

## EVALUATION OF SECONDARY ALUMINIUM DROSS IN CALCIUM ALUMINATE CEMENT

### OVREDNOTENJE SEKUNDARNE ALUMINIJEVE ŽLINDRE V KALCIJEVEM ALUMINATNEM CEMENTU

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In this study, the evaluation of aluminium dross from secondary aluminium production in calcium aluminate cement was investigated. Salt compounds were removed significantly from the secondary aluminium dross by washing. Mixtures of washed dross and quicklime were prepared to obtain a cement additive by considering the standard cement with low alumina and they were sintered at 1250 °C for 5 h. The phases of mayenite, monocalcium aluminate, grossite, gehlenite, larnite, periclase and spinel were detected in the sintered samples using an X-ray diffraction analysis. Tests of the normal consistency, setting time and compressive strength were carried out at replacement levels of (2.5, 5, 7.5, 10 and 12.5) w/% of the cement additive to determine the physical properties of the cement paste/mortar. Increasing the level of the cement additive in the commercial calcium aluminate cement (ISIDAC 40) accelerated the setting time of the mortar and decreased workability.

Keywords: secondary aluminium dross, recycling, calcium aluminate, cement

V raziskavi je opisano ovrednotenje aluminijeve žlindre nastale pri predelavi sekundarnega aluminija, ki je bila uporabljena kot dodatek aluminatnemu cementu. Spojine soli v sekundarni aluminijevi žlindri so očistili z izpiranjem z vodo. Pripravljeno mešanico tako očiščene žlindre in živega apna so žgali 5 ur pri 1250 °C ter jo uporabili kot dodatek k standardnemu cementu z nizko vsebnostjo aluminija. Z rentgensko difrakcijsko analizo so ugotovili, da sintrana mešanica vsebuje majenit, mono kalcijev aluminat, grosit, gelenit, larnit, periklaz in špinel. Izvedli so teste konsistence, časa posedanja in tlačne trdnosti pri (2,5, 5, 7,5, 10 in 12,5) w/% sintranega dodatka in določili fizikalne lastnosti mešanice cementna pasta/malta. Naraščanje vsebnosti dodatka komercialnemu Ca-Al cementu (ISIDAC 40) je pospešilo čas posedanja malte in zmanjšalo njeno sposobnost za oblikovanje.

Ključne besede: sekundarna aluminijeva žlindra, recikliranje, kalcijev aluminat, cement

## 1 INTRODUCTION

Aluminium is widely used in various industries due to its conductivity, light weight and strong corrosion resistance. Nowadays, there are two different methods in aluminium production: primary production using the raw material (bauxite ore) and secondary production using waste products.<sup>1</sup>

Primary aluminium dross including aluminium products and scraps is re-melted in secondary aluminium production. The molten aluminium in the furnace is covered by a stream of molten salt, which absorbs the non-metallic compounds in the raw material. The dark-coloured top layer, called black dross, is removed by skimming before the molten metal is absorbed. Secondary aluminium slag mainly contains compounds of Al, O, Na, N, K, Mg, Si, Cl, F and small amounts of Ti, S, Mn, V and Fe.<sup>2,3</sup> The recycling and disposal of dross, released by the aluminium industry, is amongst the most challenging problems in the world. Dross is solid waste that can cause serious environmental pollution and is hazardous to public health. A large proportion of the released dross is disposed to landfills, causing a loss of valuable

metals and polluting ground water. About 20 % of this dross is black dross and the rest is white dross released from primary aluminium production. About 95 % of this waste is landfilled each year. It is declared that almost five million tons of white and black dross are produced worldwide per year. In addition, the reaction of aluminium dross with moisture or water vapour may produce dangerous flammable and poisonous gases such as CH<sub>4</sub>, NH<sub>3</sub>, PH<sub>3</sub>, H<sub>2</sub>, H<sub>2</sub>S, etc. For these reasons, it is necessary to create a recycling mechanism for the aluminium dross produced in these industries.<sup>4-9</sup>

Secondary aluminium production requires a lower energy consumption than primary aluminium production. The energy requirement of secondary aluminium production is only 5 % of the energy used for primary aluminium production.<sup>8,10</sup> The recycling and reuse of industrial waste and by-products are also very essential in cement and concrete production. Traditional by-products such as granulated blast furnace slag, fly ash or silica fume are evaluated as cementing materials in the cement industry. Besides, various wastes are generated in aluminium refining facilities.<sup>11</sup> Pereira et al.<sup>12</sup> investigated the mechanical behaviour of Portland cement mortars containing salt slags produced from the aluminium scrap re-melted in rotary furnaces. It was reported that the me-

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**Table 1:** Typical compositions of calcium aluminate cement (w/%)<sup>15</sup>

Type of cement	Al <sub>2</sub> O <sub>3</sub>	CaO	Fe <sub>2</sub> O <sub>3</sub> + FeO	FeO	SiO <sub>2</sub>	TiO <sub>2</sub>	MgO	K <sub>2</sub> O + Na <sub>2</sub> O	SO <sub>3</sub>
40 % Al <sub>2</sub> O <sub>3</sub>	40–45	42–48	< 10	< 5	5–8	~ 2	< 1.5	< 0.4	< 0.2
50 % Al <sub>2</sub> O <sub>3</sub>	49–55	34–39	< 3.5	< 1.5	4–6	~ 2	~ 1	< 0.4	< 0.3
50 % Al <sub>2</sub> O <sub>3</sub> (low Fe)	50–55	36–38	< 2	< 1	4–6	~ 2	~ 1	< 0.4	< 0.3
70 % Al <sub>2</sub> O <sub>3</sub>	69–72	27–29	< 0.3	< 0.2	< 0.8	< 0.1	< 0.3	< 0.5	< 0.3
80 % Al <sub>2</sub> O <sub>3</sub>	79–82	17–20	< 0.25	< 0.2	< 0.4	< 0.1	< 0.3	< 0.7	< 0.2

mechanical properties of concrete are suitable when up to 10 w/ % of cement is replaced by salt slag as compared with the control concrete. In addition, Yoshimura et al.<sup>13</sup> used aluminium dross as the raw material to replace calcined alumina. Moreover, Ewais et al.<sup>14</sup> studied the use of aluminium sludge and aluminium dross in the production of calcium aluminate cement.

Calcium aluminate cements (CACs) are named differently depending on their alumina (Al<sub>2</sub>O<sub>3</sub>) content and chemical composition. Typical compositions of calcium aluminate cement are summarized in **Table 1**.<sup>15</sup> The use of cement containing 40 % of alumina is higher than the use of cement with a high alumina content; it is preferred for specific characteristics such as abrasion resistance or refractoriness. The main clinker phases of calcium aluminate cement (CAC) are CA (monocalcium aluminate: CaAl<sub>2</sub>O<sub>4</sub>), C<sub>12</sub>A<sub>7</sub> (mayenite: Ca<sub>11</sub>Al<sub>17</sub>O<sub>33</sub>) and CA<sub>2</sub> (grossite: CaAl<sub>4</sub>O<sub>7</sub>). Calcium aluminate cement with 37–54 % of Al<sub>2</sub>O<sub>3</sub> contains a substantial amount of C<sub>2</sub>AS (gehlenite: Ca<sub>2</sub>Al<sub>2</sub>SiO<sub>7</sub>), which is not hydrated. The mayenite phase (C<sub>12</sub>A<sub>7</sub>) reacts quickly with water and plays a significant role in the hydration mechanism. The reaction of the grossite phase (CA<sub>2</sub>) with water is quite slow at ambient temperature. It can be accelerated by increasing the temperature.<sup>16,17</sup>

Calcium aluminate cement (CAC) is the preferred product in various industries due to its superior properties such as fire resistance, abrasion resistance, scour resistance and acid resistance.<sup>18</sup> Also, it allows concrete applications in cold weather, being able to gain durability even at temperatures below 0 °C. In recent years, CAC has been widely used with high-priced agents as repair materials.<sup>19</sup>

The main purposes of this work are to investigate and evaluate secondary aluminium dross as an additive material in commercial calcium aluminate cement and to contribute to the recycling of these wastes in the cement industry.

## 2 EXPERIMENTAL PART

### 2.1 Materials and method

Secondary aluminium dross used in this study was evaluated as a source of Al<sub>2</sub>O<sub>3</sub>. It was supplied from Sahinler Metal Co., Turkey. Calcium oxide (quicklime) was used to increase the CaO content of the dross. Calcium oxide was supplied from Nuh Construction Prod-

ucts Co., Turkey. The dross was ground for 30 min at 1300 min<sup>-1</sup> using a Mertest LB200 ring mill. After grinding, the particle size of aluminium dross was measured with a Malvern-Mastersizer 2000 and it was ground to a size below 100 µm. A chemical analysis was carried out using a Rigaku D/MAX/2200/PC X-ray fluorescence (XRF) spectrometer. The quicklime used in this study contained 99 % CaO and 1 % MgO. The elemental composition of secondary aluminium dross obtained with the XRF analysis is given in **Table 2**. The elemental analysis and XRD indicate that unwashed dross contains mainly Al<sub>2</sub>O<sub>3</sub> (37.47 %) and salts like NaCl and KCl.

**Table 2:** XRF elemental analysis of secondary aluminium dross

Elemental composition	Amounts (w/%)
Al (Al <sub>2</sub> O <sub>3</sub> )	19.98 (37.47)
Na	15.88
K	3.77
Cl	22.57
Ca	1.41
Fe	1.94
Mg	1.72
Si	0.41
S	0.22
Others (Ti, Cr, Mn, Cu, Zn, Ba)	4.97
O	balance

**Table 3:** Chemical composition of ISIDAC 40

Chemical composition	Amounts (w/%)
SiO <sub>2</sub>	3.60
Al <sub>2</sub> O <sub>3</sub>	39.80
Fe <sub>2</sub> O <sub>3</sub>	17.05
CaO	36.20
MgO	0.65
SO <sub>3</sub>	0.04
Loss on ignition (Na <sub>2</sub> O + K <sub>2</sub> O)	0.30
Cl	0.16
S	0.0090
	0.01

The sintered samples consisting of washed aluminium dross and calcium oxide, recognised as the cement additive, were ground for 20 min at 600 min<sup>-1</sup> using a planetary mono mill. In this process, 40 tungsten carbide (WC) balls with a diameter of 10 mm were used in a 250 mL WC bowl and the ball-to-sample weight ratio

was chosen to be 20. The grinding process was dry. The commercial calcium aluminate cement (ISIDAC 40) produced by Çimsa Cement Co. (Turkey) in accordance with the EN 14647 standard<sup>20</sup> was used as the cement material. The chemical composition of ISIDAC 40 is given in **Table 3**.

## 2.2 Sample preparation

In order to carry out the experiments, cement mixtures were prepared by adding the cement additive to the commercial calcium aluminate cement (ISIDAC 40) with different proportions. The details of the mixture proportions are illustrated in **Table 4**. Silica-based CEN standard sand with a maximum grain diameter of 2 mm prepared in accordance with the EN 196-1<sup>21</sup> standard was used as the aggregate in order to obtain mortar mixtures. It was supplied from Limak Co. (Turkey).

**Table 4:** Mixture proportions (w/%)

Mix code	1	2	3	4	5	6
ISIDAC 40	100	97.5	95	92.5	90	88.5
Cement Additive	–	2.5	5	7.5	10	12.5

The setting time experiments with the prepared cement paste mixtures were carried out using a Toni Technic automatic setting time tester at an ambient temperature of  $(20 \pm 2)$  °C and 50–60 % relative humidity according to the requirements of the EN 196-3<sup>22</sup> standard. Water was added to 500-g cement samples taken from each cement mixture to get enough workability and then the setting time tests were carried out.

Compressive strengths were measured with prismatic moulds with dimensions of  $(40 \times 40 \times 160)$  mm. Mortar preparation was conducted according to the EN 14647 and EN 196-1 standards. The specimens of mortar were prepared by mixing 1350 g of CEN standard sand (silica-rich) with 500 g of cement (mixing 1–6) and 200 mL of water. For all the mixtures, the water-to-cement ratio (w/c) was kept constant at 0.4. These measurements were carried out after curing periods of 6 h and 24 h using the Toni Technic testing machine. All the specimens were kept in the curing chamber with a temperature of  $(20 \pm 1)$  °C and a minimum relative humidity of 90 %. All the specimens were demoulded after 6 h and the specimens to be tested after 6 h were tested immediately after demoulding. The specimens to be tested after 24 h were stored in the curing chamber after demoulding.

## 3 RESULTS AND DISCUSSION

In the studies on aluminium dross, it was found that a high content of aluminium oxide was present in the dross and that the main phases were aluminium metal,  $\text{Fe}_2\text{O}_3$ ,  $\text{SiO}_2$ ,  $\text{Na}_2\text{O}$ ,  $\text{AlN}$ ,  $\text{MgO}$  while the dross also contained small amounts of Ti, S, Mn, V and Fe compounds. In addition, it was reported that salts such as NaCl or KCl and

small amounts of the chloride, nitride and fluoride phases with different compositions were encountered.<sup>23–25</sup> Aluminium dross was subjected to water washing for 30 min and 60 min to remove the high chlorine content. Results of the XRF elemental analysis of secondary aluminium dross after water washing are given in **Table 5**. It was observed that the amount of chlorine in the compound was removed by increasing the washing time. After washing the aluminium dross for 60 min, the alumina content of the dross increased from 37.74 % to 68.03 % and the chlorine content of the dross decreased from 22.57 % to 0.033 %.

**Table 5:** XRF elemental analysis of the washed secondary aluminium dross (w/%)

Chemical composition	Washing for 30 min	Washing for 60 min
Al ( $\text{Al}_2\text{O}_3$ )	30.61 (57.82)	36.02 (68.03)
Na	6.60	0.07
K	1.66	0.71
Cl	9.60	0.033
Ca	2.50	3.84
Fe	1.85	2.11
Mg	2.34	2.89
Si	0.56	0.756
Ti	0.665	1.51
S	–	–
O	balance	balance

The amount of calcium oxide in the washed aluminium dross is not high enough to form calcium aluminate clinker phases of the cement additive. CaO was added to 60 min-washed aluminium dross with a CaO/dross ratio of 3/5 and mixed in for 10 min. The purpose of using this ratio was to obtain composition values close to EN 14647. The composition of the cement additive and EN 14647 limits are given in **Table 6**.

**Table 6:** Chemical composition of cement additive and EN 14647 limits (w/%)

Chemical composition	Amounts	EN 14647 limits
$\text{SiO}_2$	1.01	–
$\text{Al}_2\text{O}_3$	42.51	35-58
$\text{Fe}_2\text{O}_3$	1.89	–
CaO	40.48	–
MgO	3.37	–
$\text{TiO}_2$	1.57	–
$\text{Na}_2\text{O}+\text{K}_2\text{O}$	0.4	max. 0.40
$\text{SO}_3$	–	max. 0.50
S	–	max. 0.10
Cl	0.020	max. 0.10

In some studies on commercial low-alumina cements, it was reported that cement clinker phases consisted of the CA,  $\text{CA}_2$ ,  $\text{C}_2\text{AS}$ ,  $\text{C}_2\text{S}$ ,  $\text{C}_{12}\text{A}_7$  phases.<sup>26–30</sup> According to the XRD analysis of the produced cement additive shown in **Figure 1**, the sample sintered at 1250 °C for 5 h was

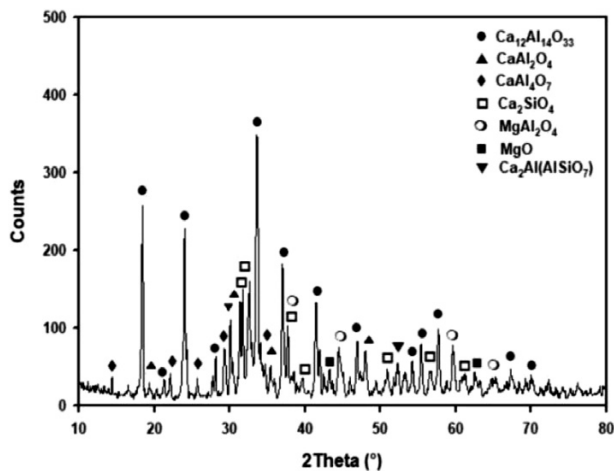


Figure 1: XRD analysis of the cement additive sintered at 1250 °C for 5 h

composed of the  $Ca_{12}Al_{14}O_{33}$  (mayenite- $C_{12}A_7$ ),  $CaAl_4O_7$  (grossite- $CA_2$ ),  $CaAl_2O_4$  (monocalcium aluminate- $CA$ ),  $Ca_2SiO_4$  (larnite- $C_2S$ ) and  $Ca_2Al(AlSiO_7)$  (gehlenite- $C_2AS$ ) phases. Along with these phases, there were small quantities of  $MgAl_2O_4$  (spinel- $MA$ ) and  $MgO$  (periclase) due to the high Mg content in the dross.

D10, D50 and D90 sizes corresponding to the particle sizes at the 10 %, 50 % and 90 % points on the cumulative distribution for the cement additive were found to be 2.05  $\mu m$ , 14.38  $\mu m$  and 39.52  $\mu m$ , respectively. It was realized that the particle size was suitable for the preparation of the cement mixture. The results of the particle size analysis are supported by the SEM image of the cement additive, given in Figure 2. It is proposed to use the cement additive in various percentages of cement. Figure 3 presents the normal consistency obtained for various percentages of the cement additive. It was observed that the workability decreased and the water requirement increased depending on the increase in the cement additive proportion in the paste. While the water requirement was 26 % for the commercial cement, it was 35.3 % for the commercial cement with 12.5 % amount

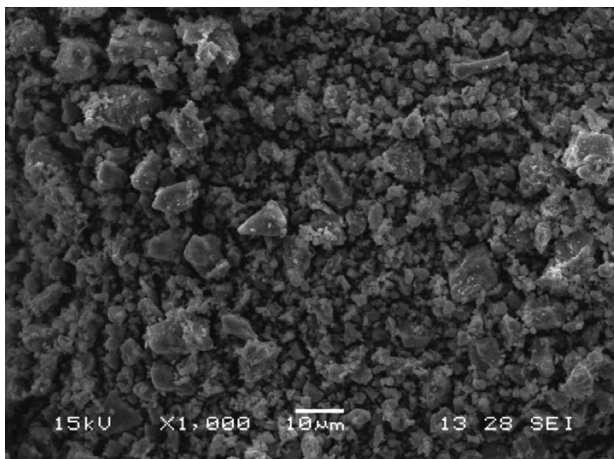


Figure 2: SEM image of the cement additive

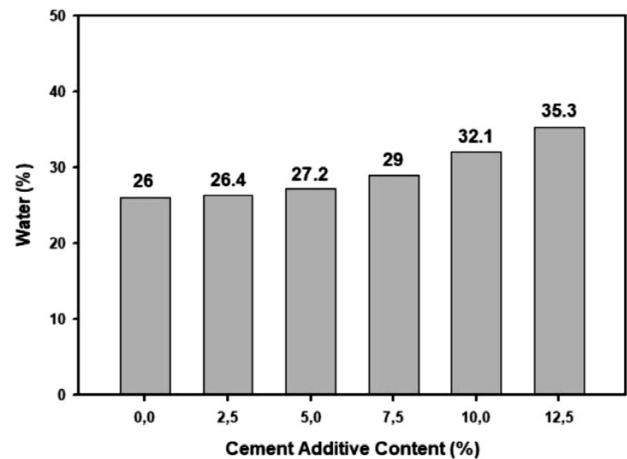


Figure 3: Variation in the normal consistency with the replacement of cement by the cement additive

of the cement additive. The percentage of the water to be added to the mix was greater for larger amounts of the cement additive.

The setting times of the cement pastes at different replacement levels are shown in Figure 4. The increasing cement additive content accelerates the hardening. According to the EN 14647 standard, the initial setting time should not be less than 90 min. It is seen from the results obtained for all the mixtures that the setting times are within the standard requirements.

According to the EN 14647 standard, the compressive strength of calcium aluminate cement, tested in accordance with the EN 196-1 standard, should not be less than 18 MPa after 6 h and 40 MPa after 24 h. For our study, the compressive strength at different replacement levels is shown in Figure 5. It was found that the increasing amount of the cement additive in the mixture adversely affected the mechanical properties. In the cement mixtures prepared with 0–12.5 w/% cement additives, the compressive strength of the concrete decreased by 65.2 % for the specimens kept for 6 h and by 58.9 %

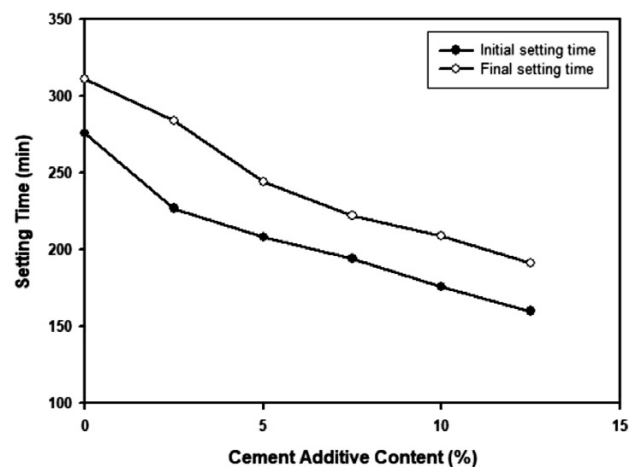


Figure 4: Relationship between the setting time and cement additive content



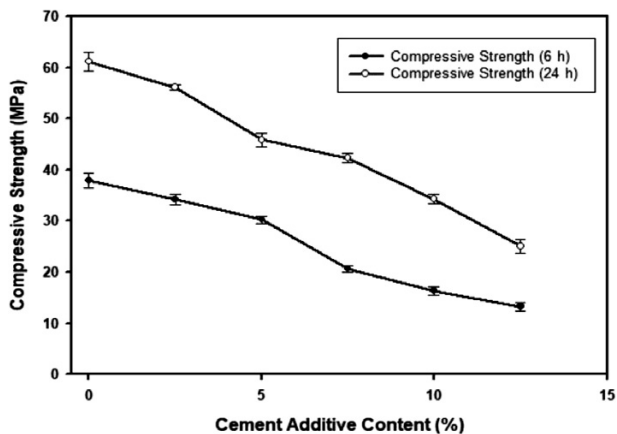


Figure 5: Relationship between the compressive strength and cement additive content

for the specimens kept for 24 h. The 7.5 w/% replacement of the commercial cement with the cement additive is selected as the optimum cement mix since it meets the requirements of the international standard.

Arimanwa et al.<sup>31</sup> investigated the effect of aluminium waste, as a supplementary cementitious material, on concrete properties. They found that the addition of aluminium dross decreased both the initial and final setting times of the cement. They also found that workability was decreased due to the water absorbed by aluminium dross from the mix. Nduka et al.<sup>32</sup> investigated the influence of aluminium dross on the mechanical and durability properties of sandcrete blocks. The water absorption was found to be increased with the increased replacement percentage of dross. Ozerkan et al.<sup>33</sup> found that the setting time was decreased up to the 15 % replacement of cementitious material.

Aluminium dross was suggested to be used in the production of eco-concrete in the study by Javali et al.<sup>34</sup> They produced several mixtures of Portland cement, aluminium dross, sand and granular iron slag. For aluminium dross, different replacement levels were considered: (5, 10, 15 and 20) w/%; the optimal level was determined to be 5 w/%. In another study by Trinet,<sup>35</sup> an increase in the amount of aluminium dross mixed into Portland cement caused a decrease in the compressive strength. These results were verified in studies by Reddy and Neeraja and Ozerkan et al.

## 5 CONCLUSIONS

It is possible to use secondary aluminium dross as the material added to cement with low alumina after washing and sintering it with quicklime at optimum conditions. The use of a high volume of the cement additive prepared from aluminium dross is not appropriate because of its high water-absorption capacity.

An increase in the cement additive content causes a decrease in both the initial and final setting times of the cement. This may be due to a higher surface area of the

cement additive. The shortening of these times reduces the workability of the mortar and therefore the addition of the cement additive should be limited. The compressive strengths decline with the increasing cement additive proportion in the cement mortar. Replacements of the commercial cement by the cement additive of up to 7.5 w/% provide for the compressive strengths of the concrete determined by the international standard requirements.

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