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Advances in Cement Research

Volatilisation of elements during clinker formation in a carbon dioxide atmosphere

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In the future, cement clinker formation is likely to take place in high temperatures and high carbon dioxide atmospheres in carbon-neutral production processes as part of, for example, electrified processes. The aim of this study was thus to compare the volatilisation of minor and trace elements during cement clinker formation in a high carbon dioxide atmosphere and a conventional combustion atmosphere. Raw meal samples were exposed, at high temperature, to the two different atmospheres, with elemental analysis performed before and after. For both atmospheres, the minor elements potassium and sulfur, and the trace elements rubidium, lead, thallium, caesium, cadmium and mercury were highly volatile. For most of the analysed elements, no difference was observed between the two atmospheres. However, volatilisation of potassium, sodium and sulfur was lower in the high carbon dioxide atmosphere. It is suggested that this should be further studied in relation to the molar ratio of sulfur to alkalis in the clinker and the effect on clinker quality.

Keywords: alkali compounds/clinkering/clinkering reactions/heavy metals/thermal behaviour

Introduction

Cement is one of the most used manufactured products in the world and, at present, no other material has the necessary quality and quantity to fully replace cement in concrete (Nwankwo *et al*., 2020). Portland cement clinker is produced by heating a carbonate-rich raw meal to around 1450°C, whereafter it is milled and mixed with additives to form the final cement product. The production process is energy intensive and the cement industry is associated with high carbon dioxide emissions (Andrew, 2019). Efforts to reduce the emissions per tonne of produced cement have already been made, for example through fuel switching and reduction of the clinker-to-cement ratio (Andrew, 2019; Benhelal *et al*., 2021; Cormos, 2022). However, to reach near-zero carbon dioxide emissions, both fuel emissions and process emissions need to be reduced. The latter originate from the thermal decomposition of the main raw material, limestone. Around 60% of carbon dioxide emissions are process emissions, while 40% are from the combustion of fuels (Schorcht *et al*., 2013). Carbon dioxide capture and subsequent storage or utilisation may be one solution to this problem as this reduces both fuel and process emissions to near zero (Bjerge and Brevik, 2014; Leilac, 2017, 2021; Tokheim *et al*., 2019; Wilhelmsson *et al*., 2018). To date, post-combustion capture is the solution that has attracted the most attention within the cement industry (Hills *et al*., 2016).

To facilitate carbon dioxide capture, the gas stream exiting the process should have a high share of carbon dioxide, preferably near 100% (MacDowell *et al*., 2010). Two ways to achieve a high concentration of carbon dioxide in the exit gas have been suggested: electrifying the production process and oxy-fuel combustion (Hökfors *et al*., 2015; Wilhelmsson and Backman, 2019). In an electrified process, the heat conventionally generated by the combustion of fuels is replaced by heat generated by electricity from fossil-free production, and the exit gas thus consists of nearly pure carbon dioxide originating from the thermal decomposition of limestone. Fuel emissions are eliminated, and thus the volume of flue gas to be captured is decreased (Wilhelmsson and Backman, 2019; Wilhelmsson *et al*., 2018). No operational industrial plant is yet fully electrified, but several studies have investigated the potential of electrical heating using different techniques, such as microwave heating, plasma heating and electrical gas heating (Buttress *et al*., 2015; Fang *et al*., 1996; Glasser, 1975;

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Kaewwichit *et al*., 2017; Long *et al*., 2002; Quéméneur *et al*., 1983; Wilhelmsson and Backman, 2019; Wilhelmsson *et al*., 2018). During oxy-fuel combustion, fuels are combusted using concentrated oxygen instead of air, significantly reducing the nitrogen input. The carbon dioxide concentration in the flue gas is expected to increase, for example from 33 vol.% to 76 vol.% (Hökfors *et al*., 2015), with the other main flue-gas components being oxygen and water. The potential of oxy-fuel combustion in cement clinker production has been investigated in several studies (Ditaranto and Bakken, 2019; ECRA, 2012; Hökfors *et al*., 2015; Zeman, 2009; Zheng *et al*., 2016). For both electrified and oxy-fuel systems, gas-tight production is essential to avoid dilution of the flue gas (Wilhelmsson *et al*., 2018).

To produce cement clinker, sources of calcium, silicon, aluminium and iron are needed. The main raw material for cement production is limestone, but other raw materials or additives (e.g. chalk, marl, clay, shale, sand, iron oxide and bauxite) are generally also needed. Industrial by-products such as blast-furnace slag, fly ash and sand are also used (Hewlett and Liška, 2019). The fuel used for the heating of the process contains ash-forming elements, and the ash contributes to the elemental composition of the cement clinker; in an electrified system the fuel is eliminated, and thus so too is the fuel ash. Since natural raw materials are utilised, minor and trace elements are included, and the origin of the raw material determines the amount and type of these elements (Bhatty, 1995).

Typically, in clinker, the elements present at concentrations <1 wt% are defined as minor elements (European Union, 2010; Gineys *et al*., 2011a), while elements at concentrations <0.02 wt% (Frandsen *et al*., 1994) or <0.01 wt% (Taylor, 1997) are defined as trace elements. In this study, major elements are defined as those present at ≥1.00 wt%, minor elements as $0.02\,\text{wt\%} \le x < 1.00\,\text{wt\%}$ and trace elements as <0.02 wt%.

During thermal processing, the minor and trace elements interact in complex ways with the main product phases of alite ($3CaO.SiO_2$ (C_3S)), belite ($2CaO.SiO_2$ (C_2S)), calcium aluminate ($3CaO.Al_2O_3$ (C_3A)), calcium ferrite ($4CaO.Al_2O_3.Fe_2O_3$ (C_4AF)), lime and periclase, influencing the formation of melt phases and solid solutions and the stability of crystalline products (Taylor, 1997). Some trace elements, such as lead and mercury, are known to volatilise at high temperatures (Bhatty, 1995; Frandsen *et al*., 1994), such as in the hot zone of the kiln. Some volatilised elements condense within the kiln system at lower temperatures, while others leave the kiln system and are, to a large extent, captured in the flue-gas cleaning equipment. Small amounts exit through the stack. The leaching properties of minor and trace elements that are retained in the cement clinker need to be considered in the final concrete product (Müllauer *et al*., 2015; Overmann *et al*., 2021). Elements identified as especially important from a pollution perspective in EU directive 2010/75 on industrial emissions (EU, 2010) are arsenic, cadmium, cobalt, chromium, copper, mercury, manganese, nickel, lead, antimony, thallium and vanadium.

Some minor and trace elements are also of importance for cement production and product quality, since they can affect clinker reactivity and cement hydration (Bhatty, 2006; Gineys *et al*., 2011a; Kolovos *et al*., 2001, 2002). Kolovos *et al*. (2001, 2002) studied the effect of foreign elements on clinker reactivity, measured by reduction of free lime content, in a chemical grade system of calcium carbonate/silicon dioxide/aluminium oxide/ferric oxide. Among the tested anions, sulfur and fluorine showed the highest reduction of the free lime content at 1450°C. Among the tested cations, tungsten, tantalum, copper, titanium and molybdenum showed the highest reduction of the free lime content at 1450°C. For the tested cations, elements with a decreased atomic radius or increased electronegativity were suggested to improve clinker reactivity.

From both emissions and product perspectives, the relationship between the volatilisation and retention of minor and trace elements is of high importance and, to a large extent, is still not understood. Furthermore, a change in the composition of the process gas, from around 20 vol.% carbon dioxide in a typical fuel-heated kiln to almost 100 vol.% carbon dioxide in, for example, an electrified process, might affect several aspects of the process, such as heat transfer, calcination temperature and clinker formation (Hökfors *et al*., 2015; Wilhelmsson *et al*., 2018). The volatilisation of minor and trace elements is known to be affected by the process atmosphere (Frandsen *et al*., 1994). Therefore, there is a need for both fundamental knowledge of volatilisation of minor and trace elements during cement clinker production, and further knowledge of how volatilisation is affected by a high carbon dioxide atmosphere.

The objective of this study was to experimentally investigate the volatilisation of 68 major, minor and trace elements during cement clinker formation in both a conventional combustion gas atmosphere and a high carbon dioxide atmosphere. Two industrial raw meals were heated in a laboratory furnace in the two different atmospheres (conventional combustion atmosphere and high carbon dioxide atmosphere). Raw meal and cement clinker samples were then analysed for elemental composition using inductively coupled plasma sector field mass spectrometry (ICP-SFMS). In addition, X-ray diffraction (XRD) was used to monitor and validate successful clinker mineral formation, and support interpretation of the ICP data.

Materials and methods

Raw meals at two industrial sites (here called A and B) in northern Europe were sampled. The compositions can be considered of industrial relevance and typical for the region, with some differences that are relevant to consider in both the clinker production process and the quality of the final product. The main differences in the elemental composition of the raw meals (reported as oxides in Table 1) were found to be in the contents of magnesium oxide, manganese(III) oxide, zinc oxide, sulfur trioxide and phosphorus

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Table 1. Chemical composition, determined by ICP-SFMS (expressed as oxides), clinker moduli and particle size ($D_{10\%}$, $D_{50\%}$ and $D_{90\%}$) of the two raw meals (A-RM and B-RM).

	A-RM	B-RM
Chemical composition		
Calcium oxide (CaO): wt%	44.91	46.45
Silicon dioxide (SiO_2): wt%	14.35	14.59
Aluminium oxide (Al_2O_3): wt%	3.48	3.97
Ferric oxide (Fe_2O_3): wt%	1.92	1.86
Magnesium oxide (MgO): wt%	2.39	0.79
Potassium oxide (K_2O): wt%	0.84	0.94
Sodium oxide (Na_2O): wt%	0.18	0.084
Titanium dioxide (TiO_2): wt%	0.20	0.14
Manganese(III) oxide (Mn_2O_3): wt%	0.074	0.42
Zinc oxide (ZnO): wt%	0.047	0.0029
Phosphorus pentoxide (P_2O_5): wt%	0.030	0.094
Sulfur trioxide (SO_3): wt%	1.09	0.28
Others (by difference)[a]	30.489	30.379
Sum	100	100
Clinker moduli		
Lime saturation factor	98.62	99.38
Silica modulus	2.66	2.50
Alumina modulus	1.81	2.13
Particle size		
$D_{10\%}$: µm	3	3
$D_{50\%}$: µm	19	30
$D_{90\%}$: µm	163	170

[a]Including elements of lesser content and non-analysed elements such as hydrogen, carbon, chlorine and oxygen

pentoxide. Furthermore, the lime and marlstones were mainly of sedimentary geological origin but, in raw meal A, some of the limestone was also of reef origin.

Dried, ground and homogenised raw meals were sampled from sampling points in air chutes before the elevators to the top cyclones. The sample size was reduced using a riffle splitter and two raw meal samples (A-RM and B-RM) were produced. Nodules with an approximate size of 1 cm^3 were hand-rolled from samples mixed with deionised water and dried at 105°C overnight. The chemical composition of the main elements in A-RM and B-RM, as determined by ICP-SFMS and expressed as oxides, are presented in Table 1 together with the clinker moduli (Taylor, 1997) and particle size ($D_{10\%}$, $D_{50\%}$ and $D_{90\%}$) measured by laser diffraction using a Panalytical Mastersizer 2000.

The experiments were performed in a laboratory tube furnace with a gas flow of 1 tube volume/min. Two different atmospheres were examined: a conventional combustion atmosphere (20 vol.% carbon dioxide, 5 vol.% oxygen, 10 vol.% water and 65 vol.% nitrogen) and a high carbon dioxide atmosphere (95 vol.% carbon dioxide and 5 vol.% oxygen). The tube furnace was preheated to 1450°C and batches of 4–5 g of raw meal nodules were heated for 40 min in an alumina crucible lined with platinum foil. After heating, the samples were quickly pushed out of the tube furnace and allowed to cool in ambient air. Four cement clinker samples were produced – two in the conventional atmosphere (called A-CC and B-CC) and two in the high carbon dioxide atmosphere (called A-CC-CO_2 and B-CC-CO_2). Each batch was weighed before and after exposure. A total of 25–30 g of clinker was produced per raw meal and atmosphere.

The chemical compositions of the raw meal and cement clinker samples were determined using ICP-SFMS, performed according to ISO 17294-2:2016 and US EPA method 200.8:1994. The samples were prepared by milling according to ISO 11464:2006, and either dissolution in acid according to EN 13656:2020 or lithium metaborate ($LiBO_2$) melting according to ASTM D3682:2013; ASTM D4503:2008. A total of 68 elements were analysed: silver (Ag), aluminium (Al), arsenic (As), gold (Au), boron (B), barium (Ba), beryllium (Be), bismuth (Bi), calcium (Ca), cadmium (Cd), cerium (Ce), cobalt (Co), chromium (Cr), caesium (Cs), copper (Cu), dysprosium (Dy), erbium (Er), europium (Eu), iron (Fe), gallium (Ga), gadolinium (Gd), germanium (Ge), hafnium (Hf), mercury (Hg), holmium (Ho), iridium (Ir), potassium (K), lanthanum (La), lithium (Li), lutetium (Lu), magnesium (Mg), manganese (Mn), molybdenum (Mo), sodium (Na), niobium (Nb), neodymium (Nd), nickel (Ni), phosphorus (P), lead (Pb), palladium (Pd), praseodymium (Pr), platinum (Pt), rubidium (Rb), rhenium (Re), rhodium (Rh), ruthenium (Ru), sulfur (S), antimony (Sb), scandium (Sc), selenium (Se), silicon (Si), samarium (Sm), tin (Sn), strontium (Sr), tantalum (Ta), terbium (Tb), tellurium (Te), thorium (Th), titanium (Ti), thallium (Tl), thulium (Tm), uranium (U), vanadium (V), tungsten (W), yttrium (Y), ytterbium (Yb) zinc (Zn) and zirconium (Zr).

XRD was used to monitor and validate successful clinker formation, as well as to support interpretation of the ICP data. Scans were collected with Cu Kα radiation using a Bruker D8 Advance instrument equipped with a Våntec-1 detector in 2θ mode. Data was collected in the 2θ range of 10–70°, with a step size of 0.02° and a sample rotation of 15 rpm. Phase identification was obtained by matching against references in the PDF-4 database (Gates-Rector and Blanton, 2019) in EVA 5.1 software, and semi-quantitative phase analysis was performed by the Rietveld method (Rietveld, 1969) using Topas-Academic 4.2 software.

Results and discussion

Clinker formation

The phase compositions of the cement clinkers, as obtained by XRD and subsequent qualitative and semi-quantitative analysis, are presented in Table 2 and the molar ratios of sulfur to alkalis in the clinkers, as determined by ICP-SFMS, are shown in Table 3 (discussed later).

The XRD patterns of the clinkers produced in the conventional atmosphere and the high carbon dioxide atmosphere are presented in Figure A1 of the online supplementary material. It is inherently challenging to produce cement clinker samples representative of industrially produced products at laboratory scale. In this work, all four samples had proper clinker composition with regard to the amount of calcium silicates. The relatively high quantity of free lime, even though the lime saturation factor was <100 for both

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Table 2. Phase composition obtained by XRD analysis for clinker exposed to conventional atmosphere (A-CC, B-CC) and high carbon dioxide atmosphere (A-CC-CO_2, B-CC-CO_2)

	A-CC	A-CC-CO_2	B-CC	B-CC-CO_2
Free lime: wt%	1.6	2.4	2.7	4.3
Quartz alpha: wt%	0.2	0.9	0.9	0.6
C_2S beta: wt%	7.3	3.7	5.0	5.3
C_3A cubic: wt%	8.7	2.3	0.8	0.1
C_3A orthorhombic: wt%	1.8	5.8	7.6	6.8
C_4AF: wt%	5.7	7.3	8.6	12.4
C_3S M_1: wt%	45.5	43.0	37.2	44.4
C_3S M_3: wt%	23.6	28.4	36.8	25.6
Periclase: wt%	4.9	4.2	0.1	0.3
Arcanite: wt%	0.0	0.9	0.0	0.0
Aphthitalite: wt%	0.8	0.9	0.3	0.3
Sum: wt%	100	100	100	100

Table 3. The molar ratio of sulfur to alkalis (SO_3/(K_2O + Na_2O)) in the clinker, as determined by ICP-SFMS

	Molar ratio of sulfur to alkalis
A-CC	0.356
A-CC-CO_2	0.300
B-CC	0.111
B-CC-CO_2	0.086

raw meals (Table 1), was likely due to coarse raw meals or the comparably short residence time in the furnace.

Previous studies have shown that a high carbon dioxide atmosphere does not affect phase composition to a great extent (ECRA, 2012; Zheng *et al*., 2016), and this was found to be the case for the main clinker phases in this work, where the differences in phase composition between the samples exposed to the high carbon dioxide atmosphere and the reference atmosphere were small. Some differences in the polymorphism of C_3A were observed, possibly linked to the sulfatisation degree, which could be interesting for future studies. In addition to the main clinker phases, the qualitative analysis indicated the presence of the minor constituents arcanite and aphthitalite. However, XRD analysis holds some limitations regarding the quantification of phases of low quantity.

Minor and trace elements can affect clinker composition, but generally the concentrations required for this are higher than those observed in typical raw meals. This is especially true regarding trace elements (e.g. Gineys *et al*., 2011b).

Volatilisation

ICP-SFMS was undertaken to assess the volatilisation of the minor and trace elements in the two raw meals after heating and to enable comparisons of heating in a simulated conventional combustion atmosphere and a high carbon dioxide atmosphere. The results are summarised in Figures 1–4. The error bars in the figures represent the reported combined analytical and sample-handling uncertainties, corresponding to two standard deviations or approximately 95% confidence intervals for the method. The clinker samples lost an average of 35.4% weight upon exposure (the average weight loss for each clinker sample can be found in Table A1 of the online supplementary material). The weight loss data for each consolidated clinker sample were used to normalise the concentrations and enable comparison with the corresponding raw meal compositions (hence the normalised concentrations for the clinker samples in Figures 1–4 and Tables 4–6 are around 35% lower than measured). Non-normalised ICP-SFMS data for all elements can be found in Table A2 of the online supplementary material.

Major elements

Major elements were defined as elements with concentrations of ≥1 wt% in the clinker samples. The results of the ICP-SFMS analysis are presented in Figure 1.

All the major elements (calcium, silicon, aluminium, iron and magnesium) presented in Figure 1 were found in the clinker

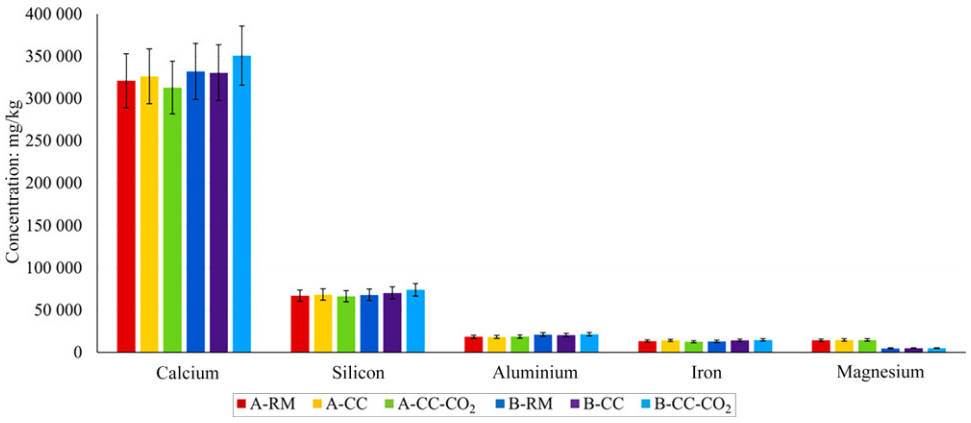

Figure 1. Concentrations of major elements in raw meals (A-RM and B-RM) and clinkers exposed to conventional atmosphere (A-CC and B-CC) and high carbon dioxide atmosphere (A-CC-CO_2 and B-CC-CO_2), measured using ICP-SFMS. Clinker concentrations normalised for weight loss

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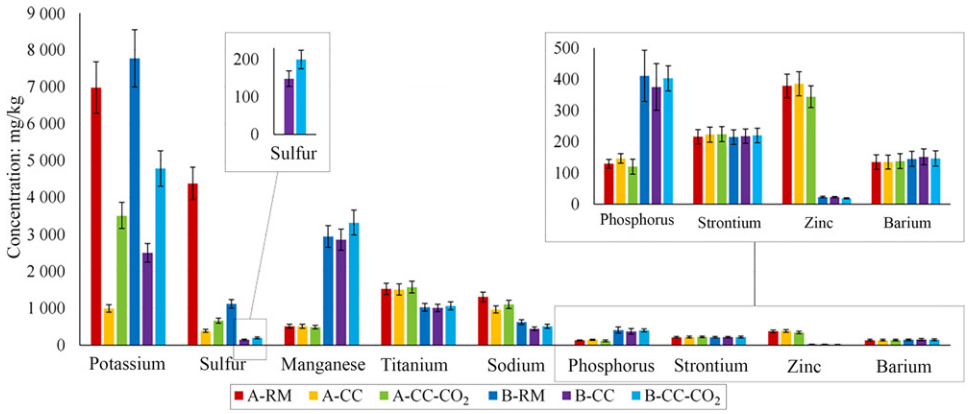

Figure 2. Concentrations of minor elements in raw meals (A-RM and B-RM) and clinkers exposed to conventional atmosphere (A-CC and B-CC) and high carbon dioxide atmosphere (A-CC-CO_2 and B-CC-CO_2), measured using ICP-SFMS. Clinker concentrations normalised for weight loss

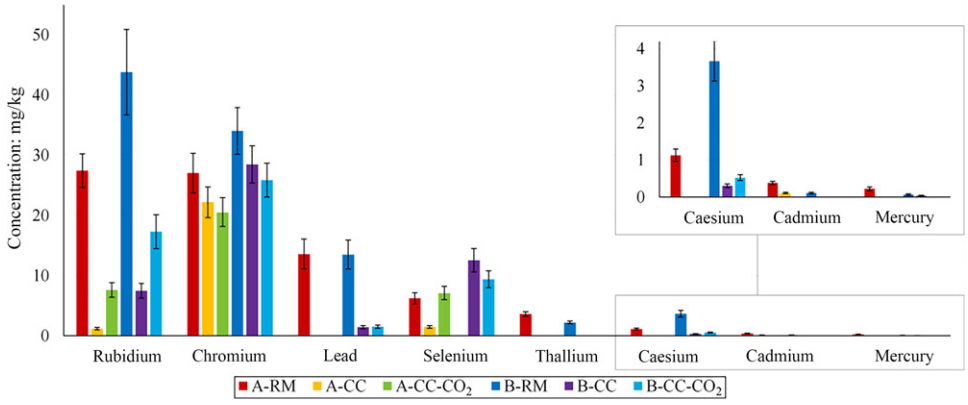

Figure 3. Concentrations of volatile trace elements in raw meals (A-RM and B-RM) and clinkers exposed to conventional atmosphere (A-CC and B-CC) and high carbon dioxide atmosphere (A-CC-CO_2 and B-CC-CO_2), as determined by ICP-SFMS. Clinker concentrations normalised for weight loss

regardless of the process atmosphere, as expected (Hewlett and Liška, 2019; Taylor, 1997). The ICP-SFMS results show that the major elements constituted 43–44 wt% of the raw meals.

Minor elements

Minor elements were defined as elements present at 0.02 wt% ≤ x < 1.00 wt% in the clinker samples. The results of the ICP-SFMS analysis are presented in Figure 2 and Table 4.

The ICP-SFMS results show that the minor elements constituted 1.4–1.6 wt% of the raw meals. Several of the minor elements (Figure 2 and Table 4) were expected to exhibit volatilisation to different degrees (Bhatty, 1995). The normalised ICP-SFMS results for the clinkers show great variation in retention between the raw meals and between the atmospheres. In the conventional atmosphere, 34 wt% of the minor elements was incorporated in the A-CC clinker, and 54% in B-CC. For the high carbon dioxide atmosphere, 53 wt% of the minor elements was incorporated in A-CC-CO_2, and 75% in B-CC-CO_2, meaning that the overall volatilisation of minor elements was lower in the high carbon dioxide atmosphere. Manganese, titanium, phosphorus, strontium and barium were all incorporated in the clinker, and no clear difference could be observed between the two different atmospheres.

As shown in Figure 2, potassium, sulfur and (to some extent) sodium were affected by the change in atmosphere. These elements exhibited lower volatilisation in the high carbon dioxide atmosphere, with the difference being especially large for potassium.

Water vapour is known to favour alkali volatilisation under some conditions (Bhatty, 1995). In the present study, the raw meals were dried prior to high-temperature exposure, and only the conventional atmosphere contained water vapour, possibly contributing to the higher volatilisation degree in the conventional atmosphere. Sulfur is known to both form volatile compounds and be incorporated as sulfates into clinker (Bhatty, 1995). For both atmospheres, the ICP-SFMS results showed low incorporation of sulfur into the clinker (see Table 4). The volatilisation of both alkali and sulfur was, however, higher in the conventional atmosphere. Bhatty (1995)

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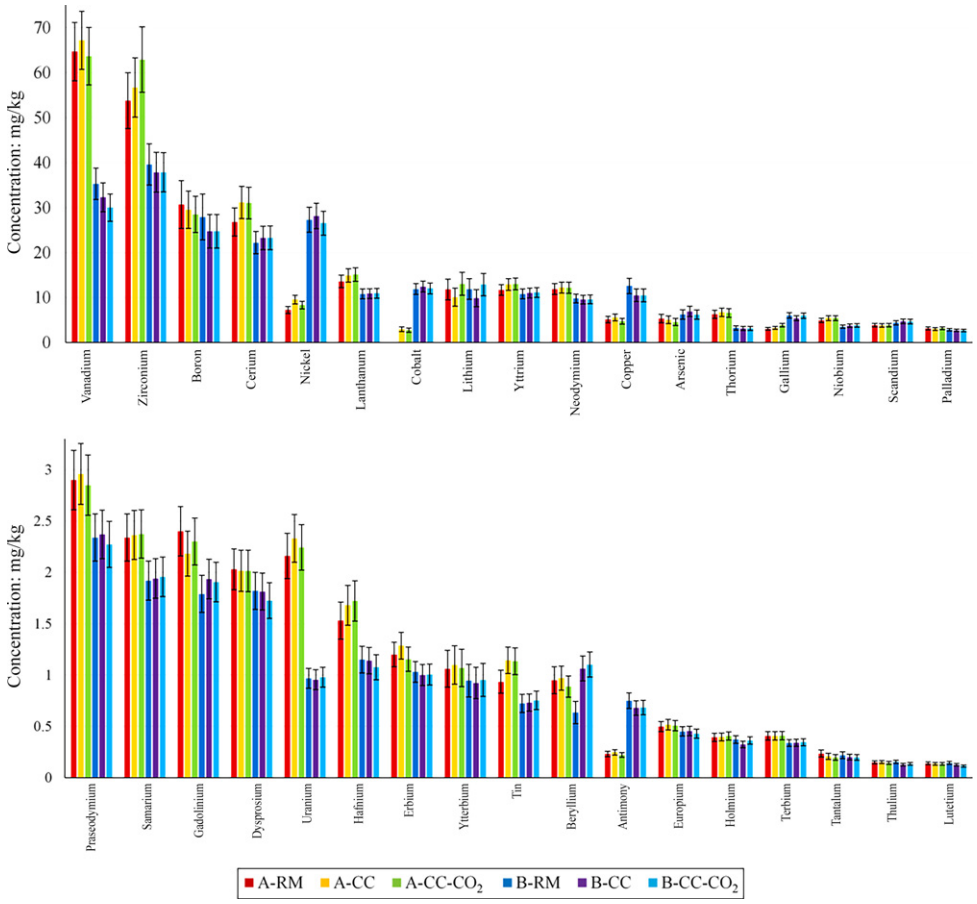

Figure 4. Concentrations of non-volatile trace elements in raw meals (A-RM and B-RM) and clinkers exposed to conventional atmosphere (A-CC and B-CC) and high carbon dioxide atmosphere (A-CC-CO_2 and B-CC-CO_2), as determined by ICP-SFMS. Clinker concentrations normalised for weight loss

suggested that water vapour in a cement kiln may affect the volatility of alkali and sulfur according to the following reaction.

$$\text{I.}\quad 2H_2O + \underset{\text{less volatile}}{2K_2SO_4} \longleftrightarrow \underset{\text{more volatile}}{4KOH} + \underset{\text{very volatile}}{2SO_2} + O_2$$

Furthermore, a high carbon dioxide atmosphere can stabilise carbonates at high temperature, possibly contributing to the higher retention of potassium and sodium at high carbon dioxide concentrations.

Alkali sulfates are important for clinker mineralogy and cement performance (Ma and Qian, 2018), and most cement producers have guidelines regarding the sulfatisation degree (molar ratio of sulfur to alkalis), which should ideally be around 1 (Cortada Mut *et al*., 2015). Excess sulfur tends to stabilise C_2S, resulting in a higher temperature needed for C_3S formation. The ICP-SFMS results indicate that the higher retention of sulfur, potassium and sodium in the high carbon dioxide atmosphere did not affect the molar ratio of sulfur to alkalis to a large extent (Table 3). All the clinkers showed a molar ratio of sulfur to alkalis <1, indicating excess alkali compared with sulfur for all the clinker compositions (Table 3). Excess alkalis are mainly incorporated into C_2S and C_3A, especially affecting the polymorphism of C_3A and resulting in more orthorhombic C_3A (Hewlett and Liška, 2019). The C_3A polymorphs presented in Table 2 show that $C_3A_{orthorhombic}/C_3A_{total}$ was higher for B-CC and B-CC-CO_2 than for A-CC and A-CC-CO_2. B-CC and B-CC-CO_2 also showed a lower molar ratio of sulfur to alkali as compared with A-CC and A-CC-CO_2. The clinker produced in the high carbon dioxide atmosphere had a higher ratio of $C_3A_{orthorhombic}/C_3A_{total}$ compared to its analogue produced in a conventional atmosphere and the molar ratio of sulfur to alkalis in the clinker was somewhat lower for the high carbon dioxide atmosphere, although the difference was small. Considering the small scale of this study, the impact on C_3A polymorphs of a dry atmosphere with a high carbon dioxide concentration compared with a conventional atmosphere should be addressed in further studies, preferably in a simulated industrial process using a counter-current reactor design, enabling the recirculation of volatile elements.

In counter-current reactors such as cement kilns, potassium, sodium, sulfur and chlorine are known to volatilise when exposed to

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Table 4. Minor elements in raw meals and clinker samples, as determined by ICP-SFMS. Clinker concentrations normalised for weight loss

	A-RM	A-CC		A-CC-CO_2		B-RM	B-CC		B-CC-CO_2	
	Concentration: mg/kg	Concentration: mg/kg	Retention: wt%	Concentration: mg/kg	Retention: wt%	Concentration: mg/kg	Concentration: mg/kg	Retention: wt%	Concentration: mg/kg	Retention: wt%
Potassium	6980	995	14	3514	50	7770	2505	32	4783	62
Sulfur	4380	386	9	665	15	1120	149	13	200	18
Manganese	513	512	100	491	96	2950	2864	97	3322	113
Titanium	1530	1512	99	1578	103	1030	1012	98	1069	104
Sodium	1310	969	74	1108	85	625	445	71	511	82
Phosphorus	130	147	113	121	93	411	375	91	403	98
Strontium	216	223	103	224	104	215	218	101	220	102
Zinc	379	386	102	344	91	23	23	99	19	83
Barium	135	135	100	138	102	145	152	105	147	101
Sum	15 573	5264	34	8184	53	14 289	7743	54	10 673	75

Table 5. Trace elements exhibiting ≥20% volatilisation in raw meals and clinker samples, as determined by ICP-SFMS. Clinker concentrations normalised for weight loss

	A-RM	A-CC		A-CC-CO_2		B-RM	B-CC		B-CC-CO_2	
	Concentration: mg/kg	Concentration: mg/kg	Retention: wt%	Concentration: mg/kg	Retention: wt%	Concentration: mg/kg	Concentration: mg/kg	Retention: wt%	Concentration: mg/kg	Retention: wt%
Rubidium	27.4	1.2	4	7.6	28	43.8	7.5	17	17.3	40
Chromium	27.0	22.2	82	20.5	76	34.0	28.4	84	25.8	76
Lead	13.6	<1	0	<1	0	13.5	1.4	11	1.5	11
Selenium	6.22	1.46	23	7.11	114	<2	12.56	—	9.40	—
Thallium	3.62	<0.05	0	<0.05	0	2.21	<0.05	0	<0.05	0
Caesium	1.13	<0.05	0	<0.1	0	3.67	0.31	8	0.53	14
Cadmium	0.383	0.109	28	<0.05	0	0.108	<0.05	0	<0.05	0
Mercury	0.223	<0.05	0	<0.05	0	0.0682	0.0384	56	<0.05	0
Sum	79.6	25.0	31	35.2	44	97.4	50.2	52	54.5	56

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Table 6. Trace elements exhibiting <20% volatilisation in raw meals and clinker samples, as determined by ICP-SFMS. Clinker concentrations normalised for weight loss

	A-RM	A-CC		A-CC-CO_2		B-RM	B-CC		B-CC-CO_2	
	Concentration: mg/kg	Concentration: mg/kg	Retention: wt%	Concentration: mg/kg	Retention: wt%	Concentration: mg/kg	Concentration: mg/kg	Retention: wt%	Concentration: mg/kg	Retention: wt%
Vanadium	64.7	67.2	104	63.7	98	35.3	32.3	91	30.0	85
Zirconium	53.8	56.7	105	62.9	117	39.6	37.9	96	37.7	96
Boron	30.7	29.5	96	28.5	93	27.9	24.7	89	24.7	89
Cerium	26.8	31.1	116	31.0	116	22.2	23.3	105	23.3	105
Nickel	7.24	9.56	132	8.35	115	27.3	28.1	103	26.5	97
Lanthanum	13.6	14.9	110	15.1	111	10.8	10.9	101	10.9	101
Cobalt	<3	2.98	—	2.75	—	11.9	12.4	104	12.0	101
Lithium	11.8	10.1	85	13.0	111	11.9	9.9	83	12.9	108
Yttrium	11.7	12.9	110	13.0	111	10.8	11.0	102	11.1	103
Neodymium	11.9	12.2	103	12.2	102	9.78	9.55	98	9.59	98
Copper	5.11	5.58	109	4.70	92	12.6	10.5	83	10.5	83
Arsenic	5.32	5.04	95	4.56	86	6.25	6.92	111	6.19	99
Thorium	6.26	6.72	107	6.59	105	3.23	3.14	97	3.15	97
Gallium	3.04	3.24	107	3.86	127	6.00	5.39	90	5.97	100
Niobium	4.92	5.43	110	5.41	110	3.52	3.75	106	3.82	108
Scandium	3.87	3.82	99	3.85	99	4.38	4.70	107	4.64	106
Palladium	3.14	3.00	95	3.18	101	2.84	2.71	95	2.67	94
Praseodymium	2.90	2.96	102	2.85	98	2.34	2.37	101	2.27	97
Samarium	2.34	2.36	101	2.37	101	1.92	1.94	101	1.96	102
Gadolinium	2.40	2.18	91	2.30	96	1.79	1.93	108	1.91	106
Dysprosium	2.03	2.02	99	2.01	99	1.82	1.81	100	1.73	95
Uranium	2.16	2.33	108	2.24	104	0.968	0.955	99	0.979	101
Hafnium	1.53	1.68	110	1.72	113	1.15	1.14	99	1.08	93
Erbium	1.20	1.29	107	1.15	96	1.03	1.00	97	1.00	98
Ytterbium	1.06	1.10	104	1.07	101	0.945	0.92	98	0.95	101
Tin	0.934	1.14	122	1.13	121	0.723	0.73	101	0.75	104
Beryllium	0.949	0.97	102	0.89	93	0.634	1.06	168	1.10	174
Antimony	0.23	0.25	107	0.22	96	0.749	0.68	91	0.68	91
Europium	0.497	0.516	104	0.507	102	0.45	0.45	101	0.43	95
Holmium	0.392	0.396	101	0.406	103	0.371	0.323	87	0.362	98
Terbium	0.406	0.406	100	0.410	101	0.338	0.340	100	0.345	102
Tantalum	0.235	0.207	88	0.196	84	0.218	0.200	92	0.198	91
Thulium	0.148	0.151	102	0.144	97	0.154	0.127	82	0.134	87
Lutetium	0.139	0.135	97	0.134	96	0.144	0.124	87	0.113	79
Sum	283.5	300.1	106	302.5	107	262.0	253.2	97	251.9	96

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high temperature and subsequently condense in the colder end of the kiln, giving rise to internal circulation and local accumulation in the kiln (Cortada Mut *et al*., 2015; Taylor, 1997). This phenomenon did not occur in the experimental setup described herein because the tube furnace was purged with gas. This accumulation in industrial kilns can be expected to lead to a higher incorporation of these elements into the clinker as compared with the results obtained in this study.

Zinc was mostly incorporated into the clinker, in accordance with previous studies (Bhatty, 1995; Hökfors and Backman, 2015) but exhibited some volatilisation in the high carbon dioxide atmosphere, although the difference between atmospheres was within the reported measurement uncertainty. Zinc can affect hydration and setting times as well as clinker formation rates (Bhatty, 2006; Bhatty, 1995), meaning that the higher volatilisation in a high carbon dioxide atmosphere can affect clinker quality, even though the concentrations were below established limits in conventional raw meals to have an effect on the final quality (Gineys *et al*., 2011a, 2011b).

Trace elements

Trace elements were defined as elements present at concentrations <0.02 wt%. For the purposes of discussion, the trace elements were divided into two categories based on the extent of volatilisation during heating. The results of the ICP-SFMS analysis for trace elements volatilised to the average extent of ≥20 wt% are shown in Figure 3 and Table 5, and those for trace elements mostly or fully incorporated in the clinker are presented in Figure 4 and Table 6. In addition, 11 trace elements were close to the detection limit or were not detected at all in either the raw meal or clinker; these elements were silver, gold, bismuth, germanium, iridium, molybdenum, rhenium, rhodium, ruthenium, tellurium and tungsten. These 11 elements, together with platinum, are thus not included in the figures, but are detailed in Table A2 of the online supplementary material. Platinum was excluded as traces of platinum foil were found in the clinker samples.

The trace elements present in concentrations above the detection limit of ICP-SFMS and exhibiting ≥20% volatilisation were cadmium, chromium, caesium, lead, rubidium, selenium, thallium and mercury. The volatile trace elements constituted 79–98 ppm of the raw meals, and some differences were observed between the atmospheres. Rubidium, and to some extent caesium, followed the same pattern as other alkali metals, as expected (Bhatty, 1995). Chromium showed a trend of being more volatile in the high carbon dioxide atmosphere, but the difference was within measurement uncertainty. The results for selenium were unexpected: selenium is expected to be volatile and not incorporated in the clinker to any great extent (Bhatty, 1995), yet it was completely incorporated in A-CC-CO_2. For clinkers produced from B-RM, more selenium was present in the clinker than the raw meal, and it is likely that there was some analytical error with the results for selenium. Lead, thallium, cadmium and mercury were all almost completely volatilised, and no difference could be detected between the atmospheres. Mercury showed a high retention in B-CC, but this was most likely due to the values being very close to the detection limit.

The trace elements present at concentrations above the detection limit of ICP-SFMS and exhibiting <20% volatilisation were vanadium, zirconium, boron, cerium, nickel, lanthanum, cobalt, lithium, yttrium, neodymium, copper, arsenic, thorium, gallium, niobium, scandium, palladium, praseodymium, samarium, gadolinium, dysprosium, uranium, hafnium, erbium, ytterbium, tin, beryllium, antimony, europium, holmium, terbium, thulium and lutetium. The less volatile trace elements constituted 260–290 ppm of the raw meals. For all the trace elements mostly or fully incorporated in clinker, all the differences between the atmospheres were within measurement uncertainty. However, it is worth noting that lithium followed the same trend as the other alkali metals, with higher volatilisation in the conventional atmosphere.

As can be seen for most trace elements, there was no significant difference between the two atmospheres, and most of the trace elements were incorporated in the clinker.

Of the 12 elements identified in EU directive 2010/75 (EU, 2010) as being especially important in relation to the prevention and control of air pollution, manganese, vanadium, nickel, cobalt, arsenic and antimony are expected to be mostly incorporated in the clinker. Mercury and thallium are highly volatile. Lead is also fairly volatile and is not expected to be incorporated in the clinker to any large extent. A major portion of chromium and copper is expected to be incorporated in the clinker; for cadmium, it depends on the conditions (Bhatty, 1995). For some of these elements, a trend of higher volatilisation in the high carbon dioxide atmosphere was observed, but the differences were within measurement uncertainty. However, as this was recurring for several of the elements in both samples, it is worth noting and investigating further in future studies.

Summary

A summary of the ICP-SFMS results is presented in Table 7. For both atmospheres, the minor elements potassium and sulfur, and the trace elements rubidium, lead, thallium, caesium, cadmium and mercury were highly volatile. The other tested trace elements were volatile to some extent, mostly retained in the cement clinker or present in levels close to or below the detection limit.

As previously noted, there was a distinguishable difference in volatilisation between the atmospheres for the minor elements, with a lower degree of volatilisation observed for the high carbon dioxide atmosphere. Potassium was responsible for a large part of this difference, followed by sulfur and sodium. In addition, for the volatile trace elements there was a trend of lower volatilisation in the

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Table 7. Summary of results as determined by ICP-SFMS. Clinker concentrations normalised for weight loss

	A-RM	A-CC		A-CC-CO_2		B-RM	B-CC		B-CC-CO_2	
	Concentration: mg/kg	Concentration: mg/kg	Retention: wt%	Concentration: mg/kg	Retention: wt%	Concentration: mg/kg	Concentration: mg/kg	Retention: wt%	Concentration: mg/kg	Retention: wt%
Major elements	434 300	441 806	102	425 619	98	438 940	440 901	100	466 021	106
Minor elements	15 573	5264	34	8184	53	14 289	7743	54	10 673	75
Volatile trace elements	79.6	25.0	31	35.2	44	97.4	50.2	52	54.5	56
Non-volatile trace elements	283.5	300.1	106	302.5	107	262	253.2	97	251.9	96
Sum	450 236	447 395	99	434 141	96	453 588	448 947	99	477 000	106

high carbon dioxide atmosphere, but this difference was much smaller and could be due to measurement uncertainty.

For most of the 68 elements analysed, no difference could be observed between the two atmospheres. With regard to implementing electrification for cement clinker production, this can be viewed as a promising initial result, as there is no clear hindrance for such a process change from the perspective of the volatilisation of minor and trace elements. Even so, further investigation of the fates of alkalis, sulfur and zinc as well as some of the trace elements included in EU directive 2010/75 (EU, 2010) is suggested, preferably in a counter-current reactor design that would enable the recirculation of volatile elements. Furthermore, it would be valuable to investigate whether the differences between atmospheres affect how some of these elements are incorporated in the clinker phases, which was beyond the scope of the present work. Future studies should also address a range of temperatures and times for clinker formation, and also compare the results for minor and trace elements with multicomponent chemical equilibrium calculations. Chemical speciation and mineral association of the minor and trace elements should also be addressed in order to facilitate mechanistical understanding of the observations.

Conclusions

The volatilisation of minor and trace elements in cement clinkers, which were produced at laboratory scale in either a conventional combustion atmosphere or a high carbon dioxide atmosphere, was analysed using ICP-SFMS. High carbon dioxide atmospheres are expected in the future when near-zero emission production is achieved by carbon dioxide capture through process electrification or oxy-fuel combustion.

For both atmospheres, the minor elements potassium and sulfur, and the trace elements rubidium, lead, thallium, caesium, cadmium and mercury were highly volatile. The other tested trace elements were either volatile to some extent, mostly retained in the cement clinker or present at levels close to or below the detection limit.

A clear trend was observed for the two atmospheres, with the volatilisation of potassium, sodium, and sulfur being lower in the high carbon dioxide atmosphere. This may affect the molar ratio of sulfur to alkalis in clinkers produced in the high carbon dioxide atmosphere, potentially affecting clinker quality.

Of the 12 elements regulated in EU directive 2010/75 (EU, 2010), manganese, vanadium, nickel, cobalt, arsenic, antimony, copper and chromium were mostly incorporated in the clinker, regardless of the atmosphere. Mercury, thallium, lead and cadmium were highly volatile for both atmospheres. For some of these elements, there was a trend of higher volatilisation in the high carbon dioxide atmosphere, but the low concentrations and relatively high measurement uncertainty suggest the need for further studies.

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The results of this study, limited to two raw meals and with idealised experimental conditions compared with industrial production, should be seen as indicative and a basis for further work. Further studies could make use of larger samples and a simulated industrial process using a counter-current reactor design that would enable the recirculation of volatile elements. It would also be interesting to include chlorine among the analysed elements, in addition to further investigating the elements exhibiting changes in volatilisation in the high carbon dioxide atmosphere. Furthermore, studies targeting chemical speciation and mineral association of the minor and trace elements are needed in order to facilitate mechanistical understanding of the observations and, ultimately, enable predictions for new raw meal mixtures.

Author contributions

Conceptualisation: A.V., K.S., B.W., M.E., M.C. and M.B. Data curation: A.V. Formal analysis: A.V., K.S., M.E., M.C. and M.B. Funding acquisition: M.E., B.W. and M.B. Investigation: A.V. and K.S. Methodology: A.V., K.S., B.W., M.C., M.E. and M.B. Project administration: M.E. M.C. and M.B. Supervision: M.E. and M.B. Visualisation: A.V., K.S. and M.B. Writing – original draught: A.V., M.E. and M.B. Writing – review and editing: A.V., K.S., B.W., M.B., M.C. and M.E.

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