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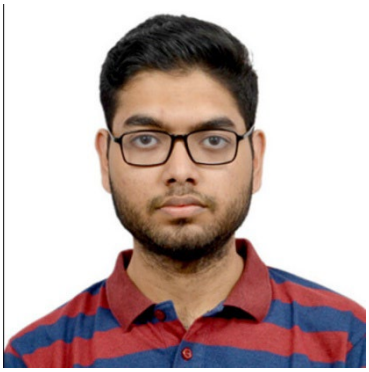
Beyond Bauxite or Alumina: A Review of Alternative Alumina Rich Sources for Calcium-Sulfo Aluminate Cement Production



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ABSTRACT

Calcium Sulfo-Aluminate (CSA) clinkers are predominantly tailored for specialized markets that require distinct properties such as accelerated strength development and shrinkage compensation, both of which are primarily associated with the presence of the ye'elimite phase. The commercial CSA clinkers constitute a high ye'elimite content from the use of raw materials such as pure alumina or bauxite, which is listed as a critical raw material by the European commission and therefore significantly increasing the production costs. This economic limitation renders CSA cement substantially more expensive than Ordinary Portland cement (OPC), thereby confining its use to small-scale application. These challenges may be addressed by exploring alternative alumina-rich industrial by-products or Al-rich residues characterized by Fe_2O_3 and SiO_2 contents below 5% and Al_2O_3 content above 60 %, which hold the potential to fully substitute pure alumina or bauxite. Therefore, maintaining the high ye'elimite content of the CSA clinkers without the use of pure alumina or bauxite. However, incorporating Al-rich residues from the aluminium industry may pose a risk to the human health during clinker production due to its high dioxin content of ~ 35 ng TEQ kg^{-1}, higher than the limit proposed by SINTEF of 10 ng TEQ kg^{-1}. Therefore, future research direction should focus on initially the degradation technologies for dioxin removal from the raw material followed by evaluating the feasibility of using Al-rich industrial residues as the sole source of alumina in CSA clinker production, aiming to optimize their processing and performance while minimizing reliance on pure alumina or bauxite.

Key words: Calcium Sulfo-Aluminate, clinker, ye'elimite, Al-rich residues.

1. INTRODUCTION

CSA cement is manufactured using primary raw materials, including limestone, bauxite, clay, and gypsum under oxidizing conditions [1, 2], whereas for Portland cement these are mostly limestone, sand, shale or clay. The chemical composition of CSA clinkers spans a wide range within the CaO-Al_2O_3-SiO_2-Fe_2O_3-SO_3 system, as illustrated in Table 1 [3]. These clinkers have lower contents of Al_2O_3 and SO_3 in comparison to OPC. In terms of mineralogy, , the dominant phase in CSA cement is ye'elimite whereas in the OPC it is the alite phase. Table2 gives an overview of the mineralogy content range in both these cements [3].This distinction contributes to the reduced CO_2 emissions associated with cements where ye'elimite serves as the principal phase, as illustrated in Table 3 [4]. Furthermore, the enthalpy required for the formation of C_3S is 1848 kJ/kg, whereas the formation of ye'elimite requires significantly less energy, approximately 800 kJ/kg [5]. This means that the formation of $C_4A_3\hat{S}$ occurs at temperatures 200–300°C lower than those necessary for the formation of C_3S [6]. A reduction in clinker temperature leads to lower energy consumption and, consequently, a decrease in CO_2 emissions. Furthermore, CSA clinker is more easily to grind compared to Portland cement due to its greater friability, which reduces the energy required for grinding [6]. This leads to a probable case for CSA cement being a low-CO_2 and less energy-intensive clinker compared to OPC. However, the main issue of CSA cement is the required alumina content, which poses a challenge in terms of price and scarcity (Figure 1). Moreover using bauxite as raw material in the manufacture of CSA cement may present economic woes due to the high cost of conventional raw materials used for the manufacturing of the CSA cement [7]. As a result, there is an urgent need to identify alternative raw material sources that will enable cost-competitive manufacturing of these cements.

Table 1 – Chemical compositional ranges, in wt%, of OPC and CSA [3].

Phase	OPC	CSA
CaO	55-75	35-45
SiO_2	15-25	5-10
Al_2O_3	2-6	25-35
Fe_2O_3	0-6	0-9
SO_3	0.3-1.5	6-30

Table 2 – Mineralogical compositional ranges, in wt%, of OPC and CSA [3]

Phase	OPC	CSA
C_3S (alite)	55-75	0-5
C_2S (belite)	10-20	0-55
C_3A, CA, $C_{12}A_7$, CA_2 (aluminates)	5-10	0-20
C_4AF (ferrites)	5-10	0-30
$C_4A_3\hat{S}$ (ye'elimite)	-	45-75

C denotes CaO, A is Al_2O_3, S is SiO_2, F is Fe_2O_3, and $\hat{S}$ is SO_3

Table 3 – CO_2 emission per tonne of the phase produced [4].

Phase	CaO,wt%	t CO_2/t of phase
C_3S	73.7	0.58
C_2S	65.1	0.51
C_3A	62.2	0.49
C_4AF	46.2	0.36
$C_4A_3\hat{S}$	36.7	0.22

C denotes CaO, A is Al_2O_3, S is SiO_2, F is Fe_2O_3, and $\hat{S}$ is SO_3

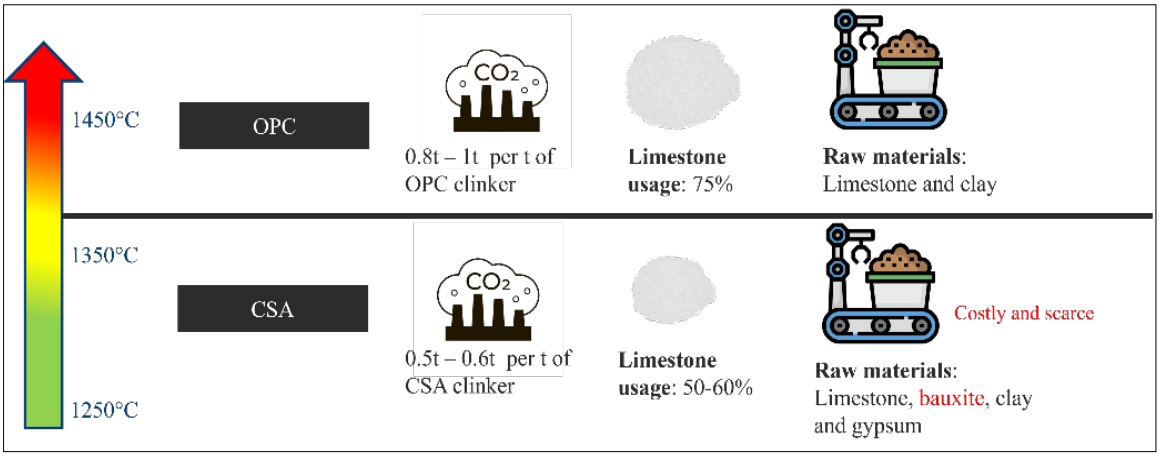

Figure 1 – CSA-A low CO_2 clinker alternative to OPC, but at what cost?

Currently there is a lack of literature that provides an overview of possible Al-rich sources having higher alumina content > 60 % that can be used as a complete replacement of pure alumina or bauxite as raw material in the CSA clinker production. Research studies have explored the use of alternative raw material such as fly ash [8-12], blast furnace slag [8, 13], bauxite residue [11, 14-18], ladle slag [19], iron-rich slags [20] in combination with pure alumina or bauxite as a alumina source for CSA clinker production. However, the complete replacement of pure alumina or bauxite is still lacking and therefore new formulations need to be developed in order to avoid the use of bauxite or pure alumina. In this review, potential sources of high alumina content (> 60 %) raw materials derived from aluminum industry for CSA clinker production and the challenges associated with their use as raw material for clinker production are discussed.

2. REVIEW OF ALTERNATIVE SOURCES OF ALUMINA: ALUMINA-RICH RESIDUES IN CSA CEMENT CLINKER

The primary challenge in advancing the research, development, and large-scale application of CSA cement, lies in reliance on alumina or bauxite to achieve the required high Al_2O_3 content (> 20%) in the clinker. These challenges can be overcome by investigating alternative alumina-rich industrial residues that have Fe_2O_3 and SiO_2 < 5 %, which have the potential to replace pure alumina completely. Various Al-rich residues have the potential to completely replace the pure alumina or bauxite due to the high Al_2O_3 > 60 % in the raw material with minor contents of Fe_2O_3 and SiO_2 < 5 %. These residues comprise aluminum anodization sludge (AAS) [21-27], pretreated black dross/ aluminum dross (Al-dross) [10, 28-36] and salt-slag by product/non-metallic product (NMP) [37, 38]. Figure 2 presents a summary of the number of publications that have utilized Al-rich residues for the synthesis of sulfo-aluminate cement clinker.

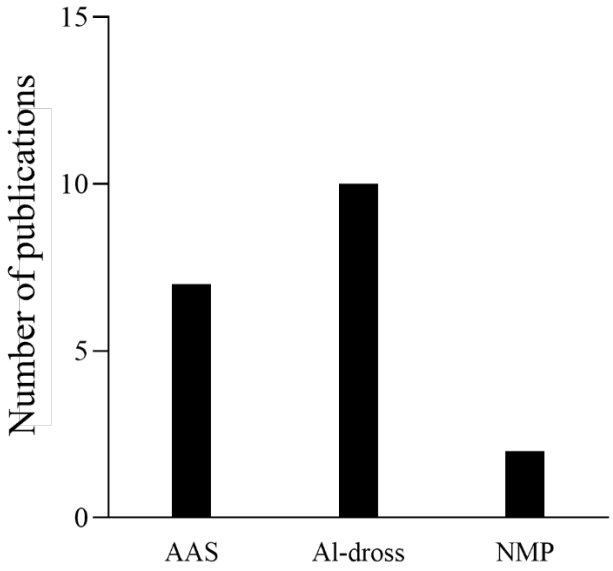

Figure 2 – Al-rich residues in synthesis of sulfo-aluminate cement.

2.1 Pretreated black dross/aluminum dross (Al-dross)

Al-dross is the most adopted Al-rich residue to synthesize sulfo-aluminate cements, based on the literature. Black dross is a hazardous waste that is discharged during secondary aluminum production. During the melting process of the aluminum scraps, the molten aluminum comes into contact with oxygen to form a layer of Al_2O_3. In addition, impurities from the scrap, non-metallic materials, small portions of metallic aluminum, addition of fluxes can agglomerate with the layer of Al_2O_3 to form black dross [42].

The black dross mostly consists of aluminum metal (10–50%), a salt-flux mixture (40–55%), and aluminum oxide (20–50%) [43]. Additionally, black dross contains minor quantities of Al_4C_3, AlP, AlAs, and Al_2S_3 along with heavy metals (Cr, Pb, Cd, and As) and salts (NaCl, KCl, NaF, KF). The reactive aluminum compounds undergo hydrolysis upon contact with water, resulting in the release of toxic gases such as NH_3, AsH_3, and H_2S, along with flammable gases including CH_4 and PH_3, whereas heavy metals and salts have the potential to leach into soil and water, leading to contamination and causing environmental pollution of both soil and water resources (Figure 3) [44]. Moreover, the accumulation of this residue has exceeded 6.5 million tons [44]. Therefore, this residue can not be disposed directly to the landfills. Hence, pretreatment is necessary through hydrometallurgical and pyrometallurgical routes for its use as a building material.

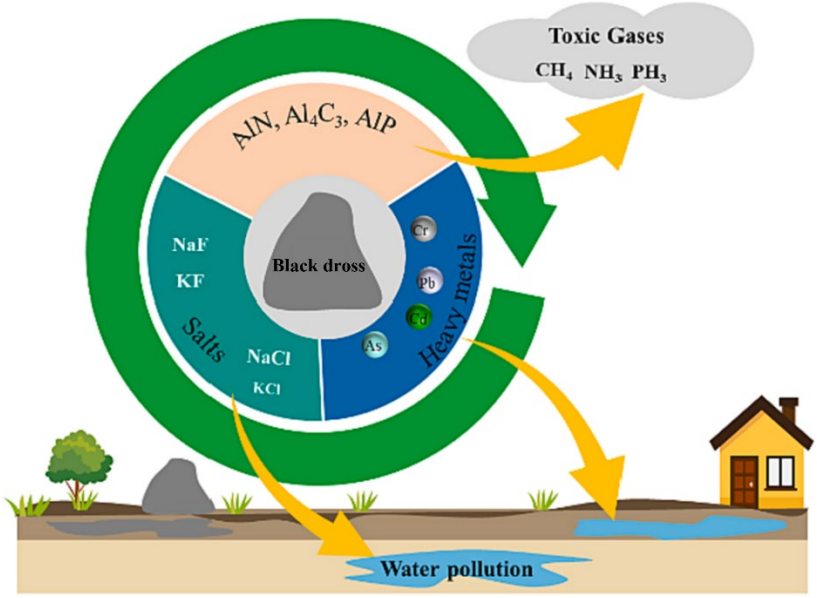

Figure 3 – Black dross- a hazardous residue [44].

The chemical composition of the pretreated Al-dross varies depending on the pretreatment method and the industry generating it. Figure 4 provides an overview of the range of chemical composition of Al-dross, adapted from [10, 28-36] and [45, 46]. In comparison to AAS, pretreated Al dross has higher Al_2O_3 contents without any presence of other oxides > 15 %. Therefore Al-dross can be a perfect alternative to bauxite or pure alumina in the synthesis of sulfo-aluminate cements. Table 4 provides an overview of the application of pretreated Al-dross in synthesizing sulfo-aluminate cement and maximizing the ye'elimite formation.

All literature studies utilizing aluminum dross for the synthesis of sulfo-aluminate clinker did not rely on pure alumina or bauxite as the source of Al_2O_3, indicating a complete substitution. The use of Al-dross as the sole Al_2O_3 source for the synthesis of sulfo-aluminate clinkers was only investigated by Ren et al. [33]. In this study, the raw meal proportion constituted of Al-dross

(17.6-41.6 %) as Al_2O_3 source, carbide slag (23.8-54.7 %) as CaO source, desulfurized gypsum (1.5-30.5 %) as CaO and SO_3 source. Additionally, magnesium tailings ranging from 9.5% to 56.9%, containing $MgCO_3$ were incorporated, offering the potential to absorb CO_2 during the carbonation process. The maximum $C_4A_3\hat{S}$ formation (80 %) was reported for raw meal proportion constituting 40.4 % Al-dross, 54.7 % carbide slag, and 4.8 % desulfurized gypsum. In other studies, the Al-dross was complemented with fly ash [28] [10, 29, 30, 34, 35], coal gangue [32], bauxite residue, Fe-slag, and blast-furnace slag [34].

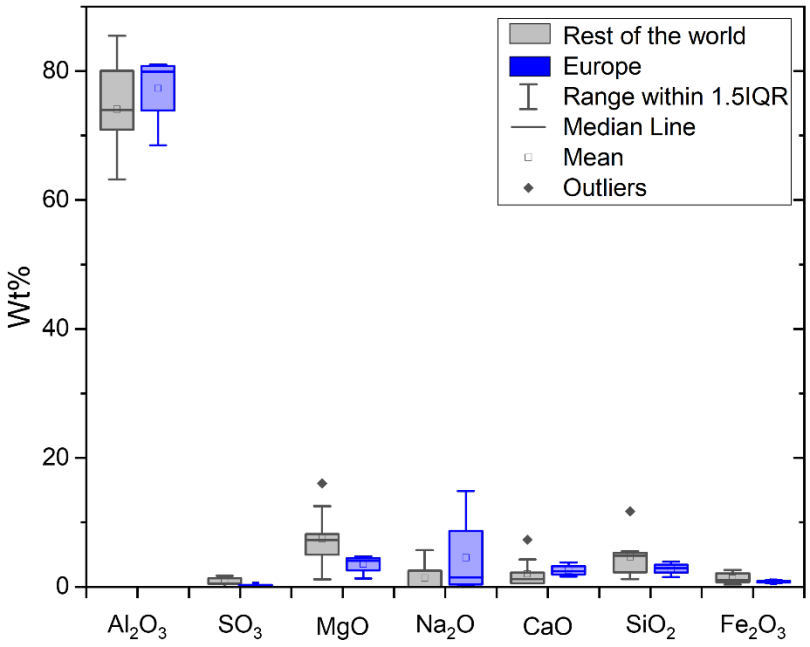

Figure 4 – Chemical composition of Al-dross, adapted from [10, 28-36] and [45, 46].

Table 4 – Overview of AAS dosage, synthesis protocol, CaO, Al_2O_3, and SO_3 oxide composition of the clinker, and the $C_4A_3\hat{S}$ content of the sulfo-aluminate clinker

Ref.	**Al-dross dosage wt%**	**Complete replacement of bauxite or pure-Al_2O_3**	**Synthesis protocol**		**C, A, and $\hat{S}$ oxide composition of the clinker wt%**			**$C_4A_3\hat{S}$ wt%**
			Temp (°C)	Dwell (min)	C	A	$\hat{S}$	
Heredia et al. [28]	45.5	Yes	1250	240	39	35	9	60
Li et al.[29]	26.7-31.1	Yes	1250	30	39-41	26-29	14-15	54-56
Ref.	**Al-dross dosage wt%**	**Complete replacement of bauxite or pure-Al_2O_3**	**Synthesis protocol**		**C, A, and $\hat{S}$ oxide composition of the clinker wt%**			**$C_4A_3\hat{S}$ wt%**
Mao et al.[30]	27.5	Yes	1275	30	40	29	14	63
Ren et al.[32]	32	Yes	1300	60	42	32	11	68
Ren et al.[33]	18-42	Yes	1260	30	29-51	20-34	2-17	43-83
Robl et al. [34]	7.9-13.5	Yes	1250-1300	60	52-57	12-17	5-7	15-19
Wu et al.[35]	25.3	Yes	1270	100	28	20	39	60

Al-dross denotes pretreated aluminum dross. C denotes CaO, A is Al_2O_3 and $\hat{S}$ is SO_3.

2.2 Aluminum Anodizing Sludge (AAS)

AAS is the second most adopted Al-rich residue for the synthesis of sulfo-aluminate cements. Anodization is an electrochemical technique that creates a protective oxide layer on the surface of aluminum alloys, enhancing their hardness and resistance to corrosion. The anodization process is typically carried out in an acidic solution, although basic solutions can also be employed in certain cases. The acidic method is the most commonly used due to its cost-effectiveness [39]. In this process, the acid gradually dissolves aluminum oxide, and the concentration of the acid is adjusted to control the oxidation rate, leading to the formation of a porous coating of controlled thickness. A significant amount of water is used during the process, which leads to the generation of substantial sludge. This sludge consists of compounds such as aluminum hydroxide, sulfate,

sodium sulfate, and calcium hydroxide. Figure 5 provides a detailed schematic of the aluminum anodization process [39].

In the EU countries, the generation of this sludge accounts for up to 100,000 t/ year [40]. Additionally, high disposal costs for the sludge have been reported, ranging from 29 to 40 USD per ton [41]. A viable and cost-effective approach to address this issue is to treat anodizing waste as a valuable commodity, to extract alumina by calcining or drying the Anodizing Aluminum Waste (AAW) to utilize the $Al(OH)_3$ content. The chemical composition of the dried AAS varies depending on the aluminum industry generating it. Figure 6 provides an overview of the range of chemical composition of dried AAS, adapted from [39] and [21-27].

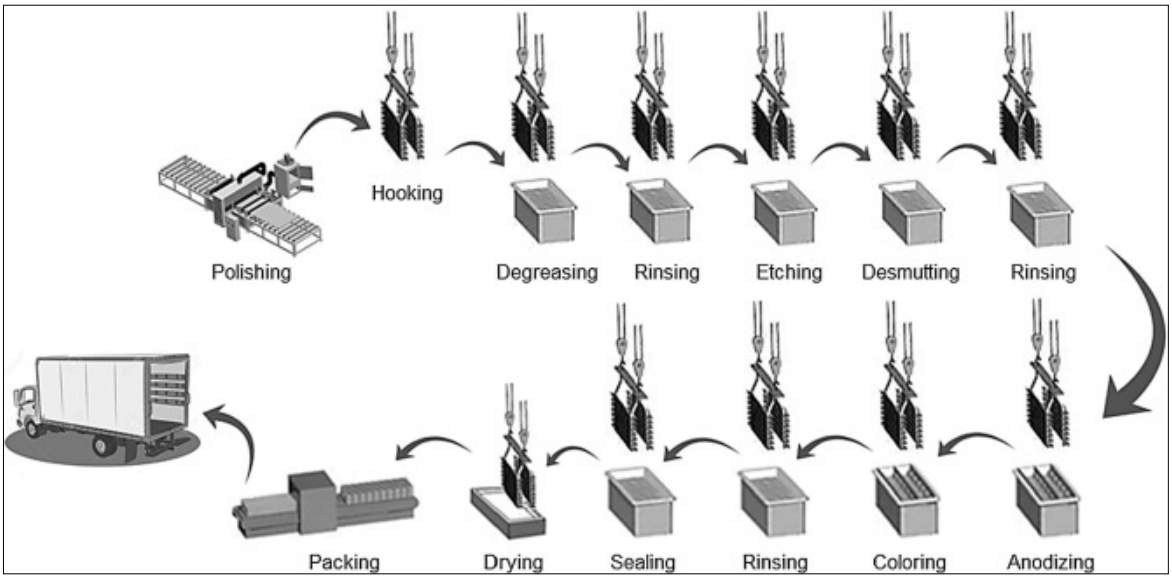

Figure 5 – Schematic picture of the anodization process of the aluminum alloy [39].

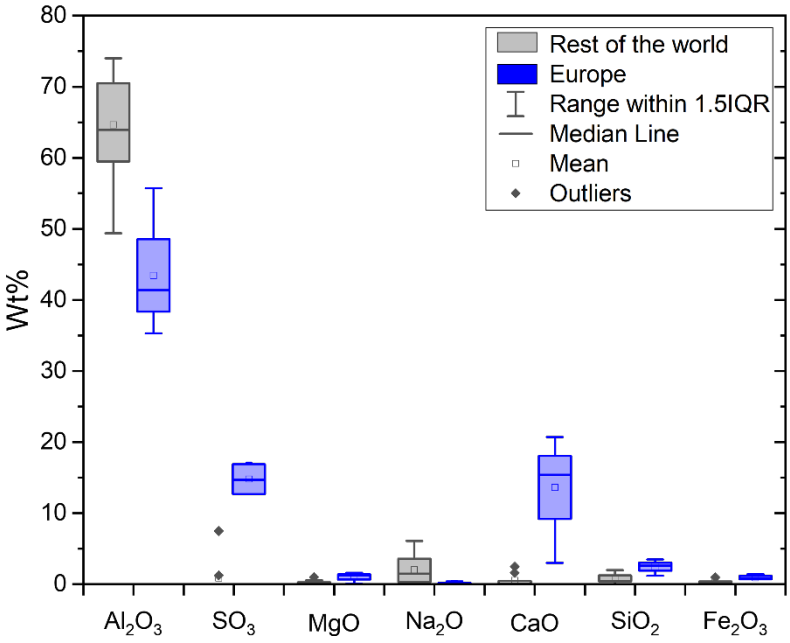

Figure 6 – Chemical composition of dried AAS, adapted from [39] and [21-27].

Based on their chemical composition, AAS can be a viable alternative to bauxite or pure alumina. Furthermore, the AAS generated in Italy contains notable quantities of calcium and sulfates, which can serve as potential sources of CaO and SO_3 for sulfo-aluminate cements. Table 5 provides an overview of the application of dried AAS in synthesizing sulfo-aluminate cement and maximizing the ye'elimite formation.

The complete replacement of bauxite or pure alumina with AAS is indeed feasible to achieve Al_2O_3 content in the clinker > 20 %. However, in the study by Costa et al. [21], the Al_2O_3 content of the clinker corresponded to 15.1 %. This is due to the high-volume incorporation of limestone (72.9 %) along with reagent grade chemicals i.e. Fe_2O_3 (1.1 %), $CaSO_4$ (7.3 %), and SiO_2 (0.2 %). Additionally, the AAS was supplemented with other Al_2O_3-containing residues, such as fly ash and bottom ash in the studies conducted by Rungchet et al. [24, 25] and Telesca et al. [26, 27]. Meanwhile Pace et al. [23] and Li et al.[22] adopted AAS as the sole Al_2O_3 source for synthesizing sulfo-aluminate cements.

Table 5 – Overview of AAS dosage, synthesis protocol, CaO, Al_2O_3, and SO_3 oxide composition of the clinker, and the $C_4A_3\hat{S}$ content of the sulfo-aluminate clinker

Ref.	**AAS dosage wt%**	**Complete replacement of bauxite or pure-Al_2O_3**	**Synthesis protocol**		**C, A, and $\hat{S}$ oxide composition of the clinker wt%**			**$C_4A_3\hat{S}$ wt%**
			Temp (°C)	Dwell (min)	C	A	$\hat{S}$	
Costa et al. [21]	18	Yes	1250	30	51	15	12	35
Li et al. [22]	42-45	Yes	1150	60	39	33	13	55
Pace et al.[23]	61	Yes	1300	120	39	36	19	68
Rungchet et al. [24]	25	Yes	Alkaline hydrothermal treatment + calcination (1050°C for 60 min)		47	26	12	76
Rungchet et al. [25]	25	Yes	Alkaline hydrothermal treatment + calcination (1050°C for 60 min)		48	26	12	75
Telesca et al. [26]	57.5	Yes	1300	120	42	31	16	58
Telesca et al.[27]	54-61	Yes	1300	120	41-42	29-32	15-17	53-62

AAS denotes aluminum anodization sludge. C denotes CaO, A is Al_2O_3 and $\hat{S}$ is SO_3.

2.3 Salt-slag by-product/ Non-Metallic Product (NMP)

During secondary aluminum production, the dross generated as a residue in the process constitutes a significant amount of metallic Al (10-50 %). To enhance the recycling efficiency of the dross, metallic Al is extracted in rotary salt furnaces through a remelting process by introducing flux into the melt, which aids in separating metallic Al from non-metallic impurities [47]. Moreover, the flux addition also lowers the melting point of oxides and protects molten Al from oxidation by creating a barrier on its surface. Combinations of NaCl and KCl serve as effective fluxing agents for the refining and recycling of aluminum from the dross. Following the melting process, the non-metallic components in the raw mix are fully absorbed by the liquid flux, which solidifies upon tapping and cooling to form what is known as salt slag or salt cake [48]. This salt slag is typically disposed in landfills. For every 1 ton of aluminum metal, 500 kg of salt slag is generated [43]. In terms of worldwide estimates, 5 million tonnes of this waste is generated annually [49]. These salt slags when disposed in landfills can come in contact with water or damp air, resulting in the formation of highly flammable gases such as hydrogen and thus classified as hazardous waste (Category H3-A and H4) in EU [49]. Therefore, end-of-life management of these wastes is becoming a challenge. A possible solution and alternative to the disposal of salt slag is the recovery of these slags to obtain insoluble salt residues, also known as the Non-Metallic Product (NMP). These residues constitute at least 60% of Al_2O_3, 3-14 wt.% of MgO, 3-12 wt.% SiO_2, 1.5-5 wt.% CaO, 1.5-3 wt.% Fe_2O_3 and minor amounts of TiO_2, Na_2O, MnO, K_2O and Cl^-< 1 wt% [50].The metallic part of the salt slag is removed by grinding and sieving whereas the water-soluble salts are separated by water leaching at selected temperature and pressure to dissolve the salts. The remaining fraction is filtered and evaporated to obtain the insoluble non-metallic part of the aluminum salt slag (NMP). These NMPs are marketed as Serox [37] and Paval by Befesa S.A, Valoxy by R.V.A, Noval by ALCAN [51], and Oxiton by B.U.S. Berzelius Umwelt-Service AG [52]. The chemical composition of the marketed NMPs is provided in Figure 7. Most of the marketed NMPs have significant Al_2O_3 content > 70 % with minor traces of MgO and SiO_2. This positions them as a viable alternative to pure alumina or bauxite for the production of sulfo aluminate cement. Moreover, the Cl content of these NMPs is < 0.1% which is below the limit for cementitious materials, according to EN-196-1. The MgO content in these NMPs is less than 10%. To ensure that it remains below 5% (the permissible limit for cementitious materials as specified by EN 196-1), acid leaching can be employed. This process converts MgO into soluble magnesium salts, which can then be separated through filtration and washing.

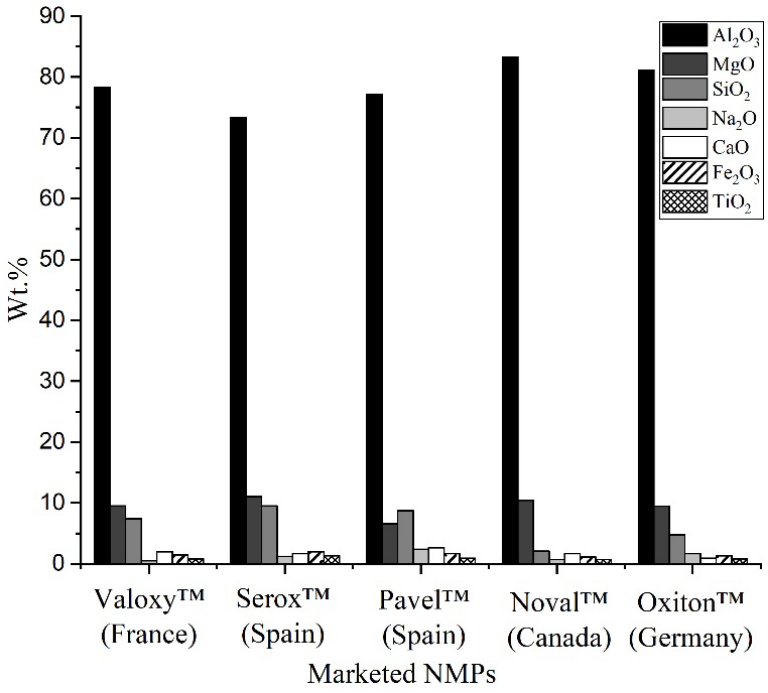

Figure 7 – Chemical composition of the marketed NMPs adapted from [51], [37, 52] and XRF results.

According to the literature, only Bayramtan et al.[37, 38] have synthesized sulfo-aluminate cement clinkers using NMP (Serox). In this study, Serox (16-23 %), ladle-furnace slag (14-33 %), ceramic waste (1.5-7 %), and glass waste (1-3 %) were incorporated in the raw-meal in which Serox, ladle-furnace slag (LFS) and ceramic waste were used as a source for Al_2O_3. The maximum $C_4A_3\hat{S}$ formation (48 %) was reported with 23 % Serox, 14 % LFS, 7 % ceramic waste, and 1 % glass waste when sintered at 1250°C for 90 min. Moreover, the presence of non-hydraulic phases was also reported such as fluorellestadite and merwinite due to the presence of MgO, F, and Cl. Table 6 provides an overview of the application of Serox in synthesizing sulfo-aluminate cement and maximizing the ye'elimite formation.

According to Table 6, the Serox and ceramic waste content needs to be at least 23 % and 7 %, respectively, to achieve an Al_2O_3 content ≥ 20 % in the clinker for achieving higher ye'elimite content more than 40 %.. This is due to the 66.4 % and 19.6 % contribution of Al_2O_3 by Serox and ceramic waste respectively in the raw meal.

Table 6 – Overview of NMP dosage, synthesis protocol, CaO, Al_2O_3, and SO_3 oxide composition of the clinker, and the $C_4A_3\hat{S}$ content of the sulfo-aluminate clinker synthesized by Bayramtan et al.[37].

Ref.	**NMP dosage wt%**	**Complete replacement of bauxite or pure-Al_2O_3**	**Synthesis protocol**		**C, A, and $\hat{S}$ oxide composition of the clinker wt%**			**$C_4A_3\hat{S}$ wt%**
			Temp (°C)	Dwell (min)	C	A	$\hat{S}$	
Bayramtan et al. [37, 38]	23	Yes	1250	90	44	20	16	48
Bayramtan et al. [37, 38]	16	Yes	1250	90	44	17	17	35
Bayramtan et al. [37, 38]	16	Yes	1250	90	43	16	19	33

NMP denotes non-metallic product (Serox in this study). C denotes CaO, A is Al_2O_3 and $\hat{S}$ is SO_3.

3. DISCUSSION AND CONCLUDING REMARKS

The utilization of these alumina-rich residues fosters an industrial symbiosis approach, facilitating collaboration between different industries to generate mutual economic and environmental advantages. The aluminium dross is the most explored substitute for pure alumina or bauxite in the production of CSA cements. One of the main reasons is the highest proportion of alumina present in the material in comparison to other oxides which correspond to < 10 wt%. Therefore, mimicking a pure alumina or bauxite would be the best. Moreover, the maximum ye'elimite reported was 68 % in the clinker when aluminium dross was incorporated as the sole source of alumina.

Pretreatment of aluminium dross can be costly and energy intensive before it can be incorporated as a raw material. Also, after pretreatment the presence of metallic Al is still above the acceptable limit for cement production. In the case of aluminium anodizing sludge, the composition of alumina can vary significantly and is not uniform depending on the location. Hence it can pose a challenge for incorporation in the raw meal of the clinker.

The non-metallic product from the salt-slag recycling is so far the best choice for the complete substitute of pure alumina or bauxite in the clinker production. The composition of these materials is uniform with less deviation between different sources. In addition, these materials pose the least heavy metal concentration and metallic alumina content in the material after the pretreatment of the salt slag. Nevertheless, these materials still find an obstacle in the valorisation of the cement clinker production due to the release of the organic chlorides when these materials are incinerated at high temperature. It is estimated that the dioxin present in the non-metallic product corresponds to ~ 35 ng/TEQ kg^{-1} based on technical data sheet of the serox product. This level is comparatively higher than the proposed limit by SINTEF of 10 ng /TEQ kg^{-1} in the residues used in the cement

clinker production [53]. These organic chlorides especially dioxins are among the most toxic chemicals known and have significant risks to human health (carcinogenic and skin damage) and the environment (contaminating ecosystems and wildlife). Eliminating dioxin from the residue would be a decisive step toward its integration as a raw material in the cement industry.

Hence more research and development on the degradation of dioxins and incorporation of this residue needs to be investigated to bring an alternative alumina source for CSA cement production into the market.

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