

Creating High-Value Eco-Friendly Materials from Industrial Coal Combustion Ash

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Abstract

Thailand is one of the largest industrialized countries in Southeast Asia and its coal-combustion ashes are mainly generated for either energy utilization or manufacturing purposes. Being one of the leading industrial conglomerates in Thailand and ASEAN, SCG is committed towards sustainable care for the environment and communities via resource and waste management system. In order to significantly reduce the conventional landfilling of coal combustion ashes as our entire organization is expected to achieve the target of "Zero-waste to Landfill" by 2012, we have developed a number of high-value eco-friendly materials for building construction applications. This paper will discuss on the present state of our industrial coal combustion ash and the challenges to effectively use coal ashes as conventional building materials. Some of the novel technical solutions to transform these waste ashes into high-value eco-friendly materials such as commercial-grade sodium silicate, zeolites A and lightweight aggregates for precast hollow-core concrete applications are further presented here.

Keywords: coal ash, sodium silicate, zeolite A, lightweight aggregate, precast concrete.

1 Introduction

Cleaner production concepts regarding activities including resource use minimization through resource recovery and reuse, eco-efficiency, and source reduction which have positive effects on the environment have been widely used in various industries including cement manufacturing [1-3] and pulp and paper industries [4-6]. Concerning these concepts, the utilization of coal ash which is a major by-product of coal-fired power plants through resource recovery has been focused by many researchers [2, 7, 8].

In Thailand, conventional electric power generators that consume fossil fuels generate more than 4 million tons of coal ash annually and less than half of it is accounted for recycling purposes. So far, the conventional approach to recycle the bulk volume of fly ash is at best, in cement and concrete products. Until now, most researchers focused on characterizing the physical performance and effects in using coal ash as pozzolanic materials for cement and concrete [9-11]. Unfortunately, this could only resolve a small fraction of the huge volume of unused fly ash. Henceforth, these fly ashes are ultimately disposed in the landfills which often pose as a serious environmental hazard.

The synthesis processes of zeolite from coal fly ash can be simply classified into direct and indirect synthesis. In the direct synthesis, silica and alumina are initially extracted from coal fly ash by the

mean of alkaline extraction under high temperature, hence resulting in the mixture of silicate and aluminate extracts which can be considered as the precursors for the zeolite synthesis. However, due to a large variety in composition of coal fly ash, the direct synthesis approach has primarily resulted in the mixed phases between zeolites and the remaining solid oxide contaminants.

Many researchers have focused on the use of coal ashes for zeolite synthesis. Wide variety of zeolites including zeolite A, phillipsite and faujasite and GIS-type zeolites were synthesized from low-rank lignite fly ash containing high amount of amorphous phases. Whereas a group of zeolites including zeolite X, phillipsite and hydroxylcancrinite were synthesized from higher rank coal fly ashes [12]. It was clearly seen that the zeolite synthesis and the structure types of zeolites are strongly affected by factors including the Si/Al molar ratio, hydrothermal treatment temperature and pressure, the presence of the structure-directing agent; type of alkaline solution, pH of mixture; and solid oxide contaminants [13-15].

Current trends have shown that the advances in the building design and materials towards environmental sustainability will determine the next generation of construction technology. From the initial design phase, architects and specialists are seeking for more ecological-friendly materials which promise to reduce energy consumption and carbon dioxide emission while the cement and concrete producers attempt to incorporate more supplementary cementitious materials (SCMs) such as industrial fly ash and ground granulated blast furnace slag (GGBS) into their final products.

Among the different types of concrete available in the market today, lightweight aggregate concrete (LWAC) is probably one of the most sustainable concrete. Over the years, LWAC has been investigated extensively and some of the well-demonstrated applications include: tall buildings, long-span bridges and off-shore floating structures [16-18]. Simply by replacing the normal weight aggregates with lightweight aggregates, a wide range of environmental-related issues in the construction industry can be specifically addressed and these include: a) Reduction in dead load which correspondingly results in lesser steel reinforcement, b) Reduced structural and column sizes which provides for more space availability, c) Improved thermal [19] and acoustic insulation for better living comfort [20] and d) Enhanced fire-retardation for security [21].

In this paper, our primary objective is to present two separate approaches to produce high-value eco-friendly materials - sodium silicate, zeolites A and artificial lightweight aggregate (LWA) from the combustion by-products of industrial coal ash, also commonly known as fly ash and bottom ash. The physical and chemical properties of the bottom ash-derived sodium silicate and zeolites are benchmarked against their commercial-grade counterparts while the fly-ash derived lightweight aggregate are characterized in accordance to the ASTM standards. The use of these eco-friendly materials in concrete applications is also presented here.

2 Materials and Methods

2.1 Industrial Coal Ash

Sub-bituminous coal fly ash and bottom ash are obtained from the Stoker boilers within our paper factories in Ratchaburi, Thailand. The ashes were separately ground and sieved to the particle size of smaller than 200 mesh (mean particle size <0.074 mm) and kept in the desiccator before use. Chemical compositions and crystallography information of the coal ashes were determined by using X-

ray Fluorescence Spectroscopy (XRF: ARL, 9400) and X-ray Diffraction Spectroscopy (XRD: Philips, X'Pert, Cu- α radiation), respectively (Table 1).

2.2 Synthesis of Zeolites Type A

Due to the interference of zeolite A formation caused by metal oxide contaminants, mainly Fe_2O_3 , CaO , and MgO ; the iron oxide (hematite) was primarily removed from raw fly ash by physical magnetic separation. Subsequently, the remaining metal oxide contaminants were removed from fly ash by the mean of acid treatment. In this stage, fly ash was refluxed with 3 M hydrochloric acid solution (9 g of hematite-free fly ash/120 ml of HCl solution) at 100°C for 6 h. The solid product obtained from this stage, consisted mainly of silica (97.7% SiO_2). The solid product was reacted with sodium carbonate to form sodium silicate at 900°C for 1 h by using the molar ratio of silica: sodium carbonate of 1:1. The obtained sodium silicate was subsequently dissolved in distilled water to produce the silica precursor, sodium silicate solution (21 wt.% SiO_2).

In order to synthesize zeolite A from sub-bituminous coal fly ash at the bench-scale order (0.5 kg of dry solid product), fly ash derived sodium silicate solution was used as the silica precursor. The certain amount of sodium aluminate was slowly added to the sodium silicate solution, followed by slow stirring at 40°C in a water bath while the pH of mixture was maintained at 11. The obtained mixture was transferred to a Teflon-lined autoclave and hydrothermally heated at 120°C for 4 h. The solid product was washed thoroughly with distilled water, dried in air at 100°C for 12 h, and then calcined at 540°C under atmospheric conditions for 6 h. In the case of the bench-scale synthesis of zeolite A from sub-bituminous coal bottom ash, a similar process to that of the fly ash was applied except that the bottom ash-derived sodium silicate solution was used as the silica precursor.

2.3 Production of Lightweight Aggregates

The chemical properties of the LWA produced from the combustion by-products of coal are shown in Table 4 and it was used directly as-received. To form the fresh pellets, liquid binder was sprayed onto the homogenous mixture during the pelletization process. Prior to high-temperature sintering, the fresh pellets were pre-heated slowly until it achieved a sintering temperature of 1000°C . This sintering process removed off any existing organic and volatile remnants within the dried pellets and formed a semi-liquid phase on the exterior surface of the dried pellets to seal off any open pores. When the elevated temperature was lowered to ambient conditions, the exterior surface of the synthetic lightweight aggregate (LWA) is solidified, harden and chemically inert while the interior structure remains structurally strong to resist high compressive load.

2.4 Lightweight Hollow-Core Precast Concrete

The chemical composition of the cement and mix design used to prepare the precast concrete samples is shown in Table 5 and Table 6, respectively. The 1-day and 28-days compressive strength of the precast concrete samples were conducted in accordance to BS EN 12390-3. For drying shrinkage tests, three samples each measuring 75mm x 75mm x 285 mm were prepared and cured in a controlled environment at 23°C with humidity of 50%, followed by testing it according to ASTM C157. Three concrete cube samples each measuring 150mm x 150 mm x 150 mm were prepared, moist-cured and subsequently subjected to abrasion-resistance tests as stipulated in ASTM C944.

Vertical heat flow measurement was conducted on precast concrete panels measuring 50cm x 50cm with a thickness of 5cm by using a Heat Flow Meter (Netzsch Instruments, HFM 436 Lambda). The thermal analysis is based on single-sided mode measurement of the steady-state heat flux through the precast concrete panels according to ASTM C1779. Infrared images of the bio-lightweight aggregates and precast concrete samples were captured by high resolution thermographic camera (InfraTec GmbH, VarioCAM) and subsequently analysed with software package IRBIS® (InfraTec GmbH).

3 Results and Discussion

3.1 Synthesis of Zeolites A

3.1.1 Indirect synthesis: preparation of zeolite A precursor from sub-bituminous coal fly ash

The formation of zeolite A from fly ash can be interfered by numerous factors [22], including the very stable silica content in the form of α -quartz in fly ash and the competitive formation of sodalite octahydrate under the higher alkaline concentration [23]. In addition, the presence of metal oxides contaminants could play significant role on the competitive formation of other zeolite types during the formative stages of the zeolite gel. Therefore, the synthesis of zeolite A from subbituminous fly ash was modified by primarily purified fly ash followed by the synthesis of zeolite A using the 2-step process via sodium silicate preparation and zeolite A synthesis.

The physical separation of iron oxide (magnetite) contaminants from fly ash was simply done by using a magnetic bar. Based on the dry weight of fly ash sample, the total weight loss after the magnetic separation process was approximately 30 wt.%. It should be noted that parts of silica and alumina were also possibly lost in the process since the iron oxide content in fly ash (including magnetite and hematite) was 8.1 wt.%. The magnetite-separated fly ash was refluxed with 3 M hydrochloric acid solution in order to remove other metal oxide contaminants. During this stage, metal oxides mainly iron, calcium and potassium oxides were removed (36 wt.% based on the dry weight of fly ash sample). The obtained solid product was then reacted with sodium carbonate powder to form sodium silicate of which easily dissolved in distilled water. This process is very promising for commercial sodium silicate production as it is not only the outstanding energy-efficient but also the environmentally friendly process. A huge savings on fuel consumption due to the lower sintering temperature (~ 200 - 300°C lower than the conventional process) and environmental impact reduction resulting from source reduction, resource recovery and reuse within this process are of great advantages.

The chemical compositions of sodium silicates obtained from this process were compared to that of the commercial one as shown in Table 2. In all cases, the presence of alumina content in sodium silicate was observed due to the fact that the silica sources of the commercial and coal ashes-derived sodium silicates were the silica sand obtained from natural source, and the mineral matter in coal itself. Since the amounts of silica and alumina were varied in each case, in order to synthesize zeolite A in the next step of process, both the fly ash- and bottom ash-derived sodium silicate were similarly dissolved in distilled water to obtain 21 wt.% SiO_2 .

3.1.2 Bench-scale synthesis of zeolite A from sub-bituminous coal fly ash and bottom ash

In this stage, fly ash and bottom ash-derived sodium silicate solutions were used as the raw materials for zeolite A synthesis. The XRD patterns of both bottom ash and fly ash-derived zeolites A products were shown in Table 2. It evidently confirmed that the selective formation of zeolite A from the bench-

scale synthesis of the fly ash and bottom ash is 72 wt.% and 94 wt.%, respectively (calculated based on the 100 wt.% crystallinity of zeolite A synthesized from pure chemicals). By using the Scherrer equation (Klug and Alexander, 1974), the calculated particle sizes of zeolite A products were 160 and 200 nm for bottom ash and fly ash, respectively.

3.1.3 Cation-exchange capacity of zeolite A

The cation-exchange capacities (CEC) of zeolite A products were shown in Table 2. It is clear that the CEC values of zeolite A products synthesized from both subbituminous fly ash and bottom ash were extremely high (4.00 and 3.77 meq/g, respectively) as compared to that of the fly ash (0.1 meq/g), bottom ash (0.03 meq/g) (Table 3). It is also observed that the CEC values of zeolite A synthesized from the subbituminous fly ash and bottom ash are comparable to that of commercial grade zeolite A (4.79 meq/g). Based on the theoretical formulae of the synthetic zeolite A as follows:

$\text{Na}_{12}(\text{A}_{112}\text{Si}_{12}\text{O}_{48})\cdot 27\text{H}_2\text{O}$, the Si/Al molar ratio of zeolite A is equal to one and the calculated CEC in terms of meq/g is 5.3. However, it should also be noted that the practical CEC value was usually lower than that of the calculated CEC. This could be attributed to the mass transfer limit during ion exchange process due to the critical aperture of zeolite A. Moreover, the higher Si/Al molar ratio observed in Tables 2 and 3 revealed the existence of the excess amorphous silica and other metal oxides contaminants in the bulk composition of zeolite A products. Regarding the heterogeneous phases of zeolite products, the practical CEC value could be strongly affected by not only the Si/Al molar ratio but also by the specific surface area and the crystallinity of zeolite A products. As the Si/Al molar ratio was theoretically similar for which samples, it was concluded that the highest CEC value was obtained from zeolite A product with a higher crystallinity and specific surface area.

3.2 Production of Lightweight Aggregates

3.2.1 Lightweight Aggregates (LWA)

By comparing between the physical properties of the normal aggregate and LWA in Table 6, it is observed that the LWA is approximately half of that compared to the normal aggregate with its loose bulk density less than 900 kg/m^3 as specified in ASTM C330, thereby classifying them as lightweight aggregate. Scanning electron microscopy (results not shown here) has revealed that the LWA has an extensive inter-connected network of both open and closed pores. As further confirmed by mercury intrusion porosimetry, the LWA has a total porosity of more than 35% with a large density of micro-pores well-dispersed throughout its structure. With increased porosity, the probability for increased pore connectivity within the concrete is more likely to increase and as water and ions penetrate through porous media, the porous LWA may reduce concrete resistance to water and chloride-ion penetration.

The excellent thermal insulation of the LWA is attributed to its spacious pockets of air volume which is contained within the voids, thereby provides for its enhanced thermal insulation. When the normal aggregate and LWA are placed in a hot oven at 100°C for 1 hour and subsequently relocated to an ambient environment, thermal infrared images (results not shown here) clearly demonstrated that the LWA possesses lower heat retention capacity.

3.2.2 Lightweight precast hollow-core concrete

The mix design for the different types of precast hollow-core concrete (normal and lightweight) is shown in Table 5. The compressive strength for all the aggregate is encouraging and they have exceeded the minimum requirement as set by ASTM and in-house quality control. For 1-day and 28-

days, the normal concrete (NC) revealed compressive strength of 46 MPa and 59 MPa, respectively (Table 7). LWAC 1 revealed 1-day and 28-days compressive strength of 43 and 51 MPa, respectively while LWAC 2 showed 1-day and 28-days compressive strength of 40 and 49 MPa, respectively. Despite the fact that LWA is more porous with higher tendency to absorb more water which often leads to higher water/cement ratio; both LWAC 1 and LWAC 2 clearly demonstrated excellent strength/weight performance.

Drying shrinkage analysis has revealed that on the 28-day, NC has moderate drying shrinkage of 350 μm while LWAC 1 and LWAC exhibits Abrasion resistance tests revealed that NC has the lowest loss, as compared to LWAC 1 and 2. However these values are still closely similar and it does not compromise on the final quality of the lightweight precast hollow-core.

For the thermal conductivity study, preliminary results for the normal precast hollow-core concrete display more than 0.3 W/m.K while both lightweight aggregate concrete 1 and 2 (Table 7) revealed lower thermal conductivity at 0.27 and 0.24 W/m.K, respectively. It should be noted that both experiments and theoretical analyses have identified two principal (systematic) errors that affect the operation of an idealized guarded hot plate apparatus such as the heat flow meter in our case. These errors are mainly edge heat flows at the periphery of the specimens and heat flow across the gap due to a thermal imbalance.

4 Conclusions

Zeolite A was successfully synthesis from sub-bituminous fly ash and bottom ash containing high crystalline silica content through the 2-stage process. Sodium silicate was firstly prepared from coal ash-derived silica by thermal fusion with sodium carbonate at 900°C for 1 h, and zeolite A was hydrothermally synthesized from the obtained sodium silicate and sodium aluminate at 120°C for 4 h, consecutively. It was found that zeolite A of 94% and 72% crystallinities with relatively high CEC values as 4.00 and 3.77 meq/g, respectively, (compared to 4.79 meq/g of commercial zeolite A) were synthesized from sub-bituminous fly ash and bottom ashes, respectively. Furthermore, since the silica fusion temperature was 200-300°C lower than conventional sodium silicate production, less fuel consumption and significant environmental impact reduction are mainly achieved. The development of our LWA and lightweight precast concrete has fulfilled the ASTM standards. Extensive laboratory analysis has confirmed LWA's excellent physical performance with 1-day precast concrete compressive strength of more than 40 MPa. By using real-time high-resolution infrared thermal imaging, we are able to observe that with a higher dosage of bio-LWA in the precast concrete, enhanced thermal insulation with excellent compressive strength can be achieved. Future work program with our LWA will be focused towards: i) in-depth analysis the physical properties of concretes made with our LWA such as water permeability, flexural strength, shrinkage and creep as well as ii) characterization with advanced state-of-the-art techniques such as computed tomography and synchrotron facilities to better understand its behavior and kinetics in-situ, especially during concrete curing process.

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Table 1. Chemical compositions and mineral contents of sub-bituminous coal fly ash and bottom ash.

Samples	SiO ₂	Al ₂ O ₃	Fe ₂ O ₃	CaO	MgO	SO ₃	LOI	Na ₂ O	K ₂ O	TiO ₂	P ₂ O ₅
Fly Ash	51.55	23.64	8.1	5.62	2.01	0.57	5.8	0.02	1.67	0.63	0.05
Bottom Ash	52.47	24.33	8.29	3.61	1.27	0.08	7.28	0.02	1.74	0.57	0.04
Samples	Quartz	Mullite	Anhydrite	Hematite	Magnetite	Lime	Calcite	Amorphous			
Fly Ash	18.18	12.66	0.77	1.27	0.96	-	-	66.47			
Bottom Ash	19.11	-	8.89	0.81	0.86	4.7	4.5	58.87			

Table 2. Chemical compositions of sodium silicate and zeolite A products prepared from various sources of silica and alumina.

	SiO ₂	Al ₂ O ₃	Fe ₂ O ₃	CaO	Na ₂ O	K ₂ O	TiO ₂	P ₂ O ₅	ZrO ₂	SrO	V ₂ O ₅
Sodium silicate	%										
Commercial	38.6	1.95	0.16	0.13	50.46	0.56	0.62	0.03	-	-	-
Fly Ash	78.57	2.56	3.32	2.11	-	4.15	8.53	-	0.56	0.09	0.1
Bottom Ash	54.69	7.29	13.45	4.24	12.62	4.55	2.67	-	0.22	0.14	-
Zeolite A	%										
Commercial	62.63	28.25	0.11	7.45	-	1.34	0.11	-	0.02	0.02	-
Fly Ash	60.71	28.6	1.09	5.14	-	1.12	3.21	-	0.08	0.04	0.02
Bottom Ash	44.22	22.09	5.3	23.99	-	2.64	1.46	-	0.04	0.11	0.01

Table 3. Selected properties of sub-bituminous coal ashes and zeolite A products.

Materials	Si/Al	Surface Area (m ² /g)	CEC (meq/g)
Fly Ash	7.41	9.9	0.1
Bottom Ash	7.33	7.6	0.03
Zeolite A			
Commercial	1.88	35.3	4.79
Fly Ash-Derived	1.8	41.2	4
Bottom Ash-Derived	1.7	36.1	3.77

Table 4. Chemical composition of Ordinary Portland Cement (OPC) III and lightweight aggregate.

Oxide(%)	OPC Type III	Lightweight Aggregate
SiO ₂	19.97	92.15
Al ₂ O ₃	5.91	2.34
Fe ₂ O ₃	3.21	1.69
CaO	63.87	1.09
MgO	1.07	0.03
SO ₃	3.79	-
Na ₂ O	0.3	< 0.01
K ₂ O	0.28	1.30
TiO ₂	0.26	0.46
P ₂ O ₅	0.05	0.82
LOI	0.9	< 0.01

Table 5. Mixture proportions normal concrete (NC) & lightweight aggregate concretes (LWAC 1 & 2).

Samples	OPC III	Rock	LWA	Sand	Limestone Powder	Superplasticizer	Water
	kg/m ³	Cc	liter				
NC	302	1000		1000	23	1000	150
LWAC 1	302	500	260	1000	23	1000	150
LWAC 2	302	-	482	1000	23	1000	150

Table 6. Physical properties of normal aggregate versus lightweight aggregate.

	Normal Aggregate	Lightweight Aggregate
Specific gravity	2.7	1.5
Loose bulk density (SSD) (kg/m ³)	1600	600-850
Adsorption (%)	-	5 – 15
Pore Size (microns)	0.03	0.12
Pore Volume (cc/g)	-	0.15
Porosity (%)	0.2	36
Thermal Conductivity (W/{m.K})	0.46	0.12 - 0.2

Table 7. Physical properties of normal concrete (NC) & lightweight concretes (LWAC 1 & 2)

	1-Day Compressive Strength	28-Days Compressive Strength	Bulk Density	Drying Shrinkage	Abrasion Resistance	Thermal Conductivity
	MPa	MPa	kg/cm ³	µm	g	W/{m.K}
NC	46	59	2450	350	0.83	0.37
LWAC 1	43	51	2100	258	1	0.27
LWAC 2	40	49	1850	508	1.4	0.24