

Slutrapport för projekt

# Utilization of pulp mill wastes in cement-based materials: Case study in cooperation with Södra

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## Återanvändning av massabruks avfall i cementbaserade materialer: En förstudie i samarbete med Södra

## Utilization of pulp mill wastes in cement-based materials: Case study in cooperation with Södra

Titel på projektet – svenska								
Ateranvändning av massabruks avfall i cementbaserade materialer: En förstudie i								
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Universitet/högskola/företag								
Rise Research Institute of Sweden								
Adress								
BOX 857, 501 15 Borås, Sweden								
Namn på projektledare								
Arezou Babaahmadi								
Namn på ev övriga projektdeltagare								
[Klicka här och skriv]								
Nyckelord: 5-7 st								
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concrete								











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## Förord

The concept of this project was a case study where viability of using pulp mill's waste products as supplementary cementitious materials was investigated.

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Södra is the chosen pulp production company to focus on in the pilot scale study and the project was defined as cooperation between CBI cement and concrete research institute and Södra.

Södra provided consultation as well as waste products in this project, while all analysis as well as practical investigation were performed at CBI cement and concrete research institute.

The project leader was Arezou Babaahmadi employed at Rise CBI research institute and the main contact at Södra Cell Värö was Andreas Martinsson who planned for all needed sample uptakes as well as consultations. Södra provided 20% of the total project budget.

All the help with arrangement of the tests and analysis performed at Rise CBI Research Institute by Gilles Plusquellec, Wolfram Oettel, Camilla Lindström as well as Linus Brander is hereby acknowledged and appreciated.

The report is written by Arezou Babaahmadi and Gilles Plusquellec.

The LCA analysis are performed by Nadia Al-Ayish at Rise CBI Research Institute.

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## Sammanfattning

Med ökad oro för CO2-utsläpp, har det resulterat till ersättning av kol med biomassa i förbränningsbaserade kraftverk. Detta skulle däremot emellertid leda till brist av flygaska, som är en av de mest välkända restprodukter från kolförbränning. Flygaska används som tillsatsmaterial i byggsektorn för att minska den stora miljöpåverkan av cementproduktionen. För att försöka hitta andra alternativa tillsatsmaterialer, syftar detta projektet till att demonstrera möjligheten att använda restprodukter från massafabriker i Sverige (barkaska och slam) i produktion av cementbaserade materialer. På grund av brist på studier för validering deras möjliga återvinningsvärde, dessa restprodukter deponeras i dag. Södra är den valda massafabriken i denna förstudie.

### Mål och resultatnytta

Mål – Detta projekt syftar till att demonstrera möjligheten med att använda restprodukter från massafabriker i Sverige (barkaska och slam) i produktion av cementbaserade materialer. Främsta syftena är följande:

 För att karakterisera restprodukter från massa- och pappersindustrin (kemisk sammansättning och variationer i tid).
För att utnyttja massabruk avfall i produktionen av bygg murbruk, marksten, bindemedel eller betong.
För att undersöka egenskaperna hos utvecklade byggmaterial i form av styrka och hållbarhet samt att jämföra de med nuvarande tillgängliga produkter på marknaden.
För att undersöka möjligheter att standardisera massafabrikers avfall som potentiella tillsatsmaterialer till cement.

**Resultatnytta/Effekter:** Resultaten från ett sådant projekt skulle i sin tur göra det möjligt att förhindra deponering av stor mängd av restprodukter från massa- och pappersindustrin och minskar cementtillverknings miljöpåverkan. Enligt statistik som inkommit från Södra Cell Värö, bara i 2016, har 1050 ton aska producerats på Värö, medan förutspått produktion i 2017 förväntas producera omkring 1500 ton. Resultaten är av högst värde för cementindustrin och byggsektorn vilket kan vara en stor motivation för dessa industrier att investera i utnyttjandet av sådana restmaterial. SLUTRAPPORT

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Worldwide increased concern of the  $CO_2$  emissions has led to replacement of coal with biomass in combustion-based power plants. However, this would cause the scarcity of fly ash, one of the most well-known rest products from coal combustion, which is used as supplementary cementitious materials in construction sector to reduce the large environmental footprint of cement production. Seeking to find alternative SCMs, this project aims to demonstrate the viability of using waste products from pulp mills in Sweden (bark ash and green liquor dreg) as supplementary cementitious materials. Due to lack of studies validating their possible recycling value these materials are landfilled today. Södra is the chosen pulp production company in the pilot scale investigations.

The aim of this project is to demonstrate potentials of pulp mills waste products to be utilized as supplementary cementitious materials. This would in turn enable prevention of landfilling large amount of rest products from pulp and paper industry and reduces worldwide cement production environmental foot prints, causing 6 % of the global carbon dioxide emissions. According to statistics received from Södra Cell Värö, in 2016, 1050 tons of ash has been produced while predicted production in 2017 is around 1500 tons. It should also be noticed that there are much more pulp and paper industries in Sweden with more or less similar production amounts.

The specific goals of the proposed study are:

1)To characterize the rest products from pulp and paper industry in terms of chemical composition and variations in time.

2)To utilize the pulp mills waste in production of construction mortars, paving stones, binder glue or mixed concrete.

3)To investigate the properties of developed construction materials in terms of strength and durability and to compare them with their current available products in the market.

4)To examine the possibilities to standardize pulp mills wastes as potential supplementary cementitious materials.

To follow the aims, up to 50 different bio ash samples in a period of 4 month has been collected in 3 different locations, where Södra plants are located (Värö, Mönsterås, Mörrum).

The bio ashes where characterized in terms of chemical composition, mineralogy and particle size distribution. Moreover, the pozzolanic reactivity as well as the activity index of this ashes compared to a reference material is investigated.

The results indicate that:

• Bio ashes are showing a relatively high consistency in chemical composition with respect to both time and location.





• The presence of several silicate minerals explains the relatively high SiO<sub>2</sub> content determined by chemical analysis.

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- Pore size distribution is relatively similar in bio-fly ashes collected from different locations and comparable with a randomly selected coal fly ash.
- Pozzolanic activity as well as activity index of the bio-flashes is close to the border of acceptance according to SS-EN\_196-5 and ASTM C311 standards, indicating good potentials for further investigations.
- The water bath cooling method causes lower chloride contents in the bio ashes. This indicates a high potential for using simple bath immersion as a leaching method to decrease the chloride and sulfate content in bio ashes.

In the next stage it is planned to:

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- To investigate potentials of bio ashes from other industries rather than pulp and paper industry.
- To investigate potential of leaching methods for decreasing chloride and sulfate contents in the bio ashes.
- To account for the durability of bio ash containing concrete in terms of carbonation, sulfate and chloride ingress.

### Inledning och bakgrund

Application of biomass ash in construction materials has been a topic of concern in the last decade for many researchers [1-7]. It is reported that flexural strength and compressive strength in mortars and concrete specimens using biomass ash is comparable to the reference material [1, 7]. Moreover, durability related factors such as frost resistance and alkali silica expansions were reported to either be comparable to the reference materials or even improved with application of biomass ash [2, 3, 7]. However, as biomass is obtained from a relatively vast number of sources, e.g. scrap lumber, forest debris, crops and certain types of waste residues, the variations in chemical composition of the biomass ash has been the major concern reported in literature. As an example in some studies [1], the sum of the contents of silicon dioxide, aluminum oxide and iron oxide is higher than 70% and the content of calcium oxide is lower than 10%, while others reported [4, 5] the same values being lower than 50% and the content of CaO being higher than 20%. Another reported major problem has been the high chloride content in some types of analyzed biomass ashes which is a hindrance for further use of such wastes in construction materials. The reason for this matter is the problems associated with chloride induced corrosion of the reinforcement in concrete constructions, which limits the allowable chloride content in cementitious binders. Moreover, it is even shown that the variation of the composition of the biomass ash is not only due to diversity of the biomass resources, but also even in case of a specific biomass for example wood ash, changes in types of trees or the combustion method and temperature would change the composition of the eventual remained ash. These are probably some of the major reasons that



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although relatively significant research has been conducted on this topic, demonstrating the viability of using these materials are still a challenge. To tackle this challenge in this project, 2 main issues are taken into consideration:

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- 1) The focus of the project is not on diverse types of biomass ash, but only on pulp mills residuals, which are free from contaminations of lead, zinc, chromium or chlorides.
- 2) The concentration is on waste products from pulp mills in Sweden (Södra as prestudy case). In this way, diversity in the type woods used in pulp and paper industry would be minimized, while at the same time Swedish construction sector can benefit from the results. It should be noted that in construction companies, the availability of raw materials in local market plays a very important role as transportation costs would increase the cost of end products. Therefore, these types of demonstration projects should be performed on locally available sources. It should be highlighted that a similar type of study in Sweden has not been performed so far.

### Genomförande

#### **Project partners**

The concept of this project is a case study where viability of using pulp mills waste products as supplementary cementitious materials is investigated. Södra is the chosen pulp production company to focus on in the pilot scale study. The work program involves a screening phase were properties of produced waste products over a period of 6 month is characterized. In parallel an experimental investigation on eventual properties of construction materials produced using pulp mills wastes will be carried out. In case of promising results Life Cycle Assessment (LCA) tools will be utilized in the project to assess environmental impacts associated with propositions in this study. This can further lead to possible standardization of new raw materials (pulp mills waste) in producing cementitious construction materials.

The project is defined as cooperation between CBI cement and concrete research institute and Södra. Södra provides consultation as well as waste products in this project, while all analysis and practical investigation will be performed at CBI cement and concrete research institute.

**Dr. Arezou Babaahmadi** is the main **project leader** in this project (15 % of total time). She is actively involved in many research projects at CBI research institute where she focuses on cement and cement-based materials hydration process both from experimental and modelling dimensions. She was announced as the researcher of year 2015 in the field of concrete technology because of the contribution of her PhD results in the field of nuclear waste management. The practical and technical laboratory work will be by active research assistants working at CBI cement and concrete research institute (55 % of total time).

The main contact at **Södra Cell Värö** is **Andreas Martinsson** (10 % of total time). He is process engineer and is responsible for the management of waste from Värö.



He would provide the needed consultation and make arrangements providing the project with needed waste products (15% of total time involvement from technical staff).

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#### Work packages

The detailed work plan of the project is organized in the following Work Packages (WP):

WP1-Project management and sample uptake

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The goal of this work package is to monitor the project progress towards goals and deliverables, as well as to manage the project budget. Moreover, in this work package the time frame, frequency as well as type of ashes that should be sampled, is planned for.

**WP2**-Screening and characterizing pulp mill waste products

In this work package chemical composition of waste products over a period of 6 month will be documented. All parameters affecting the changes in properties of waste products will be investigated.

**WP3**-Development of new construction materials using pulp mill waste

In this work package, the possibility of using pulp mill waste products in cementbased construction materials is investigated. The main properties such as strength, workability and important durability factors are accounted for. The effect of changes in properties of pulp mill waste on the properties of the produced construction materials is investigated.

WP4- Assessment of environmental impacts of developed materials (LCA analysis)

The LCA analysis is performed according to the recommendations provided by Energimyndigheten (Re-Source). The results from this part are submitted as an excel sheet which was provided by Re-Source.

The following time presented in Table 1, plan will be followed during the project time.

	Month	Month	Month	Month	Month	Month	Month	Month	Month	Month
	1	2	3	4	5	6	7	8	9	10
WP1										
WP2										
WP3			1							
WP4					l I	r I	l I	l I	l I	

#### Table 1. Project time plan



## **Resultat och diskussion**

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#### Screening

The sample uptakes have been performed in 3 different södra plants including Värö, Mörrum and Mönsterås.

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As shown in Figure 1, three different locations for sample uptake was chosen at Värö plant, which includes fly ashes, bottom ashes as well mixed fly ash and bottom ash after the cooling bath. The reason for choosing the third location was to account for the effect of water bath on the chemical composition of the ashes. The statistical frequency of the sample uptakes is also presented in Table 2.



Figure 1. Sampling locations at Värö

Provplats	Kategori	Datum - provtagning
Värö	Flygaska	2017-07-03
Värö	Flygaska	2017-07-06
Värö	Flygaska	2017-07-17
Värö	Flygaska	2017-08-09
Värö	Flygaska	2017-08-10
Värö	Flygaska	2017-08-16
Värö	Flygaska	2017-08-24
Värö	Flygaska	2017-08-25
Värö	Flygaska	2017-08-29
Värö	Flygaska	2017-08-31
Värö	Blandat Botten + Flygaska	2017-07-03
Värö	Blandat Botten + Flygaska	2017-07-06
Värö	Blandat Botten + Flygaska	2017-07-17
Värö	Blandat Botten + Flygaska	2017-08-04
Värö	Blandat Botten + Flygaska	2017-08-10



Figure 2, further presents the sampling location at Mörrum plant. In this case only a mixed fly ash+bottom ash before entering the water bath was collected.

Table 3, summarizes the sampling statistics at Mörrum and Table 4 shows the



Figure 2. Sampling locations at Mörrum



Table 3. Frequency of sample uptake at Mörrum plant

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Provplats	Kategori	Datum - provtagning
Mörrum	Bottensaska +Flygaska	2017-07-25
Mörrum	Bottensaska +Flygaska	2017-07-27
Mörrum	Bottensaska +Flygaska	2017-08-01
Mörrum	Bottensaska +Flygaska	2017-08-03
Mörrum	Bottensaska +Flygaska	2017-08-08
Mörrum	Bottensaska +Flygaska	2017-08-10

At Mönsterås plant, fly ash and bottom ash samples were gathered as presented in Table 4.

Provplats	Kategori	Datum - provtagning
Mönsterås	Flygaska	2017-07-11
Mönsterås	Flygaska	2017-07-13
Mönsterås	Flygaska	2017-07-17
Mönsterås	Flygaska	2017-07-19
Mönsterås	Flygaska	2017-07-25
Mönsterås	Bottenaska	2017-07-11
Mönsterås	Bottenaska	2017-07-13
Mönsterås	Bottenaska	2017-07-17
Mönsterås	Bottenaska	2017-07-19
Mönsterås	Bottenaska	2017-07-25

#### Table 4. Frequency of sample uptake at Mönsterås plant

#### Characterization

#### Chemical composition

The chemical composition of the ashes was accounted for by the help ICM-MS analysis.

Table 5 presents a summary of the chemical composition (wt% of the different oxides) of all the ashes that has been retrieved. Only an average of the data recovered over time and the associated standard deviation are presented in this table. The type of ash indicated as "mix" in the table represent samples where both fly ash and bottom ash are present. It is also indicated if the mix is "washed", "sieved" or "unwashed", depending on the location it has been retrieved in the plant.

From Table 5, it can be noticed that, for each oxide and ash type, the standard deviation is relatively low compared to the average value. This indicates a rather small variation of the composition over time. Only the SiO<sub>2</sub> content of the ash taken from Mörrum presents a high deviation (i.e.  $31.5 \pm 14.1$  wt% for the investigated period).



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The main oxides interesting for the cement industries  $(SiO_2, CaO \text{ and } Al_2O_3)$  are all present in these ashes, although the  $Al_2O_3$  content seems quite low. The alkali content (Na<sub>2</sub>O and K<sub>2</sub>O) is overall relatively low and should not be a concern for those specific ashes.

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The general evolution of composition with respect to Si, Al as well as Ca and C contents, in different sampling locations, is presented in Figures 3 and 4.

As expected, the type of ash and the plant of origin slightly affect the chemical composition of the ashes.  $SiO_2$  and CaO are the oxides that show the more important variation. Those oxides also present the main differences when comparing fly ashes and bottom ashes: there is less  $SiO_2$  and more CaO in the fly ashes than in the bottom ashes.

For the Värö plant, the "mix – sieved" ash has a similar composition as the fly ash. This was expected since only the fine fraction of the mix is present in the sample.

Location	Туре	SiO <sub>2</sub>		Al <sub>2</sub> O <sub>3</sub>		Fe <sub>2</sub> O <sub>3</sub>		CaO		MgO	
		Avrg.	Std.dv	Avrg.	Std.dv	Avrg.	Std.dv	Avrg.	Std.dv	Avrg.	Std.dv
Värö	Fly ash	17.7	5.7	4.0	1.0	2.2	0.3	29	4	4.3	0.5
	Mix - washed	20.9	5.9	4.9	0.9	2.3	0.4	30	5	4.7	0.6
	Mix - washed+sieved	13.5	2.9	4.1	0.6	2.1	0.3	35	4	5.4	0.5
	Bottom ash	41.7	5.0	9.3	1.6	3.1	0.6	27	4	4.2	0.5
Mönsterås	Fly ash	17.8	2.9	7.4	2.6	5.1	0.8	31	2	4.4	0.4
	Bottom ash	71.2	2.3	7.6	1.5	1.2	0.1	10	1	1.1	0.1
Mörrum	Mix - Unwashed	31.5	14.1	4.1	1.4	1.4	0.2	29	7	3.6	1.0

Table 5. Summary of the chemical composition in wt% of the ashes retrieved from the different location. The data are represented as the average value and the associated standard deviation.

Location	Туре	Na₂O		K₂O		P <sub>2</sub> O <sub>5</sub>		Cl		LOI	
		Avrg.	Std.dv	Avrg.	Std.dv	Avrg.	Std.dv	Avrg.	Std.dv	Avrg.	Std.dv
Värö	Fly ash	2.0	0.3	11.5	3.8	3.3	0.4	1.2	0.5	15.5	2.7
	Mix - washed	1.2	0.3	4.5	0.9	3.7	0.5	0.10	0.04	23.8	7.7
	Mix - washed+sieved	1.2	0.1	3.9	1.2	4.4	0.4	0.1	0.1	25.1	3.5
	Bottom ash	2.0	0.2	5.0	0.7	2.5	0.4	0.01	0.01	2.4	2.0
Mönsterås	Fly ash	1.7	0.2	5.9	0.7	3.7	0.3	0.5	0.1	14.4	1.7
	Bottom ash	2.2	0.4	5.3	0.8	0.8	0.1	0.02	0.003	0.6	0.1
Mörrum	Mix - Unwashed	1.5	0.4	6.5	1.0	2.5	0.8	0.5	0.3	16.1	5.5

To represent the evolution of the composition in time, the Figures 5A, 5B and 5C show the evolution of the chloride content of the ashes taken from Värö, Mönsterås and Mörrum, respectively. The vertical scale is a logarithmic scale. The content of chloride is one of the key parameters to know if the ash can be used in concrete. According to the Swedish standard EN 450-1:2012 (requirement for fly ash), the



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It is important to highlight the fact that a clear decrease of the chloride content is measured after washing (sieved mixed): it goes down from 1.2 wt% for the fly ash to 0.1% for the mix after washing and sieving. This indicates that a simple washing with water is enough to remove most of the chloride present in the sample. Similarly, a decrease of the K<sub>2</sub>O content is also noticed.

#### Phase analysis

The phase analysis of the various ashes has been done by X-ray diffraction, using a Rigaku Miniflex 600. All the collected ashes have been analysed, but because most of them present similar patterns, only a selection of each type is presented in this report to highlight the differences.

The diffractograms are presented in Figure 6 (fly ashes), Figure 7 (bottom ashes) and Figure 8 (mixed ashes). The coloured area in Figure 6, Figure 7 and Figure 8 indicate various identified phase, each colour being associated to one phase. No background corrections have been applied to visualize the amorphous nature of the samples: if the base-line presents a "bump", amorphous phases are present. Vice versa, a horizontal base-line indicates a crystalline sample. The more amorphous a sample is, the more reactive it should be.

It must be noted that at this stage no quantitative data are available. The results presented here are only qualitative.

The base-line of all the diffractograms is not completely horizontal. This indicates that a small portion of the sample is amorphous.

All the different ashes have 3 phases in common: quartz (SiO<sub>2</sub>), microcline (KAlSi<sub>3</sub>O<sub>8</sub>, an alkali feldspar mineral) and albite (NaAlSi<sub>3</sub>O<sub>8</sub>, a plagioclase feldspar mineral). Portlandite, calcite and calcium oxide are also detected in the fly ashes (and consequently in the mixed ashes). The presence of several silicate minerals explains the relatively high SiO<sub>2</sub> content determined by ICP-MS.

Portlandite is mainly observed for the mixed ash (Figure 8). No portlandite is detected in any of the bottom ashes. By comparing Figure 7 and Figure 8, we can see an increase of the portlandite pic only when the ashes are washed, suggesting a precipitation of portlandite during the washing.







Figure 3. Evolution of composition of ashes with respect to Si and Al content, in different locations.



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Figure 4. Evolution of composition of ashes with respect to Ca and C content, in different locations.





Figure 5. Evolution of the chloride content in function of time for the different type of ashes taken at different places (Värö, Mönsterås and Mörrum). The red bars represent the maximum chloride content according to the standard EN 450-1:2012.





Figure 6. Diffractograms of the fly ashes.



Figure 7. Diffractograms of the bottom ashes.



Figure 8. Diffractograms of the mixed ashes (fly ash + bottom ash).

#### Particle Size

The particle size distribution (PSD) results of randomly selected bio-fly ashes collected at Iggesund, Månsterås, Morrum and Värö as well as the PSD results from a randomly selected coal combustion fly ash are presented in Figure 9. As can be seen the particle size distribution of the bio ashes are relatively similar and comparable with the PSD results for the coal fly ash.

#### Pozzolanic Reactivity

Pozzolanic reactivity of the bio fly ashes are measured according to Standard SS-EN\_196-5. The results are presented in Figure 10. There is a randomly selected cement and metakaolin sample which is also added to make the comparison easier. As shown, the bio-Fly ashes are very close to the border of acceptance. This indicates that these ashes could even comply with the currently practiced standards with slight modifications.

Moreover, the activity index test is performed on randomly selected bio fly ashes according to ASTM C311 standard. The results are presented in in Figure 11. As illustrated, all the bio ashes are relatively close to the border of acceptance. Figure 12, shows the cast ash prisms after performing the compressive strength test at 28 days.



Figure 9. Particle size distribution results



Figure 10. Pozzolanic reactivity test results









Figure 12. Bio-Fly ashes after compressive strength test at 28 days





## Slutsatser, nyttiggörande och nästa steg

It is worldwide known that production of cement causes 6 % of the global carbon dioxide emissions. Moreover, according to statistics received from Södra Cell Värö, in 2016, 1050 tons of ash has been produced at Värö while predicted production in 2017 is around 1500 tons. Considering that there are much more pulp and paper industries in Sweden with similar production amounts, these materials if proven to be beneficial can to a very good extent support the construction sector to be substituting cement.

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Aiming to enable prevention of landfilling large amount of rest products from pulp and paper industry and reducing environmental foot prints due to cement production, this project was defined to investigate potentials of using bio ashes from pulp and paper industry as an alternative supplementary cementitious material.

Up to 50 different bio ash samples in a period of 4 month has been collected in 3 different locations, where Södra plants are located (Värö, Mönsterås, Mörrum).

The bio ashes where characterized in terms of chemical composition, mineralogy and particle size distribution. Moreover, the pozzolanic reactivity as well as the activity index of this ashes compared to a reference material is investigated.

The results indicate that:

- Bio ashes are showing a relatively high consistency in chemical composition with respect to both time and location.
- The presence of several silicate minerals explains the relatively high SiO<sub>2</sub> content determined by chemical analysis.
- Pore size distribution is relatively similar in bio-fly ashes collected from different locations and comparable with a randomly selected coal fly ash.
- Pozzolanic activity as well as activity index of the bio-flashes is close to the border of acceptance according to SS-EN\_196-5 and ASTM C311 standards, indicating good potentials for further investigations.
- The water bath cooling method causes lower chloride contents in the bio ashes. This indicates a high potential for using simple bath immersion leaching methods to decrease the chloride and sulfate content in bio ashes.

In the next stage it is planned to:

- To investigate potentials of bio ashes from other industries rather than pulp and paper industry.
- To investigate potential of leaching methods for decreasing chloride and sulfate contents in the bio ashes.
- To account for the durability of bio ash containing concrete in terms of carbonation, sulfate and chloride ingress.



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## **Publikationslista**

A journal publication is under preparation. The journal which the article will be sent to is not completely decided yet.

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## Projektkommunikation

RISE CBI cement and concrete research institute is in direct contact with an industrial consortium consisting of Swedish cement and concrete producing industries and is in constant contact with them. The partner consortium has excellent networks to stakeholders as well as end users. CBI has excellent contacts to the Swedish road administrations (Trafikverket) and Norwegian road authorities (Statlig Vegvesen) as well as to the Swedish Boverket via research projects and within national committees. Furthermore, the partners have a strong international network in the construction area via participation in projects funded by the European Commission. All these organizations can be informed about outcomes of the project via:

- CBI information days, a one-day conference (every year) with an audience, which consists to ca. 80 % of representatives from industry and public bodies.
- The Swedish Concrete Association (Betongföreningen), where CBI is holding the secretariat.
- CBI Nytt, a biannual information journal particularly aimed at the Swedish construction industry and by CBI Newsletters (4 times per year).
- CBI webpages.
- Conferences and lectures aimed at end users in the field.

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