



D2.1

Ash characterization and categorization

| | |
|--|--------------------------------------|
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Modification control

| VERSION | DATE | DESCRIPTION AND COMMENTS | AUTHOR |
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| 0.1 | 31.08.2022 | Planning of delivery (part 1 experimental procedures, part 2 selection of ashes, and part 3 lab results) | Lisbeth M. Ottosen. Discussed at Teams meeting 1 |
| 0.2 | 08.09.2022 | Selection of characterization methods | Teams meeting 2 (discussion leader Lisbeth M. Ottosen) |
| 0.3 | 16.01.2023 | Ashes have been selected and sampling reports | Teams meeting 6 |



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| | | made. Presented and discussed. | (discussion leader Lisbeth M. Ottosen) |
|-----|------------|---|---|
| 0.4 | 16.02.2023 | Draft version of procedures (part 1) | Commented by partners |
| 0.5 | 15.05.2023 | Partners deadline for uploads and commenting on documents in Teams folder | All partners |
| 0.6 | 31.05.2023 | Finalizing delivery report | Lisbeth M. Ottosen, Huilin Li, Ebba Schnell, Mie Tybjerg |

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About this delivery

Background

To enable the recycling of the ashes as resources in different applications, it is vital to know the qualities and variations within the different groups of ashes. The characteristics of a specific ash depend on the raw materials and the combustion process, which represents a challenge in transforming the ashes into secondary resources. A comprehensive characterization of the selected incineration ashes will be carried out in terms of chemical composition, physical and chemical properties.

The ashes will be categorized into groups with critical characteristics (e.g., chemical composition/undesired elements and reactivity) taken as criteria. The groups will be rated into different levels. Lower level of a certain group of ashes means more efforts of pre-treatment will be needed before utilization.

The delivery is essential to the whole AshCycle project. The detailed characterization and categorization of the ashes will provide information needed for different utilizations investigated in both WP3 and WP4, as well as in the pilots and replication cases in WP5. Ash characterisation testing will complement the further product testing (WP3,WP4) and environmental checks (WP6), to establish compliance for end use (demonstrators WP5).

The present delivery (D2.1) consist of three parts

1. Collection of laboratory procedures
2. Overview and information of characterized ashes
3. Results from characterisations

The ashes are categorized dependent on their leaching properties

Process in task 2.1

This task had active participation from 11 partners, and the collaboration and progress were ensured through shared documents in a Teams folder and a series of Teams meetings:

- Meeting 1. Planning and introduction (31.08.2022 14.00-15.00)
- Meeting 2. Selection of characterization methods 08.09.2022 09.00-15.30
- Meeting 3. Discussion on draft procedures (1-8) (26.9.2022 09.00-11.00)
- Meeting 4. Discussion on draft procedures (9-16) (27.09.2022 09.30-11.30)
- Meeting 5. Four last procedures discussed (04.11.2022 11.30-13.00)
- Meeting 6. Partners present selected ashes (16.01.23 09.00 – 14.30)
- Meeting 7. Draft version of procedures first characterizations (16.02.2023 09.30-11.00)
- Meeting 8. Sampling procedures, finalization of procedures (13.03.2023 14.00-15.30)
- Meeting 9. Progress and update on characterization (25.04.2023 09.30-11.00)
- Meeting 10. Last uploads at Teams delivery 2.1 (15.05.2023 13.30-14.00)
- Meeting 11. Dissemination (23.05.2023 11.00-12.00)



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The first 5 meetings focused on selection of experimental procedures in the common ash characterization program. It was important to obtain consensus on the procedures so we characterize all the ashes in the project in the same way. This is necessary in order to be able to compare results and not at least in the replicates of pilots.

From meeting 6 to 8 the ash sampling and experimental work were simultaneous to updating the characterization procedures to avoid uncertainties. Meeting 9 and 10 focused on the progress and results for the characterizations, and meeting 11 focused on dissemination of the results (we plan journal paper(s) and publication of the obtained data an open source to be submitted in the autumn 2023).

Part. 1 The characterization program

Table 1 gives an overview of the 17 methods selected to the ash characterization program. The procedures inclusive safety instructions are all compiled in part 1 of this delivery-report. Different partners have worked together on the specific procedures, and the authors are stated in connection to each procedure.

Table 1: selected methods in the characterization program

| Category | Characteristic | Method | Standard |
|----------------------|---|--|---|
| Sampling | Sampling protocol guidelines Homogenization and reduction of sample size | | EN 197-7; EN 932-1 |
| Chemical composition | Concentration of major elements Concentration of heavy metals and REEs Mineralogy and amorphous phase Estimation of organic and carbonate content Total organic content (TOC) | X-ray fluorescence (XRF) Acid digestion in HNO ₃ , HClO ₄ and HF. ICP-OES measurement X-ray powder diffraction (XRD) Loss on Ignition at 550 °C and 950 °C TOC analyzer | EN 15935:2021 EN 15936:2022 |
| Chemical properties | Water content, pH, conductivity, leaching, solubility Reactivity Reactivity - Bound Water Free CaO content Alkali dissolution | Measurements in suspensions of ash in distilled water L:S 1:10 Cumulative heat determination by isothermal calorimetry Paste samples and weight loss at 350 °C Chemical extraction and titration Dissolution in 4M NaOH and ICP-OES analysis | ASTM C1897-20 ASTM C1897-20 |
| Physical properties | Bulk density Density Particle size distribution (>250 µm) Particle size distribution (<250 µm) Specific surface area of solids | Powder volume and weight Pychnometer Sieving Laser diffractometer Gas adsorption – BET method | EN 1097-3:1998 EN 1097-6 EN 933-1:2012 ISO 13320, ISO 14487, ISO 9276- ISO 9277 |

We decided to have two characterization modes: (I) Screening, which excludes three methods (TOC, specific surface area, and mineralogy), and (II) Full characterization, which includes all methods. The screening is done for potential ashes to have a pool for selecting from in the other WPs. Full characterization is done for all ashes selected in the other WPs.

Part 2. Selected Ashes

Ashes were selected from the different partners, and all together the selection included 15 MSWI ashes, 32 wood ashes, 10 sewage sludge ashes, 27 co-combustion ashes and 1 other. Available information which can be shared



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publically on the incineration plants was filed in tables in the Teams folder. Part 2 of this report is compiled from these tables.

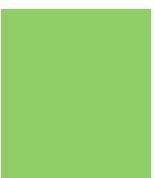
Part 3. Ash characterisation

In part 3, the sampling reports are given together with tables with the results from the experimental analysis.



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Part. 1 The characterization program



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Ash characterization procedures

WP2 – Task 1

Task 2.1 AshCycle
5-22-2023



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1 Introduction

A variety of ashes with different characteristics are produced all over Europe. The ashes are largely underutilized and often used as backfill in mines or landfilled. The ashes originate, e.g., from the combustion of biomass, sewage sludge, or waste.

In support of a resource-efficient society based on circular economy principles, secondary resources, such as ashes, must be utilized at the highest possible level. Some of these ashes contain valuable and scarce chemical elements, which should be extracted and recovered to reduce the mining of the same elements from nature. Many examples in the literature show that ashes can replace natural raw materials in the production of construction materials; however, sometimes only after pretreatment. There are many possibilities for transforming the ashes into secondary resources.

The ash characteristics depend on the fuel type, combustion type, and combustion parameters. Thus, the ashes grouped into a main group (e.g., wood ash or sewage sludge ash) vary significantly. This means that in order to exploit the full potential of the ashes as resources, it is necessary to systemize the knowledge of ash characteristics and categorize the ashes into possible use categories based on this.

In literature, there is no generally accepted scheme for the experimental characterization of ashes, and the methods often vary slightly, so a direct comparison of the results is doubtful. This calls for a common characterization scheme to compare research results obtained at different institutions.

The present collection of ash characterization procedures is a scheme developed and agreed on among the partners of the European project AshCycle – Integration of underutilized ashes into material cycles by industry-urban symbiosis (Grant No. 101058162).

The researchers who were responsible for the description of the procedure in each chapter are mentioned there. The whole group of researchers who met and decided on this characterization scheme are:

Technical University of Denmark: Lisbeth M. Ottosen (lead of the task), Gunvor M. Kirkelund, Ebba Schnell. **Technical University of Delft, The Netherlands:** Guang Ye, Boyu Chen and Farnaz Aghabeyk. **University of Oulu, Finland:** Priyadarshini Perumal & Tero Luukkonen. **University of Zagreb, Croatia:** Nina Štirmér, Ivana Carević, Jelena Šantek Bajto, Ivana Banjad Pečur and Ivan Gabrijel. **Slovenian National Building and Civil Engineering Institute:** Sabina Dolenc, Lea Žibret, Vilma Ducman & Sara Tominc. **DOK.ING ENERGO, Croatia:** Danica Maljković, Edi Kirasić, and Morana Drušković. **NEXE ltd., Croatia:** Ana Grahovac, Renata Bunjevac & Zvonko Kekez. **Beton Lučko, Croatia:** Tanja Jelenić & Suzana Hozmec. **SECO, Belgium:** Wouter Fock. **Veolia, Belgium:** Vim Vermeire & Peter De Vylder. **Mineralz B.B., The Netherlands:** Marc Brito van Zijl & Rob Bleijerveld

2 Sampling

2.1 Sampling protocol guidelines

Sabina Dolenc, ZAG

Background and principle

Sampling protocol (according to previous projects and also following standards EN 197-7 and EN 932-1)

Step 1 Sampling permission

Contact the owner of the material which will be sampled and gather permission for sampling and ask them for permission to publish the data. Determine the date of sampling.

The aim of sampling is to obtain a bulk sample that is representative of the average properties of the batch.

Step 2 Sampling details

Proper and careful sampling and sample transport is a prerequisite for an analysis that will give reliable results. The person conducting sampling needs to be informed of the aim of the sampling. Furthermore, the correct use of the specified apparatus helps to avoid biased sampling. Sampling variation caused by the heterogeneity of the batch is reduced to an acceptable level by taking an adequate number of sampling increments (a quantity of material taken from a batch by one operation of the sampling apparatus). If the material is homogenized by production processes, one large increment may be representative of the batch.

Sampling from stockpiles

Sampling increments of approximately equal size shall be taken from different points at different heights or depths, distributed over the complete stockpile. The location and number of sampling increments shall take into account the way in which the stockpile was built, its shape and the possibility of segregation within the stockpile. A gravitational separation of material on stockpiles and ponds shall be considered as well as size of deposited particles. A sampling increment shall be taken from the deepest point of each hole. Composite sample need to be composed of minimum ten sampling increments taken from the deposit or stockpile in a manner to be representative for the deposited material or area. Sampling increments are selected at random from all parts of sampling area. Procedures showed on Figure 1 can serve as guidelines to collect a representative composite sample. Exact sampling procedure and distribution of taken sample increments might depend from place to place. For materials with larger grains, a larger amount of sample shall be collected to achieve appropriate homogeneity. At places with ongoing production, an appropriate temporal factor shall be considered.

Collect at least one composite sample from each sampling location/material determined for sampling. Equipment for sampling is not predefined – please use equipment adequate for the type of the sampling material. Avoid equipment that might cause sample contamination.

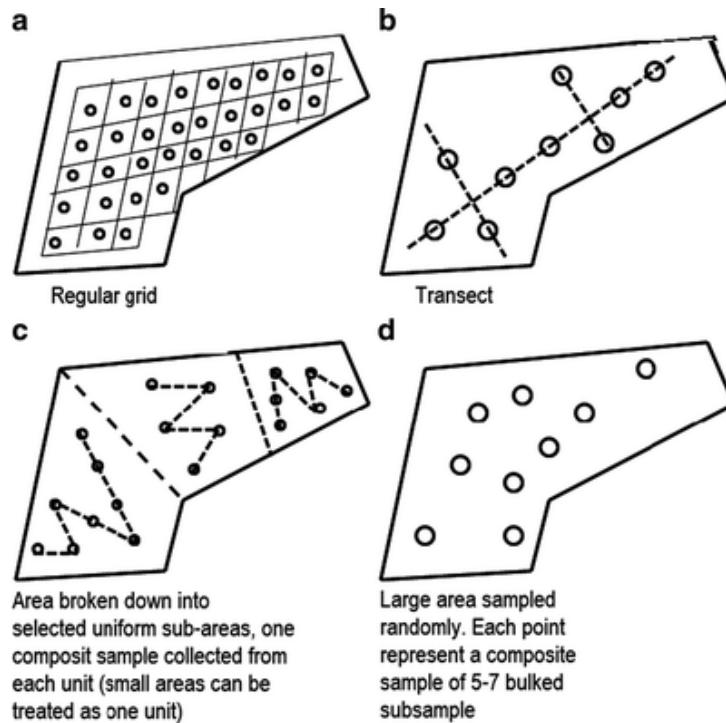


Figure 1: Some possibilities on how to get a representative composite sample.

Sampling from silo

Sampling at an outlet shall be carried out in accordance with “sampling at belt and chute discharge points”.

The silo shall be opened sufficiently to ensure a uniform material flow without segregation; this requires an aperture at least three times the diameter of the maximum grain diameter. For aggregates with lower size above 32 mm an aperture of at least 200 mm is required.

Sampling from stationary conveyor belt

All sampling increments shall be taken at the same sampling point. The material shall be taken across the complete cross section of the belt in every sampling increment.

Note: The sampling frame should be used to separate the material that is to be taken as the sampling increment at the sampling point. As an alternative to the use of the sampling frame a shovel or a flat piece of metal can be used to separate the sampling increment at both ends from the material remaining on the belt. This increment should have a length of about three times the width of the material stream on the belt or a minimum mass as calculated according to the equation 1.

Sampling at belt and chute discharge points

Note: Mechanical apparatus provides the most practical means of taking samples from belt and chute discharge points. Manual sampling should be avoided, if possible, due to both errors and the dangers involved.

The period during which the sampling is to be done shall be divided into a number of equal intervals, and a sampling increment shall be taken in the middle of each interval.

A sampling increment shall be taken by passing the sampling receptacle, e.g., the sampling box through the discharge stream in a uniform movement, making sure that the complete cross section of the stream of material is intercepted.

Step 3 Recording sampling details

Sampling procedure will depend on the material sampled (MSWI, wood ash, sewage sludge ash, co-combustion ashes, other types of biomass) and the conditions on the field (stockpile, pond, waste dump, etc.). **Therefore, a detailed description of the sampling procedure should be recorded on the ASHCYCLE sampling report template (see Appendix).**

Equipment adequate for the type of the sampling shall be used to take a sample increments, observing the following precautions as appropriate.

Use of personal protective equipment (PPE) such as gloves, helmet, boots, reflective vest etc. is required. Manual sampling with shovels or scoops etc. shall not be applied from moving objects (conveyer belts for instance). Sampling activities must in accordance with national safe work regulations!

Step 4 mixing and homogenizing the sampled ash

Subsamples need to be mixed and homogenized in the plastic container or big PVC bag on the field. **A homogenized composite sample shall be of such a size that all the tests, wherever specified, can be carried out twice.** The total quantity to be taken shall be greater than or at least equal to that required for supplying to all the laboratories concerned.

Calculate the minimum amount of sample using the equation to ensure minimum 25 kg of sample

Add the total amount of sample to the appendix: ASHCYCL sampling report template

Note: It is recommended that the minimum mass of a bulk sample be calculated in accordance with the following equation 1:

$$M = 6 \times \sqrt{D} \times \rho_b$$

where:

M = the mass of the sample, in kilograms;

D = the maximum grain size, in millimeters;

ρ_b = the loose bulk density, in megagrams per cubic metre, determined as specified in prEN 1097-3.

Step 5 Marking of samples

The samples or containers shall be clearly and durably marked.

Marking shall include:

- unique code/ID
- identification of the laboratory samples: place of sampling, date of sampling, and designation of the material – send this documentation via e-mail. Along with the shipment put only the necessary data for carrier company and customs procedure (do not mark the type of material and planned analyses in the shipment)
- permanently mark the samples and at least two different ways of sample marking is highly recommended:
 - with permanent marker on the PVC sample bag and container,
 - with a permanent marker on the sticker adhered on the PVC sample bag and container,
 - with a permanent marker on the paper sheet wrapped in PVC bag, sealed with the tape and inserted in the PVC sample bag,
 - with a sticker on a bag and/or container,
 - any other manner that will withstand transportation and handling of samples;

Regarding sample IDs following codes are suggested for different materials:

| Country codes: | Fuel codes: | Ash type codes: |
|------------------|--|----------------------------|
| FI - Finland | MSWI- Municipal Solid Waste Incineration Fly Ash | MA – Mix Ash |
| DK - Denmark | WA – Wood Ash | FA – Fly Ash |
| BE - Belgium | SSA – Sewage Sludge Ash | BA – Bottom Ash |
| NL - Netherlands | CC - Co-combustion ashes | APC – Air polluted Control |
| HR - Croatia | PSFA – Paper Sludge Fly Ash | |
| SI - Slovenia | <i>Add and describe code if needed</i> | |

Final sample ID is combination of mentioned codes with a sequence #country.fuelcode.ashtype.number. For example: #DK.WA-FA.1 (Denmark-wood ash-Fly Ash-sample 1), #NL.SSA-MA.1(Netherlands-sewage sludge-Mix Ash-sample 1), #FI.CC-BA.1(Finland-co-combustion ash-Bottom Ash-sample 1) etc.

The laboratory samples shall be packed and transported in such a way that their condition at the time of sampling is preserved. Material shall be packed in clean containers such that fineness cannot be lost during transportation. The containers shall be given an air-tight closure.

Step 6 Sampling report

Record the sample location (company/site) and fill the sampling record sheet.

Sampling report need to be prepared (appendix: Sampling report template) for each laboratory sample or for each group of laboratory samples from a single source.

The sampling report shall include:

- The sampling report identification (serial number)
- The laboratory sample identification mark(s)
- The date and place of sampling
- Size of the batch
- Sampling point or identification of the batch sampled
- The name of sampler(s)
- Mark any important notices about sampling, sampling procedure, sampling conditions etc.

Depending on the circumstances other information can be relevant.

Step 7 Photo

Ask for permission to take photographs of:

- wider sampling area (deposit, site or stockpile from where the sample was taken),
- detailed picture of the sampling micro locations
- picture of packed composite sample

ID of pictures shall follow the guidelines for sample marks with additional field indicating position of photography – for instance DK.WA-FA.1-01, DK.WA-FA.1-02 etc.

Make also some pictures during sampling procedure as they can be used in presentations and documents. If possible and legally acceptable make a short videos of sampling or overviews over sampling areas.

Step 8 Fill ash sampling template

Fill the ASHCYCLE sampling report template (see Appendix) with required data and send it by email to the receiver. Make a list of taken pictures and a folder with these photographs.

Provide these files also to the coordination T1.2 - DTU, which will afterward distribute data to all partners which will be involved in further processes.

References

Sampling protocol guidelines Version 2. RIS ALiCE: Al-rich industrial residues from mineral binders in ESEE region.

Alakangas, E., 2015. Quality guidelines for wood fuels in Finland VTT-M-04712-15. Technical report.

Park, Y., 2008. Moisture and Water Activity. In book: Handbook of Processed Meats and Poultry Analysis (pp.35-67). DOI:[10.1201/9781420045338.ch3](https://doi.org/10.1201/9781420045338.ch3)

Appendix: ASHCYCLE sampling report template

| | |
|--|--|
| Sample ID: | |
| Company/Site: | |
| Country: | |
| Owner of the material: | |
| Type of material (CODE): | |
| Sampling performed by: (person and institution) | |
| Amount of sample (kg): | |
| Date for sampling: | |

Orthophoto image of the sampling area (Google Earth for instance)
Mark the exact sampling locations on the image

| |
|---|
| Sample description, sampling procedure and observation: |
| |

| |
|---------------------|
| Photographs: |
| Ex. DK.WA-Fa.1-01 |

| |
|---------------|
| Notes: |
| |

2.2 Reduction of sample size and representative sample

Sabina Dolenec, ZAG & Wouter Fock, SECO

Gather information

Make an overview of all tests to be performed on the sample. Get information on:

- Sample mass needed for each test;
- Granulometry needed for each test
- Special conditions (air dry, oven dry, ...)

Prepare a plan

Combine the test masses with equal or similar requirements. Often, the workload can be reduced by combining the preparation for several tests. This will also reduce the amount of sample that will be wasted during preparation.

The actions will consist in a sequence of quartering and crushing/milling, possibly combined with drying.

Sample Reduction

There are two ways of reducing the sample size: “coning and quartering” and the “riffle box”.

Coning and quartering

For the reduction of a bulk sample (air dried), the “coning and quartering” protocol can be used (Figure 1 and Figure 2).

Place the bulk sample in homogenization or hand mix it in sack/bag. Put homogenized bulk sample on the working surface and mix it thoroughly by heaping it up to form a cone and turning it over with the shovel to form a new cone. Repeat this operation three times. When forming the cones, deposit each shovelful on the peak of the new cone in such a way that the material runs down all sides of the cone and is evenly distributed so that the different sizes become well-mixed. Flatten the third cone, by inserting the shovel repeatedly and vertically into the peak of the cone, to form a flat heap which has a uniform thickness and diameter. Quarter the flat heap along two diagonals intersecting at right angles. Discard one pair of opposite quarters and store them as back-up for future analysis. Shovel the other two quarters into a stockpile. Repeat the operation until the ultimate two quarters correspond to the required mass.

Use of personal protective equipment (PPE) such as glasses, mask, gloves, lab coat, etc. is suggested.

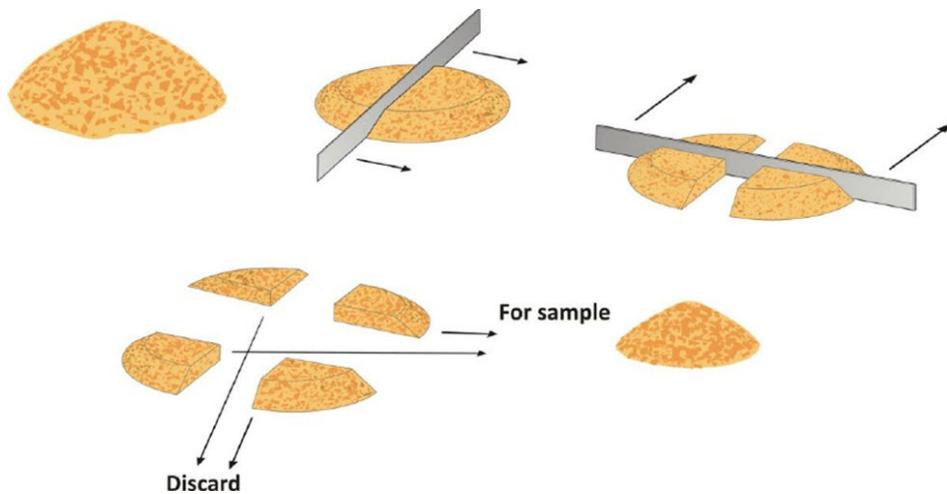


Figure 1: Coning and quartering procedure (source: Alakangas, 2015).

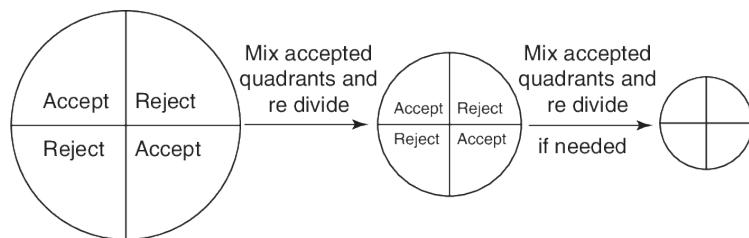


Figure 2: Reduction of a sample by quartering (source: Park, 2008).

Riffle splitter

For the reduction of a bulk sample (air dried), a riffle box can be used (Figure 3).

A riffle box is a device designed to reduce a sample aggregate to half its original size. The box contains a number of chutes discharging alternately to opposite sides. The width of the chutes varies according to the largest particle size. The volume reduction is rapid for dry material of suitable fineness.

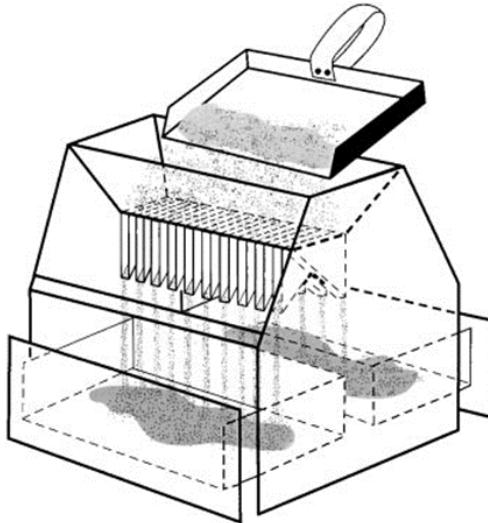


Figure 3: Riffle box (source: www.researchgate.net)

Storage

Sample needs to be packed in a PVC bag of suitable size and sealed with tape. It is desirable to put the sealed PVC bag into a plastic container of appropriate size and seal it again.

Store also the material that is discarded during the quartering or splitting process. If a crushing or milling process was inserted between two quartering or splitting processes, avoid mixing original and crushed granulometries. Store them separately with proper marking.

All required documentation for sending and customs procedure shall be provided by the institution which performed sampling (with assistance of the companies who own the material deposits).

References

Alakangas, E., 2015. Quality guidelines for wood fuels in Finland VTT-M-04712-15. Technical report.

Park, Y., 2008. Moisture and Water Activity. In book: Handbook of Processed Meats and Poultry Analysis (pp.35-67). DOI:[10.1201/9781420045338.ch3](https://doi.org/10.1201/9781420045338.ch3)

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Pretreatment of ash before use

The amount is per sample for each method after quartering.

| Method | Ash used in procedure | Amount of ash |
|--|---|------------------------------------|
| 3.1 X-ray fluorescence (XRF) | Dried at 105°C and mill/grind to 10µm (feel like flour) | 10 g |
| 3.2 Concentrations of heavy metals, REE's and Hg | Dried at 105°C and grind to < 63µm | 15 g |
| 3.3 X-ray powder diffraction (XRD) | Dried at 105°C and mill/grind to 10µm (feel like flour) | 2 g |
| 3.4 Loss on ignition | Dried at 105°C | 2.5 g |
| 3.5 Total organic content (TOC) | Dried at 40°C and sieved <250µm | 3 g |
| 4.1 Water content, pH, conductivity, Water solubility | Untreated as received ash | 20 g |
| 4.2 Reactivity (cumulative heat determination by isothermal calorimetry) | Dried at 105 °C and grind <125 µm | 10 g |
| 4.3 Reactivity (Bound water) | Dried at 105 °C and grind <125 µm | 10 g |
| 4.4 Free CaO content | Dried at 105°C and grind to < 63µm | 1-1.5 g |
| 4.5 Alkali dissolution | Dried at 105°C and grind to <10µm | 1 g |
| 5.1 Bulk density of ashes | Untreated as received ash | Depending on container and ash |
| 5.2 Density of ashes using pycnometer | Dried at 105°C and sieved <4mm | 300 g after sieving |
| 5.3 Particle size distribution (sieving >250µm) | Dried at 105°C | Depending on grain size of the ash |
| 5.4 Particle size distribution (laser <250µm) | Dried at 105°C and sieved < 250µm | 10 g |
| 5.5 Specific surface areal of solids by gas absorption (BET) | Untreated as received ash | 2 g |
| 5.6 Determination of metallic Al content | Dried at 105°C and sieved <125µm | 5-10 g |

3 Chemical composition

3.1 X-ray fluorescence (XRF) analysis

Boyu Chen, Delft University of Technology

Background and principle

X-ray Fluorescence (XRF) is a non-destructive analytical technique used for elemental analysis and chemical analysis. During the measurement, a prepared sample specimen is excited by the radiation of an X-ray, resulting in the emission of characteristic X-ray fluorescence radiation (such as K_α or K_β) for each element. The signals created by these X-rays are collected and used to determine the composition of the sample. The elements that can be detected by XRF range from sodium (Na) to uranium (U).

Special equipment

- X-ray fluorescence (XRF) spectrometer.

Chemical safety

- The person who operates the machine should have a radiation safety training for X-ray.

Reagents

- No chemical is used.

Procedure

Dry as-received ashes at 105 °C till there is no mass change.

Grind ash sample into homogeneous fine powder, same particle size as the powder used for XRD analysis (around 10 µm)

Please note: Keep track of how many kilograms of sample you take for sample preparation. If you find a big difference in XRF results between 1kg of sampling and 2kg of sampling, just take more samples until you have stable results.

Reporting of result

The XRF results should be presented in the form of oxides. The data need to be normalized to 100 wt.%. The following is the example:

Table 1 Chemical composition of MSWI bottom ash

| Compounds (wt.%) | MSWI bottom ash |
|--------------------------------|-----------------|
| SiO ₂ | 46.53 |
| CaO | 15.86 |
| Al ₂ O ₃ | 13.68 |
| Fe ₂ O ₃ | 10.79 |
| Na ₂ O | 3.20 |
| K ₂ O | 0.97 |
| SO ₃ | 1.80 |
| Cl | 0.40 |
| P ₂ O ₅ | 1.14 |
| MgO | 2.50 |
| ZnO | 0.81 |
| CuO | 0.52 |
| TiO ₂ | 1.11 |
| MnO | 0.31 |
| PbO | 0.12 |
| Cr ₂ O ₃ | 0.09 |
| BaO | 0.05 |
| NiO | 0.04 |
| SrO | 0.05 |
| SnO ₂ | 0.02 |
| Sum | 100 |

3.2 Concentrations of heavy metals, REEs and Hg

Sabina Dolenec & Lea Žibret, ZAG

Background and principle

Concentrations of heavy metals, REEs and Hg by ICP-ES/MS, provided by external laboratory: <https://www.bvna.com/mining-laboratory-services>.

Method: ULTRA TRACE BY ICP-ES/MS (MA250)

Analysed elements (59 elements) with detection limits and upper limits are shown in Figure 1.

| ULTRA-TRACE BY ICP-ES/MS | | | |
|--|------------------------------|--|-------------|
| CODE | ELEMENT | DETECTION LIMIT | UPPER LIMIT |
| MA250 | | | |
| | | Ultra-trace ICP-ES/MS, 59 elements, 0.25 g | |
| | Ag | 20 ppb | 200000 ppb |
| | Al | 0.01 % | 20 % |
| | As | 0.2 ppm | 10000 ppm |
| | Ba | 1 ppm | 10000 ppm |
| | Be | 1 ppm | 1000 ppm |
| | Bi | 0.04 ppm | 4000 ppm |
| | Ca | 0.01 % | 40 % |
| | Cd | 0.02 ppm | 4000 ppm |
| | Ce | 0.02 ppm | 2000 ppm |
| | Co | 0.2 ppm | 4000 ppm |
| | Cr | 1 ppm | 10000 ppm |
| | Cs | 0.1 ppm | 2000 ppm |
| | Cu | 0.1 ppm | 10000 ppm |
| | Dy | 0.1 ppm | 2000 ppm |
| | Er | 0.1 ppm | 2000 ppm |
| | Eu | 0.1 ppm | 2000 ppm |
| | Fe | 0.01 % | 60 % |
| | Ga | 0.02 ppm | 100 ppm |
| | Gd | 0.1 ppm | 2000 ppm |
| | Hf | 0.02 ppm | 1000 ppm |
| | Ho | 0.1 ppm | 2000 ppm |
| | In | 0.01 ppm | 1000 ppm |
| | K | 0.01 % | 10 % |
| | La | 0.1 ppm | 2000 ppm |
| | Li | 0.1 ppm | 2000 ppm |
| | Lu | 0.1 ppm | 2000 ppm |
| | Mg | 0.01 % | 30 % |
| | Mn | 1 ppm | 10000 ppm |
| | Mo | 0.05 ppm | 4000 ppm |
| | Na | 0.001 % | 10 % |
| | Nb | 0.04 ppm | 2000 ppm |
| | Nd | 0.1 ppm | 2000 ppm |
| | Ni | 0.1 ppm | 10000 ppm |
| | P | 0.001 % | 5 % |
| | Pb | 0.02 ppm | 10000 ppm |
| | Pr | 0.1 ppm | 2000 ppm |
| | Rb | 0.1 ppm | 2000 ppm |
| | Re | 0.002 ppm | 100 ppm |
| | S | 0.04 % | 10 % |
| | Sb | 0.02 ppm | 4000 ppm |
| | Sc | 0.1 ppm | 200 ppm |
| | Se | 0.3 ppm | 1000 ppm |
| | Sm | 0.1 ppm | 2000 ppm |
| | Sn | 0.1 ppm | 2000 ppm |
| | Sr | 1 ppm | 10000 ppm |
| | Ta | 0.1 ppm | 2000 ppm |
| | Tb | 0.1 ppm | 2000 ppm |
| | Te | 0.05 ppm | 1000 ppm |
| | Th | 0.1 ppm | 4000 ppm |
| | Tl | 0.001 % | 10 % |
| | Tl | 0.05 ppm | 10000 ppm |
| | Tm | 0.1 ppm | 2000 ppm |
| | U | 0.1 ppm | 4000 ppm |
| | V | 2 ppm | 10000 ppm |
| | W | 0.1 ppm | 200 ppm |
| | Y | 0.1 ppm | 2000 ppm |
| | Yb | 0.1 ppm | 2000 ppm |
| | Zn | 0.2 ppm | 10000 ppm |
| | Zr | 0.2 ppm | 2000 ppm |
| AQ200-Hg | | | |
| | Aqua Regia ICP-ES/MS, Add-on | | |
| | Hg | 0.01 ppm | 50 ppm |
| Digestion is partial for some Cr and Ba minerals and oxides of Al, Fe, Hf, Mn, Sn, Ta, Zr and REEs. Volatilization As, S, Se and Sb. | | | |

Figure 1: Analysed elements with detection limits and upper limits.

Special equipment

- ICP-ES/MS
- Laboratory equipment for acid digestion.

Chemical safety

- Nitric acid: Corrosive, flammable, and acute toxicity
- Perchloric acid: Corrosive, flammable, health hazard, serious health hazard
- Hydrofluoric acid: Corrosive and acute toxicity
- Hydrochloric acid: Corrosive and health hazard

Read the SDS before use

Reagents

Use only concentrated reagents

- Nitric acid – [HNO₃]
- Perchloric acid – [HClO₄]
- Hydrofluoric acid – [HF]
- Hydrochloric acid – [HCl]

Procedure

Take approximately 100 g of dry (105 °C) sample using a sample divider or by quartering. Sieve the portion on a 125 µm sieve until the residue remains constant. Grind the retained materials so that it completely passes the 125 µm sieve.

Sample preparation: dry sample, granulation < 63 µm

Sample needed: min. 15 g

Acid digestion: 0.25 g split is heated in HNO₃, HClO₄ and HF to fuming and taken to dryness. The residue is dissolved in HCl.

The amount of sample for analysis: 0.25 g

Prepared sample is digested to complete dryness with an acid solution of (2:2:1:1) H₂O-HF-HClO₄-HNO₃. 50% HCl is added to the residue and heated using a mixing hot block. After cooling the solutions are transferred to test-tubes and brought to volume using dilute HCl. Sample splits of 0.25g are analyzed.

Reporting of result

Results are reported in ppm (or ppb, %).



References

Schedule of services & fees:

<https://www.bvna.com/sites/g/files/zypfnx386/files/media/document/Bureau-Veritas-USD-Fee-Schedule-2022%281%29.pdf>.

3.3 X-ray powder diffraction (XRD) analysis

Lea Žibret, ZAG & Boyu Chen, Delft University of Technology & Gunvor Marie Kirkelund, DTU Sustain, University of Denmark

Background and principle

X-Ray Powder Diffraction (XRPD) is one of the most important techniques for the identification of crystalline materials. Combined with a quantitative method for determination of the amount of amorphous phase. Using an X-Ray diffractometer, X-rays are focused on a sample and the diffracted X-rays are collected into a diffractogram. Due to the minerals' unique d-spacing, and based on Bragg's law, the minerals can be identified, when comparing the diffractogram (d-spacing) of an unknown sample to the known d-spacing of known minerals from a database.

Special equipment

- X-ray diffractometer

Chemical safety

- X-rays are hazardous and precaution should be taken. Modern X-ray diffractometers comply with stringent safety regulations, so there is no chance of radiation for users and no special safety measures are needed.

External standard

- NIST standard SRM 1878b - Respirable Alpha Quartz (Quantitative X-Ray Powder Diffraction Standard). Can be bought at www.shop.nist.gov product no. 1878b

Procedure

Sample preparation

Grind sample into fine powder, around 10 µm (to have as sharp peaks as possible in the XRD). Same preparation method as for XRF.

Mill the sample mechanical and then grind by hand 5-10 minutes. The sample must feel like flour.

Measuring sample

Measure sample from 20° up to 700 2 theta or as low as the apparatus can go to include AFt and AFm peaks below 100 2 theta

Suggested settings: 45 kV and 40 mA, stepsize 0.01 degree/2 sec, approx. 20 min (can be increased, depends on the instrument and how to get good spectrum)

Interferences

Fe-bearing crystalline material give low signal by Cu-X ray tube, e.g. Goethite, Lepidocrosite, Hematite...

Qualitative phase identification

Use available software (HighScore, Topas, Eva etc.)

List the PDF card number connected to identified phases and the Database

Quantitative (QXRD) – amorphous phase determination

Measure the external standard at exactly the same time and conditions as other samples

A short description of the External standard method procedure

In HighScore (only new versions enable external standard method) first open the xrd-pattern of the external standard measured. Determine background, search peaks and find appropriate crystal modification.

Set K-factor of the quartz NIST 1878b for the standard (CustomizeàProgram SettingsàFitting/Rietveld). Then do the Rietveld refinement. The value for standard will be set to 100% and the amorphous content 0%.

Afterwards open the xrd-pattern of the sample and perform the analysis (determine background, search peaks, find appropriate crystal modifications (all peaks needs to be resolved) and perform the Rietveld refinement).

Agreement indices (makes sense only if the correct phases are selected):

- R-profile, Weighted R-profile (generally values of Weighted R-profile between 5 and 15 give acceptable results)
- Goodness of Fit (GOF): theoretically should be 1, in practice the values GOF up to around 5 are acceptable

Reporting of result

Qualitative results

Show diffractograms with identified peaks

List the PDF card number connected to identified phases and the Database

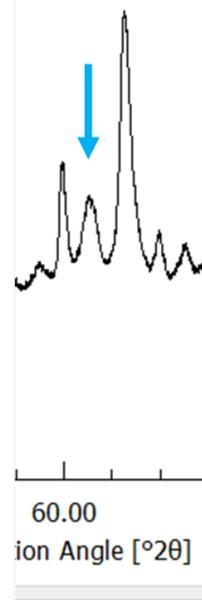
Some examples of good and bad peaks:

Not good sample preparation
The peak is not sharp

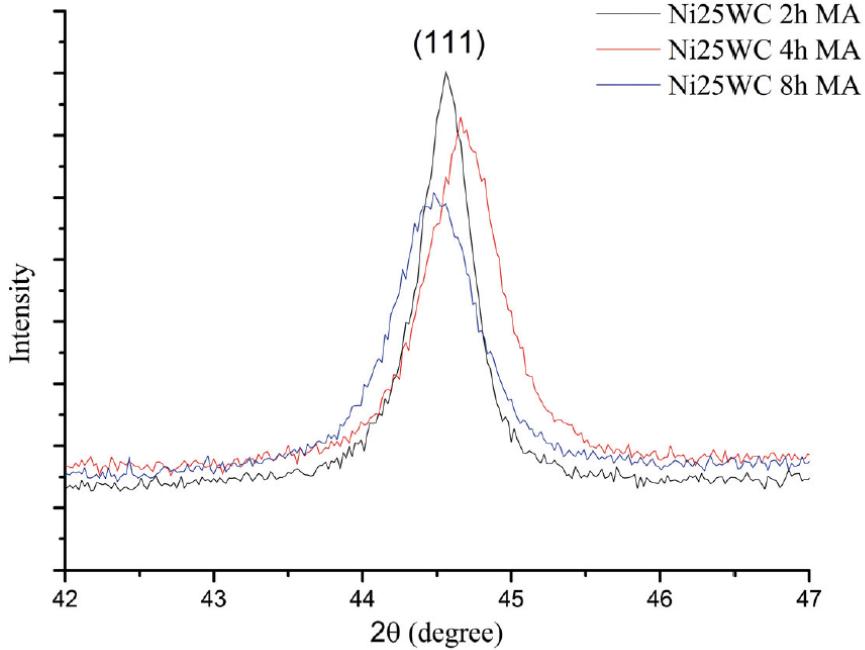


Good sample preparation
The peak is sharp

After grinding for
a longer time



Below, the black peak is preferred over the red and blue, to have sharp peaks avoid long-time or high-energy milling.



Quantitative results

% amorphous phase

Overview of AshCycle partners' XRD equipment

| Partner | XRD instrument | Software | Database | QXRD |
|--|---|--|---|-------------------------------|
| DTU | PanAlytical X'Pert PRO Θ-Θ (CuK α radiation) | HighScore | ICDD-2 | External standard |
| ZAG | PANalytical Empyrean (CuK α radiation) | HighScore | ICDD PDF4+ 2016 RDB powder diffraction file | External standard |
| TUDelft | Bruker D8 (Cu K α radiation) | Eva (Bruker software) and Profex (free software) | ICDD PDF 4+ 2022 | Internal standard (Si powder) |
| Oulu | Rigaku Smartlab (Cu-K α) | PDXL V.2 (Rigaku, Japan) | PDF-4+ 2020 | External standard |
| UNIZG – all ashes will be tested in ZAG for mineralogy | | | | |

3.4 Loss on Ignition

Lisbeth M. Ottosen, DTU Sustain, Technical University of Denmark

Background and principle

This document specifies a method for the determination of the loss on ignition (LOI) at 550 °C and 950 °C.

Sequential LOI at these two temperatures is a common and widely used method to estimate the organic and carbonate content. LOI at these two temperatures are characteristics often reported in the scientific literature dealing with incineration ashes.

Exposure time (if too short) and sample size (if too large compared to exposure time) can influence the result [1]. The exposure time and sample size of this procedure are inspired from EN 15935:2021 Soil, waste, treated biowaste and sludge – Determination of loss on ignition.

Special equipment

- Muffle furnace (1200°C)
- Porcelain crucibles

Chemical safety

No use of chemicals.

Reagents

No use of chemicals.

Procedure

Place the crucibles in a 550°C furnace for 30 min to remove dirt (1h if the furnace is cold).

Place the crucibles in a desiccator (with a desiccant) for cooling to room temperature. Use a tool such as e.g. a pincer when moving the crucible from one place to another.

Weigh the crucibles on an analytical balance with 4 digits.

Weigh 2.5 g of dry ash (dried at 105°C) into the crucible using the analytical balance.

Place the crucible in the furnace for 2 hours at 550°C.

Place the crucibles with glowed ash in a desiccator (with a desiccant) for cooling to room temperature.

Weigh the crucibles with glowed ash.

The crucible with the ash glowed at 550°C is now placed in the furnace for 2 hour at 950°C.

Place the crucibles with glowed ash in a desiccator (with a desiccant) for cooling to room temperature.

Weigh the crucibles with glowed ash.

NB: Do not touch the crucibles during the procedure. The fingers may marks that can change the weight.

For each ash in AshCycle we make three replicates.

Reporting of result

The loss of ignition is expressed as a percentage of dry matter the dry matter:

$$\% LOI_{550} = \frac{(Dw_{105} - Dw_{550})}{Dw_{105}} \cdot 100$$

$$\% LOI_{950} = \frac{(Dw_{105} - Dw_{950})}{Dw_{105}} \cdot 100$$

where:

Dw_{105} = total mass of the dried specimen after 105°C

Dw_{550} = total mass of the glowed specimen after 550°C

Dw_{950} = total mass of the glowed specimen after 950°C

From [1] is suggested:

$$\text{Estimated organic content} = LOI_{105}$$

$$\text{Estimated Carbonate content} = (LOI_{950} - LOI_{550}) \cdot 1.36$$

References

- [1] Heiri, O., Lotter, A.F., Lemcke, G. (2001) Loss on ignition as a method for estimating organic and carbonate content in sediments: reproducibility and comparability of results. Journal of Paleolimnology 25: 101–110, 2001.

3.5 Total organic content (TOC)

Sabina Dolenec & Lea Žibret, ZAG

Background and principle

Important ash property is the content of total organic carbon (TOC) that consists of both volatile carbonaceous matter and unburned carbon because it not only reflects combustion efficiency but also leads to adverse impacts on ash disposal or utilization (1). For instance, in the context of landfill, the EU Council decision (2003) requires the concentrations of TOC to be <6% for hazardous waste, <5% for non-hazardous waste, and <3% for inert waste (2).

To quantify unburned carbon (or TOC in the absence of volatile carbonaceous matter) in ash residues from coal and/or biomass combustion, methods based on thermogravimetric analysis (TGA) have been established (1, 3–6) in addition to the simple but less accurate loss-on-ignition method (7).

Comparison of results between different methods is also discussed (8–11).

EN 15936:2022 specifies two methods for the determination of TOC in sludge, treated bio-waste, soil and waste samples containing more than 0.1% carbon in relation to the dry mass: indirect procedure and direct procedure. The method can also be applied to other environmental solid matrices. In this protocol “direct procedure” is described, in which total inorganic carbon (TIC) is removed from the sample by acid.

Special equipment

- TOC analyser
- Ceramic vessels.



Figure 1: TOC analyser at ZAG.

Chemical safety

- Hydrochloric acid: Corrosive and health hazard

Read the SDS before use

Reagents

Use only concentrated reagents

- Hydrochloric acid - [HCl]

Procedure

Sample pre-treatment

Sample pre-treatment according to EN 16179 or EN 15002, if not specified (homogenization, phase/fraction separation, drying and sub-sampling).

Particle size: < 250 µm.

Drying: temperature should not exceed 40°C.

Mass of the sample

As large as possible and should be chosen so that liberated quantity of carbon dioxide lies within the working range of the equipment/calibration (for ZAG max. mass is 3 g, but for most of samples up to 1 g).

Removal of the inorganic carbon and determination of the TOC

The sample is weighted into a vessel (may be thermal treated).

The sample is carefully treated with a small volume of HCl. Add as little acid as possible; allow at least 4 h for the complete removal of the carbon dioxide.

The moisture may be partly removed by drying <40°C.

Combust sample in the carrier gas containing oxygen at temperature, high enough to convert all organic carbon into carbon dioxide (900 °C – 1500 °C; 950 °C for ZAG's instrument).

The amount of carbon dioxide released turning the combustion is measured by IR spectrometry, thermal conductivity detection or other suitable technique.

Reporting of result

The result obtained from TOC analyser is in g/kg. According to EN 15936:2022 the TOC content is reported as carbon on a dry matter basis (the water content, determined separately, is used):

$$m_{TOC,dm} = F \times m_{TOC} \frac{100}{100 - w}$$

Where:

$m_{TOC,dm}$ = the TOC content as carbon, calculated on dry matter basis expressed in % in relation to the dry mass,

m_{TOC} = the TOC content as carbon in the sample expressed in % C,

w = the water content of the dried sample as mass fraction expressed in %,

F = the dilution factor resulting from the sample preparation.

Results are obtained in % C.

References

- (1) Zhao, M.; Han, Z.; Sheng, C.; Wu, H. Characterization of Residual Carbon in Fly Ashes from Power Plants Firing Biomass. *Energy Fuels* 2013, 27, 898–907.
- (2) Kumpiene, J.; Robinson, R.; Brannvall, E.; Nordmark, D.; Bjurström, H.; Andreas, L.; Lagerkvist, A.; Ecke, H. Carbon speciation in ash, residual waste and contaminated soil by thermal and chemical analyses. *Waste Manage.* 2011, 31, 18–25.
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EN 15936:2022: Soil, waste, treated biowaste and sludge – Determination of total organic carbon (TOC) by dry combustion.

4 Chemical properties

4.1 Water content, pH, conductivity, leaching and water solubility of the ash

Lisbeth M. Ottosen, DTU Sustain, Technical University of Denmark

Background and principle

This procedure compiles different measurements into one sequence: water content, pH, conductivity, leaching and water solubility of the ash.

The water content is measured is determined as the free water, i.e., the water which evaporates at 105 °C. The liquid to solid (L:S) ratio for the procedure is 1:10 chosen from the leaching test EN 12457-2:2002, and to optimize the working process in the lab, the same L:S ratio was chosen for pH and conductivity. The dry mass of the ash that was not dissolved during the leaching is made for the calculation of the soluble fraction.

Special equipment

- Heating closet
- Analytical balance
- pH- and conductivity meter
- vacuum pump
- ICP-ES
- IC
- Whatman 42 filter paper with 0.45µm pore size

Chemical safety

- No use of chemicals

Reagents

- No use of chemicals

Procedure

Water Content

3 beakers are placed in a heating closet at 105°C overnight. The beakers are placed in a desiccator to cooldown by using a tweezer to devote moisture. They are then weighed on an analytical balance and the weighed is noted

Weigh 20 g sample on an analytic balance into each beaker, note the amount of sample and place the samples in a 105°C heating closet overnight.

Weigh the beakers with the dried sample on analytical balance after cooling in the desiccator.

Leaching L/S-10, pH, and conductivity

Take the ash from water content. Mix and crush it by hand in a mortar.

Weigh 10.00 g ash on an analytic balance into 250 ml plastic bottles.

Gently add 100.00 ml of distilled water using a volumetric pipette. Place the sample on a shaking table for 24 hours.

NOTE: Place a petri dish (one for each sample) in a heating closet at 105°C overnight for the water solubility.

Take of the samples and let them stand for 15 min before filtering.

Water solubility

Weigh the petri dish and the 0.45µm Whatman 42 filter paper on an analytical balance using a tweezer.

Filtrate the sample from leaching using a vacuum pump and without rinsing. Take a sample from the liquid without removing the filter paper and save the sample for IC and ICP. Rinse the last part of the sample in the bottle onto the filter and make sure that everything is on the filter paper.

Take the filter paper with the sample and place it on the petri dish. Place the petri dish in the heating closet at 105°C overnight.

Measure pH and conductivity on the sample for IC and ICP.

Weigh the petri dish with the dried sample on analytical balance after cooling in the desiccator.

reporting of result

Calculate the water content in %, using Equation below.

$$\% \text{ water content} = \frac{(w_w - w_d)}{(w_w - w_p)} \times 100$$

where:

w_w = total mass of the wet specimen and petri dish,

w_d = total mass of the dried specimen and petri dish

w_p = total mass of the petri dish

The result from leaching is given in mg/l and the conductivity in mS/cm.

Calculate the water solubility in g, using the Equation below.

$$\% Solubility = \frac{1 - (A - B)}{C} \cdot 100$$

Where:

A = petri dish with dry ash and petri dish

B = filter paper and petri dish

C = Amount of ash from the leaching test

4.2 Reactivity (Cumulative Heat Determination by Isothermal Calorimetry – ASTM C1897–20)

Sabina Dolenc, ZAG

Background and principle

Isothermal calorimetry is used to determine the heat of hydration of hydrating pastes composed of the supplementary cementitious materials (SCM), calcium hydroxide, calcium carbonate, potassium sulfate, and potassium hydroxide. The heat of hydration value is used to determine the chemical reactivity of the SCM.

Special equipment

- Calorimeter
- High-shear blender (ex. IKA ULTRA-TURRAX tube drive control)

Chemical safety

- Calcium hydroxide: Corrosive and health hazard
- Potassium hydroxide: Corrosive and health hazard

Read the SDS before use

Reagents

Use only Chemicals of reagent grade

- calcium hydroxide – $[Ca(OH)_2]$
- calcium carbonate – $[CaCO_3]$
- potassium sulfate – $[K_2SO_4]$
- potassium hydroxide – $[KOH]$

Procedure

Sample

Take approximately 100 g of dry ($105^{\circ}C$) sample using a sample divider or by quartering. Sieve the portion on a $125\text{ }\mu\text{m}$ sieve until the residue remains constant. Grind the retained materials so that it completely passes the $125\text{ }\mu\text{m}$ sieve.

Mixture preparation

The ratio of SCM to calcium hydroxide is 1 to 3 by mass.

The ratio of SCM to calcium carbonate is 2 to 1 by mass.

Prepare a potassium solution by dissolving 4.00 g of potassium hydroxide and 20.0 g of potassium sulfate in 1.00 L of reagent water conditioned at $23 \pm 3^{\circ}C$.

The ratio of potassium solution to solids (sum of the SCM, the calcium hydroxide, and the calcium carbonate) is 1.2 by mass.

Table gives an example of the mixture proportions for an SCM paste conforming to this standard. The mass ratio of the SCM in the fresh paste is $10\text{ g}/99\text{ g} = 0.101$. The required total mass to be mixed will depend on the type and number of tests to be performed.

| | SCM | Ca(OH)_2 | CaCO_3 | Potassium Solution |
|--------------|-------|-------------------|-----------------|--------------------|
| Mass (grams) | 10.00 | 30.00 | 5.00 | 54.00 |

Paste mixing

Weigh the designated amounts of the dry SCM, calcium hydroxide, and calcium carbonate on weighing papers, then combine and mix until a homogeneous color is achieved. Mass measurements are to be accurate to $\pm 01\text{ g}$. Store the dry mixture in an air-tight container in a storage environment at $40 \pm 2^\circ\text{C}$ until the temperature of the dry mixture is stabilized at $40 \pm 2^\circ\text{C}$.

Place and seal the potassium solution in an air-tight container in a storage environment at $40 \pm 2^\circ\text{C}$ until the solution is at $40 \pm 2^\circ\text{C}$.

Mix the dry mixture and the potassium solution at $1600 \pm 50\text{ r/min}$ for 2 min using the high-shear blender (Figure 1) so that a homogeneous paste is achieved. Alternatively, use the procedure for mixing pastes in Practice C1738/C1738M, where the dry mixture of solids is considered cementitious materials or cement and the potassium solution is considered to be mixing water. Record the time at start of mixing and use that as time zero.



Figure 1. High-shear blender: IKA ULTRA-TURRAX tube drive control

Preparation of the Apparatus

The calorimeter shall be set at 40 ± 0.5 °C for at least 16 h before test.

Prior to mixing the pastes, place the calorimeter specimen containers, lids, and pipettes in a 40 ± 2 °C storage environment until they reach 40 ± 2 °C.

Insert sealed, air-tight containers filled with 9.40 ± 0.05 g of deionized water into the reference channels of the calorimeter.

Paste

Determine the mass of the empty calorimeter specimen container to the nearest 0.01 g. Cast 15.0 g of the freshly mixed paste in the specimen container. Record the mass (m_p) of the paste to the nearest 0.01 g.

When placing the paste into the calorimeter specimen container, avoid spilling paste on the opening and outer sides of the container.

Measurement

Place each sealed, air-tight calorimeter specimen into the calorimeter measurement chambers within 10 min of the start of mixing and record the heat release at 40 ± 0.5 °C for 168 h (7 days) after mixing.

Reporting of result

Calculate the cumulative heat release (H) from 75 min until 3 days (72 h ± 10 min) and 7 days (168 h ± 10 min) after start of mixing (time zero) by integration of the recorded heat release. Calculate the cumulative heat release in units of J/(g of SCM) using Eq 1: below.

$$H_{SCM} = \frac{(H)}{m_p \times 0.101}$$

where:

H = cumulative heat release (H) from 75 min until 3 days (72 h ± 10 min) and 7 days (168 h ± 10 min) after start of mixing,

m_p = the mass of the paste,

0.101 = mass fraction of the SCM in the paste specimen.

NOTE: Calculation of cumulative heat release is started at 75 min after mixing to allow enough time for the paste and container to equilibrate at 40 ± 0.5 °C inside the calorimeter.

Report to the nearest 0.1 J/g the cumulative heat release per gram of SCM, H_{SCM} , after 72 h (3 days) and 168 h (7 days) from time of mixing.



References

ASTM C1897 – 20: Standard Test Methods for Measuring the Reactivity of Supplementary Cementitious Materials by Isothermal Calorimetry and Bound Water Measurements.

ASTM C1738/C1738M-19: Standard Practice for High-Shear Mixing of Hydraulic Cement Pastes.

Avet et al., 2022. Report of RILEM TC 267-TRM phase 2: optimization and testing of the robustness of the R3 reactivity tests for supplementary cementitious materials. *Materials and Structures*, 55.

<https://link.springer.com/article/10.1617/s11527-022-01928-6>

4.3 Reactivity (Bound water– ASTM C1897–20)

Sabina Doleneč, ZAG

Background and principle

Chemically bound water of pastes composed of the supplementary cementitious materials (SCM), calcium hydroxide, calcium carbonate, potassium sulfate, and potassium hydroxide is determined as a measure of the chemical reactivity of the SCM.

Special equipment

- Chamber furnace (350°C)
- High-shear blender (ex IKA ULTRA-TURRAX tube drive control)

Chemical safety

- Calcium hydroxide: Corrosive and health hazard
- Potassium hydroxide: Corrosive and health hazard

Read the SDS before use

Reagents

Use only Chemicals of reagent grade

- calcium hydroxide – $[Ca(OH)_2]$
- calcium carbonate – $[CaCO_3]$
- potassium sulfate – $[K_2SO_4]$
- potassium hydroxide – $[KOH]$

Procedure

Sample

Take approximately 100 g of dry (105 °C) sample using a sample divider or by quartering. Sieve the portion on a 125 µm sieve until the residue remains constant. Grind the retained materials so that it completely passes the 125 µm sieve.

Mixture preparation

The ratio of SCM to calcium hydroxide is 1 to 3 by mass.

The ratio of SCM to calcium carbonate is 2 to 1 by mass.

Prepare a potassium solution by dissolving 4.00 g of potassium hydroxide and 20.0 g of potassium sulfate in 1.00 L of reagent water conditioned at 23 ± 3 °C.

The ratio of potassium solution to solids (sum of the SCM, the calcium hydroxide, and the calcium carbonate) is 1.2 by mass.

Table gives an example of the mixture proportions for an SCM paste conforming to this standard. The mass ratio of the SCM in the fresh paste is $10\text{ g}/99\text{ g} = 0.101$. The required total mass to be mixed will depend on the type and number of tests to be performed.

| | SCM | Ca(OH)_2 | CaCO_3 | Potassium Solution |
|--------------|-------|-------------------|-----------------|--------------------|
| Mass (grams) | 10.00 | 30.00 | 5.00 | 54.00 |

Paste mixing

Weigh the designated amounts of the dry SCM, calcium hydroxide, and calcium carbonate on weighing papers, then combine and mix until a homogeneous color is achieved. Mass measurements are to be accurate to ± 0.1 g. Store the dry mixture in an air-tight container in a storage environment at 40 ± 2 °C until the temperature of the dry mixture is stabilized at 40 ± 2 °C.

Place and seal the potassium solution in an air-tight container in a storage environment at 40 ± 2 °C until the solution is at 40 ± 2 °C.

Mix the dry mixture and the potassium solution at 1600 ± 50 r/min for 2 min using the high-shear blender so that a homogeneous paste is achieved. Alternatively, use the procedure for mixing pastes in Practice C1738/C1738M, where the dry mixture of solids is considered cementitious materials or cement and the potassium solution is considered to be mixing water. Record the time at start of mixing and use that as time zero.

Paste

Within 5 min after the start of mixing, cast 15 ± 2 g of paste into an air-tight specimen container. Cast at least two paste specimens in separate containers for each curing age.

Store the sealed paste specimens at 40 ± 2 °C for 168 h (7 days) ± 3 h. If additional curing ages are to be examined, separate sealed specimens for each curing age shall be prepared and stored at 40 ± 2 °C for those times.

At the required curing age, remove the paste specimen from the container, and cut off and discard the top portion of the specimen so that at least 10 g of specimen remains for bound water measurements.

Paste Specimen Preparation

Crush the paste specimen using a mortar and pestle so that the particle sizes are not greater than about 2 mm. If the crushed paste specimen is too moist to be passed through a 2 or 2.36-mm sieve, then spread 10

± 1 g of the crushed paste evenly and no more than 10 mm thick over the surface of a petri dish, and dry in an oven at 40 °C for 24 ± 1 h. Then continue crushing.

If the entire crushed paste specimen does not pass a 2 or 2.36-mm sieve, continue crushing using a mortar and pestle until entire specimen passes the selected sieve. To minimize the production of very fine material, use several passes of the specimen through the crushing equipment. Remove the portion passing the sieve before re-crushing the remainder of the specimen. Thoroughly mix by coning from one paper to another ten times. Spread 10 ± 1 g of the mixed and crushed paste specimen evenly in a petri dish. The thickness of the ground paste shall not exceed 10 mm. Dry the final crushed paste specimen in an oven at 40 °C for 24 ± 1 h.

Measurement

Heat the furnace to a temperature of 350 ± 10 °C.

Place the empty porcelain crucible in the pre-heated furnace at 350 ± 10 °C for 1 hour ± 10 min. Cool the empty crucible down to room temperature in an empty desiccator above fresh silica gel or another suitable desiccant. Record the mass of the cooled, empty crucible (w_c) to the nearest 0.001 g.

Transfer 5 ± 0.5 g of dried crushed paste to the cooled, empty crucible. Record the total mass of the dried specimen and crucible (w_0) to the nearest 0.001 g.

Place the crucible with dried paste in the pre-heated furnace at 350 ± 10 °C for 2 hour ± 10 min. Cool the heated paste in a desiccator above fresh silica gel or other suitable desiccant for 1 hour ± 5 min. Record the total mass of the heated paste and crucible (w_h) to the nearest 0.001 g within 10 min after taking the heated paste out of the desiccator.

Reporting of result

Calculate the chemically bound water content, in g per 100 g of paste dried at 40 °C, using Equation below.

$$H_2O_{bound,dried} = \frac{(w_0 - w_h)}{w_0 - w_c} \times 100$$

where:

w_0 = total mass of the dried specimen and crucible,

w_c = mass of the cooled, empty crucible, and

w_h = total mass of the heated paste and crucible.

Report to nearest 0.01 g the bound water content in g per 100 g of paste dried at 40 °C for each of the two specimens as well as the average value.



References

ASTM C1897 – 20: Standard Test Methods for Measuring the Reactivity of Supplementary Cementitious Materials by Isothermal Calorimetry and Bound Water Measurements.

ASTM C1738/C1738M-19: Standard Practice for High-Shear Mixing of Hydraulic Cement Pastes.

4.4 Free CaO content

Ana Grahovac, NEXE d.d. & Ivana Carević, Faculty of Civil Engineering University of Zagreb

Background and principle

This is protocol for free CaO content based on EN 451-1 standard Method of testing fly ash -- Part 1: Determination of free calcium oxide content.

Special equipment

- Balance, capable of weighing to an accuracy of 0,0001 g or better.
- Test sieve, with 0.063 mm
- Mortar or pestle for grinding
- Desiccator, containing dry agent like silica gel
- Filter crucible (pore size of 0.004 mm to 0.01 mm). Alternatively, also filter funnel, in which a filter paper with the fine pores of a diameter of approximately 0.002 mm and a filter paper with medium pores of a diameter of approximately 0.007 mm can be placed, can be used.
- Flask, of 250 ml
- Spiral reflux condenser
- Absorption tube, filled with sodium hydroxide on an inorganic carrier (to protect the contents of the flask and the condenser from reacting with atmospheric carbon dioxide)

Chemical safety

- 2-butanol: flammable, and acute toxicity
- 2-propanol: flammable and health hazard
- Ethanol: flammable and health hazard
- Hydrochloric acid: Corrosive and health hazard

Read the SDS before use

Reagents

Use only reagents of analytical grade and distilled water, or water of equal purity.

- Ethyl acetoacetate (Butanoic acid, 3-oxo-ethyl ester) – [CH₃COCH₂COOC₂H₅]
- 2-Butanol – [CH₃CH(OH)CH₂CH₃]
- 2-Propanol – [(CH₃)₂CHOH]
- Indicator (0,1g of bromophenol blue in 100 ml of 100% ethanol) – [C₁₉H₁₀Br₄O₅S] & [CH₃CH₂OH]
- Hydrochloric acid (0,100 M) – [HCl]

Procedure

Preparation of test sample

Before starting the determination, treat the laboratory sample to obtain homogenous test sample. Subdivide the laboratory sample by a suitable method to obtain a subsample of about 20 g. Pass this subsample through the test sieve 0.063 mm. Grind any residue in the mortar until all the subsample passes through the sieve completely. Homogenize the total subsample and place it in the desiccator until tested.

Procedure

Weight 1 to 1.5 g of sample (subsample prepared in accordance with protocol given above) and place into the 250 ml flask and add a mixture of 12 ml butanoic acid, 3-oxo-ethyl ester and 80 ml butan-2-ol.

Fit the flask with the spiral reflux condenser and the absorption tube and boil for 3 h.

Filter the warm mixture through the filter crucible.

Wash the residue with 50 ml propan-2-ol. If the filter is cloudy, reject it and repeat the extraction with the new weighed portion of the subsample.

Add a few drops of bromophenol blue indicator to the filtrate and titrate with hydrochloric acid until the color changes to yellow.

Record the volume V of hydrochloric acid used.

In addition, to color change also other procedures for end point determination can be used (e.g. pH value, conductivity)

Reporting of result

Calculate the free CaO from the formula, expressed as a percentage by mass of the dry ash:

$$W_{CaO} = \frac{M \cdot c \cdot V}{m \cdot 100} \cdot 100$$

where:

V = volume of hydrochloric acid used for titration, in ml

C = concentration of the hydrochloric acid solution

m = mass of the test portion used, in g

M = 28.04 g/mol (1/2 of molar mass of CaO)

Express the results where two results have been obtained as the mean result, calculated to two decimals and expressed to one decimal. If the difference between determinations is more than twice the standard deviations, repeat the test and take the mean of the closest values. The standard deviation for repeatability is 0.03% by mass.



References

EN 451-1 Method of testing fly ash -- Part 1: Determination of free calcium oxide content

4.5 Alkali dissolution

Priyadarshini Perumal, University of Oulu

Background and principle

In this method, Amount of soluble aluminum (Al) and silicates (Si) in the alkaline environment is quantified. Raw material should be milled, in the size range similar to the samples used for XRD studies.

Special equipment

- Shaker mixer
- Vacuum pump
- Inductively coupled plasma optical emission spectrometry (ICP-OES)
- Nylon filter paper with 0.45 µm pore size

Chemical safety

- Sodium hydroxide: Corrosive
- Nitric acid solution: Corrosive, flammable, and acute toxicity

Read the SDS before use

Reagents

- Sodium hydroxide (4M) – [NaOH]
- Nitric acid solution (6M) – [HNO₃]

Procedure

The solubility tests will be carried out with 4M NaOH solution as alkali solvent. For each test, 1 g of milled sample is mixed with 1000 mL of alkali solvent.

The mixture is then shaken in a shaker mixer (KS 260 basic, IKA) at 150 rpm for 24 h. The solution should be filtered through a nylon filter paper with 0.45 µm pore size.

The pH of the collected solution is set below 2 with 6M nitric acid solution to prevent precipitation.

The dissolved Si and Al amounts to be determined from the collected solution by inductively coupled plasma optical emission spectrometry (ICP-OES) according to standard SFS-EN ISO 11885:2009.

Reporting of result

Results are reported as mg/l. Test is repeated twice to get an average value.



References

Sreenivasan, H., Cao, W., Hu, Y. et al. (6 more authors) (2020) Towards designing reactive glasses for alkali activation: understanding the origins of alkaline reactivity of Na-Mg aluminosilicate glasses. PLoS ONE, 15 (12). e0244621.

5 Physical properties

5.1 Bulk density

Farnaz Aghabeyk, Civil Engineering and Geosciences, Materials and Environment, TU Delft & Ivana Carević, Faculty of Civil Engineering University of Zagreb

Background and principle

The bulk density of a powder is the ratio of the mass of a powder sample to its volume, including the contribution of the interparticulate void volume (including all pores and voids). This method covers measurement of powders and non-cohesive materials.

For AshCycle project bulk density will be measured on fly ashes.

Special equipment

- funnel – allow material to flow freely from a height of 7,3 cm without risk of compaction;
- container – cylindrical cup with a known volume (for example 0.5 l (500 cm³) or 1 l (1000 cm³);
- scale – electric scale to weigh the container and the container + material inside;
- measuring tape – to measure the height between the top edge of the container and the bottom edge of the funnel;
- straightedge blade i.e. trowel or spatula – the tool helps in levelling by scrapping and removing excess material;
- rack – for installing the funnel in the required height of 7.3 cm

The station for the test prepared for conducting the density test should be considered the configuration given in figure 1.

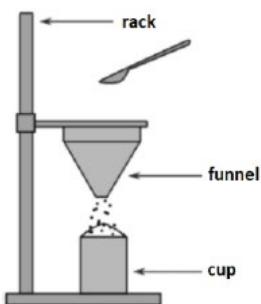


Figure 1 Prepared station for the testing of bulk density

Figure 2 Measuring the mass of the container with the material

Chemical safety

- No use of chemicals.

Reagents

- No use of chemicals.

Procedure

Place the funnel in the rack. The bottom end of the funnel should be closed by the lid.

Weigh the container and write down the weight of the cylindrical cup as m_{con} .

Place the container in the central position under the funnel.

Check the height between the top edge of the container and the bottom of the funnel – it should be 7.3 cm.

Put the material in the funnel, gently filling the material with a spoon as low as possible and as close as possible to the lid first and then to the material, which is placed already in the funnel. It is forbidden to touch the material in the funnel with a spoon.

Unplug the lid of the funnel and let the material flow freely into the container until it fills up the cup and forms a characteristic embankment slope.

If the material does not flow freely and get stuck in the funnel see **IMPORTANT NOTE**.

Once the cup is completely full the excess of powder needs to be levelled by scraping (typically with a straight edge blade).

The mass of the container with the material is measured in the balance as $m_{con}+m$ (Figure 2).

IMPORTANT NOTE: Material in powder state such as lime and cement may need a couple of trial tests before the exact test, due to the fact that these materials are easily stuck into a container because of their strong bonds between the particles forming agglomerates. Trial tests should be carried out until the material covers the surface of the funnel so thoroughly that the material can fall at once.

Lime and/or cement can get stuck inside the funnel and not fall freely, especially if the experiment is performed for the first time or no caution is taken to prevent over compaction of the material in the funnel itself. The former can be avoided by ‘priming’ the funnel first, i.e. running a few trial tests to let the material coat the inside walls of the funnel, thus allowing it fall freely, without adherence in the subsequent tests. The over compaction can be mitigated by ensuring that the powdered material is placed inside the funnel carefully and not throwing it from a height.

Reporting of result

The mass of the material is finally measured as the difference in weight of the container with material and the mass of the container itself. The density in grams per cm^3 is equal to:

$$d_m = \frac{(m_{con+m} - m_{con})}{V}$$

where:

m_{con+m} = the mass of the container with the material, in g

m_{con} = the mass of the container, in g

V = capacity of the container, in cm³

It is recommended to take the average of three independent, similar and consecutive determinations (i.e. using three different sub-samples). Based on experience, it is best to perform the test more times (i.e. >5) to establish an acceptable error margin for any given material. If the resulting values deviate from one another by more than 10 g, repeat it twice ((i.e. >5)).

References

Internal protocol for bulk density at the Civil Engineering and Geosciences, Materials and Environment, TUDelft laboratory.

D. Bjegović, V. Ukrainczyk, D. Mikulić, and Z. Rak, *Poznavanje gradiva, auditorne vježbe, praktikum, aktivna nastava*, Zagreb, 1994, in Croatian

EN 1097-3:1998 Tests for mechanical and physical properties of aggregates -- Part 3: Determination of loose bulk density and voids

5.2 Density of ashes using pycnometer

Ivana Carević, Faculty of Civil Engineering University of Zagreb

Background and principle

The density of bottom ashes is determined using method EN 1097-6 (Pycnometer) - Tests for mechanical and physical properties of aggregates -- Part 6: Determination of particle density and water absorption.

Method "*a pycnometer method for aggregate particles passing the 4 mm test sieves and retained on the 0,063 mm test sieve*" is used with some exceptions. The method is used for measurement of bottom ashes.

Different/exceptions from EN 1097-6: sieving on 0.063 mm is excluded.

Special equipment

- ventilated oven thermostatically controlled to maintain a temperature of $110 \pm 5^\circ\text{C}$
- balance, accurate to 0.1 % of the mass of the test portion
- water bath, thermostatically controlled, capable of being maintained at $22 \pm 5^\circ\text{C}$
- thermometer, accurate to $0,1^\circ\text{C}$
- test sieve, 4 mm with apertures as specified in EN 933-2
- trays, which can be heated in a ventilated oven without change in mass
- dry soft absorbent cloths
- washing equipment.
- timer
- pycnometer, consisting of a glass flask or other suitable vessel with volume between 500 ml and 1000 ml, constant to 0,5 ml for the duration of the test
- metal mould, in the form a frustum of a cone (40 ± 3) mm at the top, 90 ± 3 mm at the bottom 75 ± 3 mm high. The metal shall have a minimum thickness of 0.8 mm.
- metal tamper, of mass (340 ± 15) g and having a flat circular tamping face of diameter 25 ± 3 mm for use with the metal mould
- funnel, plain glass
- shallow tray, of non-water absorbing material having a plane bottom of area not less than 0.1 mm^2 and an edge of not less than 50 mm in height
- warm air supply, such as a hair dryer

Chemical safety

- No use of chemicals.

Reagents

- No use of chemicals.

Procedure

Record initial mass of ash (please see table 1). Sieve ash on a 4 mm and discard particles (metals, unburned wood, impurities) retained on the 4 mm sieve. Recorded mass that is discarded (mass on sieve 4 mm in table 1). The mass of the test portion of ash shall be not less than 300 g (mass after sieving in table 1).

Immerse the prepared test portion in water at $22\pm3^{\circ}\text{C}$ in the pycnometer and remove entrapped air by gently rolling and jolting the pycnometer in a tipped position. Stand the pycnometer in the water bath and keep the test portion at the temperature of $22\pm3^{\circ}\text{C}$ for $24\pm0,5$ h. At the end of soaking period, take the pycnometer from the water bath and remove remaining entrapped air by gentle rolling and jolting.

Overflow the pycnometer by adding water and place the cover on the top without trapping air in the vessel. Then dry the pycnometer on the outside and weight it M₂, record the temperature of the water.

Decant most of the water covering the test portion and empty the pycnometer into the tray.

Refill the pycnometer with the water and place the cover in position as before, then dry the pycnometer on the outside and weight it M₃, record the temperature of the water.

The difference in the temperature of the water in the pycnometer during M₂ and M₃ weighting shall not exceed 2 °C.

Spread the soaked test portion in a uniform layer over the bottom of the tray. Expose the aggregate to a gentle current of a warm air to evaporate surface moister. Stir it at frequent intervals to ensure uniform drying until no free surface moister can be seen and the aggregate particles no longer adhere to one another. Let the sample cool at room temperature while stirring it.

To assess whether the surface-dry state has been achieved, hold the metal cone mould with its largest diameter face downwards on the bottom of the tray. Fill the cone mould loosely with the part of the drying test portion. Through the hole at the top of the mould place the metal tamper on the surface of the sand. Tamp the surface 25 times by letting the tamper fall under its own weight. Gently lift the module clear of the aggregate, if the aggregate does not collapse, continue drying and repeat cone test until the collapse situation occurs at the mould removal.

Weight the saturated and surface dried test portion M₁. Dry the aggregate in a ventilated oven at a temperature $110\pm5^{\circ}\text{C}$ until it has reached a constant mass. Let it cool to ambient temperature and weight, M₄.

Reporting of result

Calculate the particle density in megagram per cubic metre in accordance with given formula:

Apparent particle density:

$$\rho_a = \rho_w \frac{M_4}{M_4 - (M_2 - M_3)} \quad (1)$$

Oven-dried particle density

$$\rho_{rd} = \rho_w \frac{M_4}{M_1 - (M_2 - M_3)} \quad (2)$$

Saturated and surface dried particle density

$$\rho_{ssd} = \rho_w \frac{M_1}{M_1 - (M_2 - M_3)} \quad (3)$$

And water absorption after immersion for 24 h:

$$WA_{24} = \rho_w \frac{100x(M_1 - M_4)}{M_4} \quad (4)$$

where:

ρ_w = density of water, in megagrams per cubic metre

M1 = mass of saturated and surface dried aggregate in the air, in grams

M2 = mass of the pycnometer containing the sample of saturated aggregate and water in grams

M3 = mass of pycnometer filled with water only, in grams

M4 = mass of the oven-dried test portion in air, in grams

Express the values of particle density to the nearest 0,01 mg/m³ and water absorption to the nearest 0,1%.

If content of unburned carbon is quite high (as given in figures below), recorded it (take a picture), because unburnt carbon is also an integral part of the ash.



Sample 1



Sample 2



Sample 3

Table 1 Test list

| Sample ID | Initial mass | Mass on sieve 4 mm | Mass after sieving on sieve 4 mm (min. 300 g) | M1 | M2 | M3 | M4 |
|-----------|--------------|--------------------|---|----|----|----|----|
| | | | | | | | |
| | | | | | | | |
| | | | | | | | |
| | | | | | | | |

References

EN 1097-6 (Pycnometer) - Tests for mechanical and physical properties of aggregates -- Part 6: Determination of particle density and water absorption

EN 933:2 Tests for geometrical properties of aggregates -- Part 2: Determination of particle size distribution -- Test sieves, nominal size of apertures

5.3 Particle size distribution (sieving >250µm)

Sabina Dolenec & Lea Žibret ZAG

Background and principle

Reference washing and dry sieving method can be used for type testing and particle size distribution of aggregates up to 90 mm nominal size (EN 933-1:2012). Dry sieving may be used for aggregates free from particles, which cause agglomeration. Principle is based on separating material into several particle size classifications of decreasing size by a series of sieves. The aperture sizes and the number of sieves are selected due to the nature of sample and the accuracy required (test sieves, specified in EN 933-2).

According to the previous investigation (1) the dry sieving method with the following series of sieves is suggested (apertures in mm): 16, 8, 4, 2, 1, 0.5, 0.25, 0.125, 0.063.

Special equipment

- Series of sieves (apertures in mm): 16, 8, 4, 2, 1, 0.5, 0.25, 0.125, 0.063.
- Laboratory sieve shaker.
- Laboratory scale.

Chemical safety

- No use of chemicals.

Reagents

- No use of chemicals.

Procedure

Minimum size of test portions

$$M = (D/10)^2$$

where:

M = minimum mass of test portion in kg,

D = maximum aggregate size in mm.

Preparation of sample

Dry the test portion in the oven at 110 ± 5 °C to a constant mass. After cooling record the mass (M1).

The main steps of dry sieving

Pour dry sample into the sieving column (decreasing aperture from top to bottom).

Shake the column manually or mechanically. Mechanical shaking is preferred (approx. 5 min).

Remove the sieves, starting with the largest aperture size and shake each sieve manually (use pan and lid to ensure no material is lost).

The material which passes each sieve should be transferred onto the next sieve.

Weight the retained material for each sieve (R1, R2, R3...).

The material, remained in the pan shall be weighted and recorded as P.

Number of replicates: 3.

Note that the effectiveness of mechanical sieving is influenced by the aggregate type, the sieving time, the loading on the sieve and the parameters of shaking movements (amplitude, frequency). The mechanical sieving time should be carefully chosen. The sieving procedure shall be considered as finished when additional sieving does not lead to a change of mass of the retained material on any sieve by more than 1.0 % by mass.

Reporting of result

Results are reported as

Mass retained on each sieve as a % of original dry mass M1.

Cumulative % of fines f passing the 0.063 mm sieve:

$$f = 100 \times P/M1$$

where:

M1 = dried mass of test portion (kg),

P = material, remained in the pan (kg).

Results will be reported in tabular (the cumulative percentages passing each sieve) and graphical form (histogram of density distribution and cumulative distribution).

References

RIS ALICE EITRM111511: DELIVERABLE D3.2, Report on the laboratory characterization of Al-rich residues sampled from selected RIS countries, 2021

EN 933-1:2012 Test of geometrical properties of aggregates – Part 1: Determination of particle size distribution – Sieving method.



EN 933-2:2020 Tests for geometrical properties of aggregates - Part 2: Determination of particle size distribution - Test sieves, nominal size of apertures.

5.4 Particle size distribution (laser <250µm)

Sabina Dolenec & Lea Žibret, ZAG

Background and principle

Guidance on instrument qualification and size distribution measurement of particles (PSD) is provided by ISO 13320 standard, which is applicable to particle sizes ranging from approximately 0.1 µm to 3 mm (can be extended above 3 mm and below 0.1 µm).

Special equipment

- Laser diffraction particle size analyzer

Chemical safety

- 2-propanol: flammable and health hazard
- Ethanol: flammable and health hazard

Read the SDS before use

Reagents

Use only reagents of reagent grade and 99,8%

- 2-Propanol – [(CH₃)₂CHOH]
- Ethanol – [CH₃CH₂OH]

Procedure

Sample preparation

Prepare a representative sample of suitable volume by using adequate sample splitting technique.

The sample should be dry (T of drying 105°C).

Large particles, which could damage the device, should be removed by sieving (suggested that dried samples are sieved through sieve 0.250 µm).

Dispersion procedure

Wet method with alcohol (2-propanol or ethanol) dispersion media.

Proper dispersion of the particles in the liquid can be generally prepared by pasting, stirring and sonication.

To avoid unrepresentative sampling (sample sedimentation) dry sample can be dispersed directly into the measuring cell (especially if the instrument allows sonication of the suspension in the measuring cell).

Measurement

Measuring range: 2000 – 0.01 μm .

Number of measurements: 3 (results should be comparable).

Reporting of result

Reported as tabular data and graphical (histogram of density distribution and cumulative distribution; example is given on Figure 1), according to ISO 9276-1.

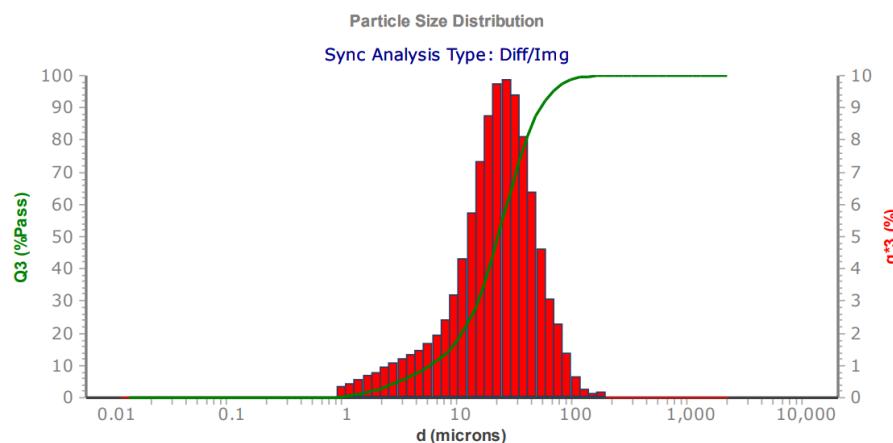


Figure 1: Example of PSD measurement results (co-combustion fly ash from Slovenian heating plant).

Reported percentiles (μm) in table: d(90), d(50), d(10).

References

ISO 13320: Particle size analysis – Laser diffraction methods.

ISO 14487: Sample preparation – Dispersing procedures for powders and liquids.

ISO 9276-1: Representation of results of particle size analysis – Part 1: Graphical representation.

5.5 Specific surface area of solids by gas adsorption — BET method

Tero Luukkainen, University of Oulu

Background and principle

These instructions are based on the standard ISO 9277. The principle of the method is to cover the studied material surface by a monolayer of physisorbed adsorbate (most often N₂ at its boiling point, 77.3 K) and to apply the BET equation to calculate the monolayer amount (unit mol/g). This is then converted into specific surface area by multiplying with molecular cross-sectional area.

Special equipment

- BET

Chemical safety

- No use of chemicals

Reagents

- No use of chemicals

Procedure

Take a representative 2 g sample of ash as received.

Remove physically adsorbed material from the sample surface by degassing. Degassing at 200°C for 24 h is recommended.

Measure the degassed sample.

Notes:

The precision of the measurement depends on the control of the following conditions.

- a) *The temperature or the p₀ value of the adsorptive should be monitored during the analysis.*
- b) *The purity of the adsorptive and any helium used to calibrate volumes or as a carrier gas should be at least 99.99 % volume fraction. If necessary, the gases should be dried and cleaned, e.g. oxygen removed from nitrogen.*
- c) *The saturation vapour pressure p₀ of the adsorptive at the measuring temperature can either be determined directly using a nitrogen vapour pressure thermometer or it can be monitored and determined by measurement of the thermostat bath temperature.*
- d) *The validity of the result depends on careful sampling and sample preparation.*

Apply either static volumetric method, flow volumetric method, gravimetric method, or carrier gas method to determine gas adsorption at equilibrium (see the standard for details).

5.6 Determination of metallic Al content

Boy Chen, Delft University of Technology

Background and principle

The presence of metallic Al in incineration ashes was reported by previous researchers. Metallic Al oxidizes under alkaline conditions in blended cement concrete and alkali-activated concrete. This redox reaction of metallic Al results in the release of hydrogen gas, which can potentially cause an increase in the volume and a decrease in the strength of hardened concrete. Therefore, it is important to determine the metallic Al content in incineration ashes prior to its addition to concrete.

The water displacement method is the most commonly used approach for measuring the metallic Al content in incineration ashes. The reaction of metallic Al and NaOH solution releases hydration gas, the volume of which is measured and subsequently used to calculate the metallic Al content in incineration ashes. Eq.1 provides a description of the chemical reaction between metallic Al and NaOH solution.

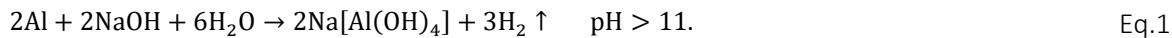


Figure 1 depicts the experimental set-up designed for measuring the metallic Al content in incineration ashes, with MSWI bottom ash serving as an example. In this configuration, MSWI bottom ash is immersed in the NaOH solution.

The hydrogen gas released from the reaction between metallic Al and NaOH solution in the three-neck round-bottom flask is first directed through a U-shaped drying tube. This is a step undertaken to eliminate the effect of water vapor. Subsequently, the increasing pressure drives hydrogen gas to displace the water within a stoppered wide-mouth glass bottle. The volume of yielded hydrogen gas is equivalent to the volume of water displaced in the graduated cylinder.

Special equipment

- Magnetic stirrer
- three-neck round-bottom flask
- U-shaped drying tube
- stoppered wide-mouth glass bottle
- graduated cylinder

Chemical safety

- Sodium Hydroxide: Corrosive

Read the SDS before use

Reagents

- Sodium hydroxide (1M) – [NaOH]

Procedure

Sample preparation

The ash sample collected from incineration plants. The ash is dried at 105° and the particle size should be smaller than 125 μm to ensure that all the metallic Al can be oxidized within 1-2 hours. Grind the ash samples to obtain a homogeneous composition if necessary.

Collect a representative sample of the incineration ashes (5-10 g).

Sodium Hydroxide (NaOH) Solution

The concentration of the NaOH solution will be dependent on the anticipated metallic Al content in the ashes, keeping in mind the reaction stoichiometry. Normally 1 M NaOH solution is good.

Reaction Set-Up:

Immerse the incineration ash sample in the NaOH solution within a three-neck round-bottom flask. Connect one neck of the flask to a U-shaped drying tube to remove the contribution of water vapor. Attach another neck to a dropping funnel, which should be closed immediately after each addition of NaOH solution to avoid unwanted escape of hydrogen gas.

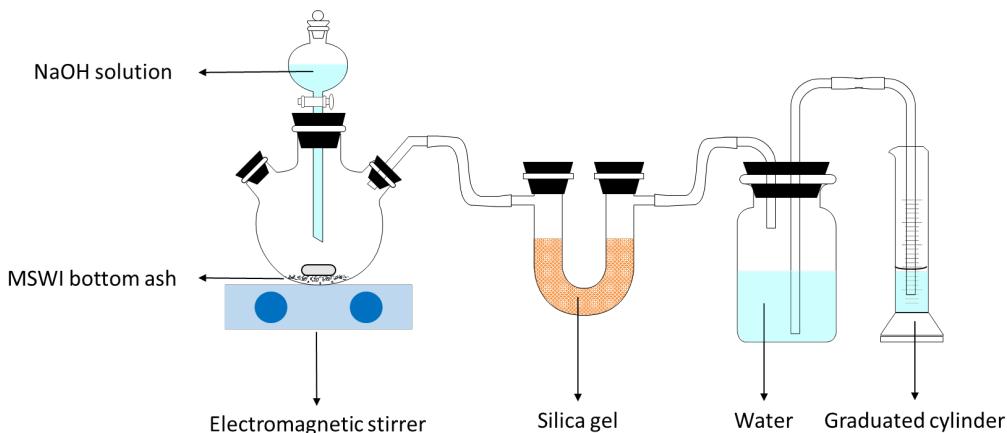


Figure 1 Set-up used for the measurement of metallic Al content in MSWI bottom ash.

Measurement of Hydrogen Gas:

Monitor the reaction between the NaOH solution and the ashes. The hydrogen gas generated will displace the water in the graduated cylinder. Record the volume of water displaced, which is equivalent to the

volume of hydrogen gas produced.

Calculation of Metallic Al Content:

Use the volume of the produced hydrogen gas to calculate the metallic Al content in the incineration ashes.

The weight percentage of metallic Al in incineration ashes (f_{Al}) using Equation below.

$$f_{Al} = \frac{2\Delta V(\frac{273}{273+T})}{3V_0} \frac{M_{Al}}{m_{BA}}$$

where:

ΔV = the volume of hydrogen gas measured at room temperature T (expressed in Celsius degree)

V_0 = denotes the molar gas volume at standard pressure and temperature ($P_0 = 101.3\text{kPa}, T_0 = 273K$)

M_{Al} = the molar mass of aluminum

m_{BA} = the mass of incineration ashes

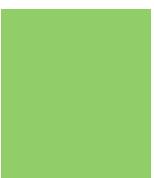
Reporting of result

The metallic Al content is reported as a weight percentage, calculated relative to the total mass of measured incineration ashes.

References

- [1] Chen B. Utilization of MSWI bottom ash as a mineral resource for low-carbon construction materials: Quality-upgrade treatments, mix design method, and microstructure analysis[J]. 2023.

Part 2. Selected Ashes



**Funded by
the European Union**

Selected ashes and details on incineration plants

(Compiled from Teams folder by Mie T. Tybjerg and Lisbeth M. Ottosen DTU (22.05.23) on behalf of the task 2.1 group participants. The tables in the Teams folder are filled out by the participants)

The tables in this report are presenting the ashes selected for the experimental work in the AshCycle project in **task 2.1**. The tables report important information on the actual incineration plants.

The ashes have been given a systematic name: #country.type.number. An example on such name is #BE.MSWI.FA.1, which is MSWI fly ash number 1 from Belgium.

All together there are 15 MSWI ashes, 32 wood ashes, 10 sewage sludge ashes, 27 co-combustion ashes and 1 other.

The information on the incineration plants collected are:

- Information given on the incineration plants (when available from the plant):
- Plant name (and if there is authorization to publish data with plant name)
- Plant type
- Fuel type
- Origin of fuel
- Incineration temperature range
- Total fuel input capacity
- Filter type
- Flue gas purification system
- Ash Types
- Amounts of ash produced each year

Not all information was available or the information could not be reported due to confidentiality. The available information are reported in the tables in the following paragraphs

Information on the different ashes and incineration plants

| | | |
|----|---|----|
| 1. | Municipal solid waste incineration (MSWI) ashes | 2 |
| 2. | Wood ashes..... | 6 |
| 3. | Sewage sludge ashes..... | 15 |
| 4. | Co-combustion ashes..... | 19 |
| 5. | Other biomass ashes..... | 30 |

1. Municipal solid waste incineration (MSWI) ashes

| Plant short name (#country.ashtype.number) | Plant name | Authorization to publish data with plant name (Yes), publish data anonymously (A) or data cannot be published (No) | Plant Type | Fuel type | Origin of fuel Household (H) Commercial (C) Industrial (I) |
|---|---------------|--|------------|-------------------------|---|
| #DK.MSWI.FA.1 | ARGO | Yes | Grate | Waste | H & C |
| #DK.MSWI.FA.4 | Amager Bakke | Yes | Grate | Waste | H & C |
| #BE.MSWI.FA.1 | IVM (Eeklo) | Yes | Grate | Waste | H, C & I |
| #BE,MSWIFA,3 | IVAGO (Ghent) | Yes | grate | Waste | H, C & I |
| #BE,MSWIFA,4 | IVBO (Bruges) | Yes | grate | Waste | H, C & I |
| #NL.MSWI.FA.1 | | | Grate | Waste | H & C |
| #NL.MSWI.FA.2 | | | Grate | Waste, hazardous waste | H & C |
| #NL.MSWI.FA.3 | | | Grate | Waste | H & C |
| #NL.MSWI.FA.4 | | | Grate | Waste and sewage sludge | H & C |
| #NL.MSWI.FA.5 | | | Grate | Waste | H & C |
| #NL.MSWI.FA.6 | | | Grate | Waste | H & C |
| #NL.MSWI.FA.7 | | | Grate | Waste | H & C |
| #NL.MSWI.FA.8 | | | Grate | Waste | H & C |
| #NL.MSWI.FA.9 | | | Grate | Waste | H & C |

| Plant short name (#country.ashtype.number) | Incineration temperature range (degrees Celcius) | Total fuel input capacity (t/year) | Filter type | Flue gas purification system Wet (W) Dry (D) |
|---|--|------------------------------------|---------------------|--|
| #DK.MSWI.FA.1 | | | Baghouse filter | W |
| #DK.MSWI.FA.4 | 950-1100 | 531000 | ESP | W |
| #BE.MSWI.FA.1 | 900 | 100000 | ESP+Baghouse filter | W & D |
| #BE,MSWIFA,3 | 1000 | 100000 | ESP | W |
| #BE,MSWIFA,4 | 850-1200 | 175000 | Baghouse filter | W |
| #NL.MSWI.FA.1 | +/-1000 | 400000 | ESP | W |
| #NL.MSWI.FA.2 | +/-1000 | 1400000 | ESP | W |
| #NL.MSWI.FA.3 | 1000-1100 | 250000 | ESP | D |
| #NL.MSWI.FA.4 | 1000-1200 | 700000 | ESP | W & D |
| #NL.MSWI.FA.5 | 1000-1200 | 700000 | ESP | W & D |
| #NL.MSWI.FA.6 | >950 | 650000 | ESP | D |
| #NL.MSWI.FA.7 | 1150 | 300000 | ESP | D |
| #NL.MSWI.FA.8 | 1000-1300 | 730000 | ESP | D |
| #NL.MSWI.FA.9 | 900-1100 | 1000000 | ESP | W |

| Plant short name (#country.ashtype.number) | Ash type | | | Possible to sample FA without air pollution control residues | Country |
|--|------------|--------------------------|---------------|--|-------------|
| | Fly ash | Air pollution residue | Bottom ash | | |
| #DK.MSWI.FA.1 | x | | | yes | Denmark |
| #DK.MSWI.FA.4 | x | | | yes | Denmark |
| #BE.MSWI.FA.1 | x | x | x | yes | Belgium |
| #BE,MSWIFA,3 | x | x | x | yes | Belgium |
| #BE,MSWIFA,4 | x | x | x | yes | Belgium |
| #NL.MSWI.FA.1 | x | x | x | yes | Netherlands |
| #NL.MSWI.FA.2 | x | x | x | yes | Netherlands |
| #NL.MSWI.FA.3 | x | x | x | yes | Netherlands |
| #NL.MSWI.FA.4 | x | x | x | yes | Netherlands |
| #NL.MSWI.FA.5 | x | x | x | yes | Netherlands |
| #NL.MSWI.FA.6 | x | 2x | x | yes | Netherlands |
| #NL.MSWI.FA.7 | x | x | x | yes | Netherlands |
| #NL.MSWI.FA.8 | x | x | x | yes | Netherlands |
| #NL.MSWI.FA.9 | x | x | x | yes | Netherlands |

| Plant short name (#country.ashtype.number) | Amount of fly ash produced (t/year) | Amount of APC residue produced (t/year) | Amount of bottom ash produced (t/year) |
|---|--|--|---|
| #DK.MSWI.FA.1 | | | |
| #DK.MSWI.FA.4 | 5900 | | 101558 |
| #BE.MSWI.FA.1 | 1300 | 1900 | 18872 |
| #BE,MSWIFA,3 | 777 | 3664 | 12711 |
| #BE,MSWIFA,4 | 2790 | 480 | 32000 |
| #NL.MSWI.FA.1 | 6400 | 2400 | 110000 |
| #NL.MSWI.FA.2 | 23500 | 3000 | 330000 |
| #NL.MSWI.FA.3 | 4200 | 4200 | 55000 |
| #NL.MSWI.FA.4 | 12000 | 11000+640 | 247500 |
| #NL.MSWI.FA.5 | 3400 | 5700 | 82500 |
| #NL.MSWI.FA.6 | 6400 | 3200+6300 | 150000 |
| #NL.MSWI.FA.7 | 4200 | 3500 | 75000 |
| #NL.MSWI.FA.8 | 6000 | 5000 | 180000 |
| #NL.MSWI.FA.9 | 19000 | 1800 | 260000 |

2. Wood ashes

| Plant #ID (#country.ashtype.no) | Plant location | Incineration technology | Fuel type | Origin of fuel |
|------------------------------------|--------------------|-----------------------------------|--|---|
| #HR.WA.1 | Slavonski Brod OIE | Grate+ pulverised fuel combustors | wood chips, wood chips with impurities | Whole trees, wood residue (including twigs and tops), wood waste including bark |
| #HR.WA.2 | Strizivojna Hrast | Grate | wood chips | Whole trees |
| #HR.WA.3 | Požega | Grate | wood chips from the whole wood, residues from wood processing (including bark) | Whole trees, wood residue (including twigs and tops), wood waste including bark |
| #HR.WA.4 | Žakanje | Grate | wood chips with impurities; wood chips from the whole wood, residues from wood processing (including bark) | Whole trees, wood residue (including twigs and tops), wood waste including bark |
| #HR.WA.5 | Karlovac | Grate | wood chips, residues from wood processing (including bark) | Whole trees, wood residue (including twigs and tops), wood waste including bark |
| #HR.WA.6 | Vinkovci (Spačva) | Grate | wood chips, wood chips from the whole wood, residues from wood processing (including bark) | Whole trees, wood residue (including twigs and tops), wood waste including bark |
| #HR.WA.7 | Gospic | Grate | wood chips, wood chips with impurities, residues from wood processing (including bark) | Whole trees, wood residue (including twigs and tops), wood waste including bark |
| #HR.WA.8 | Brinje | Grate | wood chips, wood chips with impurities, residues from wood processing (including bark) | Whole trees, wood residue (including twigs and tops), wood waste including bark |
| #HR.WA.9 | Babina Greda | bubbling fluidised bde-BFB | pure wood chips, wood chips from whole wood (example: wood chips containing bark, sapwood, needles/leaves) | Whole trees, wood residue (including twigs and tops), wood waste including bark |

| Plant #ID (#country.ashtype.no) | Plant location | Incineration technology | Fuel type | Origin of fuel |
|------------------------------------|----------------------------|--|--|--|
| #HR.WA.10 | Bjelovar | Grate | pure wood chips | timber logs |
| #HR.WA.11 | Ljubešćica | Grate | pure wood chips, wood chips from whole wood (example: wood chips containing bark, sapwood, needles/leaves) | timber logs, residues from timber harvesting, waste from the wood industry |
| #HR.WA.12 | Grubišno Polje | Biograte | pure wood chips, wood chips from whole wood (example: wood chips containing bark, sapwood, needles/leaves) | timber logs |
| #HR.WA.13 | Glina, Glina | Grate+ pulverised fuel combustors | pure wood chips, wood chips from whole wood (example: wood chips containing bark, sapwood, needles/leaves) | timber logs |
| #HR.WA.14 | Glina, Drvni Centar | Grate+ pulverised fuel combustors | pure wood chips, wood chips from whole wood (example: wood chips containing bark, sapwood, needles/leaves) | timber logs |
| #SI.WA.1 | Toplarna Nova Gorica | heat power station | wood chips | |
| #BE.WA.1 | E Wood Antwerpen | | Wood type B (non- recyclable wood waste) | wood biomass |
| #BE.WA.2 | BEB Gent (BEE) | | Wood type B (non- recyclable wood waste) | wood biomass |
| #NL.WA.1 | | fluidize bed | waste wood | |
| #NL.WA.2 | | | wood, waste wood | |
| #NL.WA.3 | | fluidize bed | wood | |
| #NL.WA.4 | | grade | waste wood, biomass | |
| #NL.WA.5 | | | waste wood, biomass | compost overflow |
| #NL.WA.6 | | | waste wood | |

| Plant #ID (#country.ashtype.no) | Type of wood used | Biomass consumption (per hour or other available data) | Temperature range (°C) | Filter |
|------------------------------------|---|---|------------------------|---|
| #HR.WA.1 | mostly beech, oak, hornbeam, poplar | 9 tonne/hour | | cyclone separator |
| #HR.WA.2 | mostly oak, hornbeam | 5-5,5 tonne per hour | 1000-1100 | cyclone separator, electrostatic filter |
| #HR.WA.3 | mostly beech, oak, hornbeam | 3 tonne per hour | 700 | cyclone separator |
| #HR.WA.4 | mostly mix (beech, spruce, fir) | 2 tonne per hour | 700-800 (max 900-1000) | cyclone separator |
| #HR.WA.5 | mix wood | 8 tonne per hour | 700 | electrostatic filter |
| #HR.WA.6 | mostly beech, oak, hornbeam, poplar, fraxinus | 1.1-2.5 tonne per hour in summer; 3-5.4 tonne per hour winter | 980 | cyclone separator |
| #HR.WA.7 | mix wood | 200 tone per day wet biomass or approximately 150 tonne per day dried biomass | 900-1050 | electrostatic filter; cyclone separator |
| #HR.WA.8 | mostly beech, abies, poplar, mix wood | 120 tonne per day | 750 | electrostatic filter |
| #HR.WA.9 | mostly beech, oak, hornbeam, poplar | 11,5 tonne per hour | 850 | electrostatic filter |

| Plant #ID (#country.ashtype.no) | Type of wood used | Biomass consumption (per hour or other available data) | Temperature range (°C) | Filter |
|------------------------------------|--|---|------------------------|-----------------------|
| #HR.WA.10 | mixed wood | 2t/h | 700-800 | cyclone separator |
| #HR.WA.11 | beech, oak, fir, mixed wood | 1,5t/h | 800-1000 | cyclone separator |
| #HR.WA.12 | mixed wood | 8t/h | 680 | electrostatic filter |
| #HR.WA.13 | mixed wood form Romania, Serbia, Croatia | 3t/h | 700-800 | cyclone separator |
| #HR.WA.14 | mixed wood form Romania, Serbia, Croatia | 7t/h | 700-800 | cyclone separator |
| #SI.WA.1 | mixed wood from Italy and Slovenia | x | 700-900 | cyclone separator |
| #BE.WA.1 | mix of wood types | | | |
| #BE.WA.2 | mix of wood types | | | |
| #NL.WA.1 | | 300000 ton/year | | |
| #NL.WA.2 | forst wood and compost overflow | | | |
| #NL.WA.3 | forest and scrap | | | |
| #NL.WA.4 | Wood type B (non-recyclable wood waste) | 150000 ton/year | >950 | Cycloon and filterbag |
| #NL.WA.5 | Wood type B (non-recyclable wood waste) | 185000 ton/year | | |
| #NL.WA.6 | | 170000 ton/year | | Filter bag |

| Plant #ID (#country.ashtype.no) | Available ash types | | | Country |
|------------------------------------|---------------------|------------|--------------------------|-------------|
| | Fly ash | Bottom ash | Mixed fly and bottom ash | |
| #HR.WA.1 | | | | HR |
| #HR.WA.2 | | | | HR |
| #HR.WA.3 | | | | HR |
| #HR.WA.4 | | | | HR |
| #HR.WA.5 | | | | HR |
| #HR.WA.6 | | | | HR |
| #HR.WA.7 | | | | HR |
| #HR.WA.8 | | | | HR |
| #HR.WA.9 | | | | HR |
| #HR.WA.10 | | | | HR |
| #HR.WA.11 | | | | HR |
| #HR.WA.12 | | | | HR |
| #HR.WA.13 | | | | HR |
| #HR.WA.14 | | | | HR |
| #SI.WA.1 | x | x | x | SI |
| #BE.WA.1 | | | | Belgium |
| #BE.WA.2 | | | | Belgium |
| #NL.WA.1 | x | | | Netherlands |
| #NL.WA.2 | x with APC | | | Netherlands |
| #NL.WA.3 | x | | | Netherlands |
| #NL.WA.4 | x | | | Netherlands |
| #NL.WA.5 | x | | | Netherlands |
| #NL.WA.6 | x | | | Netherlands |

| Plant #ID (#country.ashtype.no) | Amount of ash produced (t/year) | Expected large variation in ash properties over the year (Y-yes, N- no) | Current ash management |
|--|--|--|---|
| #HR.WA.1 | Bottom ash - 3 m3 per 10 days, Fly ash - 6 m3 per day | Unknown | it is deposited on its own landfill |
| #HR.WA.2 | 1 tonne per day | Unknown | it is deposited on its own landfill |
| #HR.WA.3 | 200 tonne per year | Unknown | it is handed over to authorized waste management companies, landfilled |
| #HR.WA.4 | 5 tonne per week or 700kg per day | Unknown | it is handed over to authorized waste management companies, landfilled |
| #HR.WA.5 | bottom ash - 400 kg every 3 days, Fly ash - 730 kg per day | Unknown | it is handed over to authorized waste management companies, this company mix ash with sewage sludge |
| #HR.WA.6 | 43,7 tonne per month | Unknown | it is handed over to authorized waste management companies, landfilled |
| #HR.WA.7 | fly ash - 1050 kg per day, bottom ash: 1m3 per 3 days | Unknown | it is deposited on its own landfill |
| #HR.WA.8 | fly and bottom - 4m3 per day | Unknown | it is handed over to authorized waste management companies, landfilled |
| #HR.WA.9 | fly ash: 1026,71 tonne/vera; bottom: 1080,34 tonne/year | Unknown | one part is used in agonomy, other landfilled |

| Plant #ID (#country.ashtype.no) | Amount of ash produced (t/year) | Expected large variation in ash properties over the year (Y-yes, N- no) | Current ash managment |
|--|---|--|--|
| #HR.WA.10 | 15-35 t/month | N | it is handed over to authorized waste management companies, landfilled |
| #HR.WA.11 | 2,5t per week, mixed / 90% bottom ash and 10% fly ash, coarse fraction | N | it is handed over to authorized waste management companies, landfilled |
| #HR.WA.12 | FLY ASH: 2020 - 365 t 2021 - 310 t BOTTOM ASH: cca. 4 tonnes every 2-3 days | N | it is handed over to authorized waste management companies, landfilled / a colaboration with agricultural company is planned in oder to declare it as a by-product |
| #HR.WA.13 | fly and bottom: 1 tonne per two-three days | Unknown | Ashes are bought it by company for City of Zadar that mixes sludges with biomass ashes in order to stabilized sludge. |
| #HR.WA.14 | fly and bottom: 1 tonne per day | Unknown | Ashes are bought it by company for City of Zadar that mixes sludges with biomass ashes in order to stabilized sludge. |
| #SI.WA.1 | 100 tonnes/year | N | Ash is turned over to the local utility company |
| #BE.WA.1 | Plant under construction | | |
| #BE.WA.2 | | | |
| #NL.WA.1 | 8700 | | immobilisation and landfill |
| #NL.WA.2 | 1000 | | immobilisation and landfill |
| #NL.WA.3 | | | |
| #NL.WA.4 | 2000 | N | immobilisation and landfill |

| Plant #ID (#country.ashtype.no) | Approximate monthly cost of ash management (transport, disposal costs, etc.) | Date of collecting | Notes |
|------------------------------------|--|----------------------------|---|
| #HR.WA.1 | 0 EUR | 8.7.2022, SONJA (UNIZG) | Only fly ash was collected. Bottom ash was hot to collect. |
| #HR.WA.2 | 0 EUR | 11-07-2022 | Two fly ashes were collected |
| #HR.WA.3 | 7500 KN per month (approximately 1000 EUR per month) | 15-07-2022 | Mix ash was collected |
| #HR.WA.4 | 710 KN per tone (without VAT) | 26-08-2022 | Mix ash was collected, BL |
| #HR.WA.5 | 0 KN/EUR | 26-08-2022 | Two ashes were collected, BL |
| #HR.WA.6 | 3000KN per month | 3.9.2022. | Mix ash was collected |
| #HR.WA.7 | unknown | 07-09-2022 | Only fly ash was collected, bottom ash was not collected. It is collected mixing with water (wet bottom ash). |
| #HR.WA.8 | unknown | 07-09-2022 | Only fly ash was collected. Bottom ash was not collected because they just had fly one in container. |
| #HR.WA.9 | 30000 KN per month | 23.9.2022. | Two ashes were collected. (bottom and fly). Silica sand is used during combustion (840,45 tonne per year) |

| Plant #ID (#country.ashtype.no) | Approximate monthly cost of ash management (transport, disposal costs, etc.) | Date of collecting | Notes |
|--|---|-------------------------------|--|
| #HR.WA.10 | unknown | 28.9.2022. | Mix ash was collected. |
| #HR.WA.11 | ca. 10.000 HRK + PDV per month | 16-09-2022 | Mix ash was collected. |
| #HR.WA.12 | 10.000 HRK/month | 28.9.2022. | Only fly ash was collected. Bottom ash is mixed with water and of high temperature when in container (it was not collected). |
| #HR.WA.13 | unknown | 17-10-2022 | Fly and bottom ashes were collected. |
| #HR.WA.14 | unknown | 17-10-2022 | Fly and bottom ashes were collected. |
| #SI.WA.1 | x | 21-07-2022 | samples available |
| #BE.WA.1 | | | |
| #BE.WA.2 | | | |
| #NL.WA.1 | | | |
| #NL.WA.2 | | | |
| #NL.WA.3 | | | |
| #NL.WA.4 | | | |
| #NL.WA.5 | | | |
| #NL.WA.6 | | | |

3. Sewage sludge ashes

| Plant name – short (#country.asstype.no) | Plant name/ wastewater treatment plants | Authorization to publish data with plant name (Yes), publish data anonymously (A) or data cannot be published (No) | Date of sampling | Plant Type |
|---|---|--|--|--|
| #DK.SSA.FA.1 | Avedøre spildevandscenter | Yes | | Fluidized bed |
| #DK.SSA.FA.2 | Lynetten Wastewater Treatment Plant | Yes | | Fluidized bed |
| #HR.SSA.1 | Karlovac | | 7.9.2022 of SS 26.9.2022 burned | Thermochemical gasification with rotary kiln reactor |
| #HR.SSA.2 | Velika Gorica | | 28.9.2022 of SS 12.10.2022 burned | Thermochemical gasification with rotary kiln reactor |
| #HR.SSA.3 | Sisak | | 24.10.2022. of SS 28.10.2022 burned | Thermochemical gasification with rotary kiln reactor |
| #HR.SSA.4 | Našice | | 14.10.2022. of SS 28.10.2022 burned | Thermochemical gasification with rotary kiln reactor |
| #HR.SSA.5 | Zagreb | | | Thermochemical gasification with rotary kiln reactor |
| #NL.SSA.1 | | | | |
| #BE.SSA.1 | SLECO | | | Fluidized bed |
| #BE.SSA.2 | Aquafin/Besix/ Indaver Gent | | | |

| Plant name – short (#country.ashtype.no) | Fuel type | Origin of fuel | Temperature range (°C) | Filter |
|---|----------------------------------|--|-----------------------------------|--------------------------------------|
| #DK.SSA.FA.1 | Sewage sludge | Municipal | ~ 850 | Electrofilter (before bag filter) |
| #DK.SSA.FA.2 | Sewage sludge | Municipal | ~ 850 | Electrofilter (before bag filter) |
| #HR.SSA.1 | Sewage sludge | Municipal | 900 | cyclone separator |
| #HR.SSA.2 | Sewage sludge | Municipal | 900 | cyclone separator |
| #HR.SSA.3 | Sewage sludge | Municipal | 900 | cyclone separator |
| #HR.SSA.4 | Sewage sludge | Municipal | 900 | cyclone separator |
| #HR.SSA.5 | Sewage sludge | Municipal | 900 | cyclone separator |
| #NL.SSA.1 | Sewage sludge | | | |
| #BE.SSA.1 | Sewage sludge combined with MSWI | Household, commercial, industrial and sludge | >850 | ESP+Baghouse filter |
| #BE.SSA.2 | Sewage sludge | Biomass after household waste water purification | | |

| Plant name – short (#country.ashtype.no) | Ash Type | | | Country |
|---|-----------------|-------------------|---------------------------------|----------------|
| | Fly ash | Bottom ash | Mixed fly and bottom ash | |
| #DK.SSA.FA.1 | x | | | DK |
| #DK.SSA.FA.2 | x | | | DK |
| #HR.SSA.1 | | HR.SSA.1 | | HR |
| #HR.SSA.2 | | HR.SSA.2 | | HR |
| #HR.SSA.3 | | HR.SSA.3 | | HR |
| #HR.SSA.4 | | HR.SSA.4 | | HR |
| #HR.SSA.5 | | x | | HR |
| #NL.SSA.1 | | | | Netherlands |
| #BE.SSA.1 | x | x | | Belgium |
| #BE.SSA.2 | | | | Belgium |

| Plant name – short (#country.ashtype.no) | Amount of ash produced (t/year) | Expected large variation in ash properties over the year (Y-yes, N- no) | Current sludge/ash management |
|---|---|--|---|
| #DK.SSA.FA.1 | 2600 | N | Storred at plans's own site. Plan is to dig it out when method for P recovery is developed. |
| #DK.SSA.FA.2 | 3500 | N | Storred at plans's own site. Plan is to dig it out when method for P recovery is developed. |
| #HR.SSA.1 | 300 tonne per month of SS produced | Unknown | SS is drived to Benkovac. |
| #HR.SSA.2 | 2000 tonne per year SS | Unknown | Until last year SS was use in agriculture (amount 200 tonne of dray matter per year) and the rest was stored at wastewater treatment facility. From this year it is not allowed to use SS in agriculture - higher cost of ladrnfilling are expected. |
| #HR.SSA.3 | 1200 tonne per year (KB 190805), monthly 100tonne, or 3,3 tonne per day | Unknown | The authorized waste manager and the authorized waste transporter take away and dispose of the sludge. In 2020, the sludge from our facility was transported to the biogas plant (incineration), this year to the Stružec compost plant. In 2019, we disposed of a small part of the sludge on agricultural land. After that, not anymore, as the Ministry of Agriculture came out with a new law and limited the use of sludge in agriculture. |
| #HR.SSA.4 | 800 tonne per year + 30 tonne per year waste oil (KB 190810) | Unknown | SS is used in agriculture, before use parametars are tested for its use in agriculture. |
| #HR.SSA.5 | | Unknown | |
| #NL.SSA.1 | | | |
| #BE.SSA.1 | 159277 | N | |
| #BE.SSA.2 | Plant under construction (operational 2026) | | |

| Plant name – short (#country.asstype.no) | Approximate monthly cost of ash/sludge management (transport, disposal costs, etc.) | Date of collecting | Notes |
|---|---|--|--|
| #DK.SSA.FA.1 | | 08-03-2023 | |
| #DK.SSA.FA.2 | | 08-03-2023 | |
| #HR.SSA.1 | 615 KN /tonne for SS management | 7.9.2022 of SS 26.9.2022 burned | SS was collected 7.9.2022 by Indelooop (150 kg of SS). It was dried on 105 °C for several days and burned on 900 °C on 26.9.2022 (from 59 kg of dried SS to 25 kg SSA) |
| #HR.SSA.2 | 200000 KN last year for SS transport and cost of landfilling (on their own facility). Approximately this year: 1000000 KN/year | 28.9.2022 of SS 12.10.2022 burned | SS was collected 28.9.2022 by Indelooop (150 kg of SS). It was dried on 105 °C for several days and burned on 900 °C on 12.10.2022 (from 39 kg of dried SS to 11 kg SSA) |
| #HR.SSA.3 | The cost of sludge disposal is expensive for us, and it ranges from HRK 800- 1500/ton of sludge (with transport). Any waste manager who disposes of sludge must additionally recover it in special facilities, and this makes the cost of disposal expensive. With transportation cost. | 24.10.2022. of SS 28.10.2022 burned | SS was collected 24.10.2022 by Indelooop (150 kg of SS). It was dried on 105 °C for several days and burned on 900 °C on 28.10.2022 (from 47 kg of dried SS to 23 kg SSA) |
| #HR.SSA.4 | 160000 KN per year | 14.10.2022. of SS 28.10.2022 burned | SS was collected 14.10.2022 by Indelooop (150 kg of SS). It was dried on 105 °C for several days and burned on 900 °C on 28.10.2022 (from 71 kg of dried SS to 34 kg SSA) |
| #HR.SSA.5 | | | In order to collect SS, approval must be obtained from Ministry of Economy and Sustainable Development. This was done for all partners in Croatia that are planning to do activities with SSA. It took an average 6 months to get these approvals. Additionally, according to the instructions of the Ministry, the DOKING company had to registered in the Registry of Waste Carriers maintained by the competent authority of the county, i.e. the City of Zagreb, and received registration number in the Registry of Waste Carriers PRV-3866. With this SSA we will continue to work in WP4. |
| #NL.SSA.1 | | | |
| #BE.SSA.1 | | | This plant is also listed under MSWI |
| #BE.SSA.2 | | | Will also do fosfor recovery |

4. Co-combustion ashes

| Plant name – short (#country.ashtype.no) | Plant name | Authorization to publish data with plant name (Yes), publish data anonymously (A) or data cannot be published (No) | Plant Type | Fuel type |
|---|---------------------------------------|---|--|---|
| #SI.CC.1 | VIPAP VIDEM KRŠKO d.d (K4) | | paper mill | combustion of coal, biomass and/or paper sludge |
| #SI.CC.2 | VIPAP VIDEM KRŠKO d.d (K5) | | paper mill | the combustion residue from a steam boiler in which de-inking fibre paper sludge, waste wood and bark are used as fuel |
| #SI.CC.3 | Ljubljana power station (TETOL) | | heat and power station | brown coal and wood biomass -wood chips (15%) |
| #SI.CC.4 | Toplarna Celje | | heat and power station | municipality waste (ligh fraction),dehydrated sewage sludge |
| #FI.CC.APC.1 | Oulun energia, OEBIO | Y | heat and power plant, circulating fluidized bed | biomass 70 % (wood chips from the whole wood, residues from wood processing, including bark), solid recovered fuel (SRF) 15 % and peat 15 % as main fuels |
| #FI.CC.APC.2 | Oulun energia, OEBIO | Y | heat and power plant, circulating fluidized bed | biomass 70 % (wood chips from the whole wood, residues from wood processing, including bark), solid recovered fuel (SRF) 15 % and peat 15 % as main fuels |
| #FI.CC.APC.3 | Oulun energia, OEBIO | Y | heat and power plant, circulating fluidized bed | biomass 70 % (wood chips from the whole wood, residues from wood processing, including bark), solid recovered fuel (SRF) 15 % and peat 15 % as main fuels |
| #FI.CC.APC.4 | Oulun energia, OEBIO | Y | heat and power plant, circulating fluidized bed | biomass 70 % (wood chips from the whole wood, residues from wood processing, including bark), solid recovered fuel (SRF) 15 % and peat 15 % as main fuels |

| Plant name – short (#country.ashtype.no) | Plant name | Authorization to publish data with plant name (Yes), publish data anonymously (A) or data cannot be published (No) | Plant Type | Fuel type |
|---|----------------------------|---|--|--|
| #FI.CC.APC.5 | Oulun energia, OEBIO | Y | heat and power plant, circulating fluidized bed | biomass 70 % (wood chips from the whole wood, residues from wood processing, including bark), solid recovered fuel (SRF) 15 % and peat 15 % as main fuels |
| #FI.CC.APC.6 | Oulun energia, OEBIO | Y | heat and power plant, circulating fluidized bed | biomass 70 % (wood chips from the whole wood, residues from wood processing, including bark), solid recovered fuel (SRF) 15 % and peat 15 % as main fuels |
| #FI.CC.APC.7 | Oulun energia, OEBIO | Y | heat and power plant, circulating fluidized bed | biomass 70 % (wood chips from the whole wood, residues from wood processing, including bark), solid recovered fuel (SRF) 15 % and peat 15 % as main fuels |
| #FI.CC.APC.8 | Oulun energia, OEBIO | Y | heat and power plant, circulating fluidized bed | biomass 70 % (wood chips from the whole wood, residues from wood processing, including bark), solid recovered fuel (SRF) 15 % and peat 15 % as main fuels |
| #FI.CC.FA.1 | Oulun energia, OEBIO | Y | heat and power plant, circulating fluidized bed | biomass 70 % (wood chips from the whole wood, residues from wood processing, including bark), solid recovered fuel (SRF) 15 % and peat 15 % as main fuels |
| #FI.CC.FA.2 | Oulun energia, OEBIO | Y | heat and power plant, circulating fluidized bed | biomass 70 % (wood chips from the whole wood, residues from wood processing, including bark), solid recovered fuel (SRF) 15 % and peat 15 % as main fuels |
| #FI.CC.FA.3 | Oulun energia, OEBIO | Y | heat and power plant, circulating fluidized bed | biomass 70 % (wood chips from the whole wood, residues from wood processing, including bark), solid recovered fuel (SRF) 15 % and peat 15 % as main fuels |

| Plant name – short (#country.ashtype.no) | Plant name | Authorization to publish data with plant name (Yes), publish data anonymously (A) or data cannot be published (No) | Plant Type | Fuel type |
|---|----------------------------|---|--|--|
| #FI.CC.FA.4 | Oulun energia, OEBIO | Y | heat and power plant, circulating fluidized bed | biomass 70 % (wood chips from the whole wood, residues from wood processing, including bark), solid recovered fuel (SRF) 15 % and peat 15 % as main fuels |
| #FI.CC.FA.5 | Oulun energia, OEBIO | Y | heat and power plant, circulating fluidized bed | biomass 70 % (wood chips from the whole wood, residues from wood processing, including bark), solid recovered fuel (SRF) 15 % and peat 15 % as main fuels |
| #FI.CC.FA.6 | Oulun energia, OEBIO | Y | heat and power plant, circulating fluidized bed | biomass 70 % (wood chips from the whole wood, residues from wood processing, including bark), solid recovered fuel (SRF) 15 % and peat 15 % as main fuels |
| #FI.CC.BA.1 | Oulun energia, OEBIO | Y | heat and power plant, circulating fluidized bed | biomass 70 % (wood chips from the whole wood, residues from wood processing, including bark), solid recovered fuel (SRF) 15 % and peat 15 % as main fuels |
| #FI.CC.BA.2 | Oulun energia, OEBIO | Y | heat and power plant, circulating fluidized bed | biomass 70 % (wood chips from the whole wood, residues from wood processing, including bark), solid recovered fuel (SRF) 15 % and peat 15 % as main fuels |
| #FI.CC.BA.3 | Oulun energia, OEBIO | Y | heat and power plant, circulating fluidized bed | biomass 70 % (wood chips from the whole wood, residues from wood processing, including bark), solid recovered fuel (SRF) 15 % and peat 15 % as main fuels |

| Plant name – short (#country.ashtype.no) | Plant name | Authorization to publish data with plant name (Yes), publish data anonymously (A) or data cannot be published (No) | Plant Type | Fuel type |
|---|----------------------------|---|---|---|
| #FI.CC.BA.4 | Oulun energia, OEBIO | Y | heat and power plant, circulating fluidized bed | biomass 70 % (wood chips from the whole wood, residues from wood processing, including bark), solid recovered fuel (SRF) 15 % and peat 15 % as main fuels |
| #FI.CC.BA.5 | Oulun energia, OEBIO | Y | heat and power plant, circulating fluidized bed | biomass 70 % (wood chips from the whole wood, residues from wood processing, including bark), solid recovered fuel (SRF) 15 % and peat 15 % as main fuels |
| #FI.CC.BA.6 | Oulun energia, OEBIO | Y | heat and power plant, circulating fluidized bed | biomass 70 % (wood chips from the whole wood, residues from wood processing, including bark), solid recovered fuel (SRF) 15 % and peat 15 % as main fuels |
| #FI.CC.BA.7 | Oulun energia, OEBIO | Y | heat and power plant, circulating fluidized bed | biomass 70 % (wood chips from the whole wood, residues from wood processing, including bark), solid recovered fuel (SRF) 15 % and peat 15 % as main fuels |
| #BE.CC.1 | SLECO | Y | Fluidized bed | Sewage sludge combined with MSWI |
| #BE,MSWIFA,2 | SLECO (Antwerp) | Y | Fluidized bed | Waste+Sludge |

| Plant name – short (#country.ashtype.no) | Incineration temperature range (degrees Celcius) | Cooling system strategy of bottom ash | Total fuel input capacity (t/year) | Filter type | Flue gas purification system |
|---|--|---|--|--|---|
| #SI.CC.1 | | | | | |
| #SI.CC.2 | | | | | |
| #SI.CC.3 | | | | | |
| #SI.CC.4 | 650-850 C | | | | |
| #FI.CC.APC.1 | 850 - 950 | water cooling | 550000 | electrostatic filter, tubular bag filter | Burnt lime and activated charcoal are fed to clean the flue gases. These end up in APC |
| #FI.CC.APC.2 | 851 - 950 | water cooling | 550000 | electrostatic filter, tubular bag filter | Burnt lime and activated charcoal are fed to clean the flue gases. These end up in APC |
| #FI.CC.APC.3 | 852 - 950 | water cooling | 550000 | electrostatic filter, tubular bag filter | Burnt lime and activated charcoal are fed to clean the flue gases. These end up in APC |
| #FI.CC.APC.4 | 853 - 950 | water cooling | 550000 | electrostatic filter, tubular bag filter | Burnt lime and activated charcoal are fed to clean the flue gases. These end up in APC |
| #FI.CC.APC.5 | 854-950 | water cooling | 550000 | electrostatic filter, tubular bag filter | Burnt lime and activated charcoal are fed to clean the flue gases. These end up in APC |
| #FI.CC.APC.6 | 855-950 | water cooling | 550000 | electrostatic filter, tubular bag filter | Burnt lime and activated charcoal are fed to clean the flue gases. These end up in APC |

| Plant name – short (#country.ashtype.no) | Incineration temperature range (degrees Celsius) | Cooling system strategy of bottom ash | Total fuel input capacity (t/year) | Filter type | Flue gas purification system |
|---|---|--|---|---|---|
| #FI.CC.APC.7 | 856 - 950 | water cooling | 550000 | electrostatic filter, tubular bag filter | Burnt lime and activated charcoal are fed to clean the flue gases. These end up in APC |
| #FI.CC.APC.8 | 857 - 950 | water cooling | 550000 | electrostatic filter, tubular bag filter | Burnt lime and activated charcoal are fed to clean the flue gases. These end up in APC |
| #FI.CC.FA.1 | 858 - 950 | water cooling | 550000 | electrostatic filter, tubular bag filter | Burnt lime and activated charcoal are fed to clean the flue gases. These end up in APC |
| #FI.CC.FA.2 | 859 - 950 | water cooling | 550000 | electrostatic filter, tubular bag filter | Burnt lime and activated charcoal are fed to clean the flue gases. These end up in APC |
| #FI.CC.FA.3 | 860 - 950 | water cooling | 550000 | electrostatic filter, tubular bag filter | Burnt lime and activated charcoal are fed to clean the flue gases. These end up in APC |
| #FI.CC.FA.4 | 861 - 950 | water cooling | 550000 | electrostatic filter, tubular bag filter | Burnt lime and activated charcoal are fed to clean the flue gases. These end up in APC |
| #FI.CC.FA.5 | 862 - 950 | water cooling | 550000 | electrostatic filter, tubular bag filter | Burnt lime and activated charcoal are fed to clean the flue gases. These end up in APC |

| Plant name – short (#country.ashtype.no) | Incineration temperature range (degrees Celcius) | Cooling system strategy of bottom ash | Total fuel input capacity (t/year) | Filter type | Flue gas purification system |
|---|--|---------------------------------------|------------------------------------|--|--|
| #FI.CC.FA.6 | 863 - 950 | water cooling | 550000 | electrostatic filter, tubular bag filter | Burnt lime and activated charcoal are fed to clean the flue gases. These end up in APC |
| #FI.CC.BA.1 | 864 - 950 | water cooling | 550000 | electrostatic filter, tubular bag filter | Burnt lime and activated charcoal are fed to clean the flue gases. These end up in APC |
| #FI.CC.BA.2 | 865 - 950 | water cooling | 550000 | electrostatic filter, tubular bag filter | Burnt lime and activated charcoal are fed to clean the flue gases. These end up in APC |
| #FI.CC.BA.3 | 866 - 950 | water cooling | 550000 | electrostatic filter, tubular bag filter | Burnt lime and activated charcoal are fed to clean the flue gases. These end up in APC |
| #FI.CC.BA.4 | 867 - 950 | water cooling | 550000 | electrostatic filter, tubular bag filter | Burnt lime and activated charcoal are fed to clean the flue gases. These end up in APC |
| #FI.CC.BA.5 | 868 - 950 | water cooling | 550000 | electrostatic filter, tubular bag filter | Burnt lime and activated charcoal are fed to clean the flue gases. These end up in APC |
| #FI.CC.BA.6 | 869 - 950 | water cooling | 550000 | electrostatic filter, tubular bag filter | Burnt lime and activated charcoal are fed to clean the flue gases. These end up in APC |
| #FI.CC.BA.7 | 870 - 950 | water cooling | 550000 | electrostatic filter, tubular bag filter | Burnt lime and activated charcoal are fed to clean the flue gases. These end up in APC |
| #BE.CC.1 | | | | | |
| #BE,MSWIFA,2 | >850 | | 640000 | ESP+Baghouse filter | wet |

| Plant name – short (#country.ashtype.no) | Ash type | | | | Possible to sample FA without air pollution control residues | Country |
|---|-----------------|------------|--------------------------------|-----------------------|---|----------------|
| | Fly ash | Bottom ash | Mixed fly and bottom ash | Air pollution residue | | |
| #SI.CC.1 | x | | | | | SI |
| #SI.CC.2 | | | 90% bottom ash and 10% fly ash | | | SI |
| #SI.CC.3 | | | Mixed fly and bottom ash | | | SI |
| #SI.CC.4 | | | Mixed fly and bottom ash | | | SI |
| #FI.CC.APC.1 | x (APC) | | | | - | Finland |
| #FI.CC.APC.2 | x (APC) | | | | - | Finland |
| #FI.CC.APC.3 | x (APC) | | | | - | Finland |
| #FI.CC.APC.4 | x (APC) | | | | - | Finland |
| #FI.CC.APC.5 | x (APC) | | | | - | Finland |
| #FI.CC.APC.6 | x (APC) | | | | - | Finland |
| #FI.CC.APC.7 | x (APC) | | | | - | Finland |
| #FI.CC.APC.8 | x (APC) | | | | - | Finland |
| #FI.CC.FA.1 | | x | | | - | Finland |
| #FI.CC.FA.2 | | x | | | - | Finland |
| #FI.CC.FA.3 | | x | | | - | Finland |
| #FI.CC.FA.4 | | x | | | - | Finland |
| #FI.CC.FA.5 | | x | | | - | Finland |
| #FI.CC.FA.6 | | x | | | - | Finland |
| #FI.CC.BA.1 | | | X (BA only) | | - | Finland |
| #FI.CC.BA.2 | | | X (BA only) | | - | Finland |

| Plant name – short (#country.ashtype.no) | Ash type | | | | Possible to sample FA without air pollution control residues | Country |
|---|----------|------------|--------------------------|-----------------------|--|---------|
| | Fly ash | Bottom ash | Mixed fly and bottom ash | Air pollution residue | | |
| #FI.CC.BA.3 | | | X (BA only) | | - | Finland |
| #FI.CC.BA.4 | | | X (BA only) | | - | Finland |
| #FI.CC.BA.5 | | | X (BA only) | | - | Finland |
| #FI.CC.BA.6 | | | X (BA only) | | - | Finland |
| #FI.CC.BA.7 | | | X (BA only) | | - | Finland |
| #BE.CC.1 | x | x | | | | Belgium |
| #BE,MSWIFA,2 | x | x | | x | yes | Belgium |

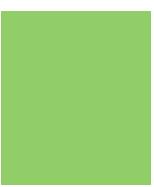
| Plant name – short (#country.ashtype.no) | Amount of fly ash produced (t/year) | Amount of APC residue produced (t/year) | Amount of bottom ash produced (t/year) | Amount of mixed fly and bottom ash produced (t/year) |
|---|--|--|---|---|
| #SI.CC.1 | | | | 3.200t/year |
| #SI.CC.2 | | | | 25.000t/year |
| #SI.CC.3 | | | | 6000t/year |
| #SI.CC.4 | | | | 2.200t/year |
| #FI.CC.APC.1 | 11200 | 2100 | 7600 | 0 |
| #FI.CC.APC.2 | 11200 | 2100 | 7600 | 0 |
| #FI.CC.APC.3 | 11200 | 2100 | 7600 | 0 |
| #FI.CC.APC.4 | 11200 | 2100 | 7600 | 0 |
| #FI.CC.APC.5 | 11200 | 2100 | 7600 | 0 |
| #FI.CC.APC.6 | 11200 | 2100 | 7600 | 0 |
| #FI.CC.APC.7 | 11200 | 2100 | 7600 | 0 |
| #FI.CC.APC.8 | 11200 | 2100 | 7600 | 0 |
| #FI.CC.FA.1 | 11200 | 2100 | 7600 | 0 |
| #FI.CC.FA.2 | 11200 | 2100 | 7600 | 0 |
| #FI.CC.FA.3 | 11200 | 2100 | 7600 | 0 |
| #FI.CC.FA.4 | 11200 | 2100 | 7600 | 0 |
| #FI.CC.FA.5 | 11200 | 2100 | 7600 | 0 |
| #FI.CC.FA.6 | 11200 | 2100 | 7600 | 0 |
| #FI.CC.BA.1 | 11200 | 2100 | 7600 | 0 |
| #FI.CC.BA.2 | 11200 | 2100 | 7600 | 0 |
| #FI.CC.BA.3 | 11200 | 2100 | 7600 | 0 |
| #FI.CC.BA.4 | 11200 | 2100 | 7600 | 0 |
| #FI.CC.BA.5 | 11200 | 2100 | 7600 | 0 |
| #FI.CC.BA.6 | 11200 | 2100 | 7600 | 0 |
| #FI.CC.BA.7 | 11200 | 2100 | 7600 | 0 |
| #BE.CC.1 | 11200 | 2100 | 7600 | 0 |
| #BE,MSWIFA,2 | 100835 | 16842 | 41600 | - |

| Plant name – short (#country.ashtype.no) | Expected large variation in ash properties over the year (Y-yes, N- no) | No. of samples from plant | Notes |
|---|--|--------------------------------------|--|
| #SI.CC.1 | yes | | recycled -Vipelex , transferred to recyclers (TERMIT) |
| #SI.CC.2 | no | | recycled - Mulex, transferred to other recyclers |
| #SI.CC.3 | yes | | recycled for degdared surfaces, (TERMIT; LUMARCI; CEP) |
| #SI.CC.4 | yes | | landfilled in CERO |
| #FI.CC.APC.1 | no | 22 | |
| #FI.CC.APC.2 | no | 23 | |
| #FI.CC.APC.3 | no | 24 | |
| #FI.CC.APC.4 | no | 25 | |
| #FI.CC.APC.5 | no | 26 | |
| #FI.CC.APC.6 | no | 27 | |
| #FI.CC.APC.7 | no | 28 | |
| #FI.CC.APC.8 | no | 29 | |
| #FI.CC.FA.1 | no | 30 | |
| #FI.CC.FA.2 | no | 31 | |
| #FI.CC.FA.3 | no | 32 | |
| #FI.CC.FA.4 | no | 33 | |
| #FI.CC.FA.5 | no | 34 | |
| #FI.CC.FA.6 | no | 35 | |
| #FI.CC.BA.1 | no | 36 | |
| #FI.CC.BA.2 | no | 37 | |
| #FI.CC.BA.3 | no | 38 | |
| #FI.CC.BA.4 | no | 39 | |
| #FI.CC.BA.5 | no | 40 | |
| #FI.CC.BA.6 | no | 41 | |
| #FI.CC.BA.7 | no | 42 | |
| #BE.CC.1 | | | |
| #BE,MSWIFA,2 | no | | |

5. Other biomass ashes

| Plant name – short #country.ashtype.no | Fuel type | Origin of fuel | Available ash types | | | Country | Amount of ash produced (t/year) |
|---|-----------------|---------------------------|---------------------|---------------|-----------------------------------|---------|---------------------------------------|
| | | | Fly ash | Bottom ash | Mixed fly and bottom ash | | |
| #NL.PSFA.1 | paper sludge | residue paperrecycling | x | | | NL | 1000 |

Part 3. Ash characterisation



**Funded by
the European Union**

Characterization results from selected ashes

(Compiled from Teams folder by Mie T. Tybjerg, Huilin Li, Ebba Schnell, and Lisbeth M. Ottosen DTU (22.05.23) on behalf of the task 2.1 group participants. The tables in the Teams folder were filled out by the participants)

The tables in this report are presenting the characterization performed for the ashes selected for the experimental work in the AshCycle project in **task 2.1**.

The first paragraph gives an overview of the characterizations performed. Different methods are used for the measurement of density and particle size distribution of fly and bottom ash, and thus the tables are separated on whether it is fly ash, bottom ash or mixed ash.

It was decided that a first screening of the ash characteristics do not necessary include XRF, TOC and BET. These methods are included in the full characterization program where for ashes selected for use in the other WPs. Except from TOC, XRF and BET only few characteristics are not yet reported for some of the ashes. The work is conducted at present, and will be available in the Teams folder for the whole project consortium within few weeks from now (before mid. June 2023). Marked in the tables are what was reported in the Teams Folder by 22.05.23.

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1. Overview of the characterization performed

1.2 Fly ashes

Table 1: Chemical composition – procedures performed on selected fly ashes

| Method / Sample ID | XRF | Heavy metals, REEs and Hg | XRD | Loss of ignition | TOC |
|--------------------|-----|------------------------------|-----|------------------|-----|
| #DK.SSA-FA.1 | | | x | x | x |
| #DK.SSA-FA.2 | | | x | x | x |
| #DK.MSIW-FA.1 | | | x | x | x |
| #DK.MSWI-FA.4 | | | x | x | |
| #HR.WA.FA.1 | x | x | x | x | |
| #HR.WA.FA.2 | x | x | x | x | |
| #HR.WA.FA.3 | x | x | x | x | |
| #HR.WA.FA.8 | x | x | x | x | |
| #HR.WA.FA.10 | x | x | x | x | |
| #HR.WA.FA.11 | x | x | x | x | |
| #HR.WA.FA.12 | x | x | x | x | |
| #HR.WA.FA.16 | x | x | x | x | |
| #HR.WA.FA.17 | x | x | x | x | |
| #HR.WA.FA.19 | x | x | x | x | |
| #SI.WA.FA.1 | x | x | x | x | |
| #SI.CC.FA.1 | x | x | x | x | |
| #SI.CC.FA.3 | x | x | x | x | |
| #SI.CC.FA.4 | x | x | x | x | |

Table 2: Chemical properties – procedures performed on selected fly ashes

| Method / Sample ID | Water Content, pH, Conductivity, Leaching, water solubility | Reactivity Calorimetry | Reactivity Bound water | Free CaO | Alkali dissolution |
|--------------------|---|------------------------|------------------------|----------|--------------------|
| #DK.SSA-FA.1 | x | | | x | x |
| #DK.SSA-FA.2 | x | | | x | x |
| #DK.MSIW-FA.1 | x | | | x | x |
| #DK.MSWI-FA.4 | x | | | | |
| #HR.WA.FA.1 | | x | x | x | |
| #HR.WA.FA.2 | | x | x | x | |

| | | | | | |
|--------------|---|---|---|---|---|
| #HR.WA.FA.3 | | x | x | x | |
| #HR.WA.FA.8 | | x | x | x | |
| #HR.WA.FA.10 | | x | x | x | |
| #HR.WA.FA.11 | | x | x | x | |
| #HR.WA.FA.12 | | x | x | x | |
| #HR.WA.FA.16 | | x | x | x | x |
| #HR.WA.FA.17 | x | x | x | x | x |
| #HR.WA.FA.19 | | x | x | x | |
| #SI.WA.FA.1 | | x | x | x | |
| #SI.CC.FA.1 | | x | x | x | |
| #SI.CC.FA.3 | | x | x | x | |
| #SI.CC.FA.4 | | x | x | x | |

Table 3: Physical properties – procedures performed on selected ashes

| Method / Sample ID | Particle size distribution by laser | Bulk density | BET | Photo |
|--------------------|-------------------------------------|--------------|-----|-------|
| #DK.SSA-FA.1 | x | | | |
| #DK.SSA-FA.2 | x | | | |
| #DK.MSIW-FA.1 | x | | | |
| #DK.MSWI-FA.4 | x | | | |
| #HR.WA.FA.1 | x | x | | x |
| #HR.WA.FA.2 | x | x | | x |
| #HR.WA.FA.3 | x | x | | x |
| #HR.WA.FA.8 | x | x | | x |
| #HR.WA.FA.10 | x | x | | x |
| #HR.WA.FA.11 | x | x | | x |
| #HR.WA.FA.12 | x | x | | x |
| #HR.WA.FA.16 | x | x | | x |
| #HR.WA.FA.17 | x | x | | x |
| #HR.WA.FA.19 | x | x | | x |
| #SI.WA.FA.1 | x | x | x | x |
| #SI.CC.FA.1 | x | x | x | x |
| #SI.CC.FA.3 | x | x | x | x |
| #SI.CC.FA.4 | x | x | x | x |

1.2 Bottom Ashes

Table 4: Chemical composition – procedures performed on selected bottom ashes

| Method / Sample ID | XRF | Heavy metals, REEs and Hg | XRD | Loss of ignition |
|--------------------|-----|---------------------------|-----|------------------|
| #HR.WA.BA.4 | x | x | x | x |
| #HR.WA.BA.7 | x | x | x | x |
| #HR.WA.BA.13 | x | x | x | x |
| #HR.WA.BA.18 | x | x | x | x |
| #HR.WA.BA.20 | x | x | x | x |
| #HR.SSA.BA.1 | x | x | x | x |
| #HR.SSA.BA.2 | x | x | x | x |
| #HR.SSA.BA.3 | x | x | x | x |
| #HR.SSA.BA.4 | x | x | x | x |
| #SI.WA.BA.1 | x | x | x | x |
| #SI.CC.BA.3 | x | x | x | x |
| #SI.CC.BA.4 | x | x | x | x |

Table 5: Chemical properties – procedures performed on selected bottom ashes

| Method / Sample ID | Reactivity Calorimetry | Reactivity Bound water | Free CaO | Alkali dissolution |
|--------------------|------------------------|------------------------|----------|--------------------|
| #HR.WA.BA.4 | x | x | x | x |
| #HR.WA.BA.7 | x | x | x | |
| #HR.WA.BA.13 | x | x | x | |
| #HR.WA.BA.18 | x | x | x | |
| #HR.WA.BA.20 | x | x | x | |
| #HR.SSA.BA.1 | x | x | x | |
| #HR.SSA.BA.2 | to be tested again | to be tested again | x | |
| #HR.SSA.BA.3 | x | x | x | |
| #HR.SSA.BA.4 | x | x | x | |
| #SI.WA.BA.1 | x | x | x | |
| #SI.CC.BA.3 | x | x | x | |
| #SI.CC.BA.4 | x | x | x | |

Table 6: Physical properties – procedures performed on selected bottom ashes

| Method / Sample ID | Density pycnometer | Particle size distribution by sieving | BET | Photo |
|--------------------|--------------------|---------------------------------------|-----|-------|
| #HR.WA.BA.4 | x | x | | x |
| #HR.WA.BA.7 | x | x | | x |
| #HR.WA.BA.13 | x | x | | x |
| #HR.WA.BA.18 | x | x | | x |
| #HR.WA.BA.20 | x | x | | x |
| #HR.SSA.BA.1 | x | x | | x |
| #HR.SSA.BA.2 | x | x | | x |
| #HR.SSA.BA.3 | x | x | | x |
| #HR.SSA.BA.4 | x | x | | x |
| #SI.WA.BA.1 | x | x | x | x |
| #SI.CC.BA.3 | x | x | x | x |
| #SI.CC.BA.4 | x | x | x | x |

1.3 Mixed ashes

Table 7: Chemical composition – procedures performed on selected mixed ashes

| Method / Sample ID | XRF | Heavy metals, REEs and Hg | XRD | Loss of ignition |
|--------------------|-----|---------------------------|-----|------------------|
| #HR.WA.MA.5 | x | x | x | x |
| #HR.WA.MA.6 | x | x | x | x |
| #HR.WA.MA.9 | x | x | x | x |
| #HR.WA.MA.14 | x | x | x | x |
| #HR.WA.MA.15 | x | x | x | x |
| #SI.CC.MA.2 | x | x | x | x |

Table 8: Chemical properties – procedures performed on selected mixed ashes

| Method / Sample ID | Water Content, pH, Conductivity, Leaching, water solubility | Reactivity Calorimetry | Reactivity Bound water | Free CaO | Alkali dissolution |
|--------------------|---|------------------------|------------------------|----------|--------------------|
| #HR.WA.MA.5 | | x | x | x | |
| #HR.WA.MA.6 | x | x | x | x | x |
| #HR.WA.MA.9 | | x | x | x | |
| #HR.WA.MA.14 | x | x | x | x | x |
| #HR.WA.MA.15 | | x | x | x | |
| #SI.CC.MA.2 | | x | x | x | |

Table 9: Physical properties – procedures performed on selected mixed ashes

| Method / Sample ID | Density pycnometer | Particle size distribution by sieving | Particle size distribution by laser | BET | Photo |
|--------------------|--------------------|---------------------------------------|-------------------------------------|-----|-------|
| #HR.WA.MA.5 | x | x | | | x |
| #HR.WA.MA.6 | x | x | | | x |
| #HR.WA.MA.9 | x | x | | | x |
| #HR.WA.MA.14 | x | x | | | x |
| #HR.WA.MA.15 | x | x | | | x |
| #SI.CC.MA.2 | x | x | x | x | x |

2. Ash sampling reports

| MSWI | | Wood | | Sewage sludge | |
|---|---|---|---|---|---|
| Plant name – short #country.ashtype.no | sampling report available (yes/no) | Plant name – short #country.ashtype.no | sampling report available (yes/no) | Plant name – short #country.ashtype.no | sampling report available (yes/no) |
| #DK.MSWI.FA.1 | yes | #HR.WA.1 | yes | #DK.SSA.FA.1 | yes |
| #DK.MSWI.FA.4 | yes | #HR.WA.2 | yes | #DK.SSA.FA.2 | yes |
| #BE.MSWI.FA.1 | no | #HR.WA.3 | yes | #HR.SSA.1 | yes |
| #BE.MSWI.FA.3 | no | #HR.WA.4 | yes | #HR.SSA.2 | yes |
| #BE.MSWI.FA.4 | no | #HR.WA.5 | yes | #HR.SSA.3 | yes |
| #NL.MSWI.FA.1 | no | #HR.WA.6 | yes | #HR.SSA.4 | yes |
| #NL.MSWI.FA.2 | no | #HR.WA.7 | yes | #HR.SSA.5 | no |
| #NL.MSWI.FA.3 | no | #HR.WA.8 | yes | #NLSSA.1 | no |
| #NL.MSWI.FA.4 | no | #HR.WA.9 | yes | #BE.SSA.1 | no |
| #NL.MSWI.FA.5 | no | #HR.WA.10 | yes | #BE.SSA.2 | no |
| #NL.MSWI.FA.6 | no | #HR.WA.11 | yes | | |
| #NL.MSWI.FA.7 | no | #HR.WA.12 | yes | | |
| #NL.MSWI.FA.8 | no | #HR.WA.13 | yes | | |
| #NL.MSWI.FA.9 | no | #HR.WA.14 | yes | | |
| | | #SI.WA.1 | no | | |
| | | #BE.WA.1 | no | | |
| | | #BE.WA.2 | no | | |
| | | #NL.WA.1 | no | | |
| | | #NL.WA.2 | no | | |
| | | #NL.WA.3 | no | | |
| | | #NL.WA.4 | no | | |
| | | #NL.WA.5 | no | | |
| | | #NL.WA.6 | no | | |

| Co-combustion | | Other biomass | |
|---|---------------------------------------|---|---------------------------------------|
| Plant name – short #country.ashtype.no | sampling report available (yes/no) | Plant name – short #country.ashtype.no | sampling report available (yes/no) |
| #SI.CC.1 | no | #NL.PSFA.1 | no |
| #SI.CC.2 | no | | |
| #SI.CC.3 | no | | |
| #SI.CC.4 | no | | |
| #FI.CC.APC.1 | yes | | |
| #FI.CC.APC.2 | yes | | |
| #FI.CC.APC.3 | yes | | |
| #FI.CC.APC.4 | yes | | |
| #FI.CC.APC.5 | yes | | |
| #FI.CC.APC.6 | yes | | |
| #FI.CC.APC.7 | yes | | |
| #FI.CC.APC.8 | yes | | |
| #FI.CC.FA.1 | yes | | |
| #FI.CC.FA.2 | yes | | |
| #FI.CC.FA.3 | yes | | |
| #FI.CC.FA.4 | yes | | |
| #FI.CC.FA.5 | yes | | |
| #FI.CC.FA.6 | yes | | |
| #FI.CC.BA.1 | yes | | |
| #FI.CC.BA.2 | yes | | |
| #FI.CC.BA.3 | yes | | |
| #FI.CC.BA.4 | yes | | |
| #FI.CC.BA.5 | yes | | |
| #FI.CC.BA.6 | yes | | |
| #FI.CC.BA.7 | yes | | |
| #BE.CC.1 | no | | |
| #BE,MSWIFA,2 | no | | |

| | |
|--|--|
| Sample ID: | #DK.SSA-FA.1 |
| Company/Site: | BIOFOS Rensningsanlæg Avedøre/Kanalholmen 28, Hvidovre |
| Country: | Denmark |
| Owner of the material: | Lisbeth M. Ottosen |
| Type of material (CODE): | SSA-FA |
| Sampling performed by: (person and institution) | Michael Erikson, BIOFOS and Ebba Schnell DTU Sustain |
| Amount of sample (kg): | 5 x 20 kg |
| Date for sampling | 08-03-2023 |



Sample description, sampling procedure and observation:

Moistened FA – a small portion was taken from the container with a excavator before the container was emptied on the stockpile. FA was filled in 5 x 20L buckets.

Procedure for moistened FA – FA is moistened during the transport from silo into the container. A belt distributes the ash across the container in a even layer (back and forth) until the container is full and is transported to the stockpiles where it is covered with gravel

Photographs:

- #DK.SSA-FA.1-01 – ash taken direct from the container
- #DK.SSA-FA.1-02 – ash taken into buckets
- #DK.SSA-FA.1-03 – moist ash from silo distributed into the container
- #DK.SSA-FA.1-04 – container
- #DK.SSA-FA.1-05 – overview of the system
- #DK.SSA-FA.1-06 – overview of the system

Notes:

| | |
|--|---|
| Sample ID: | #DK.SSA-FA.2 |
| Company/Site: | BIOFOS rensningsanlæg Lynetten/Refshalevej 250, KBH |
| Country: | Denmark |
| Owner of the material: | Lisbeth M. Ottosen |
| Type of material (CODE): | SSA-FA |
| Sampling performed by: (person and institution) | Ken Andersen BIOFOS and Ebba Schnell DTU Sustain |
| Amount of sample (kg): | 7 x 30 kg |
| Date for sampling | 08-03-2023 |

**Sample description, sampling procedure and observation:**

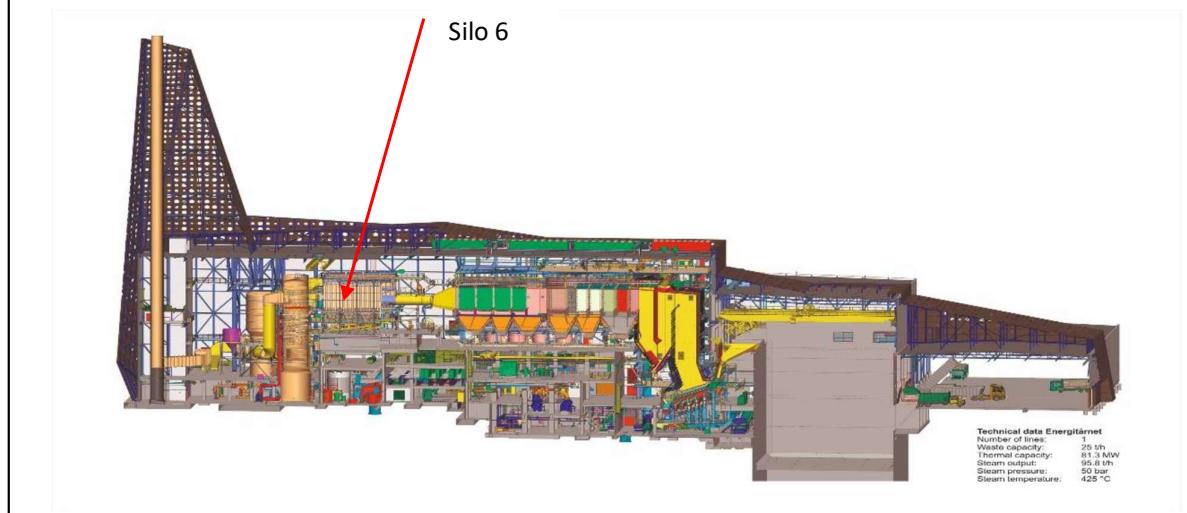
Dry FA - taken from a small outlet from Silo no. 1 by the personal due to safety into 7 x 30L buckets. The outlet was around 12 cm in diameter.

Photographs:

- #DK.SSA-FA.2-01 – oven where the ash sludge is mixed with sand and burned at 800°C
- #DK.SSA-FA.2-02 – Place where the sample is taken out from silo no. 1
- #DK.SSA-FA.2-03 – overview of the system and the place the sample is taken out
- #DK.SSA-FA.2-04 – overview of the silo system
- #DK.SSA-FA.2-05 – overview of the new system

Notes:

| | |
|--|--|
| Sample ID: | #DK.MSWI-FA.1 |
| Company/Site: | ARGO/Håndværkervej 70, 4000 Roskilde |
| Country: | Denmark |
| Owner of the material: | Lisbeth M. Ottosen |
| Type of material (CODE): | MSWI-FA |
| Sampling performed by: (person and institution) | Niels Kallehauge, ARGO and Ebba Schnell, DTU Sustain |
| Amount of sample (kg): | 5 x 10 kg |
| Date for sampling: | 17-02-2023 |



Sample description, sampling procedure and observation:

The FA was taken direct from silo no. 6 by ARGO's own personal.
Aperture was over 200 mm in size. The FA was collected in 5 x 10L buckets

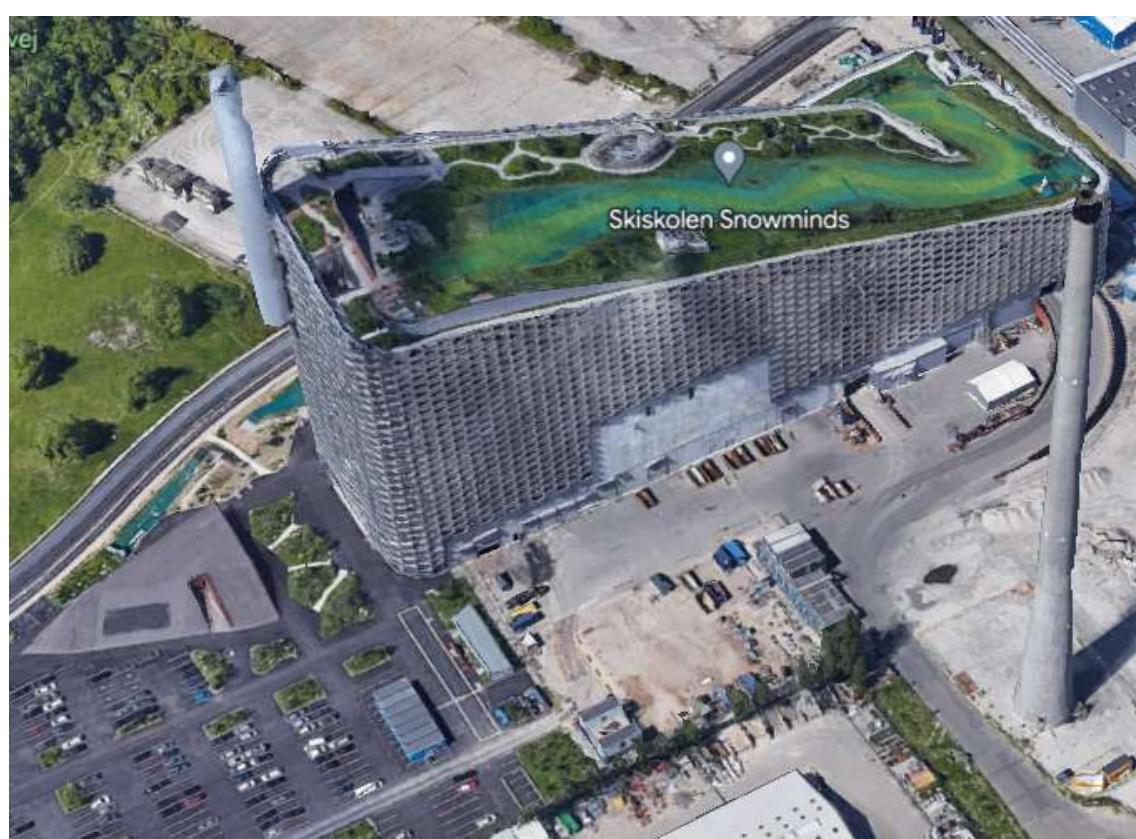
Photographs:

DK.MSWI-FA.1-01 – aperture from silo no. 6

Notes:

5 buckets marked no. 1-2-3-4-5

| | |
|--|--|
| Sample ID: | #DK.MSWI-FA.4 |
| Company/Site: | ARC, Vindmøllevej 6, 2300 KBH S |
| Country: | Denmark |
| Owner of the material: | Lisbeth M. Ottosen |
| Type of material (CODE): | MSWI-FA |
| Sampling performed by: (person and institution) | Jonas Nedenskov, ARC and Ebba Schnell, DTU Sustain |
| Amount of sample (kg): | 3 x 15 kg |
| Date for sampling | 17-04-2023 |



Sample description, sampling procedure and observation:

The FA are a mix of FA from the boiler plant and the ESP plant. The FA are mixed before sending to the silo for storage.

The FA are taken for the same outlet as the FA from the ESP Plant.

The sample was taken by ARC's own personal using a tube of 200mm in size that was connected to the plant.

The FA was collected in 3 x 20L buckets on site

Photographs:

#DK.MSWI-FA.4-01 – the Outlet for FA before storage in the silo

#DK.MSWI-FA.4-02 – the overview of the outlet
#DK.MSWI-FA.4-03 – graphic overview of the outlet

Notes:

| | |
|--|---|
| Sample ID: | #HR.WA.10 |
| Company/Site: | Energana Gospić, Smiljansko Polje 71, 53211, Gospić |
| Country: | Croatia |
| Owner of the material: | Nina Štirmer |
| Type of material (CODE): | WA-FA |
| Sampling performed by: (person and institution) | Jelena Šantek Bajto, UNIZG & Suzana Hozmec, BL |
| Amount of sample (kg): | 4 x 20 kg |
| Date for sampling | 7-9-2022 |



Sample description, sampling procedure and observation:

FA was taken directly from a closed bag in which it is usually collected and placed outside the plant, while the FA was secured from weathering.

Photographs:

#HR.WA.10 – 01 – energy plant

#HR.WA.10 – 02 – collecting the FA in a large storage bag

Notes:

FA was packed in 4 plastic bags with a volume of about 20 kg.

| | |
|--|---|
| Sample ID: | #HR.WA.14 |
| Company/Site: | BIO ENERGANA BJELOVAR d.o.o., Pakračka ulica bb, 43 000, Bjelovar |
| Country: | Croatia |
| Owner of the material: | Nina Štirmer |
| Type of material (CODE): | WA-MA |
| Sampling performed by: (person and institution) | Jelena Šantek Bajto, UNIZG & Suzana Hozmec, BL |
| Amount of sample (kg): | 5 x 20 kg |
| Date for sampling | 28-9-2022 |



| |
|--|
| Sample description, sampling procedure and observation: |
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|--|
| MA was collected directly from a closed joint container located outside the plant. |
|--|

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| Photographs: |
|---------------------|

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|---|
| #HR.WA.14 – 01 - joint container for MA |
|---|

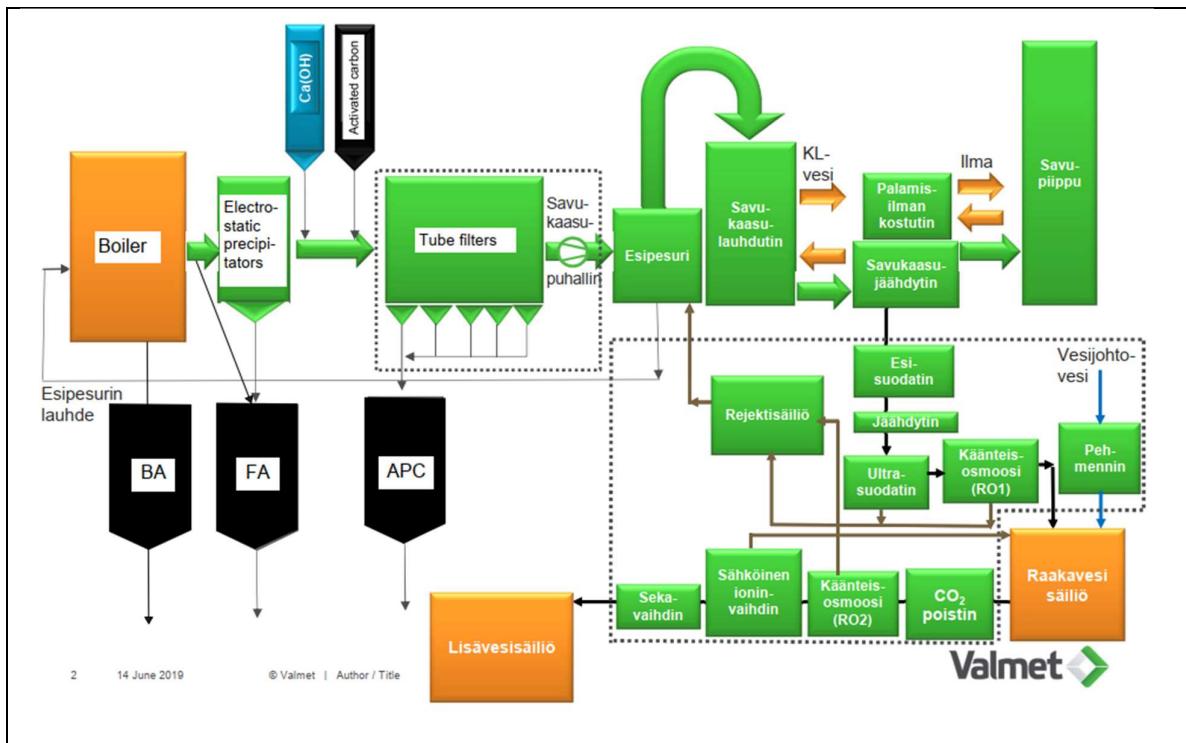
| |
|-------------------------------|
| #HR.WA.14 – 02 – energy plant |
|-------------------------------|

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| Notes: |
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|---|
| MA was packed in 4 plastic bags with a volume of about 50 kg. |
|---|

| | |
|--|---|
| Sample ID: | #FI.CC.1 |
| Company/Site: | Oulun Energia, OEBIO powerplant, Typpitie 3, 90650 Oulu |
| Country: | Finland |
| Owner of the material: | Tero Luukkonen |
| Type of material (CODE): | CC-FA, CC-APC, and CC-BA |
| Sampling performed by: (person and institution) | OEBIO powerplant personnel |
| Amount of sample (kg): | 1.5 kg of each sample |
| Date for sampling | 22 samples between: 10.10.2022 – 20.1.2023 |





Sample description, sampling procedure and observation:

Samples of fly ash (collected from electrostatic precipitators), air pollution control residue (sampled from tube filters after dosing $\text{Ca}(\text{OH})_2$ and activated carbon to fly ash), and bottom ash were collected between 10.10.2022 – 20.1.2023 on 8 occasions. Each sample is a composite sample collected over one week duration. Sampling was done the OEBIO plant personnel.

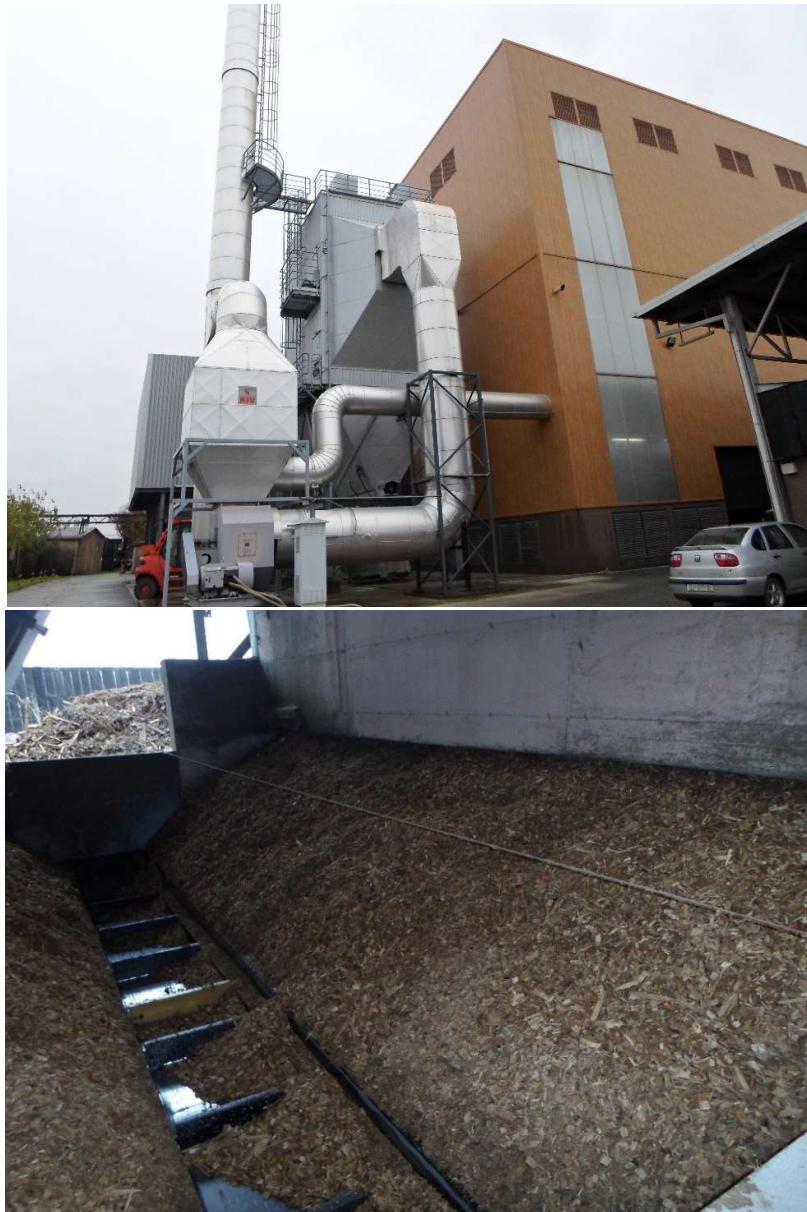
Photographs:

| |
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Notes:

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| | |
|--|--|
| Sample ID: | #HR.WA-FA.2 / #HR.WA-FA.3 / #HR.WA-BA.4 |
| Company/Site: | Strizivojna Hrast d.o.o./ B.Radića 82, 31410 Strizivojna |
| Country: | Croatia |
| Owner of the material: | Nina Štirmer |
| Type of material (CODE): | WA-FA (fine fly) / WA-FA (coarse fly) / WA-BA |
| Sampling performed by: (person and institution) | Ivana Carević, UNIZG and Strizivojna Hrast d.o.o.'s personal |
| Amount of sample (kg): | 3 x 10 kg |
| Date for sampling | 11-7-2022 |



Sample description, sampling procedure and observation:

WBA was taken by Strizivojna Hrast d.o.o.'s own personal from an individual pile for each ash type inside the plant.

Photographs:

#HR.WA-FA.2-FA.3-BA.4 - 01 – plant

#HR.WA-FA.2-FA.3-BA.4 - 02 – wood biomass

Notes:

3 bags marked HR.WA.2 (STRI fine fly) / HR.WA.3 (STRI coarse fly) / HR.WA.4 (STRI bottom)

| | |
|--|--|
| Sample ID: | #HR.WA-FA.11 |
| Company/Site: | BE-TO Brinje d.o.o., Lucane 59, 53260 Brinje |
| Country: | Croatia |
| Owner of the material: | Nina Štirmer |
| Type of material (CODE): | WA-FA |
| Sampling performed by: (person and institution) | Jelena Šantek Bajto, UNIZG; Suzana Hozmec, BL; BE-TO Brinje do.o.'s personal |
| Amount of sample (kg): | 1 x 10 kg |
| Date for sampling | 7-9-2022 |



| |
|--|
| Sample description, sampling procedure and observation: |
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|---|
| WBA was taken by BE-TO Brinje d.o.o.'s own personal from the container outside the plant. |
|---|

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| Photographs: |
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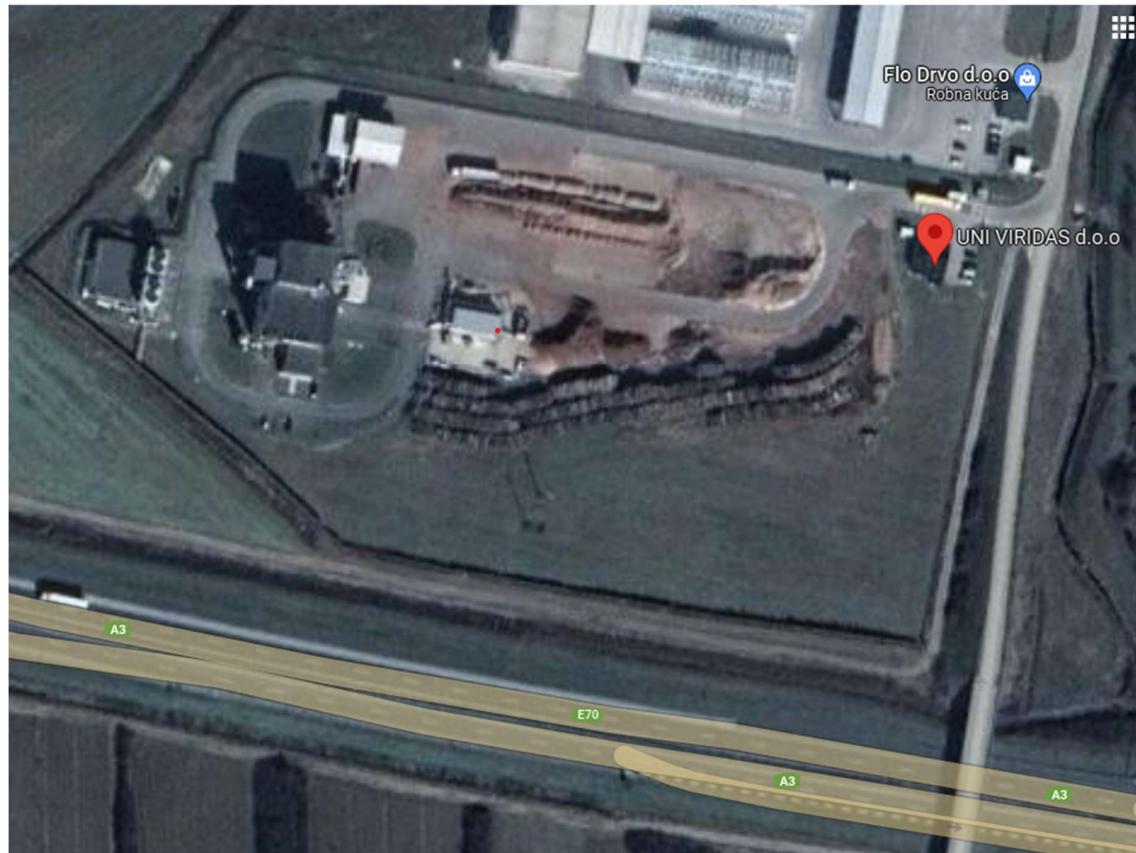
| |
|--|
| #HR.WA-MA.11 - 01 – transported ash to outside container |
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|--------------------------------|
| #HR.WA-MA.11 - 02 – stored ash |
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| Notes: |
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|--------------------------------|
| 1 bag marked HR.WA.11 (Brinje) |
|--------------------------------|

| | |
|--|--|
| Sample ID: | #HR.WA-FA.12 / #HR.WA-BA.13 |
| Company/Site: | Uni Viridas d.o.o./ 32276 Babina Greda |
| Country: | Croatia |
| Owner of the material: | Nina Štirmer |
| Type of material (CODE): | WA-FA / WA-BA |
| Sampling performed by: (person and institution) | Ivana Carević, UNIZG and Uni Viridas d.o.o.'s personal |
| Amount of sample (kg): | 2 x 10 kg |
| Date for sampling | 23-9-2022 |



| |
|--|
| Sample description, sampling procedure and observation: |
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| |
|--|
| WBA was taken by Uni Viridas d.o.o.'s own personal from the container outside the plant. |
|--|

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|---------------------|
| Photographs: |
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|---|
| #HR.WA-FA.12-BA.13 - 01 – powerplant location |
|---|

| |
|---------------|
| Notes: |
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|--|
| 2 bags marked HR.WA.12 (BG fly) and HR.WA.13 (BG bottom) |
|--|

| | |
|--|---|
| Sample ID: | #HR.WA-MA.9 |
| Company/Site: | Spačva d.d./ Duga 181, 32100 Vinkovci |
| Country: | Croatia |
| Owner of the material: | Nina Štirmer |
| Type of material (CODE): | WA-MA |
| Sampling performed by: (person and institution) | Bojan Milovanović, UNIZG and Spačva d.d.'s personal |
| Amount of sample (kg): | 1 x 10 kg |
| Date for sampling | 3-9-2022 |



Sample description, sampling procedure and observation:

WBA was taken by Spačva d.d.'s own personal from a container outside the plant.

Photographs:

#HR.WA-MA.9 - 01 – a container where WA was collected
#HR.WA-MA.9 - 02 – wood biomass and plant behind

Notes:

1 bag marked HR.WA.2 (VI, Spačva)

| | |
|--|--|
| Sample ID: | #HR.WA-FA.1 |
| Company/Site: | SLAVONIJA OIE d.o.o./Svetog Lovre 75, 35000 Slavonski Brod |
| Country: | Croatia |
| Owner of the material: | Nina Štirmer |
| Type of material (CODE): | WA-FA |
| Sampling performed by: (person and institution) | Sonja Cerković, UNIZG |
| Amount of sample (kg): | 1 x 10 kg |
| Date for sampling | 8-07-2022 |



Sample description, sampling procedure and observation:

WA-FA was taken by SLAVONIJA OIE's own personal from a pile of fly ash inside the plant and transfer to Zagreb by Sonja Cerković.

Photographs:

#HR.WA-FA.1-01 – plant SLAVONIJA OIE d.o.o.in Slavonski Brod

#HR.WA-FA.1-02 – wood biomass (preparation of wood chips)

Notes:

1 bag marked SBfly 8/07/2022

| | |
|--|---|
| Sample ID: | #HR.WA-MA.5 |
| Company/Site: | SPIN VALIS INTERNACIONAL d.o.o./Industrijska 24, 34000 Požega |
| Country: | Croatia |
| Owner of the material: | Nina Štirmer |
| Type of material (CODE): | WA-MA |
| Sampling performed by: (person and institution) | Sonja Cerković, UNIZG |
| Amount of sample (kg): | 1 x 10 kg |
| Date for sampling | 15-07-2022 |



Sample description, sampling procedure and observation:

WA-MA was taken by Sonja Cerković from container in plant and transfer to Zagreb.

Photographs:

#HR.WA-BA.1-01 –plant Spin Valis International d.o.o.

#HR.WA-MA.1-02 – container in plant for collecting MA

Notes:

1 bag marked PŽmix 15/07/2022

| | |
|--|--|
| Sample ID: | #HR.WA-MA.6 |
| Company/Site: | MAKSIM TRADE ENERGIJA d.o.o., Žakanje 58, 47276, Žakanje |
| Country: | Croatia |
| Owner of the material: | Nina Štirmer |
| Type of material (CODE): | WA-MA |
| Sampling performed by: (person and institution) | Jelena Šantek Bajto, UNIZG & Suzana Hozmec, BL |
| Amount of sample (kg): | 2 x 20 kg |
| Date for sampling | 15-07-2022 |



Sample description, sampling procedure and observation:

WA-MA was taken by Jelena Šantek Bajto and BL from container in plant and transfer to Zagreb.

Photographs:

#HR.WA-MA.1-01 – plant MAKSIM TRADE ŽAKANJE d.o.o.

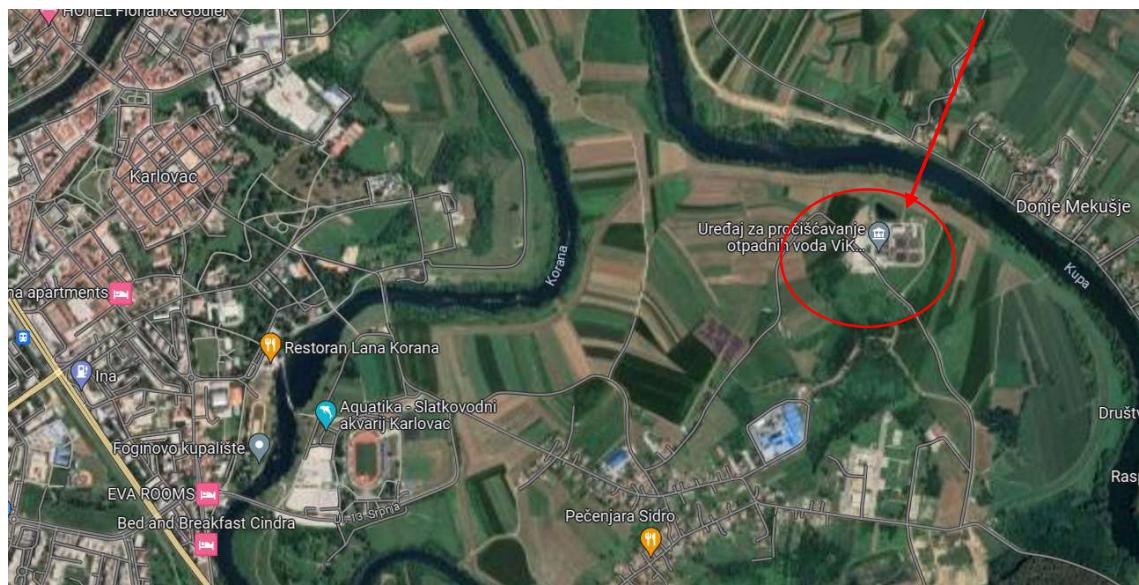
#HR.WA-MA.1-02 – container in plant for collecting MA

Notes:

1 bag marked ŽKmix 26/08/2022

| | |
|--|---|
| Sample ID: | #HR.SS.BA.1 |
| Company/Site: | Uredaj za pročišćavanje otpadnih voda ViK Karlovac, 47000,Karlovac |
| Country: | Croatia |
| Owner of the material: | Nina Štirmer |
| Type of material (CODE): | Sewage sludge - HR |
| Sampling performed by: (person and institution) | Danica Maljković, DOK and Morana Drušković, DOK |
| Amount of sample (kg): | 150 kg |
| Date for sampling | 07-09-2022 |

WWTP Karlovac





| |
|--|
| Sample description, sampling procedure and observation: |
|--|

SS was collected directly from the pile, which is protected by a roof and surrounded by a wall and is located in the circle of the wastewater treatment plant. SS is collected in plastic containers with a volume of 300 L.

| |
|---------------------|
| Photographs: |
|---------------------|

#HR.SS – location of WWTP
#HR.SS – WWTP Karlovac

| |
|---------------|
| Notes: |
|---------------|

1 buckets marked – SS KA

| | |
|--|---|
| Sample ID: | #HR.SS.BA.4 |
| Company/Site: | Wastewater treatment plant Našice |
| Country: | Croatia |
| Owner of the material: | Nina Štirmer |
| Type of material (CODE): | Sewage sludge - HR |
| Sampling performed by: (person and institution) | Danica Maljković, DOK and Morana Drušković, DOK |
| Amount of sample (kg): | 150 kg |
| Date for sampling | 14-10-2022 |





Sample description, sampling procedure and observation:

SS was collected directly from the pile, which is protected by a roof and surrounded by a wall and is located in the circle of the wastewater treatment plant is located in the circle of the wastewater treatment plant. SS is collected in plastic containers with a volume of 300 L.

Photographs:

#HR.SS – location of WWTP Našice
#HR.SS – WWTP Našice

Notes:

1 buckets marked – SS NA

| | |
|--|---|
| Sample ID: | #HR.SS.BA.3 |
| Company/Site: | Wastewater treatment plant Sisak |
| Country: | Croatia |
| Owner of the material: | Nina Štirmer |
| Type of material (CODE): | Sewage sludge - HR |
| Sampling performed by: (person and institution) | Danica Maljković, DOK and Morana Drušković, DOK |
| Amount of sample (kg): | 150 kg |
| Date for sampling | 24-10-2022 |

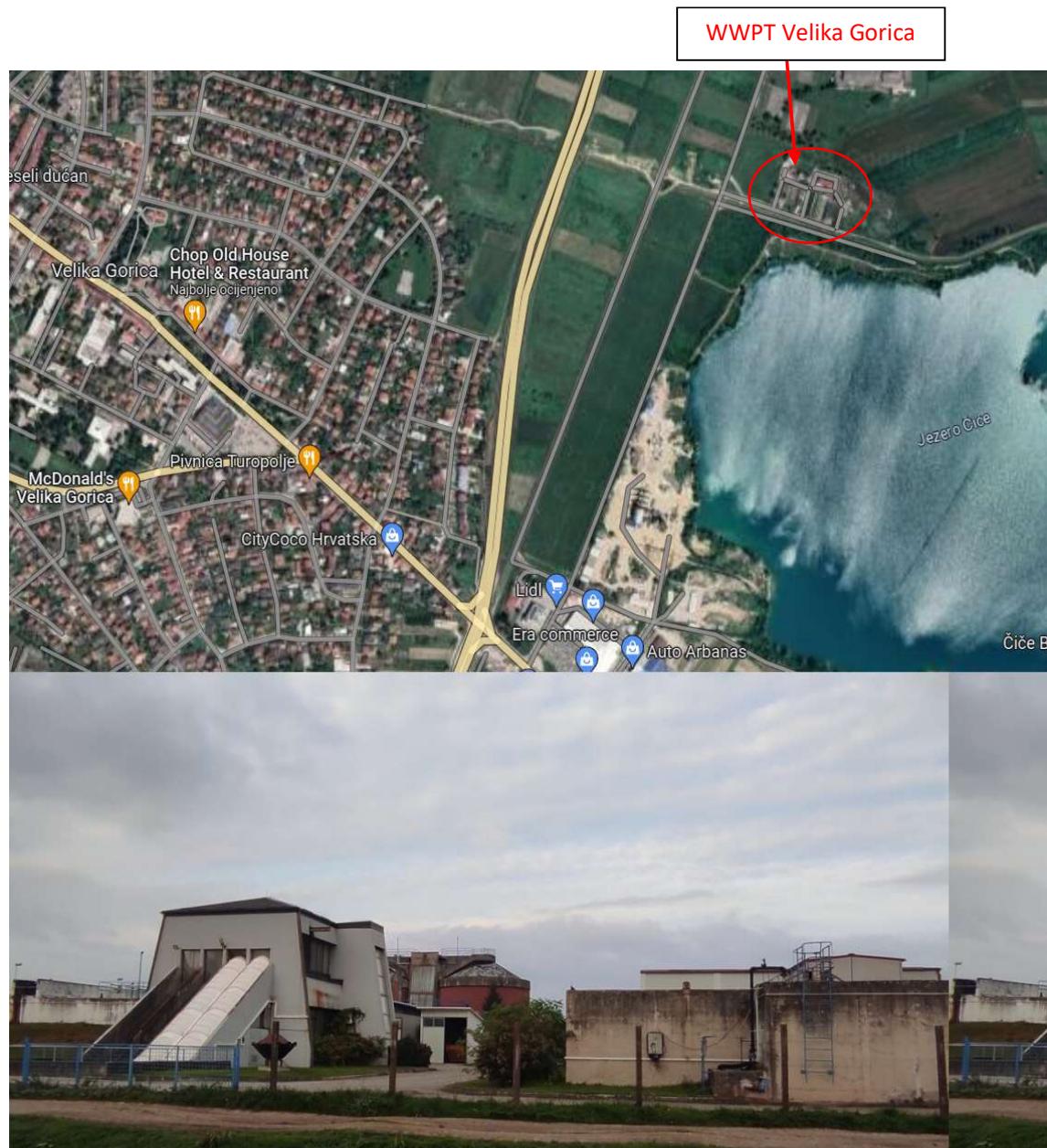


| |
|--|
| Sample description, sampling procedure and observation: |
| SS was collected directly from the pile. Sewage sludge is located in the circle of the wastewater treatment plant. SS is collected in plastic containers with a volume of 300 L. |

| |
|---------------------------------|
| Photographs: |
| #HR.SS – location of WWTP Sisak |

| |
|--------------------------|
| Notes: |
| 1 buckets marked – SS NA |

| | |
|--|---|
| Sample ID: | #HR.SS.BA.2 |
| Company/Site: | Wastewater treatment plant VG |
| Country: | Croatia |
| Owner of the material: | Nina Štirmer |
| Type of material (CODE): | Sewage sludge - HR |
| Sampling performed by: (person and institution) | Danica Maljković, DOK and Morana Drušković, DOK |
| Amount of sample (kg): | 150 kg |
| Date for sampling | 28-09-2022 |



Sample description, sampling procedure and observation:

SS was collected directly from the pile. Sewage sludge is located in the circle of the wastewater treatment plant. SS is collected in plastic containers with a volume of 300 L.

Photographs:

#HR.SS – location of WWTP Velika Gorica
#HR.SS – WWTP Velika Gorica

Notes:

1 buckets marked – SS VG

3. Results

3.1 X-ray fluorescence (XRF) analysis

Table 1. Wood fly ashes

| Compound | # | # | # | # | # | # | # | # | # | # | # | # | # | # |
|--------------------------------|------------|------------|------------|------------|-------------|-------------|-------------|-------------|-------------|-------------|------------|--------|--------|---|
| | HR.WA.FA.1 | HR.WA.FA.2 | HR.WA.FA.3 | HR.WA.FA.8 | HR.WA.FA.10 | HR.WA.FA.11 | HR.WA.FA.12 | HR.WA.FA.16 | HR.WA.FA.17 | HR.WA.FA.19 | SI.WA.FA.1 | NL WA2 | NL WA4 | |
| | wt.% | wt.% | wt.% | wt.% | wt.% | wt.% | wt.% | wt.% | wt.% | wt.% | wt.% | wt.% | wt.% | |
| SiO ₂ | 18,76 | 1,16 | 10,46 | 7,23 | 39,88 | 3,41 | 25,63 | 14,56 | 0,68 | 11,88 | 25,04 | 2,71 | 4,67 | |
| CaO | 45,79 | 49,70 | 68,62 | 52,27 | 25,19 | 50,83 | 35,82 | 42,60 | 13,96 | 55,41 | 46,15 | 6,06 | 51,87 | |
| Al ₂ O ₃ | 4,71 | 0,41 | 3,08 | 2,25 | 11,53 | 1,21 | 9,37 | 4,61 | 0,19 | 3,24 | 5,64 | 0,66 | 1,17 | |
| Fe ₂ O ₃ | 2,52 | 0,43 | 1,95 | 1,41 | 4,26 | 0,81 | 4,87 | 2,09 | 0,40 | 1,96 | 2,83 | 0,75 | 2,00 | |
| Na ₂ O | 0,92 | 2,29 | 0,42 | 0,61 | 0,26 | 0,24 | 0,82 | 0,89 | 2,26 | 0,65 | 0,70 | 30,29 | 7,90 | |
| K ₂ O | 12,30 | 23,28 | 4,87 | 16,50 | 8,23 | 23,62 | 8,10 | 9,63 | 58,73 | 11,61 | 8,39 | 12,94 | 3,67 | |
| SO ₃ | 4,27 | 12,32 | 1,06 | 7,66 | 3,16 | 7,46 | 4,99 | 13,78 | 17,94 | 5,73 | 2,25 | 15,64 | 6,87 | |
| Cl | 0,16 | 0,50 | 0,03 | 0,27 | 0,40 | 0,30 | 0,09 | 0,81 | 0,40 | 0,19 | / | 27,39 | 10,43 | |
| P ₂ O ₅ | 2,78 | 2,34 | 4,33 | 3,66 | 2,91 | 2,51 | 2,93 | 4,32 | 1,24 | 2,61 | 2,88 | 1,23 | 0,69 | |
| MgO | 3,79 | 3,36 | 4,02 | 5,73 | 2,62 | 6,80 | 6,42 | 4,78 | 1,50 | 4,43 | 4,58 | 1,07 | 1,67 | |
| ZnO | 0,06 | 0,05 | 0,01 | 0,11 | 0,10 | 0,11 | 0,09 | 0,17 | 0,12 | 0,08 | / | 0,69 | 5,16 | |
| CuO | 0,02 | 0,04 | 0,03 | 0,02 | 0,01 | 0,03 | 0,01 | 0,02 | 0,05 | 0,03 | / | 0,04 | 0,14 | |
| TiO ₂ | 0,42 | 0,04 | 0,26 | 0,18 | 0,81 | 0,09 | 0,51 | 0,31 | 0,02 | 0,27 | 0,61 | 0,17 | 1,19 | |
| MnO | 0,65 | 0,28 | 0,46 | 0,80 | 0,53 | 0,50 | 0,16 | 0,72 | 0,70 | 1,11 | 0,63 | 0,06 | 0,34 | |
| PbO | / | / | / | / | / | / | / | / | / | / | / | 0,13 | 1,23 | |
| Cr ₂ O ₃ | 0,01 | 0,01 | 0,01 | 0,01 | 0,02 | 0,01 | 0,02 | 0,01 | 0,05 | 0,01 | 0,03 | 0,02 | 0,10 | |
| BaO | 0,17 | 0,14 | 0,24 | 0,27 | 0,10 | 0,14 | 0,09 | 0,11 | 0,21 | 0,38 | 0,19 | 0,01 | 0,23 | |
| NiO | / | / | / | / | / | / | / | / | / | / | / | 0,01 | 0,02 | |

| SrO | 0,11 | 0,11 | 0,19 | 0,12 | 0,03 | 0,06 | 0,11 | 0,09 | 0,06 | 0,15 | 0,08 | 0,01 | 0,09 |
|--------------------------------|-------|-------|--------|-------|--------|-------|--------|-------|-------|-------|--------|------|------|
| SnO ₂ | / | / | / | / | / | / | / | / | / | / | / | 0,01 | 0,05 |
| As ₂ O ₃ | / | / | / | / | / | / | / | / | / | / | / | 0,01 | 0,04 |
| Sum | 97,42 | 96,43 | 100,00 | 99,08 | 100,00 | 98,11 | 100,00 | 99,46 | 98,51 | 99,71 | 100,00 | | |

Table 2. Wood bottom ashes

| Compound | # HR.WA.BA.4 | # HR.WA.MA.5 | # HR.WA.MA.6 | # HR.WA.BA.7 | # HR.WA.MA.9 | # HR.WA.BA.13 | # HR.WA.MA.14 | # HR.WA.MA.15 | # HR.WA.BA.18 | # HR.WA.BA.20 | # SI.WA.BA.1 |
|--------------------------------|--------------|--------------|--------------|--------------|--------------|---------------|---------------|---------------|---------------|---------------|--------------|
| | wt.% | wt.% | wt.% | wt.% | wt.% | wt.% | wt.% | wt.% | wt.% | wt.% | wt.% |
| SiO ₂ | 16,46 | 20,89 | 23,21 | 38,05 | 7,74 | 82,85 | 33,60 | 11,15 | 9,55 | 37,07 | 7,007 |
| CaO | 59,13 | 48,42 | 44,14 | 26,29 | 79,72 | 6,77 | 33,27 | 67,95 | 64,45 | 35,23 | 64,645 |
| Al ₂ O ₃ | 3,99 | 4,96 | 6,47 | 9,55 | 1,63 | 2,81 | 7,55 | 2,81 | 2,32 | 7,51 | 2,208 |
| Fe ₂ O ₃ | 2,06 | 2,78 | 3,31 | 6,34 | 0,97 | 1,03 | 3,02 | 1,62 | 2,01 | 3,95 | 1,025 |
| Na ₂ O | 0,73 | 1,02 | 0,69 | 0,50 | 0,00 | 0,26 | 1,18 | 0,78 | 0,00 | 0,67 | 0,157 |
| K ₂ O | 9,37 | 10,80 | 9,47 | 9,25 | 3,31 | 3,49 | 9,37 | 6,42 | 10,03 | 7,13 | 12,091 |
| SO ₃ | 0,54 | 1,23 | 2,99 | 0,59 | 1,57 | 0,24 | 1,18 | 1,42 | 1,79 | 0,28 | 0,575 |
| Cl | 0,00 | 0,00 | 0,14 | 0,01 | 0,04 | 0,00 | 0,02 | 0,00 | 0,05 | 0,00 | / |
| P ₂ O ₅ | 3,00 | 3,38 | 3,98 | 2,39 | 1,60 | 0,99 | 2,83 | 2,31 | 2,69 | 2,12 | 3,461 |
| MgO | 3,71 | 4,44 | 3,55 | 4,69 | 2,67 | 0,95 | 5,66 | 3,14 | 4,33 | 3,75 | 6,893 |
| ZnO | 0,00 | 0,02 | 0,07 | 0,03 | 0,01 | 0,02 | 0,02 | 0,03 | 0,01 | 0,01 | / |
| CuO | 0,02 | 0,02 | 0,01 | 0,01 | 0,03 | 0,00 | 0,02 | 0,02 | 0,01 | 0,01 | / |
| TiO ₂ | 0,25 | 0,32 | 0,50 | 0,61 | 0,14 | 0,11 | 0,50 | 0,20 | 0,19 | 0,45 | 0,114 |
| MnO | 0,43 | 0,97 | 1,20 | 0,52 | 0,31 | 0,03 | 0,48 | 0,97 | 1,99 | 0,82 | 1,268 |
| PbO | / | / | / | / | / | / | / | / | / | / | / |
| Cr ₂ O ₃ | 0,03 | 0,02 | 0,04 | 0,02 | 0,01 | 0,06 | 0,01 | 0,01 | 0,04 | 0,02 | 0,028 |
| BaO | 0,14 | 0,32 | 0,14 | 0,15 | 0,10 | 0,04 | 0,13 | 0,36 | 0,45 | 0,29 | 0,413 |
| NiO | / | / | / | / | / | / | / | / | / | / | / |
| SrO | 0,16 | 0,19 | 0,10 | 0,05 | 0,17 | 0,02 | 0,10 | 0,15 | 0,19 | 0,10 | 0,115 |
| SnO ₂ | / | / | / | / | / | / | / | / | / | / | / |
| As ₂ O ₃ | / | / | / | / | / | / | / | / | / | / | / |
| Sum | 100,00 | 99,74 | 100,00 | 99,04 | 100,00 | 99,65 | 98,90 | 99,31 | 100,10 | 99,39 | 100 |

Table 3. MSWI bottom ashes

| Compound | #NL MSWI BA1 |
|--------------------------------|--------------|
| | wt.% |
| SiO ₂ | 47.73 |
| CaO | 14.70 |
| Al ₂ O ₃ | 11.05 |
| Fe ₂ O ₃ | 14.12 |
| Na ₂ O | 3.93 |
| K ₂ O | 0.84 |
| SO ₃ | 1.71 |
| Cl | 0.28 |
| P ₂ O ₅ | 0.90 |
| MgO | 1.84 |
| ZnO | 0.63 |
| CuO | 0.50 |
| TiO ₂ | 0.95 |
| MnO | 0.17 |
| PbO | 0.13 |
| Cr ₂ O ₃ | 0.09 |
| BaO | 0.28 |
| NiO | 0.03 |
| SrO | 0.04 |
| SnO ₂ | 0.03 |



Table 4. MSWI fly ashes

Table 5. MSWI APC ashes

| Compound | #NL MSWI APC5 | #NL MSWI APC6A | #NL MSWI APC6B |
|--------------------------------|---------------|----------------|----------------|
| | wt.% | wt.% | wt.% |
| SiO ₂ | 7.19 | 0.42 | 0.47 |
| CaO | 21.63 | 2.34 | 39.40 |
| Al ₂ O ₃ | 1.87 | 0.13 | 0.14 |
| Fe ₂ O ₃ | 1.14 | 0.11 | 0.18 |
| Na ₂ O | 19.41 | 51.32 | 7.48 |
| K ₂ O | 8.20 | 0.26 | 0.36 |
| SO ₃ | 11.72 | 8.67 | 6.53 |
| Cl | 19.31 | 35.82 | 43.39 |
| P ₂ O ₅ | 0.86 | 0.05 | 0.02 |
| MgO | 1.05 | 0.17 | 0.47 |
| ZnO | 4.31 | 0.35 | 0.24 |
| CuO | 0.90 | 0.02 | 0.10 |
| TiO ₂ | 0.73 | 0.02 | NG |
| MnO | 0.04 | NG | 0.01 |
| PbO | 0.60 | 0.06 | 0.04 |
| Cr ₂ O ₃ | 0.05 | NG | NG |
| BaO | 0.09 | NG | NG |
| NiO | 0.02 | NG | NG |
| SrO | 0.04 | 0.00 | 0.03 |
| SnO ₂ | 0.16 | 0.04 | NG |

| | | | |
|--------------------------------|----|----|-------|
| As ₂ O ₃ | NG | NG | 0.004 |
|--------------------------------|----|----|-------|

Table 6. Sewage sludge ashes

| Compound | HR.SSA.BA.1 | HR.SSA.BA.2 | HR.SSA.BA.3 | HR.SSA.BA.4 |
|--------------------------------|-------------|-------------|-------------|-------------|
| | wt.% | wt.% | wt.% | wt.% |
| SiO ₂ | 28,01 | 21,84 | 38,68 | 41,91 |
| CaO | 19,62 | 19,25 | 9,61 | 7,43 |
| Al ₂ O ₃ | 10,87 | 7,47 | 13,8 | 13,42 |
| Fe ₂ O ₃ | 5,97 | 4,03 | 6,91 | 8,62 |
| Na ₂ O | 0,66 | 0,88 | 0,69 | 1,3 |
| K ₂ O | 1,22 | 1,51 | 2,38 | 2 |
| SO ₃ | 3,85 | 3,96 | 2,18 | 1,63 |
| Cl | 0,07 | 0,325 | 0,026 | 0,037 |
| P ₂ O ₅ | 11,5 | 6,78 | 8,81 | 6,89 |
| MgO | 1,73 | 2,45 | 3,08 | 2,3 |
| ZnO | 0,13 | 0,171 | 0,059 | 0,105 |
| CuO | 0,026 | 0,09 | 0,305 | 0,028 |
| TiO ₂ | 0,581 | 0,765 | 0,741 | 0,715 |
| MnO | 0,098 | 0,094 | 0,16 | 0,23 |
| PbO | / | / | / | / |
| Cr ₂ O ₃ | 0,066 | 0,066 | 0,051 | 0,075 |
| BaO | 0,069 | 0,085 | 0,09 | 0,116 |
| NiO | / | / | / | / |
| SrO | 0,033 | 0,022 | 0,028 | 0,029 |
| SnO ₂ | / | / | / | / |
| As ₂ O ₃ | / | / | / | / |
| Sum | 84,503 | 69,788 | 87,6 | 86,835 |

Table 7. Other ashes

| Compound | #FI.CC.BA1 | #FI.CC.FA1 | #FI.CC.APC1 | #SI.CC.FA.1 | #SI.CC.MA.2 | #SI.CC.FA.3 | #SI.CC.BA.3 | #SI.CC.FA.4 | #SI.CC.BA.4 |
|--------------------------------|------------|------------|-------------|-------------|-------------|-------------|-------------|-------------|-------------|
| | wt.% | wt.% | wt.% | wt.% | wt.% | wt.% | wt.% | wt.% | wt.% |
| SiO ₂ | 64 | 32,457 | 12,52 | 25.320 | 15.996 | 32.683 | 36.255 | 7.110 | 31.305 |
| CaO | 12,066 | 23,686 | 32,701 | 40.856 | 69.443 | 25.466 | 35.270 | 13.310 | 35.942 |
| Al ₂ O ₃ | 10 | 9,663 | 5,39 | 10.497 | 10.175 | 10.814 | 6.612 | 3.670 | 12.680 |
| Fe ₂ O ₃ | 3,175 | 7,428 | 5,355 | 10.769 | 0.529 | 14.050 | 4.552 | 1.030 | 4.197 |
| Na ₂ O | 3,014 | 2,831 | 1,776 | 0.611 | 0.241 | 0.397 | 0.657 | >15 | 3.395 |
| K ₂ O | 3,459 | 3,43 | 2,435 | 1.212 | 0.397 | 2.731 | 3.952 | 3.540 | 1.242 |
| SO ₃ | 0,411 | 7,562 | 8,106 | 2.948 | 0.343 | 3.125 | 0.135 | 8.305 | 1.632 |
| Cl | 0,056 | 1,513 | 6,234 | / | / | / | / | / | / |
| P ₂ O ₅ | 0,676 | 1,586 | 1,258 | 0.306 | 0.277 | 0.626 | 1.110 | 0.870 | 2.824 |
| MgO | 2,015 | 3,68 | 2,736 | 6.013 | 2.117 | 8.829 | 10.519 | 1.420 | 3.769 |
| ZnO | 0,423 | 0,379 | 0,054 | / | / | / | / | / | / |
| CuO | 0,246 | 0,235 | 0,294 | / | / | / | / | / | / |
| TiO ₂ | 0,673 | 1,468 | 1,12 | 0.951 | 0.313 | 0.686 | 0.419 | 0.930 | 2.362 |
| MnO | 0,349 | 0,611 | 0,46 | 0.170 | 0.036 | 0.313 | 0.362 | 0.040 | 0.220 |
| PbO | 0,04 | 0,117 | | / | / | / | / | / | / |
| Cr ₂ O ₃ | 0,121 | 0,113 | 0,119 | 0.023 | bdl | 0.012 | 0.023 | 0.050 | 0.143 |
| BaO | 0,093 | | | 0.240 | 0.040 | 0.188 | 0.088 | 0.033 | 0.245 |
| NiO | 0,03 | 0,049 | | / | / | / | / | / | / |
| SrO | 0,051 | 0,02 | 0,055 | 0.086 | 0.095 | 0.081 | 0.047 | 0.009 | 0.044 |
| SnO ₂ | | | | / | / | / | / | / | / |
| As ₂ O ₃ | | | | / | / | / | / | / | / |
| Sum | | | | 100 | 100 | 100 | 100 | 40.318 | 100 |

3.2 Concentrations of heavy metals and REEs

Table 1. Wood ashes

| | | Sample ID | HR.WA.FA.1 | HR.WA.FA.2 | HR.WA.FA.3 | HR.WA.FA.8 | HR.WA.FA.10 | HR.WA.FA.11 | HR.WA.FA.12 | HR.WA.FA.16 | HR.WA.FA.17 | HR.WA.FA.19 | #SI.WA.FA.1 |
|----------------------|----------|-----------|------------|------------|------------|------------|-------------|-------------|-------------|-------------|-------------|-------------|-------------|
| Trace/Heavy elements | Analyte: | Unit: | | | | | | | | | | | |
| | Mo | PPM | 1,5 | 2,94 | 1,04 | 2 | 2,37 | 2,46 | 1,48 | 2,85 | 4,51 | 2,73 | 2,29 |
| | Cu | PPM | 88,4 | 148,5 | 92,2 | 137,5 | 87,7 | 108,9 | 90,9 | 88,3 | 296,4 | 88,4 | 82,6 |
| | Pb | PPM | 25,9 | 45,21 | 7,97 | 33,02 | 51,31 | 22,11 | 51,31 | 72,47 | 53,78 | 21,96 | 61,62 |
| | Zn | PPM | 410,1 | 317,6 | 72 | 559,2 | 730,7 | 627,3 | 409,9 | 1351,9 | 749,6 | 434,9 | 394,1 |
| | Ag | PPB | 170 | 397 | 88 | 336 | 358 | 343 | 428 | 724 | 1044 | 280 | 458 |
| | Ni | PPM | 32,8 | 34,7 | 49 | 44,7 | 36,6 | 21,3 | 40,2 | 36,9 | 47,2 | 43,7 | 51,9 |
| | Co | PPM | 6,6 | 3,5 | 7 | 7,8 | 12,7 | 4,4 | 9,8 | 8,9 | 4,7 | 8,2 | 6,9 |
| | Mn | PPM | 2866 | 1460 | 2170 | 3848 | 2965 | 2186 | 739 | 4081 | 3361 | 5580 | 3194 |
| | As | PPM | 4,1 | 3,9 | <0.2 | 4,5 | 9,9 | 3,6 | 9,8 | 13,9 | 6 | 3,3 | 2,3 |
| | U | PPM | 0,6 | 0,2 | 0,7 | 0,7 | 1,9 | 0,3 | 1,6 | 0,8 | <0.1 | 0,7 | 0,8 |
| | Th | PPM | 2,6 | 0,4 | 2,3 | 1,9 | 8,1 | 1 | 5,9 | 3,1 | <0.1 | 2 | 3,1 |
| | Sr | PPM | 597 | 702 | 997 | 730 | 290 | 364 | 448 | 668 | 331 | 862 | 495 |
| | Cd | PPM | 7,63 | 3,1 | 1,28 | 9,23 | 5,68 | 7,97 | 8,81 | 14,98 | 9,12 | 5,6 | 12,49 |
| | Sb | PPM | 1,29 | 1,22 | 0,2 | 0,88 | 3,54 | 0,5 | 1,8 | 1,17 | 1,28 | 0,44 | 3,48 |
| | Bi | PPM | 0,15 | 0,23 | <0.04 | 0,14 | 0,29 | 0,19 | 0,37 | 0,56 | 0,19 | 0,12 | 0,26 |
| | V | PPM | 22 | 6 | 21 | 24 | 67 | 11 | 60 | 35 | 3 | 26 | 29 |
| | Cr | PPM | 25 | 50 | 73 | 35 | 72 | 13 | 61 | 36 | 112 | 27 | 91 |
| | Ba | PPM | 198 | 69 | 1408 | 134 | 259 | 257 | 259 | 53 | 41 | 57 | 1110 |
| | W | PPM | 0,8 | 0,7 | 0,5 | 0,6 | 2,8 | 0,4 | 1,2 | 1,4 | 0,8 | 0,9 | 1,5 |
| | Zr | PPM | 4,4 | 6,2 | 2,4 | 1,7 | 63,8 | 2,2 | 32,7 | 11,2 | 2,2 | 4,7 | 13,2 |
| | Sn | PPM | 1,4 | 1,7 | 0,5 | 1,3 | 3,4 | 1,1 | 2,9 | 4,6 | 2,2 | 1,2 | 2,3 |
| | Hf | PPM | 0,08 | 0,14 | 0,08 | 0,03 | 1,67 | 0,02 | 0,85 | 0,15 | 0,06 | 0,05 | 0,31 |

| | | | | | | | | | | | | | |
|-------|----|-----|--------|--------|--------|--------|--------|--------|--------|-------|-------|--------|--------|
| | Rb | PPM | 86,1 | 52,8 | 53,6 | 103,4 | 183,7 | 201,5 | 144,4 | 141,3 | 146,1 | 108,1 | 159,6 |
| | Ta | PPM | <0,1 | <0,1 | <0,1 | <0,1 | 0,7 | <0,1 | 0,4 | <0,1 | <0,1 | <0,1 | 0,2 |
| | Nb | PPM | 2,17 | 0,67 | 0,79 | 0,99 | 10,06 | 1,04 | 5,84 | 0,72 | 0,28 | 0,6 | 3,91 |
| | Cs | PPM | 2 | 2,7 | 0,6 | 2,3 | 4,3 | 4,6 | 4,3 | 3,1 | 5,4 | 2,2 | 2,2 |
| | Ga | PPM | 4,07 | 2,31 | 2,53 | 3,94 | 11,89 | 2,05 | 9,99 | 6,63 | 5,14 | 4,43 | 4 |
| | In | PPM | 0,02 | 0,02 | <0,01 | 0,02 | 0,05 | 0,01 | 0,05 | 0,11 | 0,03 | 0,02 | 0,01 |
| | Re | PPM | <0,002 | <0,002 | <0,002 | <0,002 | <0,002 | <0,002 | <0,002 | 0,003 | 0,002 | <0,002 | <0,002 |
| | Se | PPM | 1,4 | 2,2 | 1,4 | 1,5 | 1 | 1,1 | 2,3 | 3,1 | 1,5 | 1,3 | 0,8 |
| | Te | PPM | 0,07 | 0,08 | 0,13 | 0,09 | 0,07 | 0,09 | 0,08 | 0,12 | 0,07 | 0,11 | 0,05 |
| | Tl | PPM | 0,54 | 0,99 | 0,12 | 0,7 | 1,27 | 1,03 | 0,74 | 1,34 | 1,27 | 0,44 | 2,32 |
| | Hg | PPM | 0,05 | <0,01 | 0,01 | <0,01 | 0,14 | <0,01 | 0,39 | 0,05 | <0,01 | <0,01 | <0,01 |
| REEs | La | PPM | 10,8 | 1,9 | 8,7 | 10 | 29 | 5,6 | 17,9 | 14 | 0,9 | 11,2 | 11,7 |
| | Sc | PPM | 2,9 | 0,5 | 2,8 | 2,7 | 7,3 | 1,2 | 7,2 | 3,8 | 0,3 | 3,1 | 3,3 |
| | Y | PPM | 5,7 | 1,4 | 6,2 | 5,7 | 12,4 | 2,9 | 11,6 | 7,9 | 1,3 | 6,9 | 6,5 |
| | Ce | PPM | 21,15 | 3,57 | 16,92 | 18,59 | 56,71 | 8,71 | 35,59 | 27,3 | 1,85 | 21,78 | 23,29 |
| | Pr | PPM | 2,4 | 0,4 | 1,9 | 2 | 6 | 1,1 | 4 | 3,1 | 0,3 | 2,5 | 2,5 |
| | Nd | PPM | 9,1 | 1,6 | 7,2 | 7,8 | 22,8 | 4,1 | 15,6 | 12 | 1,4 | 9,8 | 9,9 |
| | Sm | PPM | 1,7 | 0,3 | 1,4 | 1,5 | 4,1 | 0,8 | 3 | 2,3 | 0,3 | 1,9 | 1,9 |
| | Eu | PPM | 0,3 | <0,1 | 0,3 | 0,3 | 0,8 | 0,1 | 0,6 | 0,5 | <0,1 | 0,4 | 0,3 |
| | Gd | PPM | 1,5 | 0,3 | 1,3 | 1,3 | 3,3 | 0,7 | 2,6 | 2 | 0,3 | 1,7 | 1,6 |
| | Tb | PPM | 0,2 | <0,1 | 0,2 | 0,2 | 0,4 | <0,1 | 0,3 | 0,3 | <0,1 | 0,2 | 0,2 |
| | Dy | PPM | 1,1 | 0,2 | 1,1 | 1,1 | 2,3 | 0,5 | 2,1 | 1,5 | 0,2 | 1,3 | 1,2 |
| | Ho | PPM | 0,2 | <0,1 | 0,2 | 0,2 | 0,4 | <0,1 | 0,4 | 0,3 | <0,1 | 0,2 | 0,2 |
| | Er | PPM | 0,5 | 0,1 | 0,6 | 0,5 | 1,2 | 0,3 | 1,1 | 0,7 | 0,1 | 0,6 | 0,6 |
| | Tm | PPM | <0,1 | <0,1 | <0,1 | <0,1 | 0,2 | <0,1 | 0,1 | <0,1 | <0,1 | <0,1 | <0,1 |
| | Yb | PPM | 0,5 | 0,1 | 0,6 | 0,5 | 1,1 | 0,2 | 1 | 0,7 | <0,1 | 0,6 | 0,6 |
| | Lu | PPM | <0,1 | <0,1 | <0,1 | <0,1 | 0,1 | <0,1 | 0,1 | <0,1 | <0,1 | <0,1 | <0,1 |
| Other | Li | PPM | 13,8 | 10 | 9,4 | 16 | 44,7 | 10,5 | 36,8 | 19,1 | 32,2 | 16,3 | 14,5 |

| | Be | PPM | <1 | <1 | <1 | <1 | 2 | <1 | 2 | 1 | <1 | <1 | <1 |
|--|----|-----|-------|-------|-------|-------|-------|-------|-------|-------|--------|-------|-------|
| | Fe | % | 0,79 | 0,22 | 0,75 | 0,77 | 2,2 | 0,37 | 2,13 | 1,1 | 0,2 | 0,97 | 1,22 |
| | Ca | % | 22,52 | 26,21 | 36,26 | 25,26 | 14,81 | 23,9 | 15,27 | 23,73 | 7,35 | 28,47 | 20,65 |
| | P | % | 0,915 | 1,148 | 1,573 | 1,421 | 0,953 | 0,952 | 0,897 | 1,549 | 0,633 | 1,003 | 0,803 |
| | Mg | % | 2,07 | 1,63 | 2,53 | 1,97 | 1,72 | 2,76 | 2,74 | 2,27 | 0,87 | 1,91 | 1,8 |
| | Ti | % | 0,107 | 0,019 | 0,091 | 0,111 | 0,319 | 0,047 | 0,199 | 0,101 | 0,01 | 0,108 | 0,196 |
| | Al | % | 1,7 | 0,29 | 1,47 | 1,56 | 4,82 | 0,69 | 4,4 | 2,28 | 0,13 | 1,84 | 2,02 |
| | Na | % | 0,391 | 0,687 | 0,357 | 0,238 | 0,362 | 0,119 | 0,56 | 0,446 | 0,945 | 0,256 | 0,32 |
| | K | % | 5,36 | 4,51 | 3,66 | 4,54 | 6,41 | 6,15 | 5,34 | 6,79 | >10.00 | 5,35 | 4,96 |
| | S | % | 0,96 | 3,04 | 0,29 | 1,28 | 0,86 | 1,34 | 0,92 | 2,8 | 4,35 | 1,04 | 0,59 |

Table 2. Wood Bottom ashes

| | | | | | | | | | | | | | |
|------------|-----|--------|--------|--------|--------|--------|--------|--------|--------|--------|--------|--------|------|
| | Nb | PPM | 1,01 | 1,66 | 0,78 | 3,68 | 0,97 | 1,1 | 3,75 | 0,86 | 0,23 | 1,57 | 0,77 |
| Cs | PPM | 0,9 | 1,2 | 1,2 | 2,4 | 0,5 | 0,8 | 1,8 | 0,7 | 1 | 1,4 | 2,1 | |
| Ga | PPM | 2,32 | 3,55 | 2,9 | 7,09 | 1,62 | 2,04 | 6,1 | 2,62 | 1,91 | 5,02 | 1,3 | |
| In | PPM | <0,01 | <0,01 | <0,01 | 0,01 | <0,01 | <0,01 | <0,01 | <0,01 | <0,01 | <0,01 | <0,01 | |
| Re | PPM | <0,002 | <0,002 | <0,002 | <0,002 | <0,002 | <0,002 | <0,002 | <0,002 | <0,002 | <0,002 | <0,002 | |
| Se | PPM | 1,3 | 0,7 | 0,8 | 0,5 | 1,6 | <0,3 | 1 | 0,9 | 0,8 | 0,8 | 0,8 | |
| Te | PPM | 0,12 | 0,1 | 0,09 | <0,05 | 0,17 | <0,05 | 0,09 | 0,07 | 0,1 | 0,47 | 0,07 | |
| Tl | PPM | <0,05 | 0,24 | 0,25 | 0,06 | 0,16 | <0,05 | 0,16 | 0,26 | 0,2 | <0,05 | 0,12 | |
| Hg | PPM | <0,01 | 0,01 | 0,01 | <0,01 | 0,02 | <0,01 | <0,01 | <0,01 | 0,04 | <0,01 | 0,01 | |
| REEs | La | PPM | 8,3 | 9,3 | 14,8 | 23,5 | 4,2 | 4,6 | 20,2 | 7,5 | 8,1 | 17,4 | 4,7 |
| | Sc | PPM | 2,5 | 2,9 | 3 | 6,9 | 1,2 | 1,5 | 4,8 | 2,2 | 2,5 | 4,9 | 1,2 |
| | Y | PPM | 5,9 | 6,7 | 8,9 | 15,8 | 2,9 | 2,3 | 12,8 | 5,2 | 6,2 | 11,4 | 4,4 |
| | Ce | PPM | 16,22 | 17,55 | 24,86 | 44,29 | 8,01 | 9,61 | 39,12 | 14,13 | 14,24 | 31,93 | 7,36 |
| | Pr | PPM | 1,7 | 2 | 2,7 | 4,8 | 0,9 | 1 | 4,3 | 1,6 | 1,7 | 3,6 | 0,9 |
| | Nd | PPM | 6,9 | 7,6 | 10,2 | 18,1 | 3,5 | 4,1 | 17,1 | 6,1 | 6,4 | 14,3 | 3,5 |
| | Sm | PPM | 1,4 | 1,5 | 2 | 3,4 | 0,7 | 0,8 | 3,4 | 1,2 | 1,3 | 2,8 | 0,7 |
| | Eu | PPM | 0,3 | 0,3 | 0,4 | 0,7 | 0,1 | 0,1 | 0,6 | 0,2 | 0,3 | 0,6 | <0,1 |
| | Gd | PPM | 1,3 | 1,4 | 1,9 | 3,1 | 0,6 | 0,6 | 2,9 | 1,1 | 1,3 | 2,6 | 0,7 |
| | Tb | PPM | 0,2 | 0,2 | 0,3 | 0,4 | <0,1 | <0,1 | 0,4 | 0,1 | 0,2 | 0,3 | <0,1 |
| | Dy | PPM | 1,1 | 1,2 | 1,6 | 2,6 | 0,5 | 0,5 | 2,3 | 0,9 | 1,1 | 2,1 | 0,6 |
| | Ho | PPM | 0,2 | 0,2 | 0,3 | 0,5 | <0,1 | <0,1 | 0,4 | 0,2 | 0,2 | 0,4 | 0,1 |
| | Er | PPM | 0,6 | 0,6 | 0,8 | 1,4 | 0,3 | 0,2 | 1,2 | 0,5 | 0,6 | 1,1 | 0,3 |
| | Tm | PPM | <0,1 | <0,1 | 0,1 | 0,2 | <0,1 | <0,1 | 0,2 | <0,1 | <0,1 | 0,1 | <0,1 |
| | Yb | PPM | 0,6 | 0,6 | 0,8 | 1,3 | 0,3 | 0,2 | 1,1 | 0,4 | 0,5 | 1,1 | 0,3 |
| | Lu | PPM | <0,1 | <0,1 | 0,1 | 0,2 | <0,1 | <0,1 | 0,2 | <0,1 | <0,1 | 0,1 | <0,1 |
| Other main | Li | PPM | 9 | 14,7 | 17,9 | 31,1 | 5,9 | 15,3 | 18,8 | 11,3 | 9,7 | 16,2 | 4,6 |
| | Be | PPM | <1 | 1 | <1 | 2 | <1 | <1 | 1 | <1 | <1 | 1 | <1 |
| | Fe | % | 0,75 | 1,11 | 0,98 | 3,76 | 0,38 | 0,43 | 1,44 | 0,71 | 0,91 | 1,58 | 0,48 |

| | | | | | | | | | | | | | |
|--|----|---|-------|-------|-------|-------|--------|-------|-------|-------|-------|-------|-------|
| | Ca | % | 33,53 | 27,67 | 33,29 | 16,36 | >40,00 | 4,18 | 18,3 | 33,65 | 32,03 | 20,99 | 29,24 |
| | P | % | 1,051 | 1,204 | 1,376 | 1,152 | 0,603 | 0,334 | 0,978 | 1,016 | 1,126 | 0,724 | 1,058 |
| | Mg | % | 2,16 | 2,52 | 2,9 | 2,83 | 1,56 | 0,61 | 3,25 | 1,8 | 3,03 | 2,16 | 2,79 |
| | Ti | % | 0,097 | 0,115 | 0,144 | 0,269 | 0,058 | 0,049 | 0,171 | 0,101 | 0,075 | 0,15 | 0,04 |
| | Al | % | 1,39 | 1,89 | 1,85 | 3,95 | 0,68 | 0,9 | 3,11 | 1,34 | 1,26 | 3,08 | 0,84 |
| | Na | % | 0,353 | 0,358 | 0,131 | 0,307 | 0,162 | 0,146 | 0,723 | 0,207 | 0,185 | 0,369 | 0,096 |
| | K | % | 6,25 | 4,02 | 4,56 | 6,3 | 2,4 | 2,78 | 7,46 | 4,24 | 4,45 | 6,07 | 6,22 |
| | S | % | 0,18 | 0,3 | 0,39 | 0,12 | 0,38 | <0,04 | 0,28 | 0,26 | 0,29 | 0,08 | 0,19 |

Table 3. Sewage sludge ashes

| | | Sample ID | | | | |
|----------------------|----------|-----------|-------------|-------------|-------------|-------------|
| Trace/Heavy elements | Analyte: | Unit: | HR.SSA.BA.1 | HR.SSA.BA.2 | HR.SSA.BA.3 | HR.SSA.BA.4 |
| | Mo | PPM | 24,2 | 12,47 | 9,2 | 6,64 |
| | Cu | PPM | 269,5 | 577,4 | 349,6 | 281,7 |
| | Pb | PPM | 107,63 | 95,36 | 59,44 | 44,32 |
| | Zn | PPM | 1165,8 | 1023,8 | 949,2 | 918,7 |
| | Ag | PPB | 3863 | 8265 | 2694 | 3312 |
| | Ni | PPM | 122,3 | 127,7 | 93,8 | 70,5 |
| | Co | PPM | 24,8 | 12,5 | 14,6 | 14,9 |
| | Mn | PPM | 512 | 593 | 978 | 1371 |
| | As | PPM | 5,7 | 3,5 | 7 | 6,9 |
| | U | PPM | 6,2 | 3,5 | 7,1 | 5 |
| | Th | PPM | 4,7 | 3,8 | 9,3 | 8 |
| | Sr | PPM | 260 | 151 | 225 | 250 |
| | Cd | PPM | 0,29 | 1,85 | 0,23 | 0,26 |
| | Sb | PPM | 12,44 | 92,81 | 13,1 | 9,82 |
| | Bi | PPM | 2,08 | 4,51 | 0,76 | 1,02 |
| | V | PPM | 78 | 41 | 85 | 85 |
| | Cr | PPM | 238 | 161 | 144 | 108 |
| | Ba | PPM | 208 | 252 | 423 | 277 |
| | W | PPM | 30,7 | 3,3 | 4,3 | 2,8 |
| | Zr | PPM | 93,9 | 27,2 | 50,2 | 38,6 |
| | Sn | PPM | 34,6 | 36,6 | 13 | 12,3 |
| | Hf | PPM | 2,12 | 0,18 | 1,3 | 0,74 |
| | Rb | PPM | 41 | 40,2 | 91,7 | 89,5 |
| | Ta | PPM | 0,4 | <0,1 | 0,7 | 0,4 |
| | Nb | PPM | 5,82 | 1,75 | 9,3 | 7,4 |

| | | | | | | |
|---------------------|----|-----|-------|--------|-------|-------|
| | Cs | PPM | 2,8 | 2,5 | 6,2 | 5,2 |
| | Ga | PPM | 9,61 | 8,11 | 15,22 | 14,51 |
| | In | PPM | 0,05 | 0,05 | 0,02 | 0,02 |
| | Re | PPM | 0,003 | <0.002 | 0,005 | 0,003 |
| | Se | PPM | 3,6 | 3,7 | 2,8 | 3 |
| | Te | PPM | <0.05 | <0.05 | <0.05 | <0.05 |
| | Tl | PPM | 0,29 | 0,15 | 0,28 | 0,31 |
| | Hg | PPM | 0,01 | 0,03 | 0,05 | 0,04 |
| REEs | La | PPM | 17,2 | 15,9 | 31,4 | 26,9 |
| | Sc | PPM | 5,4 | 4,4 | 10,4 | 10,3 |
| | Y | PPM | 10,3 | 7,4 | 15,1 | 15,2 |
| | Ce | PPM | 32,25 | 31,58 | 60,52 | 53,45 |
| | Pr | PPM | 3,5 | 2,8 | 6,7 | 6 |
| | Nd | PPM | 13,4 | 11,2 | 26,2 | 23,6 |
| | Sm | PPM | 2,5 | 2,2 | 5 | 4,5 |
| | Eu | PPM | 0,5 | 0,4 | 1 | 0,9 |
| | Gd | PPM | 2,1 | 1,9 | 4,2 | 3,8 |
| | Tb | PPM | 0,3 | 0,2 | 0,5 | 0,5 |
| | Dy | PPM | 1,5 | 1,4 | 3 | 2,9 |
| | Ho | PPM | 0,3 | 0,3 | 0,5 | 0,5 |
| | Er | PPM | 0,7 | 0,7 | 1,4 | 1,4 |
| | Tm | PPM | <0.1 | <0.1 | 0,2 | 0,2 |
| | Yb | PPM | 0,7 | 0,8 | 1,3 | 1,2 |
| | Lu | PPM | <0.1 | <0.1 | 0,2 | 0,2 |
| Other main elements | Li | PPM | 26,7 | 19,5 | 41,3 | 37,7 |
| | Be | PPM | 1 | <1 | 2 | 2 |
| | Fe | % | 3,72 | 2,15 | 4,33 | 4,79 |
| | Ca | % | 12,69 | 10,79 | 5,66 | 5,08 |

| | | | | | | |
|--|----|---|-------|-------|-------|-------|
| | P | % | 4,145 | 2,17 | 3,144 | 2,585 |
| | Mg | % | 1,22 | 1,89 | 2,12 | 1,52 |
| | Ti | % | 0,234 | 0,139 | 0,391 | 0,313 |
| | Al | % | 4,96 | 3,65 | 6,58 | 6,19 |
| | Na | % | 0,376 | 0,493 | 0,714 | 0,721 |
| | K | % | 0,7 | 0,83 | 1,81 | 1,63 |
| | S | % | 0,96 | 1,41 | 0,69 | 0,66 |

Table 4. Other ashes

| | | Sample ID | #SI.CC.FA.1 | #SI.CC.MA.2 | #SI.CC.FA.3 | #SI.CC.BA.3 | #SI.CC.FA.4 | #SI.CC.BA.4 |
|----------------------|----------|-----------|-------------|-------------|-------------|-------------|-------------|-------------|
| Trace/Heavy elements | Analyte: | Unit: | | | | | | |
| | Mo | PPM | 8,19 | 2,92 | 2,92 | 2,33 | 12,58 | 15,14 |
| | Cu | PPM | 348,4 | 452,2 | 52,9 | 73,5 | 709,8 | 3850,3 |
| | Pb | PPM | 268,14 | 105,15 | 29,07 | 17,58 | 1248,52 | 766,42 |
| | Zn | PPM | 2224,3 | 160,4 | 277,3 | 109,2 | 9007,6 | 4958,5 |
| | Ag | PPB | 844 | 1048 | 5262 | 157 | 13227 | 7086 |
| | Ni | PPM | 95,1 | 10,9 | 71,4 | 34,7 | 38,9 | 184,6 |
| | Co | PPM | 44,9 | 2,6 | 52,1 | 14,6 | 15,6 | 137,5 |
| | Mn | PPM | 1085 | 184 | 1815 | 2312 | 378 | 1413 |
| | As | PPM | 24,8 | 2,1 | 23 | 3,1 | 13,8 | 6,6 |
| | U | PPM | 2,4 | 2,8 | 1,6 | 2,1 | 0,8 | 2,3 |
| | Th | PPM | 8,1 | 10,9 | 5,7 | 4,5 | 1,5 | 5,2 |
| | Sr | PPM | 663 | 822 | 524 | 385 | 171 | 384 |
| | Cd | PPM | 2,88 | 0,55 | 1,91 | 0,16 | 71,06 | 17,14 |
| | Sb | PPM | 8,35 | 3,07 | 1,38 | 0,65 | 708,07 | 234,98 |
| | Bi | PPM | 2,63 | 1,26 | 0,49 | 0,06 | 26,14 | 3,27 |
| | V | PPM | 79 | 14 | 66 | 39 | 13 | 33 |
| | Cr | PPM | 124 | 27 | 56 | 66 | 350 | 761 |
| | Ba | PPM | 176 | 348 | 284 | 673 | 40 | 425 |
| | W | PPM | 4,3 | 2,9 | 1,9 | 61,4 | 8,4 | 41,8 |
| | Zr | PPM | 104,6 | 107,5 | 57 | 71,2 | 31,1 | 186,4 |
| | Sn | PPM | 18,5 | 9,2 | 2,7 | 5,5 | 324,6 | 90 |
| | Hf | PPM | 2,68 | 2,58 | 1,49 | 1,77 | 0,31 | 3,67 |
| | Rb | PPM | 45,9 | 22,6 | 66,7 | 65,6 | 77,5 | 20,3 |
| | Ta | PPM | 0,7 | 0,6 | 0,6 | 0,6 | <0.1 | 0,2 |

| | | | | | | | | |
|---------------------|----|-----|-------|--------|-------|--------|-------|--------|
| | Nb | PPM | 7,71 | 4,99 | 5,87 | 5,48 | 1,46 | 3,06 |
| | Cs | PPM | 3,5 | 2 | 3,6 | 1,7 | 5,5 | 1,1 |
| | Ga | PPM | 13,39 | 10,45 | 11,38 | 4,82 | 5,62 | 9,82 |
| | In | PPM | 0,07 | 0,04 | 0,05 | 0,01 | 0,79 | 0,12 |
| | Re | PPM | 0,006 | <0.002 | 0,002 | <0.002 | 0,009 | <0.002 |
| | Se | PPM | 3,7 | 0,9 | 3,4 | 0,7 | 4,5 | 1,1 |
| | Te | PPM | 0,18 | 0,07 | 0,14 | 0,06 | 0,32 | <0.05 |
| | Tl | PPM | 0,59 | 0,11 | 0,77 | 0,06 | 1,14 | <0.05 |
| | Hg | PPM | 0,6 | <0.01 | 0,38 | <0.01 | 4,51 | 0,02 |
| REEs | La | PPM | 23,9 | 27,1 | 18,5 | 15,9 | 5,8 | 18,7 |
| | Sc | PPM | 7,3 | 2,4 | 8,7 | 4,6 | 1,1 | 3 |
| | Y | PPM | 18,2 | 8,6 | 16,7 | 11,1 | 3,7 | 11,2 |
| | Ce | PPM | 50,71 | 58,2 | 37,14 | 30,25 | 11,69 | 37,42 |
| | Pr | PPM | 5,4 | 5,7 | 4,1 | 3,2 | 1,2 | 3,2 |
| | Nd | PPM | 20,7 | 20,2 | 16 | 12,6 | 5,2 | 11,7 |
| | Sm | PPM | 3,9 | 3,4 | 3,1 | 2,4 | 0,6 | 1,8 |
| | Eu | PPM | 0,8 | 0,7 | 0,7 | 0,5 | 0,2 | 0,5 |
| | Gd | PPM | 3,7 | 2,5 | 3 | 2,2 | 1 | 1,9 |
| | Tb | PPM | 0,5 | 0,3 | 0,4 | 0,3 | 0,1 | 0,3 |
| | Dy | PPM | 3,1 | 1,8 | 2,6 | 2 | 0,5 | 1,4 |
| | Ho | PPM | 0,6 | 0,3 | 0,5 | 0,4 | <0.1 | 0,3 |
| | Er | PPM | 1,7 | 0,8 | 1,5 | 1,2 | 0,3 | 4,4 |
| | Tm | PPM | 0,2 | 0,1 | 0,2 | 0,2 | <0.1 | 0,1 |
| | Yb | PPM | 1,5 | 0,7 | 1,4 | 1 | 0,3 | 0,8 |
| | Lu | PPM | 0,2 | <0.1 | 0,2 | 0,1 | <0.1 | 0,1 |
| Other main elements | Li | PPM | 27,2 | 12,1 | 25,5 | 17 | 31,1 | 53 |
| | Be | PPM | 3 | <1 | 3 | 2 | <1 | 1 |
| | Fe | % | 6,09 | 0,29 | 6,97 | 2,42 | 0,72 | 2,29 |

| | | | | | | | | |
|--|----|---|-------|-------|-------|-------|-------|-------|
| | Ca | % | 23,95 | 38,48 | 12,74 | 20,14 | 9,24 | 20,93 |
| | P | % | 0,118 | 0,108 | 0,2 | 0,378 | 0,363 | 0,612 |
| | Mg | % | 3,1 | 1,03 | 4,12 | 5,26 | 0,72 | 1,92 |
| | Ti | % | 0,452 | 0,149 | 0,256 | 0,212 | 0,534 | 0,357 |
| | Al | % | 4,98 | 4,71 | 4,58 | 2,94 | 1,75 | 5,55 |
| | Na | % | 0,34 | 0,149 | 0,181 | 0,381 | 19,04 | 2,32 |
| | K | % | 0,89 | 0,29 | 1,75 | 2,9 | 3,33 | 0,89 |
| | S | % | 0,94 | 0,12 | 0,87 | 0,05 | 3,04 | 0,4 |

3.3 X-ray powder diffraction (XRD) analysis

Table 1. Wood fly ashes

| Mineral | Sample ID | HR.WA.FA.1 | HR.WA.FA.2 | HR.WA.FA.3 | HR.WA.FA.8 | HR.WA.FA.10 | HR.WA.FA.11 | HR.WA.FA.12 | HR.WA.FA.16 | HR.WA.FA.17 | HR.WA.FA.19 | #SI.WA.FA.1 |
|------------------------|--------------|------------|------------|------------|------------|-------------|-------------|-------------|-------------|-------------|-------------|-------------|
| | Detected (x) | wt.% | wt.% | wt.% | wt.% | wt.% | wt.% | wt.% | wt.% | wt.% | wt.% | wt.% |
| Amourphos phase % QXRD | | 64,1 | 88,9 | 89,9 | 88,1 | 93 | 88,4 | 75 | 68,5 | 70,1 | 62,2 | 72,1 |
| Calcite | | 21,7 | 3,8 | 2,7 | 5,9 | 1,2 | 5,8 | 6,5 | 6,1 | 1,5 | 18 | 17,1 |
| Lime | | 4 | 1,8 | 5,6 | 1,9 | 1,2 | 2 | 3 | 9 | 1,2 | 10,2 | 0,1 |
| Portlandite | | 0,9 | 1,6 | 0,6 | 0,8 | 0,2 | 0,9 | 0,5 | 1,9 | 1 | 0,9 | 2,2 |
| Arcanite | | | 3,3 | | 1,6 | | 1,9 | 0,6 | 4,1 | 15,6 | 3,2 | 1,1 |
| Aphthitalite | | | 0 | | 0 | | | | | | 0 | 0 |
| Apatite | | 0,2 | 0,6 | | 0,6 | | 0,3 | | | | | 0 |
| Quartz | | 5,1 | | 0,3 | 0,7 | 3,7 | 0,4 | 8,2 | 5,1 | | 3,6 | 4,8 |
| Brucite | | | | | 0 | | 0 | | | | | 0 |
| Periclase | | | | 0,7 | 0,4 | 0,2 | | 0,8 | 1,6 | | 1,2 | 0,8 |
| Feldpar | | | | | | | | | | | | 0 |
| Anhydrite | | | | | | | | | 3,1 | | 0 | 0 |
| Perovskite | | | | | | 0,1 | 0,3 | 0,3 | 0,6 | | 0,7 | 0,8 |
| Fairchildite | | 2,3 | | | | | | | | | | 0 |
| Plagioclases | | 0 | | | | 0,2 | | 2,2 | | | | 0,2 |
| Hematite | | | | 0,2 | | | | | | | | 0 |
| Illite/ Muscovite | | | | | | | | | | | | 0 |
| Mayenite | | | | | | | | | | | | 0,5 |
| Gehlenite | | | | | | | | 0,7 | | | | 0 |
| Melilite | | | | | | | | | | | | 0 |
| Dolomite | | 1,7 | | | | 0,2 | | 0,5 | | 6,7 | | 0,3 |

Table 2. Wood bottom ashes

Table 3. Sewage sludge ashes

| Mineral | Sample ID | HR.SSA.1 | HR.SSA.2 | HR.SSA.3 | HR.SSA.4 |
|------------------------|--------------|----------|----------|----------|----------|
| | Detected (x) | wt.% | wt.% | wt.% | wt.% |
| Amourphos phase % QXRD | | 81,1 | 78,9 | 72,3 | 76,1 |
| Calcite | | 1,4 | 0,4 | | |
| Lime | | 0,9 | | | |
| Portlandite | | | | | |
| Arcanite | | | | | |
| Aphthitalite | | | | | 0,2 |
| Apatite | | | 2,3 | 0,4 | 0,2 |
| Quartz | | 7,2 | 9,4 | 12,8 | 12,5 |
| Brucite | | | | | |
| Periclase | | | | | |
| Feldpar | | 2,8 | 4 | | 6,1 |
| Anhydrite | | | | | |
| Perovskite | | | | | |
| Fairchildite | | | | 0,6 | 0,7 |
| Plagioclases | | | | 6,2 | |
| Hematite | | | | | |
| Illite/ Muscovite | | 3,9 | 1,8 | 2,9 | 3,9 |
| Mayenite | | | | | |
| Gehlenite | | | 3,2 | | |
| Wadeite | | | | | |
| Melilite | | | | 1,5 | |
| Dolomite | | | | | |
| K-Feldspar | | | | 3,3 | |
| Akermanite | | 2,7 | | | 0,3 |

| | | | | | |
|--------|--|-----|-----|-----|-----|
| Halite | | | | | |
| Gypsum | | | | | |
| sum | | 100 | 100 | 100 | 100 |

Table 4. Other ashes

| Mineral | Sample ID | FI.CC.BA1 | FI.CC.FA1 | FI.CC.APC1 | #SI.CC.FA.1 | #SI.CC.MA.2 | #SI.CC.FA.3 | #SI.CC.BA.3 | #SI.CC.FA.4 | #SI.CC.BA.4 |
|-------------------------------|--------------|-----------|-----------|------------|-------------|-------------|-------------|-------------|-------------|-------------|
| | Detected (x) | | | | wt.% | wt.% | wt.% | wt.% | wt.% | wt.% |
| Amourphos phase % QXRD | | | | | 68.4 | 61.8 | 90.6 | 95.2 | 66.9 | 72.1 |
| Calcite | | | | | 1.1 | 16.1 | 0.8 | 0.7 | 1.0 | 4.3 |
| Lime | | | | x | 6.1 | 5.5 | 0.6 | 0 | 0.0 | 1.6 |
| Portlandite | | | | | 0.4 | 4.2 | 0.2 | 0.7 | 0.2 | 0.3 |
| Arcanite | | | | | 0 | 0 | 0 | 0 | 0 | 0 |
| Aphthitalite | | | | | 0 | 0 | 0 | 0 | 3.0 | 0 |
| Apatite | | | | | 0 | 0 | 0 | 0 | 0 | 0 |
| Quartz | | x | x | x | 6.0 | 0.2 | 3.2 | 2.2 | 0.0 | 7.9 |
| Brucite | | | | | 0 | 0 | 0 | 0 | 0 | 0 |
| Periclase | | | | | 1.7 | 0 | 1.3 | 0.2 | 0 | 0.9 |
| Feldpar | | | | | 0 | 0 | 0 | 0 | 0 | 0 |
| Anhydrite | | | x | x | 1.9 | 0 | 0.6 | 0 | 1.7 | 0.4 |
| Perovskite | | | | | 0 | 0 | 0 | 0.1 | 1.2 | 0 |
| Fairchildite | | | | | 0 | 0 | 0.4 | 0 | 0 | 0 |
| Plagioclases | | | | | 0 | 0 | 0 | 0 | 0 | 0 |
| Hematite | | | | x | 1.8 | 0 | 1.1 | 0 | 0 | 1.1 |
| Illite/ Muscovite | | | | | 0 | 0 | 0 | 0 | 0 | 0 |
| Mayenite | | | | | 1.4 | 2.4 | 0 | 0 | 0.5 | 1.6 |
| Gehlenite | | | | | 2.6 | 2.8 | 0 | 0 | 0 | 5.5 |
| Melilite | | | | | 0 | 0 | 0 | 0.6 | 0 | 0 |
| Dolomite | | | | | 0 | 0 | 0 | 0 | 0 | 0 |
| Akermanite | | | | | 0 | 0 | 0.2 | 0.3 | 0 | 0 |
| Larnite | | | | | 4.4 | 6.7 | 0 | 0 | 0 | 4.3 |
| Brownmillerite | | | | | 4.2 | 0 | 1.0 | 0 | 0 | 0 |

| | | | | | | | | | |
|--------------|---|---|---|---|-----|---|---|------|---|
| Halite | | | | 0 | 0 | 0 | 0 | 24.5 | 0 |
| Sylvite | | | | 0 | 0 | 0 | 0 | 1.0 | 0 |
| Talc | | | | 0 | 0.3 | 0 | 0 | 0 | 0 |
| Anorthite | x | x | | | | | | | |
| Orthoclase | x | | | | | | | | |
| Anorthoclase | x | | | | | | | | |
| Devitrite | | | x | | | | | | |
| KCl | | x | x | | | | | | |
| Rutile | | | | x | | | | | |
| NaCl | | | x | | | | | | |

3.4 Water content, pH, conductivity, Leaching and water solubility of the ash

Table 1. Sewage sludge ashes

| Ash Short name | Water content (%) | pH | Conductivity (mS/cm) | Solubility (%) | As (µg/l) | Cd (µg/l) | Cr (µg/l) | Cu (µg/l) | Ni (µg/l) | Pb (µg/l) | Se (µg/l) | Zn (µg/l) | Cl (µg/l) | NO ₃ ⁻ (µg/l) | SO ₄ ²⁻ (µg/l) |
|----------------|-------------------|------|----------------------|----------------|-----------|-----------|-----------|-----------|-----------|-----------|-----------|-----------|-----------|-------------------------------------|--------------------------------------|
| DK.SSA.FA.1 | 23,19 | 8,05 | 1,32 | 1,54 | 35,19 | 1,22 | 6,05 | 10,53 | 0 | 10,89 | 25,21 | 7,21 | 13257 | 5752 | 775979 |
| DK.SSA.FA.2 | 0,03 | 9,85 | 1,46 | 1,25 | 41,25 | 1,4382 | 6,31 | 7,39 | 2,87 | 13,12 | 95,06 | 6,14 | 1100114 | 2694 | 856365 |
| HR.SSA.BA.1 | | 12,6 | | | <0,11 | <0,07 | <0,09 | <0,10 | <0,06 | <0,18 | | <0,21 | 530 | | 106 |

Table 2. MSWI Fly ashes

| Ash Short name | Water content (%) | pH | Conductivity (mS/cm) | Solubility (%) | As (µg/l) | Cd (µg/l) | Cr (µg/l) | Cu (µg/l) | Ni (µg/l) | Pb (µg/l) | Se (µg/l) | Zn (µg/l) | Cl (µg/l) | NO ₃ ⁻ (µg/l) | SO ₄ ²⁻ (µg/l) |
|----------------|-------------------|-------|----------------------|----------------|-----------|-----------|-----------|-----------|-----------|-----------|-----------|-----------|-----------|-------------------------------------|--------------------------------------|
| DK.MSWI.FA.1 | 0,47 | 11,08 | 24,30 | 17,16 | 49,68 | 1,60 | 986,09 | 10,44 | 5,35 | 42,36 | 22,15 | 130,60 | 7391818 | 2509 | 3368909 |
| DK.MSWI.FA.4 | 0,11 | 10,69 | 43,93 | 37,70 | 32,36 | 3,68 | 359,64 | 5,74 | 0,00 | 148,56 | 101,06 | 95,87 | 13448722 | 9915 | 8071499 |

3.5 Reactivity of Cumulative Heat Determination by Isothermal Calorimetry – ASTM C1897-20

Table 1. Different ashes

| | Heat at 3 days | Heat at 7 days |
|-------------|----------------|----------------|
| Sample ID | J/g of SCM | J/g of SCM |
| HR.WA.FA.1 | 95,06 | 131,99 |
| HR.WA.FA.2 | 52,94 | 61,79 |
| HR.WA.FA.3 | 57,67 | 76,77 |
| HR.WA.FA.8 | 51,24 | 66,54 |
| HR.WA.FA.10 | 118,81 | 189,16 |
| HR.WA.FA.11 | 54,42 | 65,43 |
| HR.WA.FA.12 | 152,18 | 226,56 |
| HR.WA.FA.16 | 58,55 | 95,49 |
| HR.WA.FA.17 | 27,33 | 32,79 |
| HR.WA.FA.19 | 90,73 | 128,55 |
| HR.WA.BA.4 | 119,27 | 144,07 |
| HR.WA.MA.5 | 119,78 | 153,09 |
| HR.WA.MA.6 | 107,12 | 154,91 |
| HR.WA.BA.7 | 58,85 | 89,98 |
| HR.WA.MA.9 | 90,23 | 107,47 |
| HR.WA.BA.13 | 26,58 | 41,43 |
| HR.WA.MA.14 | 68,18 | 115,15 |
| HR.WA.MA.15 | 87,75 | 114,39 |
| HR.WA.BA.18 | 109,68 | 135,64 |
| HR.WA.BA.20 | 73,36 | 108,83 |
| HR.SSA.BA.1 | 188,9 | 215,15 |
| HR.SSA.BA.3 | 160,5 | 214,2 |

| | | |
|-------------|--------|--------|
| HR.SSA.BA.4 | 165,92 | 224,14 |
| SI.WA.FA.1 | 72.45 | 101.19 |
| SI.WA.BA.1 | 89.62 | 107.49 |
| SI.CC.FA.1 | 221.34 | 259.05 |
| SI.CC.MA.2 | 143.38 | 156.89 |
| SI.CC.FA.3 | 172.15 | 211.78 |
| SI.CC.BA.3 | 45.81 | 78.33 |
| SI.CC.FA.4 | 62.74 | 79.44 |
| SI.CC.BA.4 | 365.12 | 392.98 |

3.6 Reactivity of Bound Water– ASTM C1897-20

Table 1. Different ashes

| Sample ID | Bound water (g/100g of dried paste) | | |
|-------------|-------------------------------------|------|---------|
| | 1 | 2 | Average |
| HR.WA.FA.1 | 1,82 | 1,94 | 1,88 |
| HR.WA.FA.2 | 1,96 | 1,88 | 1,92 |
| HR.WA.FA.3 | 2,02 | 2,07 | 2,04 |
| HR.WA.FA.8 | 2,17 | 2,68 | 2,42 |
| HR.WA.FA.10 | 3,65 | 3,63 | 3,64 |
| HR.WA.FA.11 | 2,77 | 2,72 | 2,74 |
| HR.WA.FA.12 | 4,25 | 3,99 | 4,12 |
| HR.WA.FA.16 | 2,55 | 2,57 | 2,56 |
| HR.WA.FA.17 | 1,63 | 1,79 | 1,71 |
| HR.WA.FA.19 | 2,72 | 2,8 | 2,76 |
| HR.WA.BA.4 | 2,47 | 2,45 | 2,46 |
| HR.WA.MA.5 | 2,75 | 2,78 | 2,76 |
| HR.WA.MA.6 | 2,64 | 2,74 | 2,69 |
| HR.WA.BA.7 | 2,5 | 2,5 | 2,5 |
| HR.WA.MA.9 | 1,6 | 1,65 | 1,62 |
| HR.WA.BA.13 | 1,13 | 1,12 | 1,13 |
| HR.WA.MA.14 | 1,96 | 1,93 | 1,94 |
| HR.WA.MA.15 | 2,28 | 2,25 | 2,26 |
| HR.WA.BA.18 | 2,15 | 2,27 | 2,21 |
| HR.WA.BA.20 | 2,37 | 2,32 | 2,35 |
| HR.SSA.BA.1 | 2,77 | 2,99 | 2,88 |

| | | | |
|--------------|------|------|------|
| HR.SSA.BA.3 | 3,79 | 3,28 | 3,53 |
| HR.SSA.BA.4 | 3,98 | 3,9 | 3,94 |
| SI.WA.FA.1 | 2,81 | 2,37 | 2,59 |
| SI.WA.BA.1 | 1,66 | 1,8 | 1,73 |
| SI.CC.FA.1 | 4,64 | 4,41 | 4,52 |
| SI.CC.MA.2 | 5 | 4,75 | 4,87 |
| SI.CC.FA.3 | 3,75 | 3,65 | 3,7 |
| SI.CC.BA.3 | 3,86 | 3,53 | 3,7 |
| SI.CC.FA.4 | 2,13 | 2 | 2,06 |
| SI.CC.BA.4 | 4,38 | 4,65 | 4,52 |
| DK.SSA.FA.1 | 4,16 | 4,27 | 4,22 |
| DK.SSA.FA.2 | 3,67 | 3,57 | 3,62 |
| DK.MSWI.FA.1 | 3,44 | 3,47 | 3,46 |

3.7 Free CaO content

Table 1. Different ashes

| Free CaO (w.%) | | |
|----------------|---------|---------|
| Sample ID | Average | st.dev. |
| HR.WA.FA.1 | 8,83 | 0,02 |
| HR.WA.FA.2 | 24,6 | 0,03 |
| HR.WA.FA.3 | 26,46 | 0,03 |
| HR.WA.FA.8 | 15,8 | 0,03 |
| HR.WA.FA.10 | 0,33 | 0,02 |
| HR.WA.FA.11 | 18,14 | 0,02 |
| HR.WA.FA.12 | 0,20 | 0,02 |
| HR.WA.FA.16 | 15,34 | 0,03 |
| HR.WA.FA.17 | 15,53 | 0,03 |
| HR.WA.FA.19 | 0,20 | 0,02 |
| HR.WA.BA.4 | 17,38 | 0,02 |
| HR.WA.MA.5 | 8,26 | 0,02 |
| HR.WA.MA.6 | 8,50 | 0,02 |
| HR.WA.BA.7 | 4,23 | 0,03 |
| HR.WA.MA.9 | 45,75 | 0,02 |
| HR.WA.BA.13 | 0,37 | 0,02 |
| HR.WA.MA.14 | 6,73 | 0,02 |
| HR.WA.MA.15 | 20,65 | 0,02 |
| HR.WA.BA.18 | 22,42 | 0,02 |
| HR.WA.BA.20 | 7,38 | 0,02 |
| HR.SSA.BA.1 | 2,11 | 0,02 |
| HR.SSA.BA.2 | 0,19 | 0,02 |
| HR.SSA.BA.3 | 0,50 | 0,02 |

| | | |
|--------------|-------|------|
| HR.SSA.BA.4 | 0,00 | 0,00 |
| SI.WA.FA.1 | 4,61 | 0,02 |
| SI.WA.BA.1 | 16,74 | 0,02 |
| SI.CC.FA.1 | 8,06 | 0,01 |
| SI.CC.MA.2 | 20,63 | 0,02 |
| SI.CC.FA.3 | 5,11 | 0,01 |
| SI.CC.BA.3 | 13,24 | 0,03 |
| SI.CC.FA.4 | 0,74 | 0,01 |
| SI.CC.BA.4 | 4,37 | 0,01 |
| DK.SSA.FA.1 | 0,11 | 0,00 |
| DK.SSA.FA.2 | 0,11 | 0,00 |
| DK.MSWI.FA.1 | 8,83 | 0,20 |
| FI.CC.APC1 | 0,1 | |
| FI.CC.APC2 | 0,11 | |
| FI.CC.APC3 | 0,1 | |
| FI.CC.APC4 | 0,11 | |
| FI.CC.APC5 | 0,1 | |
| FI.CC.APC6 | 0,1 | |
| FI.CC.APC8 | 0,11 | |
| FI.CC.FA1 | 1,99 | |
| FI.CC.FA2 | 1,09 | |
| FI.CC.FA3 | 2,78 | |
| FI.CC.FA4 | 2,72 | |
| FI.CC.FA5 | 2,52 | |
| FI.CC.FA6 | 1,43 | |
| FI.CC.FA8 | 1,21 | |
| FI.CC.BA1 | 3,55 | |
| FI.CC.BA2 | 3,26 | |

| | | |
|-----------|-------|--|
| FI.CC.BA3 | 6,05 | |
| FI.CC.BA4 | 5,59 | |
| FI.CC.BA5 | 2,42 | |
| FI.CC.BA6 | 22,49 | |
| FI.CC.BA7 | 8,26 | |
| FI.CC.BA8 | 6,39 | |

3.8 Bulk density

Table 1. Different ashes

| Sample ID | bulk density, kg/m ³ | | | | bulk density, kg/m ³ | |
|---------------|---------------------------------|--------|--------|-------|---------------------------------|-----------|
| | 1 | 2 | 3 | 4 | AVERAGE | deviation |
| HR.WA.FA.1 | 473,5 | 466,7 | 468,9 | 472,3 | 470,35 | 2,70 |
| HR.WA.FA.2 | 255,2 | 249,6 | 252,7 | | 252,50 | 2,29 |
| HR.WA.FA.3 | 613 | 622,6 | 622,2 | | 619,27 | 4,43 |
| HR.WA.FA.8 | 380,6 | 384,3 | 379,1 | | 381,33 | 2,19 |
| HR.WA.FA.10 | 529,9 | 528,4 | 526,2 | | 528,17 | 1,52 |
| HR.WA.FA.11 | 213,4 | 214,4 | 214,4 | | 214,07 | 0,47 |
| HR.WA.FA.12 | 567,9 | 561,1 | 568,4 | | 565,80 | 3,33 |
| HR.WA.FA.16 | 534,8 | 527,3 | 534,4 | | 532,17 | 3,45 |
| HR.WA.FA.17 | 133,2 | 131,3 | 132,2 | | 132,23 | 0,78 |
| HR.WA.FA.19 | 443,9 | 447,6 | 449,4 | | 446,97 | 2,29 |
| #SI.WA.FA.1 | 590,0 | 592,0 | 591,0 | | 591,00 | 0,82 |
| #SI.CC.FA.1 | 738,0 | 728,0 | 732,0 | | 732,67 | 4,11 |
| #SI.CC.FA.3 | 464,0 | 473,0 | 474,0 | | 470,33 | 4,50 |
| #SI.CC.FA.4 | 360,0 | 358,0 | 359,0 | | 359,00 | 0,82 |
| #NL MSWI FA 1 | 465.68 | 497.00 | 513.80 | | 492.16 | 19.94 |
| #NL MSWI FA 2 | 491.22 | 496.60 | 495.00 | | 494.27 | 1.95 |
| #NL MSWI FA 3 | 741.11 | 729.60 | 713.20 | | 727.97 | 9.92 |
| #NL MSWI FA 4 | 401.41 | 360.60 | 398.00 | | 386.67 | 16.01 |
| #NL MSWI FA 5 | 587.79 | 584.80 | 594.20 | | 588.93 | 3.40 |
| #NL MSWI FA 6 | 235.43 | 257.80 | 250.20 | | 247.81 | 8.04 |
| #NL MSWI FA 7 | 300.36 | 297.20 | 305.60 | | 301.05 | 3.00 |

| | | | | | | |
|-----------------|--------|--------|--------|--|--------|------|
| #NL MSWI FA 9 | 613.16 | 629.80 | 619.80 | | 620.92 | 5.92 |
| #NL MSWI APC 6A | 394.59 | 370.00 | 377.40 | | 380.66 | 8.92 |
| #NL MSWI APC 6B | 686.86 | 668.60 | 672.00 | | 675.82 | 6.87 |
| #NL MSWI APC 5 | 345.17 | 348.00 | 361.60 | | 351.59 | 6.21 |
| #NL WA 2 | 257.40 | 255.60 | 265.00 | | 259.33 | 3.53 |
| #NL WA 4 | 594.51 | 609.80 | 588.80 | | 597.70 | 7.68 |

3.9 Density of ashes using pycnometer

Table 1. Different ashes

| Sample ID | ρ_a [kg/m ³] | ρ_{rd} [kg/m ³] | ρ_{ssd} [kg/m ³] | WA ₂₄ [%] |
|--------------|-------------------------------|----------------------------------|-----------------------------------|----------------------|
| HR.WA.BA.4 | 3,15 | 2,84 | 2,94 | 3,49 |
| HR.WA.MA.5 | 3,05 | 2,77 | 2,86 | 3,33 |
| HR.WA.MA.6 | 2,80 | 2,45 | 2,58 | 5,03 |
| HR.WA.BA.7 | 2,66 | 2,63 | 2,64 | 0,43 |
| HR.WA.MA.9 | 2,57 | 2,15 | 2,32 | 7,54 |
| HR.WA.BA.13 | 2,62 | 2,56 | 2,58 | 0,77 |
| HR.WA.MA.14 | 2,61 | 2,55 | 2,57 | 0,94 |
| HR.WA.MA.15 | 2,70 | 1,85 | 2,16 | 17,09 |
| HR.WA.BA.18 | 3,01 | 2,93 | 2,96 | 0,92 |
| HR.WA.BA.20 | 2,64 | 2,56 | 2,59 | 1,16 |
| HR.SSA.BA.1 | 2,36 | 1,57 | 1,90 | 21,52 |
| HR.SSA.BA.2 | 1,73 | 1,04 | 1,44 | 38,44 |
| HR.SSA.BA.3 | 1,70 | 1,42 | 1,58 | 11,86 |
| HR.SSA.BA.4 | 2,15 | 1,10 | 1,59 | 44,07 |
| SI.WA.BA.1 | 0.6 | 0.57 | 0.63 | 9.68 |
| SI.CC.MA.2 | 0.65 | 0.51 | 0.72 | 39.65 |
| SI.CC.BA.3 | 0.62 | 0.59 | 0.64 | 8.72 |
| SI.CC.BA.4 | 0.64 | 0.58 | 0.68 | 16.41 |
| #NL MSWI BA1 | 2.54 | 1.95 | 2.18 | 11.77 |
| #NL MSWI BA1 | 2.26 | 2.06 | 2.15 | 4.33 |
| #NL MSWI BA1 | 2.38 | 2.07 | 2.20 | 6.27 |
| FI.CC.APC1 | 362,5 | | | |
| FI.CC.APC2 | 351,5 | | | |
| FI.CC.APC3 | 441,1 | | | |

| | | | | |
|------------|--------|--|--|--|
| FI.CC.APC6 | 383,8 | | | |
| FI.CC.APC7 | 416,8 | | | |
| FI.CC.APC8 | 363 | | | |
| FI.CC.FA1 | 574,9 | | | |
| FI.CC.FA2 | 388,8 | | | |
| FI.CC.FA3 | 532,2 | | | |
| FI.CC.FA4 | 522,7 | | | |
| FI.CC.FA5 | 513,5 | | | |
| FI.CC.FA6 | 425,6 | | | |
| FI.CC.FA8 | 413,7 | | | |
| FI.CC.BA1 | 1408,8 | | | |
| FI.CC.BA2 | 1352,1 | | | |
| FI.CC.BA3 | 1437,9 | | | |
| FI.CC.BA14 | 1427,9 | | | |
| FI.CC.BA5 | 1362,2 | | | |
| FI.CC.BA6 | 1422,7 | | | |
| FI.CC.BA8 | 1394,2 | | | |

3.10 Particle size distribution (sieving >250µm)

Table 1. Different ashes

| Sample ID | Sieve aperture (mm) - Results (% passing) | | | | | | | | | |
|-------------|---|-------|-------|------|------|------|------|------|-------|-------|
| | < 0,063 | 0,063 | 0,125 | 0,25 | 0,5 | 1 | 2 | 4 | 8 | 16 |
| HR.WA.BA.20 | 0,2 | 5,7 | 12,4 | 25,6 | 46,7 | 73,6 | 89,2 | 95,5 | 98,1 | 100,0 |
| HR.WA.MA.6 | 0,2 | 22,8 | 51,9 | 68,0 | 84,7 | 97,0 | 99,6 | 99,8 | 99,9 | 100,0 |
| HR.WA.BA.13 | 0,3 | 1,1 | 1,9 | 3,5 | 9,8 | 74,2 | 98,0 | 98,9 | 99,5 | 99,2 |
| HR.WA.BA.18 | 0,2 | 23,6 | 54,9 | 72,1 | 82,9 | 91,7 | 96,5 | 98,3 | 99,5 | 100,0 |
| HR.WA.MA.15 | 0,3 | 16,1 | 47,1 | 73,0 | 85,3 | 95,1 | 98,9 | 99,7 | 100,0 | 100,0 |
| HR.WA.BA.7 | 0,2 | 2,7 | 9,1 | 18,1 | 46,3 | 73,3 | 84,6 | 91,4 | 95,7 | 100,0 |
| HR.WA.MA.9 | 0,2 | 10,3 | 52,5 | 66,8 | 77,9 | 91,0 | 97,8 | 99,0 | 99,5 | 100,0 |
| HR.WA.BA.4 | 0,2 | 11,2 | 29,0 | 43,2 | 60,0 | 75,7 | 86,9 | 92,6 | 96,3 | 99,6 |
| HR.WA.MA.5 | 0,2 | 17,8 | 43,9 | 50,2 | 59,0 | 77,8 | 92,2 | 97,4 | 99,2 | 100,0 |
| HR.WA.MA.10 | 0,2 | 15,7 | 40,6 | 59,8 | 70,5 | 80,1 | 90,2 | 96,5 | 98,6 | 99,8 |
| HR.SSA.BA.1 | 0,5 | 1,3 | 2,0 | 3,2 | 5,9 | 12,1 | 30,6 | 84,1 | 99,9 | 100,0 |
| HR.SSA.BA.2 | 1,1 | 9,8 | 16,4 | 19,8 | 24,5 | 38,1 | 69,5 | 93,0 | 99,5 | 99,9 |
| HR.SSA.BA.3 | 0,5 | 1,3 | 2,3 | 2,8 | 4,2 | 12,1 | 39,9 | 86,4 | 99,9 | 100,0 |
| HR.SSA.BA.4 | 0,6 | 2,4 | 3,7 | 4,6 | 6,6 | 14,6 | 40,3 | 80,7 | 98,9 | 100,0 |
| SI.WA.FA.1 | 0,2 | 78,3 | 91,1 | 95,9 | 97,3 | 98,6 | 99,4 | 99,8 | 100 | 100 |
| SI.WA.BA.1 | 0 | 59,7 | 73 | 79,5 | 88,5 | 92,4 | 95,9 | 98,9 | 100 | 100 |
| SI.CC.FA.1 | 0 | 74 | 83,7 | 89,9 | 96,1 | 98,2 | 98,7 | 98,9 | 100 | 100 |
| SI.CC.MA.2 | 0 | 22,6 | 29,2 | 37,4 | 42,6 | 66,1 | 94 | 100 | 100 | 100 |
| SI.CC.FA.3 | 0 | 67,3 | 78,8 | 89 | 94,3 | 97,1 | 98,4 | 99,1 | 100 | 100 |
| SI.CC.BA.3 | 0 | 10,2 | 14,3 | 20 | 31,4 | 52,8 | 78,6 | 89,3 | 95,7 | 100 |
| SI.CC.FA.4 | 0 | 85,6 | 95,4 | 98,9 | 99,5 | 99,7 | 100 | 100 | 100 | 100 |
| SI.CC.BA.4 | 0 | 23,1 | 37,2 | 55,1 | 68,7 | 79,1 | 84,9 | 88,9 | 92,4 | 100 |

3.11 Particle size distribution (laser >250µm)

Table 1. PSD Value for Different ashes

| | | Value (µm) | | |
|-------------|------------------------------|-----------------|-----------------|-----------------|
| Sample ID | Fly ash/Bottom ash/Mixed ash | D ₁₀ | D ₅₀ | D ₉₀ |
| HR.WA.FA.1 | FA | 2,42 | 12,20 | 55,00 |
| HR.WA.FA.2 | FA | 1,73 | 7,23 | 21,83 |
| HR.WA.FA.3 | FA | 5,21 | 45,98 | 199,48 |
| HR.WA.FA.8 | FA | 2,07 | 9,79 | 36,83 |
| HR.WA.FA.10 | FA | 3,07 | 24,89 | 148,60 |
| HR.WA.FA.11 | FA | 1,97 | 8,91 | 28,45 |
| HR.WA.FA.12 | FA | 3,39 | 17,63 | 93,10 |
| HR.WA.FA.16 | FA | 2,65 | 16,83 | 101,61 |
| HR.WA.FA.17 | FA | 1,32 | 4,63 | 12,30 |
| HR.WA.FA.19 | FA | 2,47 | 13,04 | 59,05 |
| SI.WA.FA.1 | FA | 8,83 | 31,86 | 228,10 |
| SI.WA.BA.1 | BA | 5,22 | 27,47 | 239,10 |
| SI.CC.FA.1 | FA | 4,63 | 32,49 | 234,80 |
| SI.CC.MA.2 | MA | 1,78 | 11,88 | 228,90 |
| SI.CC.FA.3 | FA | 4,99 | 28,40 | 207,10 |
| SI.CC.BA.3 | BA | 4,73 | 44,30 | 235,50 |
| SI.CC.FA.4 | FA | 5,12 | 17,97 | 88,13 |
| SI.CC.BA.4 | BA | 4,37 | 31,21 | 197,40 |

Table 2. Different ashes from Slovenia

| I.WA.FA.1 | | | SI.WA.BA.1 | | | SI.CC.FA.1 | | | SI.CC.MA.2 | | | SI.CC.FA.3 | | | SI.CC.BA.3 | | | SI.CC.FA.4 | | | SI.CC.BA.4 | | |
|-----------|-----------|------------|------------|-----------|------------|------------|-----------|------------|------------|-----------|------------|------------|-----------|------------|------------|-----------|------------|------------|-----------|------------|------------|-----------|------------|
| d (µm) | q3 (%) | Q3 (%P) | d (µm) | q3 (%) | Q3 (%P) | d(µm) | q3 (%) | Q3 (%P) | d (µm) | q3 (%) | Q3 (%P) |
| 2000 | 0 | 100 | 2000 | 0 | 100 | 2000 | 0 | 100 | 2000 | 0 | 100 | 2000 | 0 | 100 | 2000 | 0 | 100 | 2000 | 0 | 100 | 2000 | 0 | 100 |
| 1674 | 0 | 100 | 1674 | 0 | 100 | 1674 | 0 | 100 | 1674 | 0 | 100 | 1674 | 0 | 100 | 1674 | 0 | 100 | 1674 | 0 | 100 | 1674 | 0 | 100 |
| 1408 | 0 | 100 | 1408 | 0 | 100 | 1408 | 0 | 100 | 1408 | 0 | 100 | 1408 | 0 | 100 | 1408 | 0 | 100 | 1408 | 0 | 100 | 1408 | 0 | 100 |
| 1184 | 0 | 100 | 1184 | 0 | 100 | 1184 | 0 | 100 | 1184 | 0 | 100 | 1184 | 0 | 100 | 1184 | 0 | 100 | 1184 | 0 | 100 | 1184 | 0 | 100 |
| 995,6 | 0 | 100 | 995,6 | 0 | 100 | 995,6 | 0 | 100 | 995,6 | 0 | 100 | 995,6 | 0 | 100 | 995,6 | 0 | 100 | 995,6 | 0 | 100 | 995,6 | 0 | 100 |
| 837,2 | 0 | 100 | 837,2 | 0 | 100 | 837,2 | 0 | 100 | 837,2 | 0 | 100 | 837,2 | 0 | 100 | 837,2 | 0 | 100 | 837,2 | 0 | 100 | 837,2 | 0 | 100 |
| 704 | 0 | 100 | 704 | 0 | 100 | 704 | 0 | 100 | 704 | 0 | 100 | 704 | 0 | 100 | 704 | 0 | 100 | 704 | 0 | 100 | 704 | 0 | 100 |
| 592 | 0 | 100 | 592 | 0 | 100 | 592 | 0 | 100 | 592 | 0 | 100 | 592 | 0 | 100 | 592 | 0 | 100 | 592 | 0 | 100 | 592 | 0 | 100 |
| 497,8 | 0 | 100 | 497,8 | 0 | 100 | 497,8 | 0 | 100 | 497,8 | 0 | 100 | 497,8 | 0 | 100 | 497,8 | 0 | 100 | 497,8 | 0 | 100 | 497,8 | 0 | 100 |
| 418,6 | 1,95 | 100 | 418,6 | 0 | 100 | 418,6 | 0 | 100 | 418,6 | 0 | 100 | 418,6 | 0 | 100 | 418,6 | 0 | 100 | 418,6 | 0 | 100 | 418,6 | 0 | 100 |
| 352 | 2,38 | 98,05 | 352 | 1,74 | 100 | 352 | 2,31 | 100 | 352 | 1,59 | 100 | 352 | 2,22 | 100 | 352 | 1,67 | 100 | 352 | 0 | 100 | 352 | 0,57 | 100 |
| 296 | 3,77 | 95,67 | 296 | 6,79 | 98,26 | 296 | 2,78 | 97,69 | 296 | 5,49 | 98,41 | 296 | 3,01 | 97,78 | 296 | 5,71 | 98,33 | 296 | 0 | 100 | 296 | 4,42 | 99,43 |
| 248,9 | 3,7 | 91,9 | 248,9 | 5,29 | 91,47 | 248,9 | 4,92 | 94,91 | 248,9 | 5,59 | 92,92 | 248,9 | 4,44 | 94,77 | 248,9 | 8,32 | 92,62 | 248,9 | 1,18 | 100 | 248,9 | 3,86 | 95,01 |
| 209,3 | 3,23 | 88,2 | 209,3 | 2,4 | 86,18 | 209,3 | 4,52 | 89,99 | 209,3 | 2,82 | 87,33 | 209,3 | 5,23 | 90,33 | 209,3 | 6,47 | 84,3 | 209,3 | 1,33 | 98,82 | 209,3 | 3,31 | 91,15 |
| 176 | 2,34 | 84,97 | 176 | 1,84 | 83,78 | 176 | 3,64 | 85,47 | 176 | 1,34 | 84,51 | 176 | 3,17 | 85,1 | 176 | 5,89 | 77,83 | 176 | 1,17 | 97,49 | 176 | 3,28 | 87,84 |
| 148 | 2,16 | 82,63 | 148 | 2,01 | 81,94 | 148 | 2,99 | 81,83 | 148 | 0,48 | 83,17 | 148 | 2,69 | 81,93 | 148 | 4,18 | 71,94 | 148 | 1,5 | 96,32 | 148 | 2,92 | 84,56 |
| 124,5 | 2,7 | 80,47 | 124,5 | 2,29 | 79,93 | 124,5 | 3,2 | 78,84 | 124,5 | 0,57 | 82,69 | 124,5 | 2,27 | 79,24 | 124,5 | 3,17 | 67,76 | 124,5 | 2,32 | 94,82 | 124,5 | 3,17 | 81,64 |
| 104,7 | 3,05 | 77,77 | 104,7 | 2,55 | 77,64 | 104,7 | 3,56 | 75,64 | 104,7 | 1 | 82,12 | 104,7 | 2,32 | 76,97 | 104,7 | 2,65 | 64,59 | 104,7 | 2,52 | 92,5 | 104,7 | 3,45 | 78,47 |
| 88 | 3,4 | 74,72 | 88 | 2,57 | 75,09 | 88 | 3,63 | 72,08 | 88 | 1,31 | 81,12 | 88 | 2,62 | 74,65 | 88 | 2,69 | 61,94 | 88 | 2,32 | 89,98 | 88 | 3,7 | 75,02 |
| 74 | 3,57 | 71,32 | 74 | 2,97 | 72,52 | 74 | 3,69 | 68,45 | 74 | 1,6 | 79,81 | 74 | 3,09 | 72,03 | 74 | 2,88 | 59,25 | 74 | 2,18 | 87,66 | 74 | 4 | 71,32 |
| 62,23 | 3,87 | 67,75 | 62,23 | 3,36 | 69,55 | 62,23 | 3,9 | 64,76 | 62,23 | 2 | 78,21 | 62,23 | 3,58 | 68,94 | 62,23 | 3,16 | 56,37 | 62,23 | 2,21 | 85,48 | 62,23 | 4,28 | 67,32 |
| 52,33 | 4,34 | 63,88 | 52,33 | 3,82 | 66,19 | 52,33 | 4,12 | 60,86 | 52,33 | 2,45 | 76,21 | 52,33 | 3,97 | 65,36 | 52,33 | 3,34 | 53,21 | 52,33 | 2,77 | 83,27 | 52,33 | 4,33 | 63,04 |
| 44 | 4,85 | 59,54 | 44 | 4,25 | 62,37 | 44 | 4,31 | 56,74 | 44 | 2,83 | 73,76 | 44 | 4,31 | 61,39 | 44 | 3,48 | 49,87 | 44 | 3,54 | 80,5 | 44 | 4,38 | 58,71 |
| 37 | 5,47 | 54,69 | 37 | 4,62 | 58,12 | 37 | 4,45 | 52,43 | 37 | 3,1 | 70,93 | 37 | 4,58 | 57,08 | 37 | 3,56 | 46,39 | 37 | 4,57 | 76,96 | 37 | 4,41 | 54,33 |
| 31,11 | 6,16 | 49,22 | 31,11 | 4,9 | 53,5 | 31,11 | 4,52 | 47,98 | 31,11 | 3,24 | 67,83 | 31,11 | 4,77 | 52,5 | 31,11 | 3,6 | 42,83 | 31,11 | 5,82 | 72,39 | 31,11 | 4,41 | 49,92 |

| | | | | | | | | | | | | | | | | | | | | | | | |
|-------|------|-------|-------|------|-------|-------|------|-------|-------|------|-------|-------|------|-------|-------|------|-------|-------|------|-------|-------|------|-------|
| 26,16 | 6,73 | 43,06 | 26,16 | 5,07 | 48,6 | 26,16 | 4,52 | 43,46 | 26,16 | 3,26 | 64,59 | 26,16 | 4,75 | 47,73 | 26,16 | 3,59 | 39,23 | 26,16 | 7,09 | 66,57 | 26,16 | 4,39 | 45,51 |
| 22 | 6,81 | 36,33 | 22 | 5,12 | 43,53 | 22 | 4,44 | 38,94 | 22 | 3,22 | 61,33 | 22 | 4,85 | 42,98 | 22 | 3,53 | 35,64 | 22 | 8,07 | 59,48 | 22 | 4,32 | 41,12 |
| 18,5 | 6,38 | 29,52 | 18,5 | 5,02 | 38,41 | 18,5 | 4,28 | 34,5 | 18,5 | 3,16 | 58,11 | 18,5 | 4,73 | 38,13 | 18,5 | 3,43 | 32,11 | 18,5 | 8,27 | 51,41 | 18,5 | 4,2 | 36,8 |
| 15,56 | 5,35 | 23,14 | 15,56 | 4,8 | 33,39 | 15,56 | 4,05 | 30,22 | 15,56 | 3,16 | 54,95 | 15,56 | 4,53 | 33,4 | 15,56 | 3,29 | 28,68 | 15,56 | 8,09 | 43,14 | 15,56 | 4,02 | 32,6 |
| 13,08 | 4,12 | 17,79 | 13,08 | 4,47 | 28,59 | 13,08 | 3,77 | 26,17 | 13,08 | 3,24 | 51,79 | 13,08 | 4,25 | 28,87 | 13,08 | 3,13 | 25,39 | 13,08 | 7,13 | 35,05 | 13,08 | 3,78 | 28,58 |
| 11 | 3,02 | 13,67 | 11 | 4,06 | 24,12 | 11 | 3,44 | 22,4 | 11 | 3,43 | 48,55 | 11 | 3,9 | 24,62 | 11 | 2,94 | 22,26 | 11 | 5,88 | 27,92 | 11 | 3,5 | 24,8 |
| 9,25 | 2,18 | 10,65 | 9,25 | 3,6 | 20,06 | 9,25 | 3,09 | 18,96 | 9,25 | 3,67 | 45,12 | 9,25 | 3,52 | 20,72 | 9,25 | 2,73 | 19,32 | 9,25 | 4,63 | 22,04 | 9,25 | 3,18 | 21,3 |
| 7,78 | 1,61 | 8,47 | 7,78 | 3,11 | 16,46 | 7,78 | 2,73 | 15,87 | 7,78 | 3,93 | 41,45 | 7,78 | 3,12 | 17,2 | 7,78 | 2,51 | 16,59 | 7,78 | 3,6 | 17,41 | 7,78 | 2,83 | 18,12 |
| 6,54 | 1,24 | 6,86 | 6,54 | 2,64 | 13,35 | 6,54 | 2,39 | 13,14 | 6,54 | 4,14 | 37,52 | 6,54 | 2,72 | 14,08 | 6,54 | 2,28 | 14,08 | 6,54 | 2,82 | 13,81 | 6,54 | 2,49 | 15,29 |
| 5,5 | 0,99 | 5,62 | 5,5 | 2,2 | 10,71 | 5,5 | 2,06 | 10,75 | 5,5 | 4,18 | 33,38 | 5,5 | 2,33 | 11,36 | 5,5 | 2,05 | 11,8 | 5,5 | 2,25 | 10,99 | 5,5 | 2,16 | 12,8 |
| 4,63 | 0,83 | 4,63 | 4,63 | 1,8 | 8,51 | 4,63 | 1,76 | 8,69 | 4,63 | 4,2 | 29,2 | 4,63 | 1,96 | 9,03 | 4,63 | 1,81 | 9,75 | 4,63 | 1,83 | 8,74 | 4,63 | 1,86 | 10,64 |
| 3,89 | 0,72 | 3,8 | 3,89 | 1,47 | 6,71 | 3,89 | 1,49 | 6,93 | 3,89 | 4 | 25 | 3,89 | 1,62 | 7,07 | 3,89 | 1,59 | 7,94 | 3,89 | 1,49 | 6,91 | 3,89 | 1,59 | 8,78 |
| 3,27 | 0,64 | 3,08 | 3,27 | 1,18 | 5,24 | 3,27 | 1,25 | 5,44 | 3,27 | 3,68 | 21 | 3,27 | 1,33 | 5,45 | 3,27 | 1,38 | 6,35 | 3,27 | 1,21 | 5,42 | 3,27 | 1,35 | 7,19 |
| 2,75 | 0,57 | 2,44 | 2,75 | 0,95 | 4,06 | 2,75 | 1,05 | 4,19 | 2,75 | 3,26 | 17,32 | 2,75 | 1,07 | 4,12 | 2,75 | 1,17 | 4,97 | 2,75 | 0,99 | 4,21 | 2,75 | 1,15 | 5,84 |
| 2,313 | 0,52 | 1,87 | 2,313 | 0,77 | 3,11 | 2,313 | 0,87 | 3,14 | 2,313 | 2,8 | 14,06 | 2,313 | 0,84 | 3,05 | 2,313 | 0,99 | 3,8 | 2,313 | 0,8 | 3,22 | 2,313 | 0,98 | 4,69 |
| 1,945 | 0,46 | 1,35 | 1,945 | 0,61 | 2,34 | 1,945 | 0,72 | 2,27 | 1,945 | 2,33 | 11,26 | 1,945 | 0,67 | 2,21 | 1,945 | 0,82 | 2,81 | 1,945 | 0,64 | 2,42 | 1,945 | 0,84 | 3,71 |
| 1,635 | 0,42 | 0,89 | 1,635 | 0,5 | 1,73 | 1,635 | 0,58 | 1,55 | 1,635 | 1,91 | 8,93 | 1,635 | 0,52 | 1,54 | 1,635 | 0,67 | 1,99 | 1,635 | 0,53 | 1,78 | 1,635 | 0,72 | 2,87 |
| 1,375 | 0,36 | 0,47 | 1,375 | 0,41 | 1,23 | 1,375 | 0,47 | 0,97 | 1,375 | 1,54 | 7,02 | 1,375 | 0,41 | 1,02 | 1,375 | 0,55 | 1,32 | 1,375 | 0,44 | 1,25 | 1,375 | 0,61 | 2,15 |
| 1,156 | 0,11 | 0,11 | 1,156 | 0,24 | 0,82 | 1,156 | 0,38 | 0,5 | 1,156 | 1,23 | 5,48 | 1,156 | 0,24 | 0,61 | 1,156 | 0,43 | 0,77 | 1,156 | 0,37 | 0,81 | 1,156 | 0,52 | 1,54 |
| 0,972 | 0 | 0 | 0,972 | 0,25 | 0,58 | 0,972 | 0,12 | 0,12 | 0,972 | 0,98 | 4,25 | 0,972 | 0,12 | 0,37 | 0,972 | 0,34 | 0,34 | 0,972 | 0,33 | 0,44 | 0,972 | 0,44 | 1,02 |
| 0,818 | 0 | 0 | 0,818 | 0,18 | 0,33 | 0,818 | 0 | 0 | 0,818 | 0,79 | 3,27 | 0,818 | 0,13 | 0,25 | 0,818 | 0 | 0 | 0,818 | 0,11 | 0,11 | 0,818 | 0,37 | 0,58 |
| 0,688 | 0 | 0 | 0,688 | 0,15 | 0,15 | 0,688 | 0 | 0 | 0,688 | 0,64 | 2,48 | 0,688 | 0,12 | 0,12 | 0,688 | 0 | 0 | 0,688 | 0 | 0 | 0,688 | 0,21 | 0,21 |
| 0,578 | 0 | 0 | 0,578 | 0 | 0 | 0,578 | 0 | 0 | 0,578 | 0,41 | 1,84 | 0,578 | 0 | 0 | 0,578 | 0 | 0 | 0,578 | 0 | 0 | 0,578 | 0 | 0 |
| 0,486 | 0 | 0 | 0,486 | 0 | 0 | 0,486 | 0 | 0 | 0,486 | 0,35 | 1,43 | 0,486 | 0 | 0 | 0,486 | 0 | 0 | 0,486 | 0 | 0 | 0,486 | 0 | 0 |
| 0,409 | 0 | 0 | 0,409 | 0 | 0 | 0,409 | 0 | 0 | 0,409 | 0,3 | 1,08 | 0,409 | 0 | 0 | 0,409 | 0 | 0 | 0,409 | 0 | 0 | 0,409 | 0 | 0 |
| 0,344 | 0 | 0 | 0,344 | 0 | 0 | 0,344 | 0 | 0 | 0,344 | 0,26 | 0,78 | 0,344 | 0 | 0 | 0,344 | 0 | 0 | 0,344 | 0 | 0 | 0,344 | 0 | 0 |
| 0,289 | 0 | 0 | 0,289 | 0 | 0 | 0,289 | 0 | 0 | 0,289 | 0,21 | 0,52 | 0,289 | 0 | 0 | 0,289 | 0 | 0 | 0,289 | 0 | 0 | 0,289 | 0 | 0 |
| 0,243 | 0 | 0 | 0,243 | 0 | 0 | 0,243 | 0 | 0 | 0,243 | 0,17 | 0,31 | 0,243 | 0 | 0 | 0,243 | 0 | 0 | 0,243 | 0 | 0 | 0,243 | 0 | 0 |
| 0,204 | 0 | 0 | 0,204 | 0 | 0 | 0,204 | 0 | 0 | 0,204 | 0,14 | 0,14 | 0,204 | 0 | 0 | 0,204 | 0 | 0 | 0,204 | 0 | 0 | 0,204 | 0 | 0 |

| | | | | | | | | | | | | | | | | | | | | | | | |
|------------|---|---|------------|---|---|------------|---|---|------------|---|---|--------|---|---|--------|---|---|------------|---|---|------------|---|---|
| 0,172 | 0 | 0 | 0,172 | 0 | 0 | 0,172 | 0 | 0 | 0,172 | 0 | 0 | 0,172 | 0 | 0 | 0,172 | 0 | 0 | 0,172 | 0 | 0 | 0,172 | 0 | 0 |
| 0,145 | 0 | 0 | 0,145 | 0 | 0 | 0,145 | 0 | 0 | 0,145 | 0 | 0 | 0,145 | 0 | 0 | 0,145 | 0 | 0 | 0,145 | 0 | 0 | 0,145 | 0 | 0 |
| 0,122 | 0 | 0 | 0,122 | 0 | 0 | 0,122 | 0 | 0 | 0,122 | 0 | 0 | 0,122 | 0 | 0 | 0,122 | 0 | 0 | 0,122 | 0 | 0 | 0,122 | 0 | 0 |
| 0,102 | 0 | 0 | 0,102 | 0 | 0 | 0,102 | 0 | 0 | 0,102 | 0 | 0 | 0,102 | 0 | 0 | 0,102 | 0 | 0 | 0,102 | 0 | 0 | 0,102 | 0 | 0 |
| 0,086 | 0 | 0 | 0,086 | 0 | 0 | 0,086 | 0 | 0 | 0,086 | 0 | 0 | 0,086 | 0 | 0 | 0,086 | 0 | 0 | 0,086 | 0 | 0 | 0,086 | 0 | 0 |
| 0,072 | 0 | 0 | 0,072 | 0 | 0 | 0,072 | 0 | 0 | 0,072 | 0 | 0 | 0,072 | 0 | 0 | 0,072 | 0 | 0 | 0,072 | 0 | 0 | 0,072 | 0 | 0 |
| 0,061 | 0 | 0 | 0,061 | 0 | 0 | 0,061 | 0 | 0 | 0,061 | 0 | 0 | 0,061 | 0 | 0 | 0,061 | 0 | 0 | 0,061 | 0 | 0 | 0,061 | 0 | 0 |
| 0,051 | 0 | 0 | 0,051 | 0 | 0 | 0,051 | 0 | 0 | 0,051 | 0 | 0 | 0,051 | 0 | 0 | 0,051 | 0 | 0 | 0,051 | 0 | 0 | 0,051 | 0 | 0 |
| 0,043 | 0 | 0 | 0,043 | 0 | 0 | 0,043 | 0 | 0 | 0,043 | 0 | 0 | 0,043 | 0 | 0 | 0,043 | 0 | 0 | 0,043 | 0 | 0 | 0,043 | 0 | 0 |
| 0,036 | 0 | 0 | 0,036 | 0 | 0 | 0,036 | 0 | 0 | 0,036 | 0 | 0 | 0,036 | 0 | 0 | 0,036 | 0 | 0 | 0,036 | 0 | 0 | 0,036 | 0 | 0 |
| 0,03 | 0 | 0 | 0,03 | 0 | 0 | 0,03 | 0 | 0 | 0,03 | 0 | 0 | 0,03 | 0 | 0 | 0,03 | 0 | 0 | 0,03 | 0 | 0 | 0,03 | 0 | 0 |
| 0,025 5 | 0 | 0 | 0,0255 | 0 | 0 | 0,0255 | 0 | 0 | 0,025 5 | 0 | 0 | 0,025 5 | 0 | 0 |
| 0,021 5 | 0 | 0 | 0,0215 | 0 | 0 | 0,0215 | 0 | 0 | 0,021 5 | 0 | 0 | 0,021 5 | 0 | 0 |
| 0,018 1 | 0 | 0 | 0,0181 | 0 | 0 | 0,0181 | 0 | 0 | 0,018 1 | 0 | 0 | 0,018 1 | 0 | 0 |
| 0,015 2 | 0 | 0 | 0,0152 | 0 | 0 | 0,0152 | 0 | 0 | 0,015 2 | 0 | 0 | 0,015 2 | 0 | 0 |
| 0,012 8 | 0 | 0 | 0,0128 | 0 | 0 | 0,0128 | 0 | 0 | 0,012 8 | 0 | 0 | 0,012 8 | 0 | 0 |

Table 3. Different ashes from Denmark

| #DK.MSWI-FA.1 | | | #DK.MSWI-FA.4 | | | #DK.SSA-FA.1 | | | #DK.SSA-FA.2 | | |
|---------------|----------|--------|---------------|-------|---------|--------------|-------|--------|--------------|----------|---------|
| d(µm) | q3(%) | Q3(%P) | d(µm) | q3(%) | Q3(%P) | d(µm) | q3(%) | Q3(%P) | d(µm) | q3(%) | Q3(%P) |
| 2000 | 0 | 100 | 2000 | 0 | 100 | 2000 | 0 | 100 | 2000 | 0 | 100 |
| 1905,46 | 0 | 100 | 1905,461 | 0 | 100 | 1905,461 | 0 | 100 | 1905,461 | 0 | 100 |
| 1659,59 | 0 | 100 | 1659,587 | 0 | 100 | 1659,587 | 0 | 100 | 1659,587 | 0 | 100 |
| 1445,44 | 0 | 100 | 1445,44 | 0 | 100 | 1445,44 | 0 | 100 | 1445,44 | 0 | 100 |
| 1258,93 | 0 | 100 | 1258,925 | 0 | 100 | 1258,925 | 0 | 100 | 1258,925 | 0 | 100 |
| 1096,48 | 0 | 100 | 1096,478 | 0 | 100 | 1096,478 | 0 | 100 | 1096,478 | 0 | 100 |
| 954,99 | 0 | 100 | 954,9926 | 0 | 100 | 954,9926 | 0 | 100 | 954,9926 | 0 | 100 |
| 831,76 | 0 | 100 | 831,7638 | 0 | 100 | 831,7638 | 0 | 100 | 831,7638 | 0 | 100 |
| 724,44 | 0 | 100 | 724,436 | 0 | 100 | 724,436 | 0,054 | 100 | 724,436 | 0,007071 | 100 |
| 630,96 | 0,163913 | 100 | 630,9573 | 0,000 | 100 | 630,9573 | 0,411 | 99,946 | 630,9573 | 0,388329 | 99,9929 |
| 549,54 | 0,921118 | 99,84 | 549,5409 | 0,000 | 100 | 549,5409 | 1,361 | 99,536 | 549,5409 | 1,525385 | 99,6046 |
| 478,63 | 1,750786 | 98,91 | 478,6301 | 0,000 | 100 | 478,6301 | 2,343 | 98,175 | 478,6301 | 2,703675 | 98,0792 |
| 416,87 | 2,633261 | 97,16 | 416,8694 | 0,000 | 100 | 416,8694 | 3,518 | 95,832 | 416,8694 | 4,108079 | 95,3755 |
| 363,08 | 3,497219 | 94,53 | 363,0781 | 0,134 | 100,000 | 363,0781 | 4,660 | 92,314 | 363,0781 | 5,482839 | 91,2675 |
| 316,23 | 4,312312 | 91,03 | 316,2278 | 1,633 | 99,866 | 316,2278 | 5,673 | 87,654 | 316,2278 | 6,714515 | 85,7846 |
| 275,42 | 5,013664 | 86,72 | 275,4229 | 2,986 | 98,233 | 275,4229 | 6,421 | 81,980 | 275,4229 | 7,634321 | 79,0701 |
| 239,88 | 5,576726 | 81,71 | 239,8833 | 4,396 | 95,247 | 239,8833 | 6,855 | 75,560 | 239,8833 | 8,172604 | 71,4358 |
| 208,93 | 5,963175 | 76,13 | 208,9296 | 5,624 | 90,851 | 208,9296 | 6,958 | 68,704 | 208,9296 | 8,290786 | 63,2632 |
| 181,97 | 6,178396 | 70,17 | 181,9701 | 6,607 | 85,227 | 181,9701 | 6,775 | 61,747 | 181,9701 | 8,028112 | 54,9724 |
| 158,49 | 6,22516 | 63,99 | 158,4893 | 7,255 | 78,620 | 158,4893 | 6,386 | 54,972 | 158,4893 | 7,471758 | 46,9443 |
| 138,04 | 6,122985 | 57,76 | 138,0384 | 7,557 | 71,365 | 138,0384 | 5,864 | 48,586 | 138,0384 | 6,705155 | 39,4725 |
| 120,23 | 5,895609 | 51,64 | 120,2264 | 7,517 | 63,808 | 120,2264 | 5,297 | 42,721 | 120,2264 | 5,844954 | 32,7674 |
| 104,71 | 5,562352 | 45,75 | 104,7129 | 7,179 | 56,291 | 104,7129 | 4,725 | 37,424 | 104,7129 | 4,95401 | 26,9224 |
| 91,2 | 5,156999 | 40,18 | 91,20108 | 6,623 | 49,112 | 91,20108 | 4,196 | 32,699 | 91,20108 | 4,114316 | 21,9684 |
| 79,43 | 4,700953 | 35,03 | 79,43282 | 5,924 | 42,489 | 79,43282 | 3,715 | 28,504 | 79,43282 | 3,353114 | 17,8541 |
| 69,18 | 4,228022 | 30,33 | 69,1831 | 5,178 | 36,564 | 69,1831 | 3,294 | 24,789 | 69,1831 | 2,704469 | 14,501 |

| | | | | | | | | | | | |
|-------|----------|-------|----------|--------|--------|----------|--------|--------|----------|----------|---------|
| 60,26 | 3,755955 | 26,1 | 60,25596 | 4,446 | 31,387 | 60,25596 | 2,923 | 21,495 | 60,25596 | 2,166631 | 11,7965 |
| 52,48 | 3,306093 | 22,34 | 52,48075 | 3,785 | 26,941 | 52,48075 | 2,598 | 18,571 | 52,48075 | 1,738322 | 9,6299 |
| 45,71 | 2,887154 | 19,04 | 45,70882 | 3,218 | 23,156 | 45,70882 | 2,308 | 14,973 | 45,70882 | 1,403313 | 7,8916 |
| 39,81 | 2,505767 | 16,15 | 39,81072 | 2,752 | 19,937 | 39,81072 | 2,047 | 13,665 | 39,81072 | 1,145219 | 6,4882 |
| 34,67 | 2,163779 | 13,64 | 34,67369 | 2,379 | 17,185 | 34,67369 | 1,808 | 11,618 | 34,67369 | 0,946807 | 5,343 |
| 30,2 | 1,858626 | 11,48 | 30,19952 | 2,079 | 14,806 | 30,19952 | 1,588 | 9,810 | 30,19952 | 0,791809 | 4,3962 |
| 26,3 | 1,59 | 9,62 | 26,30268 | 1,833 | 12,727 | 26,30268 | 1,387 | 8,222 | 26,30268 | 0,668932 | 3,6044 |
| 22,91 | 1,352076 | 8,03 | 22,90868 | 1,622 | 10,894 | 22,90868 | 1,200 | 6,835 | 22,90868 | 0,567679 | 2,9355 |
| 19,95 | 1,145223 | 6,68 | 19,95262 | 1,435 | 9,272 | 19,95262 | 1,030 | 5,635 | 19,95262 | 0,483174 | 2,3678 |
| 17,38 | 0,963184 | 5,53 | 17,37801 | 1,258 | 7,837 | 17,37801 | 0,875 | 4,605 | 17,37801 | 0,409844 | 1,8846 |
| 15,14 | 0,807251 | 4,57 | 15,13561 | 1,094 | 6,579 | 15,13561 | 0,737 | 3,730 | 15,13561 | 0,346605 | 1,4748 |
| 13,18 | 0,671829 | 3,76 | 13,18257 | 0,938 | 5,485 | 13,18257 | 0,613 | 2,993 | 13,18257 | 0,290314 | 1,1282 |
| 11,48 | 0,55888 | 3,09 | 11,48154 | 0,796 | 4,548 | 11,48154 | 0,507 | 2,379 | 11,48154 | 0,241187 | 0,8379 |
| 10 | 0,46374 | 2,53 | 10 | 0,668 | 3,752 | 10 | 0,414 | 1,872 | 10 | 0,196839 | 0,5967 |
| 8,71 | 0,388292 | 2,07 | 8,709636 | 0,561 | 3,084 | 8,709636 | 0,338 | 1,458 | 8,709636 | 0,157928 | 0,3998 |
| 7,59 | 0,328601 | 1,68 | 7,585776 | 0,473 | 2,523 | 7,585776 | 0,276 | 1,120 | 7,585776 | 0,123487 | 0,2419 |
| 6,61 | 0,284013 | 1,35 | 6,606934 | 0,406 | 2,050 | 6,606934 | 0,227 | 0,844 | 6,606934 | 0,094284 | 0,1184 |
| 5,75 | 0,250176 | 1,07 | 5,754399 | 0,355 | 1,645 | 5,754399 | 0,187 | 0,617 | 5,754399 | 0,024135 | 0,0241 |
| 5,01 | 0,223551 | 0,82 | 5,011872 | 0,316 | 1,290 | 5,011872 | 0,155 | 0,430 | 5,011872 | 0 | 0 |
| 4,37 | 0,197981 | 0,59 | 4,365158 | 0,282 | 0,974 | 4,365158 | 0,125 | 0,275 | 4,365158 | 0 | 0 |
| 3,8 | 0,171381 | 0,4 | 3,801894 | 0,245 | 0,692 | 3,801894 | 0,099 | 0,149 | 3,801894 | 0 | 0 |
| 3,31 | 0,136331 | 0,22 | 3,311311 | 0,2042 | 0,447 | 3,311311 | 0,0499 | 0,0499 | 3,311311 | 0 | 0 |
| 2,88 | 0,087465 | 0,09 | 2,884031 | 0,1493 | 0,243 | 2,884031 | 0 | 0 | 2,884031 | 0 | 0 |
| 2,51 | 0 | 0 | 2,511886 | 0,0934 | 0,093 | 2,511886 | 0 | 0 | 2,511886 | 0 | 0 |
| 2,19 | 0 | 0 | 2,187762 | 0 | 0 | 2,187762 | 0 | 0 | | | |
| 1,91 | 0 | 0 | 1,905461 | 0 | 0 | | | | | | |
| 1,66 | 0 | 0 | 1,659587 | 0 | 0 | | | | | | |

3.12 Specific surface area of solids by gas adsorption – BET Method

Table 1. Different ashes

| Sample ID | Specific surface area [m ² /g] |
|------------|---|
| SI.WA.FA.1 | 43.47 |
| SI.WA.BA.1 | 3.49 |
| SI.CC.FA.1 | 4.15 |
| SI.CC.MA.2 | 88.95 |
| SI.CC.FA.3 | 5.75 |
| SI.CC.BA.3 | 42.08 |
| SI.CC.FA.4 | 14.4 |
| SI.CC.BA.4 | 2.06 |