CHARACTERIZATION SCEME FOR ASHES TO SUPPORT THE UTILIZATION IN DIFFERENT CONSTRUCTION MATERIALS

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Abstract

A variety of ashes 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 recycled. Some of these ashes contain scarce chemical elements, which should be 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. To scale such recycling of ashes it is necessary to be able to directly link the ash characteristics and construction material properties. In literature, there is no generally accepted scheme for the characterization of ashes. Different methods are used by different researchers, and if the same methods are used, they often vary slightly. As consequence, a direct comparison of the results is doubtful. This calls for a common

characterization scheme to support the scaling of the recycling. This paper suggests such a scheme. It is based on literature and experiences from the project partners in the EU-financed project AshCycle.

Keywords: Ash ; construction materials ; recycling ; characterization

1. INTRODUCTION

Combustion processes are important to the energy generation in the EU and during the last decades, different fuels have gained increased importance due to the phasing out of coal. Waste-to-energy and biomass-fired plants are the two main groups replacing coal-fired power plants. Next to energy, an output from the combustion is the non-combustible inorganic residues in the form of ashes. The European Green Deal stimulates the recycling of finite resources and underlines a zero-pollution ambition. In support of this, the ashes must be considered as resources, but at the same time, their use should not cause environmental pollution. Still, current practice is generally to classify the ashes as waste and disposal in landfills or use them as backfill in abandoned mines (e.g. municipal solid waste incineration fly ash MSWI-FA [1]). This leads to significant losses of metals, nutrients, rare earth elements (REEs), and industrially valuable minerals in the ashes. Moreover, the cost of landfill and backfill is high.

In support of a resource-efficient society based on circular economy principles, secondary resources such as ashes must be recycled at the highest possible level. Utilization of ashes as cement replacement in concrete is also beneficial in relation to lowering the carbon footprint of concrete. Some of these ashes contain valuable and scarce chemical elements, which should be extracted and recovered to reduce the mining of the same chemical elements from nature. Also, many examples in the literature show that ashes have the potential to replace natural raw materials in the production of construction materials; however, sometimes only after pretreatment. Still, the recycling of the chemical elements from the ashes and the ashes themselves is limited.

The Horizon Europe project AshCycle "Integration of underutilized ashes into material cycles by industry-urban symbiosis" was initiated to develop and strengthen the recovery of important chemical elements and use of the ashes in construction materials. The project is led by the University of Oulu, and it has 28 partners (7 universities and 21 industry partners). Collaboration in such a large consortium, researching many different ashes, calls for common procedures to characterize the ashes to compare and discuss results between partners. In literature, there is no generally accepted scheme for the experimental characterization of ashes. In addition, the procedures reported for the same methods often vary slightly, so a direct comparison of the obtained results is doubtful.

In addition to support direct comparison of the results obtained in the AshCycle project, the developed characterization scheme could support a general scaling of the use of ashes in construction materials. Such scaling entails the ability to link ash characteristics and construction material properties. To overcome this task, a large amount of experimental data is needed. For the comparison of these data, it would be highly beneficial if the ash characterization was performed in the same way, and thus implementing the scheme generally in the research conducted would support the scaling.

This paper presents the ash characterization scheme developed and agreed on among the partners who were involved in this exact task (the authors) on behalf of the AshCycle project. The scheme was developed through in-depth discussions in a series of online meetings in 2023. We hope that this scheme can inspire other researchers to use the same procedures so we in the future can enhance a direct comparison of research between published works, and potentially the scheme can be helpful in possible future norm work.

2. IMPORTANT INFORMATION ON COMBUSTION PLANT

2.1 Ash type

To compare experimental results obtained with different ashes, it is necessary to have consistency in the reporting. First, the ashes should preferably be grouped after the type of fuel, e.g., wood ash or sewage sludge ash. In case of co-combustion of fuels, the name should inform of this, e.g. co-combustion ash from sewage sludge and waste.

Secondly, precision is important when reporting the type of ash. Bottom ashes are the coarse incombustible part that is collected from the bottom of furnaces, i.e., they consist of the ash particles, which are too large to be carried in the flue gases. Fly ash is defined as the solid residue from the combustion process that is entrained in the flue gas and subsequently captured by particulate filtration equipment. An air pollution control (APC) residue comprises a mixture of fly ash and air pollution control reagents added to the system to further clean the flue gas, typically lime and carbon. It is important to distinguish between the ash types because they have distinctly different characteristics. For instance, the particle size is generally larger for bottom ashes than fly ashes and APC residues. Bottom ash particles in MSW combustion systems are in the size range of 0.5 – 4 mm, while fly ashes collected in grate systems are often reported to be rich in sub-micron ash particles with peaks at around 0.2 up to 0.5mm [2]. Also related to the content of heavy metals, several researchers have reported generally higher heavy metal concentrations in the fly ashes than in the bottom ashes (e.g. for wood ashes [3–5]) and similarly, the content of alkalies is higher in wood fly ash than in wood bottom ash [6]. Based on a T-test, [7] showed that an MSWI fly ash and an MSWI-APC residue were significantly different, with a 99% confidence level e.g. for pH, conductivity, loss on ignition, and Cl content.

2.2 Combustion method and parameters

The *combustion method* should be reported as it influences the ash characteristic. For example, [8] found that the most important factor influencing the ash characteristics was the combustion method in a study on the characterization of wood ashes of different types (bottom ashes, fly ashes, and mixed bottom and fly ashes) from different plants and chemometric modeling (multivariate model Partial Least Square). The combustion method was found similarly important as the ash type [8].

A part of solid fuels is inorganic, and this ash-forming matter varies strongly among different *fuel types* [2]. The ash composition depends largely on the type of fuel, but also within the fuel type categories, there is a strong dependency between the ash characteristics and the exact fuel. Examples are that for wood ash characteristics it is important which parts of the tree are included in the fuel, as e.g. [9] reported that was Pb mainly stored in the stem of the investigated trees, whereas Zn and Ni, and to a lesser extent Cd and Cu, were translocated to aerial parts of the trees for a forest stand in North-eastern France. The characteristics of sewage sludge ash strongly depend on the chemicals that were used to precipitate phosphorous at the wastewater treatment plant (typically Fe and/or Al-based chemicals) [10].

The *combustion temperature* is essential to the ash mineralogy. For example, a combustion temperature of 1000 - 1200 °C, as in grate-fired boilers, often results in the formation of calcium silicates in wood ashes whereas the low temperature in the fluidized bed combustion (850 °C) does not [11]. Also, the *flue gas temperature* influences the characteristics of fly ash particles. With higher flue gas temperatures, ash particles are softer/contain a higher fraction of melt [2].

Other information on the actual plant can widen the possibility of extracting and comparing findings across different publications such as geographical information, the amount of ash produced every year, and fuel capacity (t/day) for the specific combustion facility.

2.3 Aging of ashes

Ashes are chemically unstable, and the characteristics can be influenced during e.g. storage. For example, under moist conditions and ambient air wood ash undergo hydration and carbonation, i.e. using Ca salts as an example hydration $CaO + H_2O \rightarrow Ca(OH)_2$ and carbonation ($Ca(OH)_2 + CO_2 \rightarrow CaCO_3 + H_2O$) [12]. Similarly, MSWI APC residues carbonates [13]. Other reactions can also take place such as the formation of gypsum and ettringite was reported during the hydration of wood fly ash [3]. In accordance with this, it is important to report if the ash is sampled directly after combustion or after aging. It should also be reported how the ash is stored after sampling in order for the reader to evaluate if ash mineralogy may have changed (which is not the case if it is stored dry and in a closed container).

2.4 Summary of important information to be reported

Based on the considerations above, we suggest that the following is reported together with experimental results when dealing with ashes:

- Group of ash (e.g. wood ash, sewage sludge ash)
- Type of ash (bottom ash, fly ash, or APC)
- Type of combustion plant
- Fuel type
- Temperature range during combustion (and flue gas temperature when available)
- Filter type (fly ash and APC residue)
- Possible aging
- Storing conditions in the lab
- Information on the specific plant if allowed.

3 FULL CHARACTERIZATION SCHEME

The full characterization scheme decided on in the AshCycle project is presented in Table 1. The methods in the categories (Table 1) are briefly described in in the following paragraphs: ash sampling (3.1), chemical composition (3.2), chemical properties (3.3), and physical properties (3.4).

Table 1: Overview of procedures in the ash characterization scheme.

Category	Characteristic	Method	Standard
Sampling	Sampling protocol guidelines Homogenization/reduction of sample size		EN 197-7 + 932-1
Chemical composition	Concentration of major elements	X-ray fluorescence (XRF)	
	Concentration of heavy metals and REEs	Acid digestion and ICP-OES	
	Mineralogy and amorphous phase	X-ray powder diffraction (XRD)	
	Organic and carbonate content	LoI at 550 °C and 950 °C	EN 15935:2021
	Total organic content (TOC)	TOC analyzer	EN 15936:2022
Chemical properties	Water content, pH, conductivity, leaching, solubility	Measured in a suspension	
	Reactivity	Isothermal calorimetry	ASTM C1897-20
	Reactivity - Bound Water	Paste and weight loss at 350 °C	
	Free CaO content	Chemical extraction, titration	ASTM C1897-20
	Alkali dissolution	Dissolution in 4M NaOH, ICP-OES	

Physical properties	Bulk density	Powder volume and weight	EN 1097-3:1998
	Density	Pycnometer	EN 1097-6
	Particle size distribution (>250 µm)	Sieving	EN 933-1:2012
	Particle size distribution (<250 µm)	Laser diffractometer	ISO 13320, 14487, 9276
	Specific surface area of solids	Gas adsorption – BET method	ISO 19277

The characterization scheme includes methods that enable the evaluation of possible valuable elemental resources (nutrients, metals, or REEs) and mineralogy/chemical compounds, which are beneficial when used as raw materials in the production of construction materials. For the evaluation of the possible recycling in construction materials, it is also important to know the chemical properties not at least the reactivity and the physical properties.

Many of the selected methods follow standards, and the specific standards are given in Table 1. Procedures for the methods, which do not follow a standard, are available at https://data.dtu.dk/

3.1 Representative sample and sampling protocol

To obtain a bulk sample that is representative of the average properties of the batch the procedure for homogenization/reduction of sample size in EN 197-7 + 932-1 should be followed.

Sampling data should be documented in a sampling protocol:

- 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. and if possible add a photo of the place of the sampling

See the AshCycle sampling report template at https://data.dtu.dk/

3.2 Chemical composition

Concentrations of chemical elements. Two methods are used for the measurement of concentrations of chemical elements: (I) X-ray Fluorescence (XRF) is an 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 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). (II) Acid digestion and ICP where the ash sample is in an acid solution of (2:2:1:1) H2O-HF-HClO4-HNO3 and the concentrations are measured at ICP-OES. The two methods are used for measuring concentrations of macro- and microelements, respectively.

Mineralogy and amorphous phase. 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.

The organic and carbonate content. Sequential LOI at 550 °C and 950 °C (EN15935) 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.

T*otal organic carbon (TOC).* TOC consists of both volatile carbonaceous matter and unburned carbon. It is relevant because it not only reflects the combustion efficiency, but high TOC also leads to adverse impacts on ash recycling. EN 15936 specifies two methods for the determination of TOC in different environmental solid matrices, an indirect procedure and a direct procedure. We have selected the direct procedure in which total inorganic carbon (TIC) is removed from the sample by acid.

3.3 Chemical properties

Water content, pH, conductivity, leaching, and water solubility is a procedure that compiles different measurements into one sequence: The water content is measured as the free water, i.e., the water that evaporates at 105 °C. Dried ash is suspended in distilled water at a liquid-to-solid (L:S) ratio of 1:10 (chosen from the leaching test EN 12457-2). After filtration, the pH and conductivity are measured with electrodes, and the concentration of relevant chemical elements is measured with ICP. The dry mass of the ash that was not dissolved is measured to calculate the soluble fraction.

Chemical reactivity. Isothermal calorimetry is used to determine the heat of hydration of hydrating pastes composed of 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 (ASTM C1897-20), and we suggest using it for these alternative ashes.

Bound water. Chemically bound water of pastes composed of the SCM, calcium hydroxide, calcium carbonate, potassium sulfate, and potassium hydroxide is determined as a measure of the chemical reactivity of the SCM.

Calcium oxide content. Free CaO content is measured via titration as described in EN 451-1.

Alkaline dissolution. In this method, the amount of soluble aluminium (Al) and silicates (Si) in the alkaline environment is quantified.

3.4 Physical properties

The bulk density (fly ash) 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 (EN 1097-3) covers the measurement of powders and non-cohesive materials. For the AshCycle project, the bulk density for fly ashes is measured in a funnel/cup system and using a pycnometer for the bottom ashes.

Density (bottom ash) is determined using method EN 1097-6 based on a Pycnometer

Particle size distribution. Measured with laser diffractometer for particles <250µm (according to EN 1097-3:1998) and for particles >250µm by sieving (EN 1097-6).

Specific surface area. The method is based on gas adsorption and is based on the standard ISO 9277. The principle is to cover the studied material surface with 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 a specific surface area by multiplying with the molecular cross-sectional area.

4 RECYCLING OPTIONS EVALUATED ON BASIS OF CHARACTERIZATION

A goal of the AshCycle project is to be able to evaluate the recycling options for a specific ash based on the characterization. A sub-goal is to develop a systematic approach that can support a general scaling of the use of ashes in construction materials. When scaled, it will not be possible to go through a time-consuming test program with lab productions of the construction materials and subsequent measurements of the properties with every new batch of ash with changed characteristics. It will be necessary to evaluate the use of the specific ash based on its characteristics.

During the AshCycle project, the recycling options, which are investigated experimentally, are recovery of elemental resources or use in construction materials: fired clay bricks, rammed earth blocks, concrete, or alkali activated materials. From the experiences gained with the experimental production of the construction materials, a methodology based on ash characterization as drafted in Figure 1 can be used to choose good recycling options.

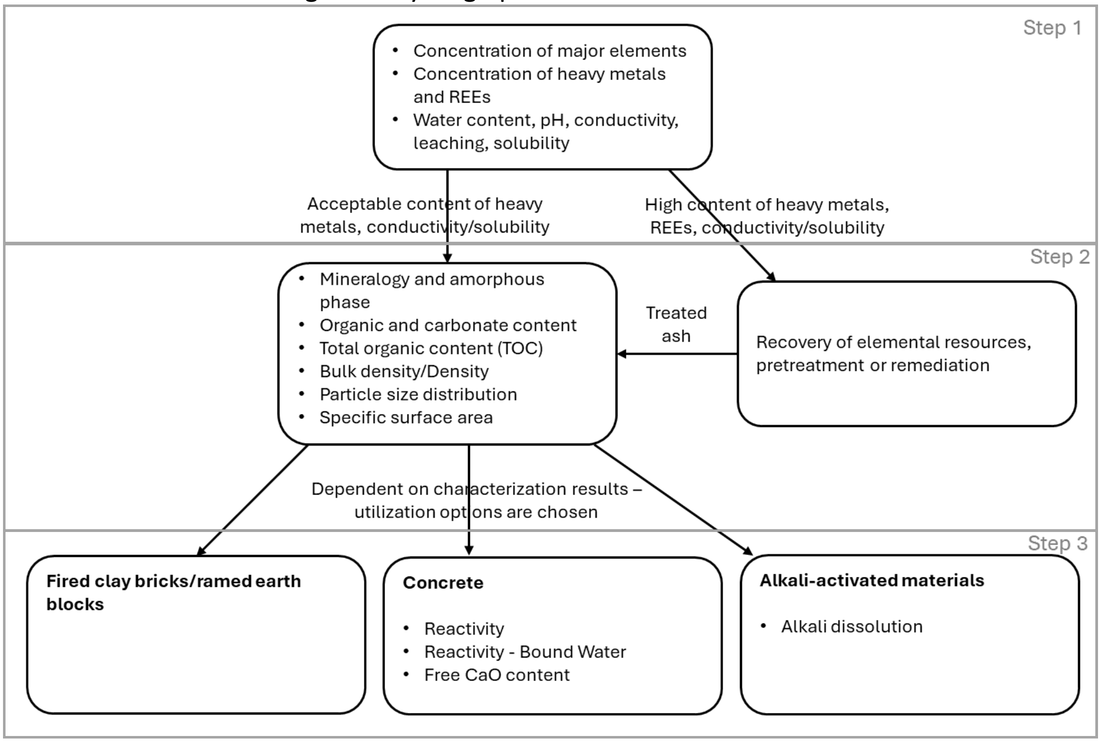

Figure 1: Ash characterization methods used a three-step evaluation on their possible beneficial use in construction materials.

In the first step, it is evaluated based on the chemical composition of the ash has content of valuable chemical elements, which should be recovered for recycling. Also, it should be evaluated if the content of heavy metals is too high for environmentally sound use in construction materials, so treatment is needed (remediation or recovery). What is a too high content will depend on national limiting values, however, such limits are not yet available, because the use of these ashes in construction materials has yet not been standardized or implemented at a larger scale. In the project, we will add to the background knowledge needed to set limiting values as we are measuring leaching from the construction materials produced. A high content of soluble salts is unwanted in all the tested construction materials. Thus, if the conductivity and solubility point to this, pretreatment of the ash before use in construction materials is probably needed, i.e., recovery or removal of these salts.

In the second step, a more thorough characterization of the ash is performed on the raw or treated ash. This characterization supports the evaluation of which construction materials the ash may be suitable for. Finally, in the third step, there are specific additional characterization methods (for concrete and alkali activated materials) to be performed. These are to be used in e.g. determining the possible concrete class and in choosing the mix design.

5. CONCLUSIONS

To enable the recycling of different ashes as resources in construction materials it is vital to be able to evaluate the possible use (or need for pretreatment before the use) based on the ash characteristics. The characteristics of a specific ash depend on the fuel and the combustion process/parameters. The ash characteristics vary hugely, even within each group of ashes (e.g. wood ashes or MSWI ashes). This is a challenge when aiming at recycling the ashes in construction materials, because the characteristics strongly influences the properties of the construction material.

It is necessary to develop a systematic approach for choosing the recycling option for a specific ash based on the characteristics, and further also to specify the use of the ash (e.g. make a proper mix design) based on the characteristics. A systematic approach is necessary to scale the use of the ashes in construction materials. When scaled, it will not be possible to go through a test program with lab productions of the construction materials and subsequent measurements of the properties with every new batch of ash with changed characteristics, such as it is carried out in the scientific literature.

To be able to link directly the ash characteristics to construction material properties a large amount of data must be compiled and evaluated e.g. using AI. In such a task it is beneficial if the same ash characterization scheme is used in the experimental work done to eliminate the influence on the result from the different methods used. This paper suggests such a systematic characterization scheme.

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