



# Recycling of desulfurization slag for the production of autoclaved aerated concrete



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## HIGHLIGHTS

- Desulfurization slag has the potential to produce autoclaved aerated concrete.
- The foaming of mortars is retarded due to the insufficient alkalinity.
- Using 0.17 M NaOH<sub>(aq)</sub> or calcining desulfurization slag can improve mortar foaming.
- Improving foaming results in the products with stable physical properties.
- The formation and morphology of tobermorite are affected by the slag added.

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## ABSTRACT

The purpose of this study was to reuse desulfurization (De-S) slag in the production of autoclaved aerated concrete (AAC). The non-magnetic fraction of De-S slag is rich in calcium and silicon compounds, and therefore suitable to be an alternative raw material for AAC production. The calcium compounds in the De-S slag used in this study were Ca(OH)<sub>2</sub>, CaCO<sub>3</sub>, γ-Ca<sub>2</sub>SiO<sub>4</sub>, and CaF<sub>2</sub> rather than CaO, and they affected the foaming of AAC mortars due to the insufficient alkalinity. Using 0.17 M NaOH<sub>(aq)</sub> to replace water or calcining the De-S slag in advance both improved the foaming of mortars, and thus obtained AAC products with stable properties. The formation and morphology of tobermorite in AAC were altered by the addition of De-S slag, probably due to the foreign ions introduced by the slag. The changes in tobermorite should also be related to the decline in the compressive strength of AAC specimens.

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## 1. Introduction

Iron- and steel-making is fundamental to modern society, because it provides the raw materials for buildings, vehicles, and various articles for daily use. In an integrated steel mill, iron is extracted from iron ores and then refined into steel, and at the same time several kinds of slags are generated in different processes. During steelmaking, the liquid iron is treated in order to meet the sulfur specification of steel (typically <0.03%), and a desulfurizer, which is normally composed of lime (CaO, ~90%) and fluorspar (CaF<sub>2</sub>, ~10%), is used in the secondary refining process. A layer of slag floating on the liquid iron then forms, and sulfur is removed from the liquid iron by a slag-liquid metal reaction described in Eq. (1) [1,2].



Afterwards, the resulting slag, called desulfurization slag (abbreviated to De-S slag), is discharged as a waste and then transported to treatment facilities. Some studies [3,4] reported that De-S slag is primarily composed of excessive lime and entrapped iron, and the desulfurization products (CaS and Al<sub>2</sub>O<sub>3</sub>), residual fluorspar, and graphite are also present. Although De-S slag normally contains considerable amount of iron and iron oxides, it is not often recycled in steel manufacturing because of the high sulfur content, so reusing DS in cement raw materials, agricultural applications, or wastewater treatments [2,5,6] is a much more feasible and common approach. In Taiwan, about 400,000 tons of De-S slag are generated annually, and most of this is reused in backfilling and controlled low-strength materials, recycling methods that have few economic benefits and could harm the environment.

De-S slag usually has small particle sizes, with Sheng et al. [2] reporting that particles smaller than 0.15 mm accounted for over 60% of the material. Many studies indicate that the major constituents of De-S slag normally include iron (Fe) and iron oxides (FeO, Fe<sub>2</sub>O<sub>3</sub>, and Fe<sub>3</sub>O<sub>4</sub>), calcium oxide (CaO), silicon dioxides

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(SiO<sub>2</sub>), aluminum oxide, and magnesium oxide (MgO) [3,7,8], and Hwang [9] stated that the metallic iron accounted for 54.1 vol% of the total iron in De-S slag. Ma and Houser [7] reported that the use of weak magnetic separation coupled with selective particle size screening can effectively separate the iron-rich particles from De-S slag, and so obtain a product with low levels of impurities for reuse. After removing iron-rich constituents, the amount of CaO in De-S slag can be increased to 48–69 wt% [8,10], and some researchers use such slag to replace fine aggregate in cement-based concrete materials [10,11]. However, Ho et al. [10] reported that the level of volume expansion of a cement mortar increased along with the amount of De-S slag used, and many studies have suggested that the volume expansion caused by free CaO and MgO from a slag can raise serious concerns with regard to the resulting properties of a construction material [12–14].

While De-S slag has high potential to be reused as a construction material based on its main components, characteristics such as its high powder content and potential volume expansion severely restrict its use. Nevertheless, it could be suitable to reuse De-S slag in the production of autoclaved aerated concrete (AAC), because the disadvantages listed above can be reversed and profitably utilized. AAC is an inorganic building material that is traditionally made from lime powder (5–30 wt%), silica sand (60–70 wt%), cement (5–35 wt%), and water [15–17]. A chemical foaming process is normally used to make AAC lightweight, and an autoclave curing process is employed to drastically increase the mechanical strength of AAC. Under the high-pressure steam conditions in an autoclave, CaO can react with SiO<sub>2</sub> and H<sub>2</sub>O to form crystalline hydrates, namely tobermorite, and this reaction contributes to the increase in mechanical strength of AAC [18,19]. Since De-S slag is rich in CaO and SiO<sub>2</sub> and has small particle sizes, it should be able to directly replace lime powder and silica sand to produce AAC, and the free CaO would be consumed during the autoclave curing, thus eliminating the problem of volume expansion. In addition, AAC has several functional properties, such as thermal insulation, fire resistance, and soundproofing, which make it a high-value building material [17], and hence reusing De-S slag to produce AAC can not only promote resource recycling, but also obtain valuable products.

However, several studies have noted that using industrial wastes as AAC raw materials, such as iron and copper tailings, blast furnace slag, and coal bottom ash, may affect the properties and microstructures of the resulting AAC products [15,20–23]. Huang et al. [20] used blast furnace slag and copper tailings as AAC raw materials, and found that some foreign ions, such as Mg and Al, changed the morphology and microstructures of the AAC products. Cai et al. [15] reused iron tailings for AAC production and reported that an increase in the amount of these wastes reduced the compressive strength of AAC, a result that is attributed to the decrease in the amounts of calcium silicate hydrates (C-S-H) and tobermorite. While De-S slag seems suitable for AAC production, little is known about its influence on the process parameters and product properties. Accordingly, the aim of this research is to partially replace lime and silica sand with De-S slag for AAC production, and to examine the effects on the foaming process and the properties of the resulting AAC products.

## 2. Materials and methods

### 2.1. Materials

The De-S slag used in this study was generated by an integrated steel mill in Kaohsiung, Taiwan. Hot De-S slag was carried with slag pots and transported to a treatment yard, and then water was poured to cool the slag. Afterwards, the cooled De-S slag

was crushed, sieved, and magnetically separated in a slag treatment plant in order to recover any large chunks of iron. The residues from the slag treatment plant were sampled and used as the research subject in this study. The moisture and ash contents, loss on ignition (LOI), magnetic and non-magnetic proportions, particle size distribution, and chemical composition of the treated De-S slag were analyzed immediately after sampling.

### 2.2. Preparation of AAC specimens

The preparation of AAC specimens included three basic steps, as follows:

#### (1) Raw material mixing

First, lime (CaO, Merck, greater than 97%), silica (SiO<sub>2</sub>, Alfa Aesar, 99.5%), cement (ordinary Portland cement, ASTM Type I), and the De-S slag were precisely weighed in accordance with the experimental design (Table 1). Because the De-S slag already contained some CaO and SiO<sub>2</sub>, the amounts of lime and silica added were adjusted according to the chemical composition of the slag, thus keeping the Ca/Si ratio of the raw mixes constant. The mix proportions of the raw materials of the AAC specimens are listed in Table 1. These raw materials were then put into a steel bowl and mixed with an electric blender until the mix had become homogeneous.

#### (2) Foaming and pre-curing

In order to make AAC mortars foam, aluminum powder (Alfa Aesar, 99.5%) was used as a foaming agent, and the amount added was 0.5 wt%. Water or a NaOH aqueous solution (0.17 M) was then added into the raw mixes at a specific water-solids ratio of 0.70 L/kg, and then blended to mortars as soon as possible using an electric blender at about 120 rpm. The mortar was poured into cast-iron molds (5-cm cubes) and then stood for foaming. After standing for 30 min the foaming of the mortars was finished, and the materials bulging out of the molds were then scraped off. The molds with AAC specimens were immediately put into a moist closet for pre-curing, and the relative humidity and temperature were set at 95% and 25 °C, respectively.

#### (3) Autoclave curing

After pre-curing in the moist closet for 24 h, the molds were removed and the hardened specimens were obtained. In the final step, an autoclave with a proportional-integral-derivative controller was employed to complete the hydration reactions of the hardened specimens, and thus AAC products were obtained. The steam pressure in the autoclave was controlled at 12 atm (the corresponding temperature was about 189 °C), and the curing time was 16 h.

### 2.3. Material testing and analysis methods

After sampling, the moisture and ash contents and LOI of the De-S slag were immediately analyzed. The LOI value was

**Table 1**  
Mix proportions of raw materials of AAC specimens.

Raw mix	Weight proportion (wt%)			
	Silica	Lime	Cement	De-S slag
Reference	70.0	25.0	5.0	0.0
DS-05	68.1	21.9	5.0	5.0
DS-10	66.2	18.8	5.0	10.0
DS-15	64.3	15.8	4.9	15.0
DS-20	62.4	12.7	4.9	20.0
DS-30	58.6	6.5	4.9	30.0
DS-40	54.8	0.4	4.8	40.0

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