypsum, CaSO ₄ H ₂ O	Yeelimite, Ca ₃ Al ₆ O ₁₂ CaSO ₄ /3CaO 3Al ₂ O ₃ CaSO ₄

CFA – During Storage

During storage of CFA, alterations, with respect to a prolonged storage time, in terms of free lime, pH, electric conductivity, water content and temperature in the CFA changes as follows:

Free lime in stored CFA

When water is added to the CFA, free lime reacts and hydrates instantaneously. The amount of free lime with respect to storage time decreases with time at the surface at a much larger scale than in the centre of the storage pile. Amount of added water as well as higher temperatures is an important factor in the slaking process. This is especially visible when storing CFA indoors at higher temperatures. More water added and higher temperatures – more free lime is slaked, *figure II*.

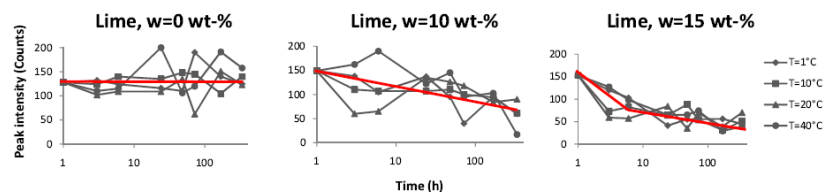


Figure II. Slaking process of lime with respect to water content and temperature [17].

pH in stored CFA

pH is lowered especially in the top stored CFA. Type of CFA also influences the development of the pH-value. The more water added the lower the pH becomes and this is in particular visible in the indoor stored CFA.

Electric conductivity in stored CFA

The electrical conductivity from non-lignite CFA is immediately lowered but begins to elevate after 1 – 2 months of storing. This is in particular visible from top stored CFA. Development of electric conductivity in centre stored CFA is more uniform and does not alter as much with respect to storage time. More water added lowers the electric conductivity.

Water content in stored CFA

Water content changes with storage time but only when CFA is stored outdoors. Precipitation raises the water content in the CFA and especially at the time of spring. Water content seems to lower as the amount of free lime in the CFA is higher because available water is used in the slaking process of free lime. Ettringite, gypsum and calcite formation is accelerated by a higher water content, *figure III*.

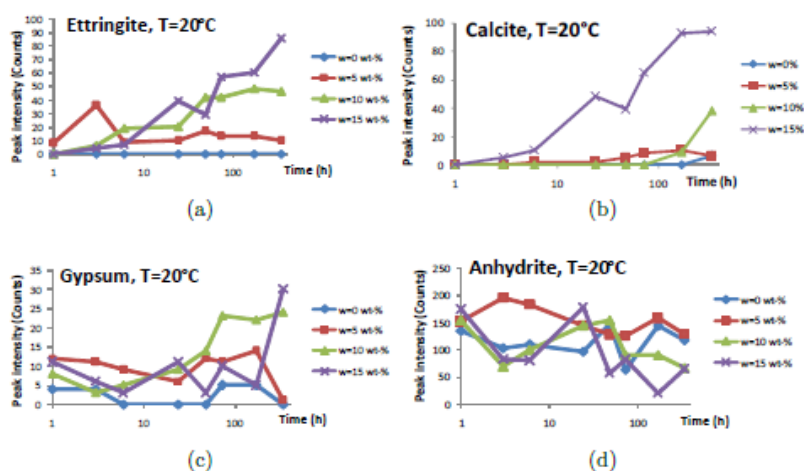


Figure III. Formation of ettringite, calcite, gypsum and anhydrite with respect to water content [17]

Particle size distribution

The particle size distribution becomes coarser when water is added, which indicate slaking of the free lime. Even if the stored CFA is activated i.e. milling, the activated CFA does not become as finely divided as prior to storing. The more free lime in the CFA, the coarser it becomes prior to activation, *table VII*. d_{50} , is a measurement expressed in mm when 50% of the CFA particles passes a given mesh in the sieving process. In the case of lignite CFA, 50% of the stored CFA has a smaller mean size than 4 mm before activation.

Table VII. Degree of fineness before/after activation of CFA through milling, $W_n = 10-12\%$

CFA	d_{50} [mm]	Activated d_{50} [mm]	Efficiency
Biomass	0,09	0,073	1,23
Peat	0,07	0,044	1,59
Lignite	4,00	0,063	63,5

Temperature development

As soon as water is added the temperature is raised instantaneously. The temperature depends on the amount of free lime in the CFA. An elevated temperature was registered in each storage pile up to two weeks after water addition. More water added made the temperature raise less, *figure IV*. This was likely caused by an increased ettringite formation at lower water contents. Ettringite formation results in initial higher temperature development than slaking of free lime [15, 16]. Temperatures reached more than 70 degrees and in the case of lignite CFA, temperatures were not registered during its peak.

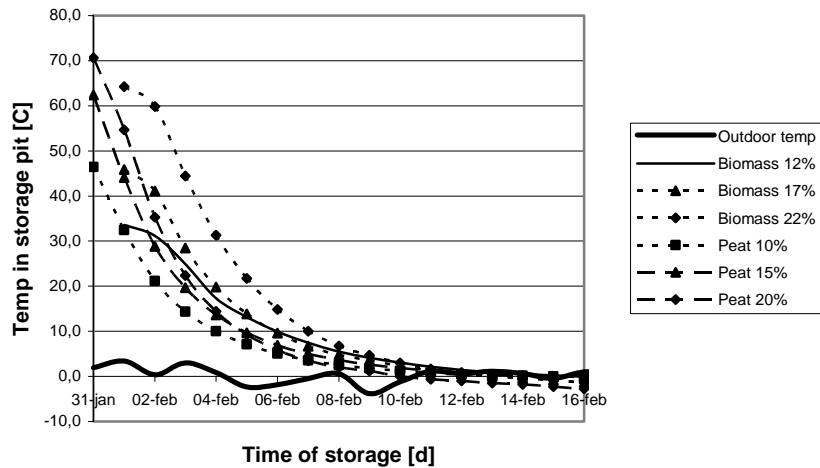


Figure IV. Temperature development in FA from biomass and peat during the two first weeks of storage.

s/s-treated sediments

When CFA was extracted from the storage piles at 7, 14, 28, 56, 84, 112 and 168d of storing, s/s-treated samples were made with CC, GGBS and CFA. Since mineral alterations occur in the CFA with storing time, this will result in varying results, i.e. variations of uniaxial strength of s/s-treated sediments.

Biomass CFA

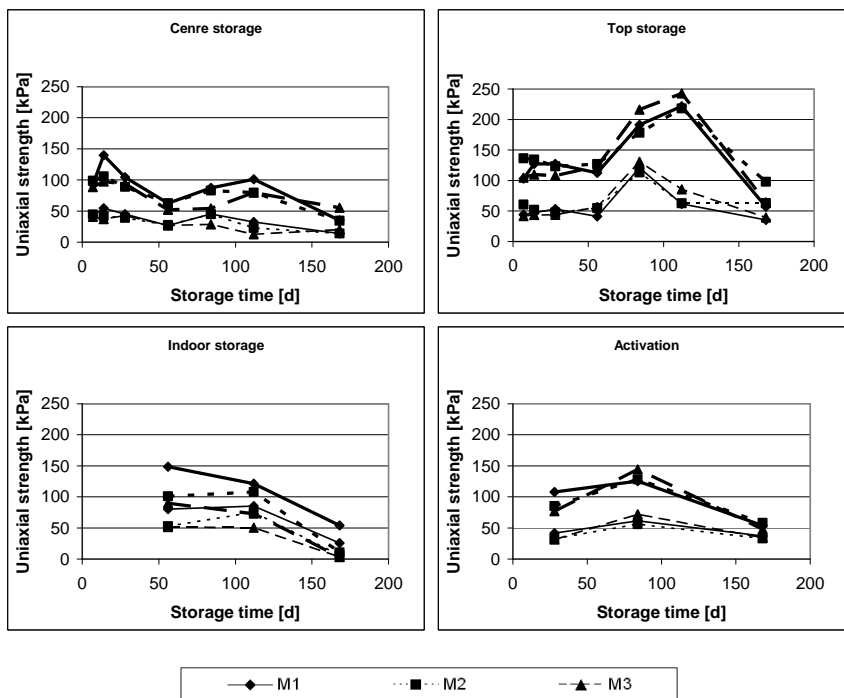


Figure V. Development of uniaxial strength in s/s-treated sediments with stored CFA from biomass as part binder with respect to storage time of CFA. 28 and 91d (bold) curing. M1 = 12 % water content, M2 = 17 % water content, M3 = 22 % water content.

Centre stored CFA. After 28d of curing the usage of centre stored CFA give test samples with lower uniaxial strength compared to when the CFA is top stored. The trend is that the usage of centre stored CFA is slowly lowering the uniaxial strength of the test samples as the storage time proceeds. Uniaxial strength is elevated after 91d of curing (pozzolanicity) with roughly 1,5 times. The pozzolanicity is not affected by storage time

Top stored CFA. The uniaxial strength of s/s-treated sediments with top stored CFA is higher than the uniaxial strength attained with the usage of centre stored CFA. An elevation of the uniaxial strength is clearly visible at a storage time of 84 – 112d. After 91d of curing uniaxial strength is elevated by roughly 1, 5 times. This effect is however lowered at the end of the storage time.

Indoor storage. After 28d curing the uniaxial strength is higher when indoor stored CFA is used. The pozzolanity is however adversely affected. At the end of the storage time low uniaxial strength is obtained. Higher water content in CFA lowers attained uniaxial strength.

Activation. Activation of CFA has a positive effect when samples are cured for 91d. After 28d of curing no effect from it can be seen. A lowering of the uniaxial strength is obtained at the end of storage time but it is not lowered as much as when the CFA is stored indoors.

Peat CFA

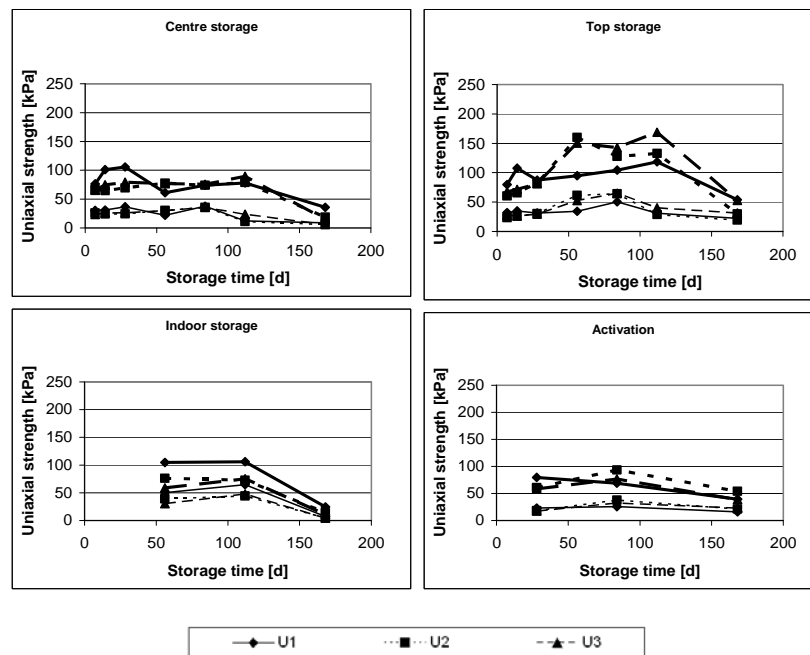


Figure VI. Development of uniaxial strength in s/s-treated sediments with stored CFA from peat as part binder with respect to storage time of CFA. 28 and 91d (bold) curing. U1 = 10 % water content, U2 = 15 % water content, U3 = 20 % water content.

Centre stored CFA. After 28d of curing the usage of centre stored CFA give test samples with lower uniaxial strength compared to when the CFA is top stored. The trend is that the usage of centre stored CFA is slowly lowering the uniaxial strength of the test samples as the storage time proceeds. Uniaxial strength is elevated after 91d of curing (pozzolanity) with roughly 1,8 times. The pozzolanity is not affected by storage time

Top stored CFA. The uniaxial strength of s/s-treated sediments with top stored CFA is higher than the uniaxial strength attained with the usage of centre stored CFA. An elevation of the uniaxial strength is clearly visible at a storage time of 56 – 112d especially with respect to 91d curing. After 91d of curing uniaxial strength is elevated by roughly 1,8 times. This effect is however lowered at the end of the storage time. Pozzolanity is adversely affected by top storing. Higher water content in CFA raise attained uniaxial strength.

Indoor storage. After 28d curing the uniaxial strength is higher when indoor stored CFA is used compared to centre stored CFA. The pozzolanity is adversely affected. At the end of the storage time low uniaxial strength is obtained. Higher water content in CFA lowers attained uniaxial strength.

Activation. Activation of CFA has a slight positive effect when samples are cured for 91d. After 28d of curing no effect from it can be seen. A lowering of the uniaxial strength is obtained at the end of storage time but it is not lowered as much as when the CFA is stored indoors.

Lignite CFA

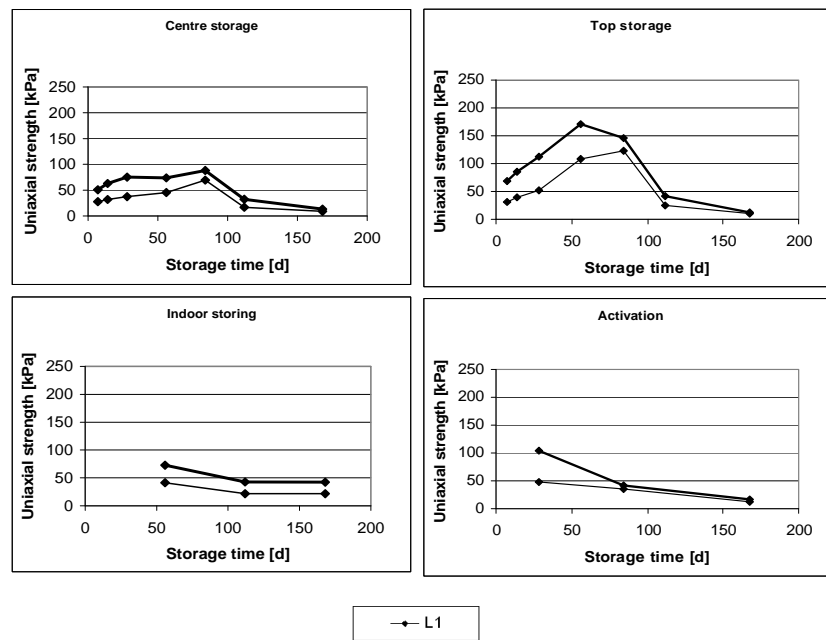


Figure VII. Development of uniaxial strength in s/s-treated sediments with stored CFA from lignite as part binder with respect to storage time of CFA. 28 and 91d (**bold**) curing. L1 = 10% water content

Centre stored CFA. After 28d of curing the usage of centre stored CFA give test samples with lower uniaxial strength compared to when the CFA is top stored. The trend is that the usage of centre stored CFA is slowly lowering the uniaxial strength of the test samples as the storage time proceeds. Uniaxial strength is elevated after 91d of curing (pozzolanity) but to a low extent. The pozzolanity is adversely affected by storage time.

Top stored CFA. The uniaxial strength of s/s-treated sediments with top stored CFA is higher than the uniaxial strength attained with the usage of centre stored CFA. The trend is that the usage of centre stored CFA is slowly lowering the uniaxial strength of the test samples as the storage time proceeds. An elevation of the uniaxial strength is clearly visible until a storage time of 84d. After this the attained uniaxial strength is clearly lowered. After 91d of curing higher uniaxial strength is attained but the pozzolanity is adversely affected by storage time.

Indoor storage. After 28d curing the uniaxial strength is similar when indoor stored CFA is used compared to centre stored CFA. The pozzolanity remains unaffected with respect to time.

Activation. Initially, activation of CFA has a positive effect when samples are cured for 91d. This effect is however almost eliminated as storage time proceeds. A lowering of the uniaxial strength is obtained at the end of storage time.

DISCUSSION

Strong time dependant variations of the stored CFA was visible throughout the storage time, both in terms of measurements on the CFA alone and when CFA was used as a partial cement replacement within the s/s-treatment method. Atmospheric exposure and type of CFA had the highest influence on obtained uniaxial strength on s/s-treated soil. After a storage time of the CFA for more than 112 days, the obtained uniaxial strength was significantly lowered.

Water content in stored CFA

Outdoor storing gave an addition of water of roughly 500l/storage pile. This caused the water content to rise significantly in the outdoor stored CFA with time. Water content was always lower at the top layer of each storage pile compared to the centre. This lowering was likely caused by an elevated vaporization and carbonization [3].

The amount of free lime in the storage piles seems to have an effect of the water content. Higher amount of free lime mean that more water may be bound into the material, thus lowering the amount of detectable water. The evolution of minerals such as ettringite, calcite and gypsum are triggered by

an elevated amount of water, *figure III*. The context of water addition is that more water causes more chemical reactions and mineralogical transformations to occur.

Temperature in stored CFA

The influence of temperature was clearly visible when comparing CFA stored indoors and outdoors. CFA stored outdoors were exposed to much lower temperatures. The reactions in the CFA were enhanced by higher temperatures. This was particularly visible when evaluating the pozzolanic effects. Indoor stored CFA provided a much smaller augmentation of uniaxial strength in s/s-treated soil samples between 28 to 91d of curing. The results from XRD analysis couldn't however clearly find a difference but this is likely due to the fact that the XRD analysis does not see amorphous phases, important for reactivity. However, s/s-treatment clearly showed these differences and the results are supported by several reports [10, 12, 13]. Portlandite was found to be particularly vulnerable to higher temperatures. Since more portlandite is created during higher temperatures, more pozzolanic reactions may occur before the CFA is used in s/s-treatment.

Atmospheric exposure to stored CFA

A very noticeable difference was found between CFA from the top and centre of each storage pile. Carbonation occurs in CFA exposed to the atmosphere, lowering the pH, making the CFA more dry and hindering eventual ettringite mineralization. When used in s/s-treatment, top stored CFA gave rise to much higher uniaxial strength compared to the usage of centre stored CFA. When pH is elevated in the top stored CFA when mixed with CC and GGBS, the formation of ettringite might be triggered since no ettringite is formed at pH <10,5. Top stored CFA was also found, especially for biomass CFA, to provide a much less pozzolanic addition at the end of the storage period. The reason for this is probably due to a lowered extent of portlandite in the top stored CFA. Carbonization was higher for FA with higher amounts of free lime.

Storage time of CFA

It seems that after a storage time of 112d of the CFA, the efficiency as a part binder in s/s-treatment begins to deteriorate. Lignite CFA is clearly adversely affected by storage time and all CFA is showing significant deterioration at the last extraction day. But CFA from biomass and peat are increasingly getting better when used as a part binder until the very last extraction date. The difference hereby cannot only be laid on the CFA, the possible deterioration of the sediments with time should furthermore be considered.

Activation of stored CFA

Activation of stored CFA enhances the pozzolanicity of the CFA. This is due to the fact that the CFA becomes more finely divided after activation. Thus, more free surfaces are available for pozzolanic reactions.

CONCLUSIONS

Three different CFA from biomass, peat and lignite firing have been stored with an addition of water both outdoors and indoors. The CFA have with increased storage time been extracted and analyzed. The CFA has also been used in an actual application, i.e. s/s-treatment of sediments.

- Temperature during storage should be kept low. Lower temperatures preserve in particular pozzolanic properties of the CFA.
- Results indicate mineral transformations in CFA during storage due to the addition of water. Amount of water should be kept low. Higher water contents trigger mineralogical transformations such as slaking of free lime.
- Very high temperatures (60 - 70°C) are to be expected when storing CFA. This could cause handling problems.
- Carbonation of the CFA exposed to CO₂ can be a positive factor since it seems to preserve ettringite for latter use. CFA exposed to CO₂ will give higher uniaxial strength in s/s-treated sediments if used as a part binder compared with the usage of CFA not affected by CO₂.
- Storage time seems to affect the pozzolanicity of the CFA but only after 4-6 months. A prolonged storage time also adversely affects the CFA, i.e. lower uniaxial strength in s/s-treated sediment after 28d of curing.
- Activation of CFA has a positive effect on the pozzolanicity but not after 6 months of storing. Activation does not affect the uniaxial strength after 28d of curing.

- Lignite CFA possessed strong ettringite forming characteristics, which make its use as a binder without the addition of cement interesting.

The results are highly dependant on the type of CFA and soil used, hence laboratory work should always be performed prior to use of a stored CFA in a large scale s/s-treatment project.

Stored CFA may be reused as a partial binder in s/s-treatment.

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