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Recycling of Biomass Bottom Ash from Fluidized Bed Combustion in Portland Cement Manufacturing: A Case Study Promoting the Circular Economy

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Abstract

Biomass bottom ash (BBA) constitutes about 20% of the by-products created during 100% biomass combustion in power and combined heat and power plants, with thus far limited utilization being reported. Our research is a case study from Poland examining the possibility of using BBA as a corrective additive to raw meal for the purpose of Portland clinker cement (PCC) production. BBA consists of mostly SiO_2 (up to 86% mass). It has low alkalis content (on average 0.68% of Na_2O and 2.68% of K_2O) and low radioactivity indices, rendering it a safe material with no emission hazard. Morphological analysis confirmed that quartz grains in BBA, due to heating and cooling in a fluidized bed, underwent polymorphic transitioning twice, leading to multiple grain defects, which resulted in improved grindability and reactivity of BBA compared to natural quartz sand. A real-time industrial test was conducted for 5 weeks in one of Poland's cement plants. BBA was fed to the rotary kiln (in proportion of 7% additive) as a substitution for natural sand (silica-bearing corrective additive) during the PCC manufacturing process, without any ash pretreatment or valorization being necessary. It was found that the addition of BBA to raw meal caused improvement of its sinterability (7% increase in clinkerization index). Results of this case study implement circular economy principles by permanently bounding a troublesome waste, i.e., BBA, into the structure of PCC without compromising the quality of the resultant cement (according to EN 197-1).

Keywords Portland cement clinker (PCC), Biomass bottom ash, Raw material, Circular economy, Silica carrier, Corrective additive, Bubbling fluidized bed (BFB)

1 Introduction

Portland cement clinker (PCC) is the primary constituent of widely produced cements, commonly referred to as Ordinary Portland Cement (OPC). These cements are some of the most manufactured industrial products globally. Since 1995, global production of PCC has tripled, reaching 4 billion Mg in 2022 (Cembureau, 2024). The cement industry accounts for approximately 7–8% of global CO_2 emissions (Andrew, 2018; Barbhuiya et al., 2024; Lehne & Preston, 2018; Pales & Leung, 2018), making it the second-largest source of anthropogenic CO_2 emissions, following only the energy sector. As the global population grows, urbanizes, and as emerging economies rapidly develop infrastructure, the demand for cement continues to rise. By 2050, the global building floor area is projected to reach 415.5 billion m^2, compared to 223.4 billion m^2 in 2015. This growth in infrastructure will

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inevitably lead to further increases in CO_2 emissions (Dean et al., 2016).

The production of 1 ton of PCC results in the emission of approximately 1 ton of CO_2. The majority of CO_2 emissions during cement production (approximately 60%) are attributed to process emissions, which are unavoidable and result from the calcination of limestone to produce oxides (CaO) and CO_2. The remaining 40% of CO_2 emissions results from the combustion of fossil fuels to heat raw materials in a rotary kiln at temperatures around 1450 °C to form clinker (Cavalett et al., 2024). The significant environmental cost of CO_2 emissions and the high energy consumption involved in the manufacturing process have driven the cement industry to explore more sustainable and cost-effective production methods. Various strategies have been proposed to reduce CO_2 emissions from cement production (Fennell et al., 2021; Uliasz-Bocheńczyk & Mokrzycki, 2022; Zhaurova et al., 2021), with one of the most common approaches being the use of alternative fuels or the incorporation of various types of industrial waste at different stages of the cement production process. These substitutions not only reduce costs, including avoided emissions costs, but also contribute to a lower carbon footprint for the cement produced, while promoting circular economy principles.

Portland cement clinker is typically produced from a raw material mix containing the key oxides CaO, SiO_2, Al_2O_3, and Fe_2O_3. Natural deposits with the required composition for clinker production are rare, so it is generally necessary to blend multiple ingredients. Industrial wastes, such as granulated blast furnace slag, fly ash from coal combustion, and silica fume, depending on their chemical and physical properties, can be utilized as ingredients in the raw meal (Karim et al., 2014). While the type, quantity, and properties of industrial wastes and by-products that can be co-grinded with PCC to produce common cements are governed by the EN 197-1 (European Standard, 2011), EN 197-5 (European Standard, 2021a), and EN 197-6 (European Standard, 2021b) standards, raw materials used to produce PCC are not similarly regulated.

A variety of industrial wastes can be used in PCC production, as long as they do not contain excessive amounts of undesirable substances, such as Cl^-, Na_2O, K_2O, and P_2O_5, which could compromise the quality of the final product. These wastes often serve as by-products that supplement and optimize the deficiency of specific oxides in the raw mix (e.g., SiO_2, Al_2O_3, or Fe_2O_3) and sometimes function as mineralizers that facilitate the sintering process during firing in the rotary kiln. The use of industrial waste in Portland clinker production has been investigated in several studies. Chandler et al. (1997) suggested that MSWI fly ash could replace limestone and CaO-bearing materials, though this was not confirmed in practice. Lam et al. (2011) found that only MSWI bottom ash and sewage sludge ash were suitable up to 6% due to their chemical composition, but required pretreatment to reduce chlorides, salts, alkalis, and heavy metals. Li et al. (2016) and Kleib et al. (2021) also confirmed the potential use of MSWI bottom ash after treatment, noting possible issues like delayed setting time. Other studies explored alternative materials such as sediments from hydroelectric reservoirs (Faure et al., 2017), iron ore tailings (Luo et al., 2016), and construction and demolition waste (Kara et al., 2017), the latter effective at 3.9 wt%. Bădănoiu et al. (2022) and Xie and Xi (2002) demonstrated the viability of using soda-lime waste glass to adjust silica content in raw mixes for clinker production. Despite ongoing research, the use of biomass bottom ash (BBA) in Portland clinker production remains largely unexplored, especially at the industrial scale. Most studies have focused on biomass fly ash (BFA) (e.g., Buruberri et al., 2015; Modolo et al., 2013; Pedreño-Rojas et al., 2024), with BBA receiving limited attention. In our previous study (Gawlicki et al., 2018), we showed that BFA, which makes up ~ 80% of combustion residues in fluidized bed boilers, is unsuitable for clinker production due to high alkali, chlorine, and phosphorus levels, making it non-compliant with EN 197-1 (European Standard, 2011) and EN 206 (European Standard, 2021b) standards. In contrast, BBA, accounting for the remaining ~ 20%, has recently gained interest. While often landfilled, it contains significant sand content (Pels et al., 2005) and has seen limited use in road construction and landscaping. However, impurities like chlorides and metals still hinder its broader application. Schlupp et al. (2023b) demonstrated that properly treated BBA can be used as aggregate in mortar without adversely affecting cement hydration. Jensen et al. (2025) also broadly revised the potential of replacing BBA from CFB and BFB as a sand substitute in concrete and mortars, providing in-depth characteristics of these materials, particularly regarding the crucial issue of the durability properties of the final products, including ASR.

Despite these promising findings, the direct utilization of BBA as a raw material in Portland clinker production has not been widely explored, particularly in real-time industrial trials. This research gap arises from the complexities of conducting industrial-scale studies, which require extensive preparation, logistical coordination, and collaboration with cement plants to integrate their equipment for real-world testing.

The primary aim of this study is to address this research gap by evaluating the feasibility of using biomass bottom ash (BBA) as a corrective additive in the raw meal for Portland cement clinker (PCC) production. Specifically, this research focuses on BBA's potential to compensate for silicon dioxide (SiO_2) deficiencies in the raw mix during the cement production process. The study includes both laboratory analysis and industrial-scale trials, serving as a proof of concept in a live cement plant environment.

Prior to the industrial trials, BBA underwent comprehensive assessment, including chemical, physical, and mechanical property analyses, alongside an in-depth study of its phase composition. These evaluations aimed to determine BBA's suitability as a corrective additive in the raw meal. The impact of BBA substitution on the sinterability of the raw meal was also investigated by calculating the clinkerization index (CI), which provides insights into its behavior during the clinker formation process.

As a proof of concept, approximately 500 tons of BBA was introduced into the industrial clinker sintering process without any pretreatment or valorization. The resulting cement was evaluated for key performance indicators, such as compressive strength and initial setting time. This comprehensive approach aims to assess whether BBA can be effectively incorporated into the cement production process, contributing to a more sustainable and cost-effective method for producing cement with a lower environmental footprint.

2 Materials and Methods

2.1 Biomass Bottom Ash (BBA)—Origin and Sampling Protocol

The material under examination in our research comprised biomass bottom ash (BBA) originating from a combined heat and power (CHP) facility located in Poland. This is a commercial plant that uses bubbling fluidized bed (BFB) technology (at 850 °C) to combust a mix of wood pellets with an additional fuel source consisting of at least 20% agricultural biomass, such as rapeseed pomace, sunflower seeds, and straw. Prior to industrial tests, the biomass bottom ash had to be evaluated for its chemical composition and homogeneity, as this aspect may be the main obstacle to utilizing waste in cement technology. Six individual samples, each weighing 20 kg, were collected and delivered every 2 weeks for 3 months from the CHP facility, then analyzed in an accredited laboratory using the methods presented in Sect. 2.2 and summarized in Table 1. Moreover, an initial batch of biomass bottom ash (approximately 30 tons) was delivered to the cement plant prior to carrying out industrial tests to verify the absence of metallic impurities in the waste, which could potentially harm the rotary kiln installation. The purity of the initial batch of BBA with respect

Table 1 Methodology and methods summary

Methods summary		
Biomass Bottom Ash (BBA)—Laboratory analysis	Industrial test—Portland cement clinker (PCC) sintering	Resultant clinker (PCC) and cement examination
		
XRF SEM–EDS Granulometric analysis Loss of ignition Free CaO Reactive CaO Alkalis Natural radioactivity density using pyknometer Bulk density (loose, compacted) Finess of the ash and its grindability Sinterability of raw material using clinkerization index (CI)	First reference period (RP-1)- sintering clinker with standard raw mix including quarts sand as corrective additive Proper industrial test (*T*)- sintering clinker with raw mix (tab) adjusted with 6.9% biomass bottom ash addition as a substitution of quarts sand Second reference period (RP-2), after proper industrial test- sintering clinker with standard raw mix including quarts sand For each trial- composition of raw meals was determined via XRF followed by, free CaO, alkalis, LOI, Cl^-. LSF, SM, AM	LSF, SM, AM, C_3S, C_2S, C_3A, C_4AF Finess, compressive strength and initial setting time for all obtained cements Leaching of heavy metals (Cr, Pb, As, Zn, V) from all obtained clinkers and cements (from all industrial periods RP-1, T and RP-2)

to metallic elements was accepted by the cement plant. Following this, a full tranche of waste, approximately 500 tons of BBA, was transported to the cement plant using self-unloading trucks. This tranche of biomass bottom ash was then fed into the industrial process of clinker sintering without any valorization or pretreatment.

2.2 Analytical Methods

Chemical composition of bulk BBA, resultant Portland clinker cement as well as final cement was determined via X-ray fluorescence, according to EN-196-2 (European Standard, 2013) using WD-XRF ZSX Primus II Rigaku Spectrometer and SQX Calculation program. Free CaO was determined according to EN 451-1 (European Standard, 2017), and reactive CaO was calculated using EN 197-1 (European Standard, 2011). Alkalis, SO_3 content chloride expressed as Cl^- and moreover LOI was determined according to EN 196-2 (European Standard, 2013).

Biomass bottom ash was also studied via SEM/EDS (energy dispersive X-ray spectroscopy) analysis using Quanta 200 FEG SEM to check the BBA for presence of other ingredients not included in previously mentioned analysis and to track morphology of the BBA grains. The mineral composition of the samples was determined using X-ray powder diffraction (XRD) with the Debye–Scherrer method. XRD patterns were recorded on a Rigaku SmartLab X-ray diffractometer under the following conditions: CuKα radiation, graphite monochromator, tube voltage of 45 kV, and tube current of 200 mA. Step scanning was performed with a step size of 0.050° 2θ and a counting time of 1 s per step. The samples were prepared using the'front loading'method. The interplanar spacing values obtained from the XRD patterns were used to identify the mineral phases present in the samples, using data from the International Centre for Diffraction Data (ICDD) and the XRAYAN software Density of BBA via pyknometer method. Bulk density—loose and compacted were—also analyzed.

Furthermore, natural radioactivity of bottom ash was performed via semiconductor gamma-ray spectroscopy, and activity concentration index (I) was calculated according to the following equations, based on European Commission requirements (1999):

$$I = \frac{C_{\mathrm{Ra}}}{300\mathrm{Bqkg}^{-1}} + \frac{C_{\mathrm{Th}}}{200\mathrm{Bqkg}^{-1}} + \frac{C_{\mathrm{k}}}{3000\mathrm{Bqkg}^{-1}}, \quad (1)$$

where C_{Ra}, C_{Th}, C_{K} are the activity concentration of radium, thorium and potassium in the building material.

Sinterability of raw material using clinkerization index (CI) according to the method developed by Blaise et al. (1971) was then calculated according to the equation:

$$\mathrm{CI} = \frac{600}{C_0 + 2C_1 + 2C_2 + 3C_3 + 4C_4 + 4C_5 + 2C_6}, \quad (2)$$

where C_0, C_1, C_2, C_3, C_4, C_5 and C_6 stand for the content of unbound—free CaO in appropriately prepared samples of raw meal roasted for 20 min at temperatures of 1000, 1100, 1200, 1300, 1350, 1400 and 1450 °C, respectively.

Phase composition of PCC (C_3S, C_2S, C_3A and C_4AF) as well as clinker compositional modules (lime saturation factor LSF, silicate modulus SM, alumina modulus AM) were calculated using Bogue equations (Lawrence, 2003).

Finally aqueous leaching of selected heavy metals (Cr, Pb, As, Zn and V) from PCC and resultant cements according to EN 12457-4 (European Standard, 2002) (L/S = 10, 1 dm^3, leaching time 24 h, filtered through 45 µm membrane, leachates analyzed via ICP-MS).

All of the above tests were performed by accredited laboratories, to ensure the quality of the obtained results. The methodological approach for our study is summarized in Table 1.

3 Results and Discussion

3.1 Physical, Chemical and Phase Characteristics of Bulk BBA

Variability in the chemical composition of biomass bottom ash (BBA) was the primary concern before conducting the industrial tests. Over a period of 2 months, samples were analyzed to assess the homogeneity of the waste and its suitability as a silicon-bearing corrective additive in Portland cement clinker production. The results of these analyses (shown in Table 2) confirm that the BBA can be considered a sufficiently homogeneous material for industrial application, with a high SiO_2 content ranging from 74 to 86% by mass, which makes it a suitable corrective silicon-bearing raw material for PCC production. The secondary constituents of BBA include CaO (ranging from 5.8 to 9.2%) and Al_2O_3 (from 3.3 to 6.2%). Other components are present at less than 5% per oxide. The alkali content in the BBA was also relatively low, with Na_2O ranging from 0.4 to 1.1% and K_2O from 2.4 to 2.9%. This should not be considered a limiting factor for BBA utilization in PCC production, as it will be introduced into the raw meal at a rate of approximately 7%.

The differences in the chemical composition of individual samples were deemed insignificant in terms of variability within the main oxides.

As depicted in Table 2, the BBA was predominantly composed of the following oxides: $SiO_2 > CaO > Al_2O_3 > K_2O > Fe_2O_3$. The obtained results are generally consistent with those reported by other authors (Cabrera et al., 2021; Carević et al., 2019; Tarelho

Table 2 The chemical composition of biomass bottom ash

Parameter (% mass)	BBA1	BBA2	BBA3	BBA4	BBA5	BBA6	Average $n=6$
LOI	1.54	1.60	2.74	3.04	1.70	1.87	2.08
SiO_2	85.76	79.02	73.85	79.84	80.95	77.94	80.56
Al_2O_3	3.31	6.17	6.17	3.64	4.71	4.26	4.71
Fe_2O_3	1.4	2.03	3.04	1.72	2.17	2.44	2.13
CaO	5.82	6.64	9.19	8.79	6.47	6.13	7.17
MgO	1.14	1.58	1.55	1.54	1.58	1.69	1.51
SO_3	0.56	0.28	0.28	0.27	0.24	0.45	0.35
Cl^-	0.04	0.05	0.04	0.02	0.02	0.05	0.04
Na_2O	0.41	0.73	1.11	0.51	0.77	0.56	0.68
K_2O	2.80	2.80	2.92	2.35	2.58	2.65	2.68

et al., 2015; Vassilev et al., 2010) studying wood combustion bottom ash. However, our BBA, derived from a fluidized bed boiler, contained a significantly higher SiO_2 content than reported in the aforementioned studies, exceeding 80% by mass. This is likely due to the use of sand as a bed material in the fluidized bed technology, which influences the composition of the BBA, resulting in a higher SiO_2 content (Jensen et al., 2025; Schlupp et al., 2023a, 2023b; Tarelho et al., 2015).

Moreover, the sum of oxides relevant for the pozzolanic reaction (SiO_2, Al_2O_3, and Fe_2O_3) reached an average content of 84.7%, which is higher than values reported by other authors (Carević et al., 2019; Lessard et al., 2017; Li et al., 2012; Schlupp et al., 2023a, 2024). BBA derived from other combustion technologies most likely does not meet the 70% threshold of pozzolanic oxides required by EN 450-1 (European Standard, 2012).

It is, however, worth noting that the high content of pozzolanic oxides in the studied BBAs does not directly translate into their pozzolanic reactivity since, as shown by the XRD analysis (Fig. 1A), BBA consists mostly of crystalline silica, with no amorphous silica detected by this method, owing it to being possibly inert. This observation aligns with other studies (Carević et al., 2019; Jensen et al., 2025; Sigvardsen et al., 2019). Therefore, further tests on the pozzolanic reactivity of BBA, e.g., Frattini test (Sigvardsen et al., 2021) should be conducted to draw definitive conclusions on this matter.

Also phase analysis (depicted in Fig. 1A) confirmed that the quartz is the main constituent of BBA. The remaining phases found in ash were illite, arcanite, sylvite, goethite, anhydrite as well as calcium aluminum oxide.

Further SEM–EDS analysis (depicted in Fig. 1B) showed that the surface layer of BBA grains contains substantial amounts of calcium (Ca) and potassium (K), likely a result of biomass combustion. This finding aligns with the observations of other authors, who report that combustion at 850 °C causes the biomass fuel to melt and agglomerate on the surface of bottom sand grains, forming K and Ca coatings on bed material particles (Anicic et al., 2018; Gatternig & Jürgen, 2015; Jensen et al., 2025).

The loss on ignition (LOI) in the studied BBA ranged from 1.54 to 3.04%, which also meets the requirements of EN 450-1 (European Standard, 2012). The amount of unburned particles (LOI) in our BBA is higher than in bottom ash from coal combustion but is comparable to BBA from fluidized bed combustion, which ranges from 1.27 to 2.08%, as reported by Modolo et al. (2013) or 1.6–2.56% as reviewed by Jensen et al. (2025).

Since the BBA was intended as a raw material for building materials, it underwent regulatory control as per building material standards. The key physical parameters of BBA, with respect to its prospective end use, are shown in Table 3.

The radionuclide activity index (I), a dimensionless parameter that combines the specific activities of Ra-226, Th-232, and K-40 normalized to their respective reference levels, was determined to be 0.76 ± 0.05 for the BBA. This value does not exceed the threshold of $I \leq 1$, classifying the ash as a safe raw material for use in buildings intended for human habitation or livestock, in accordance with Directive, 2013/59/EURATOM (2014). The concentrations of radionuclides (except for potassium) in the BBA were significantly lower than those reported by the European Commission (1999) as typical activity concentrations in coal fly ash, which are 650 Bq/kg for 40 K, and 100 Bq/kg for 232Th and 226Ra. The overall activity concentration of fly ash from coal combustion, typically observed in member states, exceeds the safe level of 1, reaching 1.31. It was also found that approximately 94% of the BBA mass was composed of particles between 0.5 and 5 mm. BBA is a fine-grained material, primarily dominated by fractions of 0.5–1 mm (~ 45%) and 1–2 mm

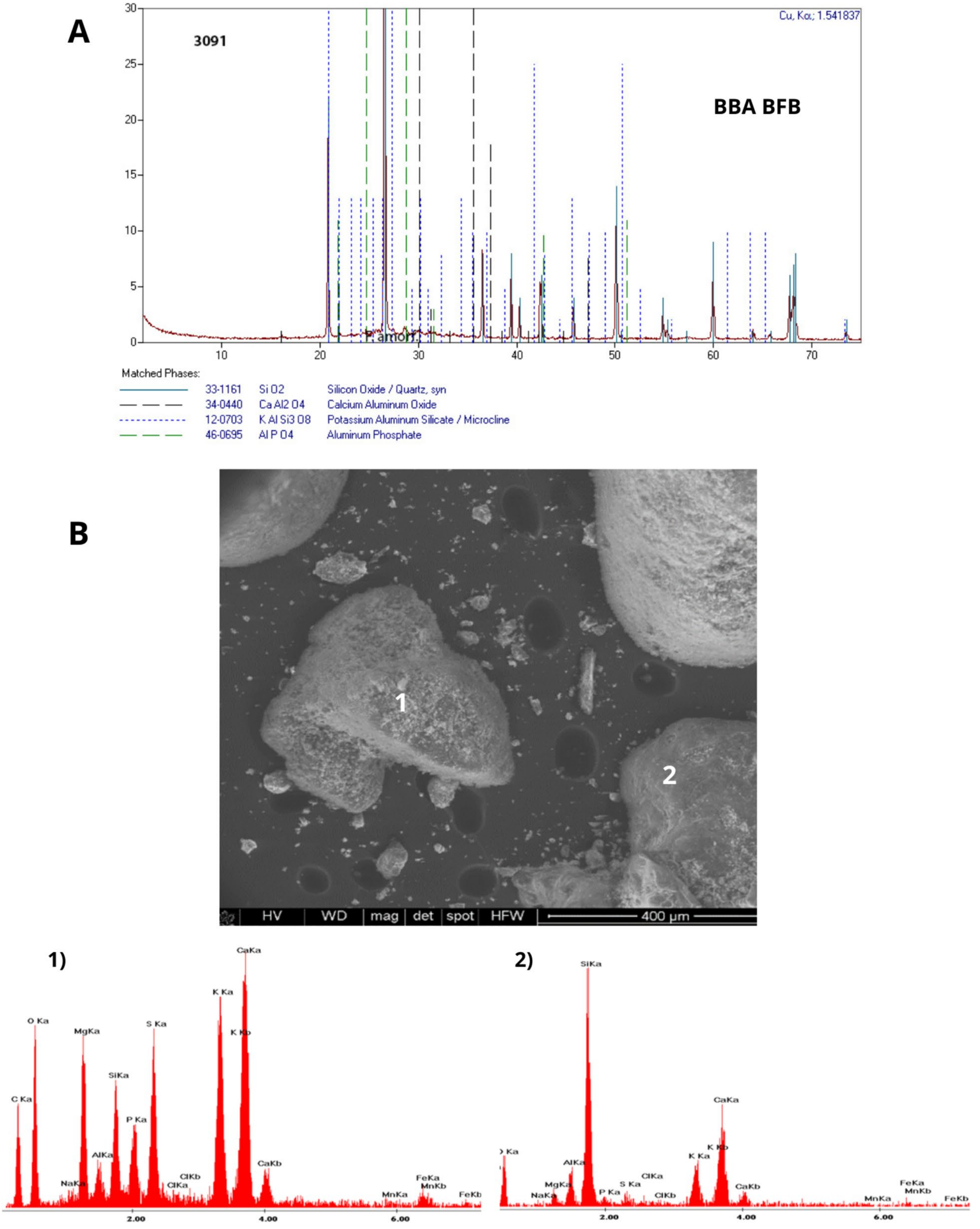


Fig. 1 Phase composition analysis of BBA using XRD (**A**) and SEM–EDS (**B**)

Table 3 Key physical parameters of BBA

BBA granulometry		**Threshold value**	**Quartz sand—reference data**
Grain size (mm)	**(% mass)**		
> 5	2.9	"Rule of thumb": quartz > 63 µm < 2%; quartz > 45 µm < 2.5%	In the opinion of cement materials specialists, the content in the raw material mix fired into Portland clinker should be minimized. This applies especially to large grains (Lafarge Canada Inc 2002).
5–2	16.7		
2–1	32.4		
1–0.5	44.8		
< 0.5	3.2		
Bulk density (kg/m^3)			
Loose	Compacted		
1456	1510	~ 1440 to 1500 (loose) ~ 1600 to 1700 (compacted)	Neville (2011)
Particle density (g/cm^3)	2.63	2.65	Deer et al. (1992)
Radionuclei activity concentration (Bq/kg)			
^{40}K	1982.13 ± 110.00		Directive 2013/59/EURATOM (2014)
^{226}Ra	18.61 ± 3.30		
^{232}Th	8.98 ± 1.91		
I (dimensionless quantity)	0.76 ± 0.05	≤ 1	

(~ 32%). The particle size distribution of fluidized bed ash does not differ significantly from the bed material used in the power plant; however, combustion generally increases the proportion of fine grains in the BBA compared to the boiler sand before combustion (Schlupp et al., 2024).

The particle density of the bottom ash, excluding pores, measured by the pycnometric method, was 2.63 g/cm^3, which is consistent with the density of natural quartz sand. These findings align with those of other authors, who reported the density of BBA from wood biomass combusted in fluidized bed boilers to be between 2.28 g/cm^3 (Schlupp et al., 2023a) and 2.67 g/cm^3 (Modolo et al., 2013).

Furthermore, the bulk density of the waste, which is an effective indicator of a material's handling characteristics, showed that the loose bulk density (mass per unit volume, including air between particles) of BBA was 1456 kg/m^3, while its compacted bulk density was 1510 kg/m^3.

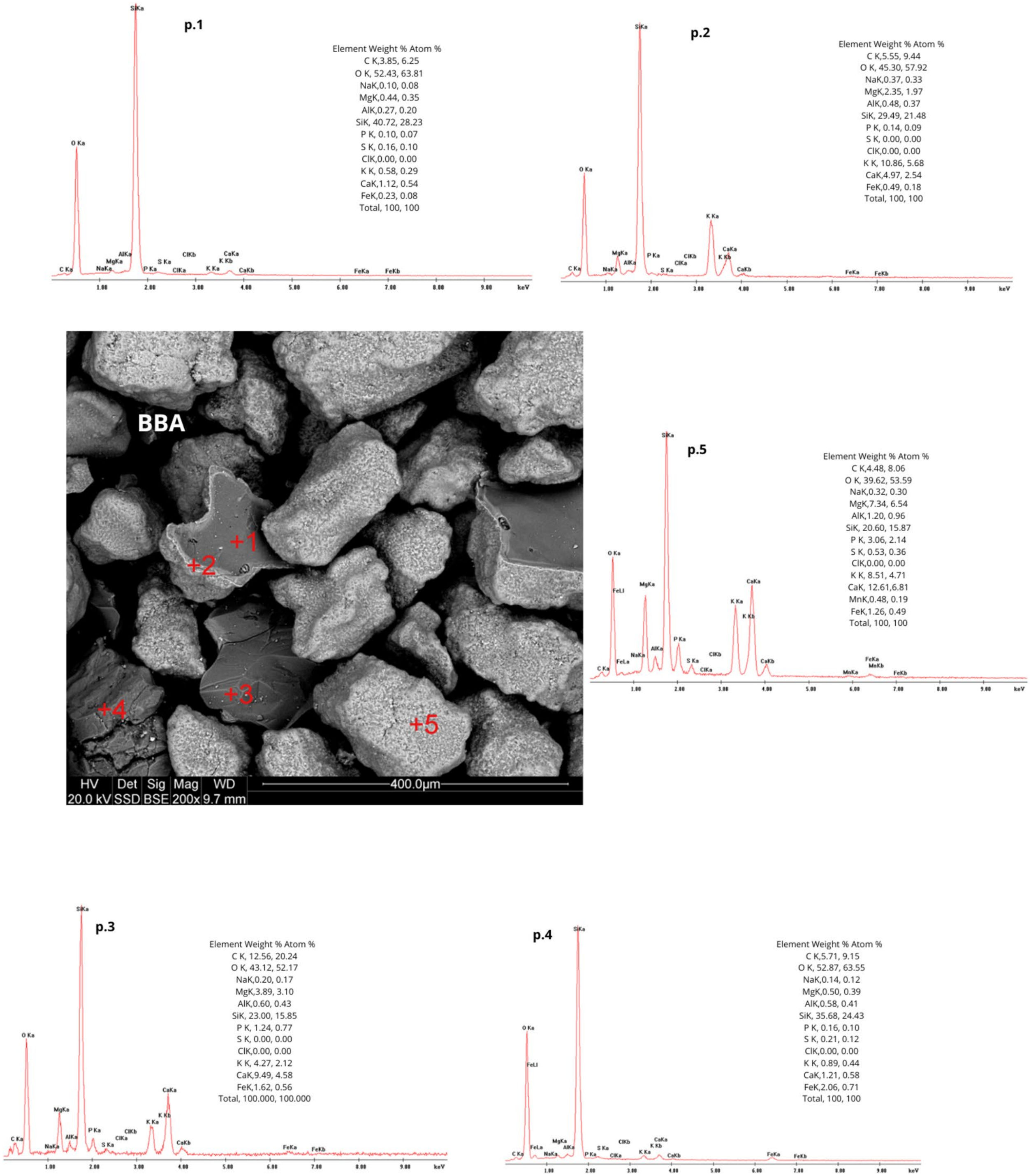
3.2 Morphology and Grindability of BBA

In addition to particle size, chemical composition, and phase composition, the crystalline form of silica in the raw materials is a critical parameter influencing the sinterability of the raw clinker meal. To assess the impact of SiO_2 transformations on the surface of ash grains, scanning electron microscope (SEM) observations were conducted on several BBA samples. Selected SEM images from grain morphology analysis are presented in Fig. 2.

Generally, the BBA grains were found to be irregular in shape and size, with defective, angular surfaces with a sticky coating of calcium and potassium as depicted in Fig. 1B. BBA is a material that has undergone prolonged exposure to temperatures of 850 °C before being cooled to ambient temperature. Heating causes the quartz grains, which make up approximately 85% of the BBA mass, to undergo polymorphic transformations twice: first from α-quartz to β-quartz at 573 °C, and then, after removal from the fluidized bed boiler and subsequent cooling, a reverse transformation from β-quartz to α-quartz. This phenomenon of quartz inversion is well known and described (Fraser 1986). As a result, the structure of BBA is less dense and more prone to cracking compared to more stable natural α-quartz sand. These transformations can increase the number of structural and macro-defects visible on the surfaces of BBA grains, thereby influencing its grindability and the reactivity of SiO_2 during the sintering process and Portland cement clinker (PCC) formation. These defects are clearly visible in Fig. 2. Literature, such as Schlupp et al., (2023a, 2023b) and Jensen et al., (2025) also reports that the surface of boiler sand grains is modified by combustion temperatures in fluidized bed boilers, with multiple cracks appearing on the surface of BBA grains.

Since BBA was considered as an alternative to natural quartz sand in the raw meal, to evaluate grindability of waste, we focused on comparing the specific surface area of BBA with natural quartz sand. The specific surface area was determined using the Blaine method after grinding the materials in a laboratory ball mill for predetermined periods (5 kg feed, grinding times of 5, 10, 15, 20, and 25 min). The results are presented in Table 4.

Our results confirmed that the grindability index of biomass bottom ash (BBA) has improved to that of



p.1

Element Weight % Atom %
C K,3.85, 6.25
O K, 52.43, 63.81
NaK,0.10, 0.08
MgK,0.44, 0.35
AlK,0.27, 0.20
SiK, 40.72, 28.23
P K, 0.10, 0.07
S K, 0.16, 0.10
ClK,0.00, 0.00
K K, 0.58, 0.29
CaK,1.12, 0.54
FeK,0.23, 0.08
Total, 100, 100

p.2

Element Weight % Atom %
C K,5.55, 9.44
O K, 45.30, 57.92
NaK,0.37, 0.33
MgK,2.35, 1.97
AlK,0.48, 0.37
SiK, 29.49, 21.48
P K, 0.14, 0.09
S K, 0.00, 0.00
ClK,0.00, 0.00
K K, 10.86, 5.68
CaK,4.97, 2.54
FeK,0.49, 0.18
Total, 100, 100

p.5

Element Weight % Atom %
C K,4.48, 8.06
O K, 39.62, 53.59
NaK,0.32, 0.30
MgK,7.34, 6.54
AlK,1.20, 0.96
SiK, 20.60, 15.87
P K, 3.06, 2.14
S K, 0.53, 0.36
ClK,0.00, 0.00
K K, 8.51, 4.71
CaK, 12.61,6.81
MnK,0.48, 0.19
FeK,1.26, 0.49
Total, 100, 100

p.3

Element Weight % Atom %
C K, 12.56, 20.24
O K, 43.12, 52.17
NaK,0.20, 0.17
MgK,3.89, 3.10
AlK,0.60, 0.43
SiK, 23.00, 15.85
P K, 1.24, 0.77
S K, 0.00, 0.00
ClK,0.00, 0.00
K K, 4.27, 2.12
CaK,9.49, 4.58
FeK,1.62, 0.56
Total, 100.000, 100.000

p.4

Element Weight % Atom %
C K,5.71, 9.15
O K, 52.87, 63.55
NaK,0.14, 0.12
MgK,0.50, 0.39
AlK,0.58, 0.41
SiK, 35.68, 24.43
P K, 0.16, 0.10
S K, 0.21, 0.12
ClK,0.00, 0.00
K K, 0.89, 0.44
CaK,1.21, 0.58
FeK,2.06, 0.71
Total, 100, 100

Fig. 2 SEM images of SiO_2 grain morphology of BBA

natural quartz sand. This, in turn, can positively impact heat consumption in the rotary kiln during clinker production, thereby enhancing kiln efficiency.

This finding is particularly important because the particle size distribution and homogeneity of the raw materials used in Portland cement clinker (PCC) production are known to influence the formation of mineral phases (Anicic et al., 2018). Inconsistent or undesirable mineral phase formation can occur if the raw material is not properly ground. Therefore, the better grindability of

Table 4 Comparison of BBA fineness with standard quartz sand

Grinding time (min)	Specific surface area (cm^2/g)	
	BBA	Quarts sand
0	720	600
5	1270	1080
10	1730	1610
15	2310	2230
20	2910	2760
25	3260	3070

BBA compared to natural sand is highly advantageous. It is also worth mentioning that quartz, due to its crystalline structure and high melting point, is one of the most challenging minerals to process in raw meal. The use of quartz sand in cement plants presents challenges because of its high hardness (7 on the Mohs scale), making it difficult to grind. In contrast, the BBA derived from fluidized bed boilers, due to its structural defects mainly resulting from the double polymorphic transformation of quartz tends to be more prone to fragmentation. This characteristic enhances its grindability and increases its reactivity.

3.3 Sinterability of Raw Meals with BBA

To assess the effect of BBA substitution on the sinterability of the raw meal, the clinkerization index (CI) was calculated. A higher CI indicates better sinterability of the raw meal. According to this principle, our results show that the clinkerization index for the raw meal containing natural quartz sand was 2.6, while the raw meal containing BBA had a higher CI of 2.8.

As far as we are aware, no comparable studies have been conducted on the sinterability of raw meals with the addition of BBA from fluidized bed boilers, or any other BBA from different combustion technologies. Therefore, we are unable to provide direct comparisons with the findings of other authors. Based on our results, we can, however, conclude that replacing natural quartz sand with biomass bottom ash (BBA) increases the clinkerization index by more than 7%.

This improved sinterability (susceptibility to clinkerization), similar to the improved grindability, could positively influence heat consumption in the rotary kiln during clinker production, thereby improving the overall efficiency of the kiln.

3.4 Results of Industrial-Scale Testing of PCC Sintering in a Cement Plant

Industrial-scale testing of Portland cement clinker sintering with the addition of BBA was conducted in a rotary kiln operating with the dry process, with a capacity of approximately 900 tons of PCC per day. The standard raw meal used at the cement plant to produce PCC consisted of limestone from a nearby mine (the primary source of CaO), natural quartz sand (the main source of silica), calcareous fly ash (supplying Al_2O_3 and SiO_2), and steel slag (added primarily to achieve the required Fe_2O_3 content).

The composition of the raw meal during the entire testing period was continuously monitored by an automatic feed control system, which was equipped with an online X-ray fluorescence (XRF) analyzer. This system allowed for real-time corrections to be made to the surplus of individual raw materials as needed.

To evaluate the feasibility of using biomass bottom ash as a corrective additive in the raw meal and to gather sufficient data for drawing reliable conclusions, the experiment was divided into three phases:

1. First reference period (RP-1): the initial phase, prior to the industrial test, in which standard raw meal with natural quartz sand was used for clinker sintering (lasting 7 days).
2. Industrial test period (T): the phase during which bottom ash replaced quartz sand in the clinker (lasting 14 days).
3. Second reference period (RP-2): the phase following the industrial test, when the standard mix, including quartz sand, was reintroduced for clinker production (lasting 14 days).

Samples of raw meal, clinker, and cement were collected during each of the three periods for comparison purposes. These samples were analyzed in an accredited laboratory according to the methodology outlined in Table 1.

The industrial-scale testing, which involved the sintering of PCC, was conducted over a total of 5 weeks. Biomass bottom ash for the test period (T) was delivered in wet form using self-unloading dump trucks and was fed into the mill, completely replacing the natural sand while maintaining the original sand-to-raw meal ratio. An online XRF EDA analyzer (with 5-min analysis intervals of the raw meal) was continuously used to monitor and control the proportion of individual raw materials in the meal, allowing for automatic corrections whenever deviations from the setpoint were detected.

The chemical compositions of the raw materials used during the industrial-scale testing, as well as the final composition of the raw meal during each test period, are presented in Table 5.

During the test period (*T*), biomass bottom ash (BBA) was introduced into the raw meal, maintaining a ratio of 6.9% (Table 6), which allowed for the complete removal of

Table 5 Composition of raw materials and raw meals during industrial test

Raw materials

Content (%)	BBA	Quartz sand	Limestone	Calcareous fly ash	Steel slag
LOI	2.10	1.30	39.46	2.81	4.95
SiO_2	79.88	90.93	5.97	41.89	23.48
Al_2O_3	4.80	2.29	1.32	21.43	5.51
Fe_2O_3	2.07	0.61	0.57	5.01	17.81
CaO	7.38	2.67	49.24	19.21	33.01
MgO	1.48	0.40	0.69	1.33	6.46
SO_3	0.33	0.20	0.26	1.73	0.37
Na_2O	0.71	0.14	0.14	0.20	0.22
K_2O	2.69	1.16	0.32	0.22	0.43

Raw meal composition during industrial test (% mass)

Industrial test periods	BBA	Quartz sand	Limestone	Calcareous fly ash	Steel slag
RP-1	–	3.2	84.5	5.4	6.9
T	6.9	–	81.0	5.7	6.4
RP-2	–	6.3	78.2	8.3	6.8

quartz sand from the meal. This substitution also resulted in a reduction in the amount of limestone required compared to the first reference period (RP-1), during which only natural quartz sand was used. In the final test period (RP-2), natural quartz sand was reintroduced to correct the raw meal, maintaining a ratio of 6%. During this period, we observed an even greater reduction in the amount of limestone (78.5%) required. However, this reduction cannot be attributed to the sand correction but rather is due to the higher calcium carbonate content of the limestone delivered from the quarry for the purposes of the testing.

The composition of the raw meals, resulting PCCs, module values, and phase content during the subsequent industrial test periods are presented in Table 6.

The results revealed that the raw meal containing BBA showed an increase in lime saturation factor (LSF) compared to both reference periods. Additionally, slightly lower residue amounts were observed on the 0.09 mm and 0.2 mm sieves in this meal, further confirming the improved grindability of BBA compared to natural quartz sand.

There was no significant difference in the chemical composition of the Portland cement clinker (PCC) produced from raw meal containing natural quartz sand (RP-1 and RP-2) compared to that produced from raw meal containing the BBA additive (*T*).

The content of free lime, a key indicator of the sinterability of raw meal, did not change significantly throughout the entire industrial testing period, confirming the enhanced sinterability of raw meal containing BBA. This was further corroborated by the fact that no significant changes were observed in the silicon and aluminum modules (see Table 6).

Table 7 presents the basic performance characteristics of Portland cements (CEM I 42.5 N) produced from the PCC generated during the industrial test, in accordance with EN 197-1 (European Standard, 2011). To produce the Portland cements (OPCs), the PCC samples were ground in a ball mill with 4% gypsum and 4% limestone to a specific surface area of 3200 cm^2/g, as determined by the Blaine method. The three resultant Portland cements (CEM RP-1, CEM T, and CEM RP-2) were then tested for their basic performance characteristics, including compressive strength after 2 and 28 days, initial setting time, and soundness. Compressive strength was measured on cement mortar specimens prepared according to EN 196-1. For each cement type and curing time, three specimens were tested to calculate average strength and standard deviation.

The results of these analyses, presented in Table 7, showed no significant differences in the cements prepared from PCC with the addition of BBA compared to those manufactured from standard raw meal using quartz sand. The mechanical properties of all obtained cements (CEM RP-1, CEM T, and CEM RP-2) showed an initial setting time of more than 60 min (ranging from 150 to 165 min), early compressive strength after 2 days exceeding 10 MPa (ranging from 17.2 to 18.4 MPa), standard

Table 6 Composition of raw meals and resultant PCCs

	Raw meals composition (% mass)		
	RP-1	**T**	**RP-2**
Particles > 0.09 mm	16.5	15.1	15.8
Particles > 0.2 mm	3.7	2.5	3.2
LOI	33.2	33.4	33.3
SiO_2	13.7	13.7	14.0
Al_2O_3	3.24	3.14	3.14
Fe_2O_3	2.21	2.21	2.10
CaO	45.6	45.6	45.4
MgO	0.88	0.95	0.96
SO_3	0.62	0.57	0.54
Cl^-	0.20	0.16	0.16
Module value			
LSF	104.9	105.1	102.7
SM	2.5	2.6	2.7
AM	1.5	1.4	1.5
	PCC composition (% mass)		
	RP-1	**T**	**RP-2**
CaO	66.17	66.06	65.91
SiO_2	22.01	21.60	22.10
Al_2O_3	5.11	5.10	5.27
Fe_2O_3	3.48	3.47	3.44
MgO	2.20	2.44	2.47
SO_3	0.26	0.20	0.14
CaO_{free}	2.03	2.09	2.03
Na_2O	0.34	0.38	0.33
K_2O	0.18	0.33	0.28
Module value			
LSF	94.65	96.08	93.73
SM	2.56	2.52	2.54
AM	1.47	1.47	1.53
Phase content (%)			
C_3S	53.82	56.43	51.34
C_2S	22.50	19.37	24.64
C_3A	7.64	7.64	8.16
C_4AF	10.59	10.56	10.46

Table 7 Performance of CEM I prepared from PCC produced during the industrial test phase, according to EN 197-1

Cement type	Compressive strength (Mpa) ± SD*		Setting time (min) ± SD*		Soundness (expansion) (mm)
	After 2 days	After 28 days	Initial	Final	
CEM RP-1	17.5 ± 0.7	42.5 ± 1.1	165 ± 8	305 ± 15	0.0
CEM T	18.2 ± 0.8	44.4 ± 1.2	150 ± 9	285 ± 9	0.0
CEM RP-2	18.4 ± 0.7	45.7 ± 1.7	150 ± 8	260 ± 12	0.0

*Results presented as mean ± standard deviation ($n = 3$)

compressive strength after 28 days ranging from 42.5 to 45.7 MPa, and no sign of soundness (0 mm).

The analysis of the mechanical and physical properties confirmed that the tested cements met the standard requirements for Portland cement type CEM I 42.5 N as per EN 197-1 (European Standard, 2011). The substitution of quartz sand with BBA in the clinker sintering process did not negatively affect the properties of the resultant ordinary Portland cement.

The inclusion of industrial waste in the raw meal prompted us to conduct tests to assess the potential leaching of heavy metals from both the initial PCCs and the resulting cements. The results, presented in Table 8, show that metals such as Cr, Pb, As, Zn, and V were immobile and exhibited minimal leaching, with concentrations not exceeding 0.004 mg/dm^3. Additionally, the concentrations of Co, Ni, Cu, Cd, Tl, and Sb were all below the detection limit of the analytical method (ICP-MS), which was set at 0.00005 mg/dm^3.

The immobilization of heavy metals and arsenic within the clinker and cement matrix is a well-documented process, primarily resulting from their incorporation into stable crystalline phases such as calcium silicates and aluminates during the high-temperature clinkerization process (above 1450 °C). This chemical binding effectively reduces the leachability and environmental mobility of these potentially harmful elements. Numerous studies have confirmed this phenomenon, highlighting the effectiveness of cement matrices in stabilizing heavy metals and metalloids. According to Bobrowski et al. (1997), the immobilization of heavy metals in cement matrices occurs primarily through their incorporation into the crystalline structure of clinker minerals during the high-temperature clinkerization process. Heavy metals are chemically bound within the calcium silicate and aluminate phases, such as alite, belite, and calcium aluminate phases, which reduces their solubility and mobility. Additionally, the hydration products of cement, such as calcium silicate hydrate (C–S–H) and ettringite, can adsorb and physically encapsulate heavy metals, further stabilizing them within the hardened cement matrix. These mechanisms collectively contribute to the effective chemical immobilization and reduced leaching of heavy metals, making cement an effective material for the safe encapsulation of industrial wastes containing toxic metals.

Table 8 Leaching of heavy metals from the PCC and resultant cements

Material	Heavy metals concentration (mg/dm^3)				
	Cr	Pb	As	Zn	V
PCC RP-1	0.004	0.001	0.005	0.001	0.001
PCC T	0.003	0.001	0.004	0.001	0.001
PCC RP-2	0,003	0.002	0.005	0.002	0.002
CEM RP-1	0.004	0.001	0.003	0.001	0.001
CEM T	0.003	0.001	0.003	0.001	0.002
CEM RP-2	0.003	0.001	0.003	0.002	0.002

Our findings are consistent with this body of research and are further supported by leaching tests conducted as part of the present study, which demonstrate minimal release of heavy metals and arsenic from the produced cement.

The results obtained from this research extend beyond laboratory-scale studies and simply cannot be directly compared to existing literature, as no similar research has been conducted. These findings should be considered as proof of concept in a live production environment within a working cement plant, spanning a full 5-week period. The industrial-scale test fully confirms the feasibility of using BBA as a substitute for natural quartz sand, which is traditionally added to the raw meal in PCC manufacturing. The results demonstrate clearly that this substitution can be made without compromising the performance parameters of the resultant cements in any way (see Table 7).

4 Conclusions

Based on the findings of this industrial-scale case study, several key conclusions can be drawn regarding the feasibility, performance, and sustainability of using biomass bottom ash (BBA) as a silica-bearing additive in Portland cement clinker production:

1. Polymorphic transformations α to β-quartz at 573 °C and back upon cooling reduce structural stability of BBA, increasing grain defects, enhancing BBA's grindability.
2. The addition of approximately 7 wt% of biomass bottom ash (BBA) to the raw meal successfully replaced 100% of natural quartz sand as a silica source in clinker production.
3. This substitution:
    - Simplifies the production process (no chemical pretreatment or valorization required),
    - Improves grindability compared to natural quartz,
    - Increases the clinkerization index by 7%, indicating better sinterability.
4. The final cement product remained fully compliant with EN 197-1 standards, with no significant changes in phase composition or performance properties.

5. This case study demonstrates the potential of BBA as a sustainable raw material in the Portland cement clinker (PCC) production process, supporting circular economy practices.
6. Given the widespread use of biomass combustion in fluidized bed boilers across Europe, the findings have strong international applicability.
7. Limitations for commercial implementation include:
    - Stable and sufficient availability of BBA,
    - Consistent chemical composition and low impurity levels (especially metallic scraps contaminants which can vary significantly between installations) and limited heavy metals and As concentrations,
    - Economic factors such as price and transportation costs,
    - Adaptability to specific technological conditions of individual cement plants.

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Author contributions
Elżbieta Jarosz-Krzemińska: conceptualization, data curation, investigation, methodology, supervision, resources, validation, writing—original draft preparation, writing review and editing, visualization (figure and table preparation), supervision, funding acquisition. Marek Gawlicki: conceptualization, methodology, writing review, supervision.

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Data availability
All data generated or analyzed during this study are included in this published article.

Declarations

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