



## Research article

## Simultaneous sulfur dioxide and mercury removal during low-rank coal combustion by natural zeolite

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## ABSTRACT

Sulfur dioxide (SO<sub>2</sub>) and trace metal such as mercury emission during combustion of low-rank coal cause a significant impact on the environment and health. Flue gas at coal-fired power stations is one of the main sources for the emissions of SO<sub>2</sub> and mercury metal. Low-cost and sustainable technologies for the removal of SO<sub>2</sub> and mercury from flue gas have become increasingly important nowadays. This paper presents the study of simultaneous removal of SO<sub>2</sub> and mercury over natural zeolite as an adsorbent in briquette and pulverized. The research is focused on evaluating adsorbent to coal optimum ratio towards adsorption performance on SO<sub>2</sub> and mercury removal. The experiments on the removal of SO<sub>2</sub> and mercury were carried out using horizontal electric furnace with different combustion temperature and adsorbent ratio. SO<sub>2</sub> in the flue gas as the result of the combustion process which exits from the outlet was analyzed using Gas Combustion and Emission Analyzer (E4400, E-Instrument). Mercury metal residues in the bottom ash were analyzed using NIC Mercury SP Analyzer. An increasing zeolite adsorbent SO<sub>2</sub> content in flue gas decreased. The optimum SO<sub>2</sub> removal was determined on 4% zeolite adsorbent ratio. It also has been found that 8% natural zeolite ratio to low-rank coal show optimum condition to adsorb mercury for all temperature condition for both briquette and pulverized conditions.

## 1. Introduction

Coal is a majority source of energy for industry and becomes the preference for many investors because it can lower the operational cost and is highly available in Indonesia. The Indonesian coal deposit of 70% is considered low-rank coal. Coal as an energy alternative is widely used as the electric source in the industries producing cement and fertilizer. Possible future rises in the price of natural gas and the demand for ammonia and its derivatives especially for the fertilizer industry motivate the consideration of coal as a feedstock [1, 2, 3]. Indonesia's low-rank coal is a source of energy with low economic value because it has high water content, low calorific value, and difficult to be exported. Coal is considered low rank due to its high moisture, ash, sulfur, nitrogen, trace metals, and low carbon content, especially exhibiting high volatile and low heating value [4, 5, 6, 7, 8]. The utilization of low-rank coal in technology can be generally found in combustion, liquidation, gasification,

and pyrolysis. Most of the power plants utilize low-rank coal with direct combustion technology.

Sulfur dioxides (SO<sub>2</sub>) emission during coal combustion to the environment generally comes from power plants as the primary energy sector. Power generator using coal and industrial activities are the major source of SO<sub>2</sub> emissions [9, 10, 11]. Coal combustion also generates nitrogen oxide (NO) which will produce nitrogen dioxide (NO<sub>2</sub>) in the atmosphere as another air pollutant, and pollution control was studied [12, 13, 14]. SO<sub>2</sub> and NO<sub>2</sub> emission produced during coal combustion causes acid rain and leads to problems such as corrosion. Several health problems and diseases such as throat disease, eyes, nose irritation, cough, and headache appear as the result of short exposure to this polluted air due to sulfur and nitrogen oxides content especially for people with asthma [15, 16, 17]. Coal combustion also produces ash contains trace metals that are harmful and has a carcinogenic effect during combustion. Several kinds of research show that zeolite can be used in controlling SO<sub>2</sub>

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and tracing metals emission [18, 19, 20, 21, 22, 23]. The use of zeolite both natural and synthetic as an adsorbent is more advantageous compared to carbon-based adsorbent due to its thermal tolerance and resistance to acid and easily regenerated and low cost.

Power plant using coal as fuel is the most important mercury emission contributor to the air because of its high volatile and corrosion potential by SO<sub>2</sub> and NO<sub>2</sub> [24, 25, 26]. Sub-micron mercury particles are emitted during coal combustion [9, 10, 11] and there are few research related to trace element especially mercury. The study on mercury classification modelling and its trace element characteristic has been studied by [27, 28, 29, 30, 31].

Some of the heavy metals (trace element) found in coal such as Hg, Pb, As, Ba, Be, B, Cd, Cr, Ta, Se, and Mo emitted are not dependent only on the amount of its content in the coal [27]. The emission may be an advanced combination of (a) the period of bed use and fatigue condition of the metal, (b) combustion temperature, (c) ash content and its composition, (d) fuel density, and (e) chlorine and sulfur content concerning trace metal volatility. This study stressed mercury released and captured during coal combustion in briquette and pulverized condition. During coal combustion, the mercury element in coal is totally vaporized in the vapor phase, since the species in high vapor pressure at typical boiler exit temperature. Although, mercury (Hg) concentration in coal is typically very low, important attention is stressed on its emission since it is highly toxic and harmful to human health. Several previous works have been done to capture SO<sub>2</sub> and trace metals during low-rank coal combustion and the effect of other species such as chlorine on its capture mechanism [32, 33, 34, 35, 36, 37, 38]. Although several studies have been conducted to see desulfurization and trace metal capture of low-rank coal, the simultaneous desulfurization, and mercury adsorption during combustion of low-rank coal using zeolite is still very limited. The advantage using of natural zeolite adsorbent is due to its low cost and wide availability.

Zeolite which is a polar adsorbent in general can be distinguished into two, namely natural zeolite and synthetic zeolite where zeolite is generally a three-dimensional structured silica-alumina crystal, formed from tetrahedral alumina and silica with cavities inside containing metal ions, usually alkaline or alkaline soil and water molecules that can move freely. Empirically, the zeolite molecular formula is M<sub>x/n</sub>(AlO<sub>2</sub>)<sub>x</sub>(SiO<sub>2</sub>)<sub>y</sub>.zH<sub>2</sub>O. Zeolite structure is so far known to vary. Natural zeolite usually contains cations K<sup>+</sup>, Na<sup>+</sup>, Ca<sup>2+</sup>, or Mg<sup>2+</sup> whereas synthetic zeolite usually contains only K<sup>+</sup> or Na<sup>+</sup> cations. In natural zeolite, the presence of water molecules in pores and free oxides on the surface such as Al<sub>2</sub>O<sub>3</sub>, SiO<sub>2</sub>, CaO, MgO, Na<sub>2</sub>O, K<sub>2</sub>O can cover the pores or active sites of the zeolite to decrease the adsorption capacity and catalytic properties of the zeolite [39, 40]. Under normal circumstances, the vacuum in the zeolite crystals is filled with free water molecules that form a spatial around the cation. When the crystal is heated for several hours, usually at a temperature of 250–900 °C, then the zeolite crystals have the function of absorbing gas or liquid. The absorbance of zeolite depends on the amount of vacuum and surface area. Adsorption on the zeolite itself occurs on the surface of the adsorbent pore. Zeolite particles have three types of pores, *macropore* and *micropore* (with sizes of >50nm and <2nm, respectively). Between them is mesopore. *Macropore* is the entrance into the particles to the *micropore*. Macropore does not contribute to the large surface area of zeolite adsorbents. In contrast, *micropore* is the cause of the large surface area of zeolite adsorbents. *Such micropores* are mostly formed during the activation process. It is at this micropore that most adsorption events occur. To analyze the microstructure (microstructure) of adsorbents using the method of scanning electron microscope (SEM) micrometer scale to nanometer, and method Thin Slice (thin incision) millimeter scale to get a visual picture of pore structure and percentage of mineral content in rocks with petrography test.

Bentonite is a term on clay containing montmorillonite, in the world of trade and belongs to the dioctahedral group. Bentonite can be divided into 2 groups based on the content of aluminum silicate hydrous, namely *activated clay* and *fuller's earth*. *Activated clay* is clay that lacks the power

of paint, but its paint ability can be increased through certain processing. Meanwhile, *fuller's earth* is used in *fulling* or cleaning woolen materials from fat. While based on the type, bentonite is divided into two, namely:

(1) *Wyoming Type* (Na-bentonite – *Swelling bentonite*)

Na-bentonite has the power to expand up to eight times when dipped in water and remains dispersed for some time in the water. In a dry state white or *cream*, in wet and exposed to sunlight will be shiny. Comparison of soda and lime is high, the colloidal suspension has a pH: 8.5–9.8, cannot be activated, the exchange position is occupied by sodium ions (Na<sup>+</sup>).

(2) *Mg*, (Ca-bentonite – *non-swelling bentonite*)

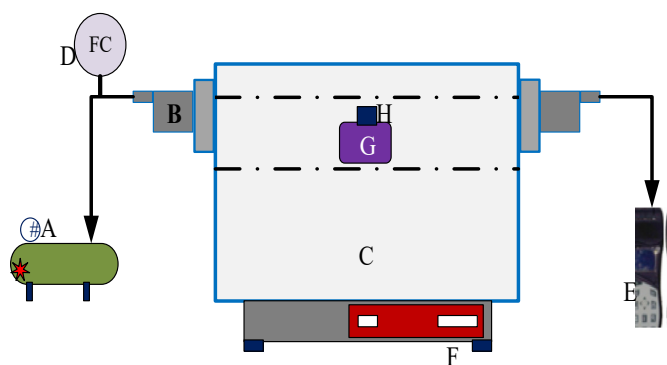
This type of bentonite is less inflated when dipped in water, and remains dispersed in water, but naturally or after activation has good sucking properties. The comparison of na and ca content is low, the colloidal suspension has a pH: 4–7. The ion exchange position is more occupied by calcium and magnesium ions. In dry conditions are rapid slaking, gray, blue, yellow, red, and brown. The use of bentonite in the refining process of cooking oil needs to be activated first [41]. Bentonite clay is very interesting to research because this clay has a layered structure with the ability to expand (*swelling*) and has cations that can be exchanged [42]. Although bentonite clay is very useful for adsorption, its adsorption capabilities are limited [43]. The use of clay as an adsorbent has several advantages because clay especially bentonite type has an inter-layer structure that can be modified to improve its properties. Besides, the use of clay as adsorbents can be regenerated [44].

Kaolin is a clay mineral with a 1:1 layer structure with a base unit consisting of a Tetrahedral Sheet SiO<sub>4</sub> and an octahedral sheet with Al<sub>3</sub> as an octahedral. Kaolin is usually located as a pure kaolinite mineral or related mineral e.g., halloysite, nacrite, and dikrit which are joined by other minerals such as smectic, mica, quartz, and feldspar as impactors. Kaolin impregnator materials especially quartz have a composition of SiO<sub>2</sub> and are about the same size as kaolin in an aggregated state so that the purification process using sifting is less effective. Kaolin minerals have a multi-layered pseudo-hexagonal structure with a size of 1–10 μm. In general, individuals of kaolin are usually composed of 10–50 layers of alumina-silica with a layer thickness of several tens of nanometers to several micrometers [45].

This research aims to reduce SO<sub>2</sub> and trace metal emission especially mercury during coal combustion for both pulverized and briquette form by adding natural zeolite adsorbent. The adsorbent will capture the sulfur and trace metal remain in ash instead of flying into the air. The use of non-carbon adsorbent such as zeolite, bentonite, kaolin, and lime powder is also capable to absorb Hg, Pb, Cd, Na as well as several volatile and non-volatile metal [46]. Natural zeolite has water molecules in the pores and free oxides on the surfaces such as Al<sub>2</sub>O<sub>3</sub>, SiO<sub>2</sub>, CaO, MgO, Na<sub>2</sub>O, K<sub>2</sub>O. The free oxides can cover the pores or active sites of the zeolite and can reduce the adsorption capacity.

## 2. Experiment procedure

The experiment used coal from Kaway XVI of West Aceh Sub-District in Aceh Province, Indonesia, and the natural adsorbent (zeolite) was obtained from West Java Province, Indonesia. Coal has low rank if it has high moisture content of 8.83%, sulfur content of 0.38%, and ash of 5.4% ash (db) with *calorific value* of 5904 cal/gr [47]. The size of coal and zeolite was crushed and screened to 60 mesh using a crusher and ball mill, then was briquetted using a briquette holding machine without binder, while the rest of the sample was left in pulverized condition. The mercury content in the ash and sulfur were analyzed. The adsorbent was then added with various concentration ratios from 4 to 12% of the weight and formed into briquette and pulverized form. The briquette was molded in coal briquette molding equipment with pressure level of 10 ton/cm<sup>2</sup> (SNI 047, 2006).



**Figure 1.** Experimental Apparatus Setup. A. Compressor; B. Stainless steel Reaction Tube; C. Electrical Furnace; D. Flow Meter; E. Emission analyzer; F. Panel; G. Ceramic Boat; H. Sample.

An electrical horizontal furnace was used to conduct experiments with the *reaction tube* made of 3-inch diameter stainless steel tube with an airflow of 1.4 times *stoichiometric air ratio* ( $\lambda$ ) as shown in Figure 1. The length of reaction tube is 1 m, and the heat is kept/maintained using isolation. The sample briquette and pulverized form was burned using excessive air in various temperatures, while airflow rate was managed by stoichiometric air ratio and placed in *ceramic sampling boat*. The airflow rate was adjusted and regulated with calibrated flowmeter equipped with an analyzer to measure the resulting  $\text{SO}_2$  concentration.

The size of coal and natural zeolite as an adsorbent were reduced using ball mill and then screened by 60 mesh with the compositions of 2, 4, 6, 8, and 10% of the sample weight. Coal and zeolite samples in briquette or pulverized form were then placed in sampling boat and fed the furnace. The airflow rate was set based on sample weight and set at excessive air to ensure combustion process complete and control using flowmeter calibrated with wet gas meter.

Combustions were conducted for 30 min in temperatures of 600 °C, 700 °C, and 800 °C, respectively. The concentration of  $\text{SO}_2$  emission resulted during combustion was measured using *Industrial gas combustion and emission analyzer* (E4400, E instrument). Adsorption efficiency of  $\text{SO}_2$  for briquette and pulverized form was measured by comparing  $\text{SO}_2$  released by the combustion with and without adsorbent addition. Heavy metal released and then captured by adsorbent zeolite was measured by analyzing residual ashes from the combustion left in the ceramic boat for both treatments analyzed using *NIC Mercury SP Analyser*. The experiment was then repeated for different variable condition.

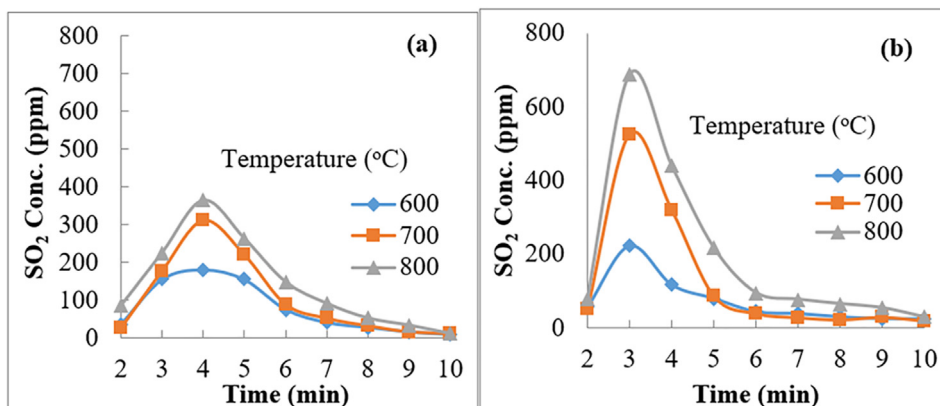
### 3. Result and discussion

#### 3.1. Effect of temperature and adsorbent on $\text{SO}_2$ emission

$\text{SO}_2$  emissions from coal combustion mainly depend on the sulfur content in the coal. Effect of operating condition shows the profile of  $\text{SO}_2$  generated during combustion as shown in Figure 2. The oxidation process occurred on surface of coal particle, i.e., a coal which was finely crushed, and consequently exposing more surfaces for a given weight. It reacted more rapidly with oxygen than with a similar sample for both in briquette and pulverized forms. Figure 2 shows  $\text{SO}_2$  profile during coal combustion at different temperatures without adsorbent for pulverized and briquette cases. The figure shows  $\text{SO}_2$  emissions in ppm increased with the increasing of temperature, and then appeared to decrease after 4 min and 3 min for briquette and pulverized forms, respectively. The decrease of  $\text{SO}_2$  emission after 4 and 3 min might be caused by the decrease of sulfur content in coal.

The experiment shows the increase of  $\text{SO}_2$  which was burned earlier as volatile matter combustion. The  $\text{SO}_2$  profile after 4 min tended to decrease due to fixed carbon combustion and the sulfur content in fixed carbon was low.  $\text{SO}_2$  emission for pulverized case was higher than briquette case for all temperatures. Higher  $\text{SO}_2$  emission for pulverized due to particles was smaller than briquette form. In briquette conditions, it took time to reach the combustion temperature (flash point), so it was not fast and the oxygen diffuse factor into the briquette was slower than the pulverized condition because the surface area was smaller. The small particle gave large surface area and easily made oxygen diffusion. The result of pulverized form gave more complete combustion compared to briquette form. Complete combustion gave more  $\text{SO}_2$  emission compared to incomplete combustion. The smaller sorbent particles gave higher  $\text{SO}_2$  reduction and CaO conversion, even it appeared in the pulverized coal combustion [37]. In pulverized conditions, the diffuse rate of  $\text{SO}_2$  gas into adsorbents was easier because the surface area was larger.

The effect of adsorbent on  $\text{SO}_2$  adsorption during coal combustion at different temperature for briquette and pulverized case at 4 min is shown in Figure 3. The figure shows effect adsorption of  $\text{SO}_2$  by adsorbent decreased  $\text{SO}_2$  emission as the adsorbent concentration increased. Similar tendency is shown on higher  $\text{SO}_2$  concentration for pulverized form compared to briquette form. The result also suggests smaller particle will give higher  $\text{SO}_2$  concentration due to complete combustion. The same phenomena are also shown higher temperature will give a higher concentration of  $\text{SO}_2$  due to more complete combustion process.



**Figure 2.**  $\text{SO}_2$  profile during coal combustion at different temperatures without adsorbent for (a) briquette and (b) pulverized cases.

Optimum adsorption condition is shown on 4% adsorbent concentration at 700 °C combustion for pulverized condition.

The result also shows the ability adsorbent on SO<sub>2</sub> adsorption in briquette form was better than pulverized form. Since volatilized and emitted in vapor phase, the increasing temperature will increase the SO<sub>2</sub> concentration. Coal in pulverized form is easily burned due to higher surface area than briquette form, resulting in higher SO<sub>2</sub> concentration. The optimum adsorption capacity was determined at 6% for briquette and pulverized 8% form, respectively.

### 3.2. Effect of temperature and adsorbent on mercury adsorption

Figure 4 shows the influence of adsorbent ratio toward mercury content in bottom ash at various combustion temperatures. The result shows that zeolite adsorbent had a proportional effect on adsorption capability of mercury. The concentration of mercury remained in bottom ash (sampling boat) as the zeolite adsorbent ratio increased. The result suggests zeolite adsorbent had significant effect on mercury adsorption, and optimum adsorbent ability to adsorb mercury was observed at 8% ratio for both briquette and pulverized forms. After 10% of optimum adsorbent ratio, adsorption capability of adsorbent to adsorb mercury decreased for all temperatures and started to befall beyond 10% ratio.

Effect of temperature had inverse influence over mercury content in bottom ash. The concentration of mercury in bottom ash decreased as the combustion temperature increased due to more volatile property of mercury in higher temperature. This is because the increase of

temperature during combustion released mercury in the more volatile form and rate of diffusion volatile mercury to adsorbent was slower than devolatilization rate of mercury from coal. In briquette conditions, some briquettes were not perfectly burned; thus, some mercuries were left in the ash and unburned carbon. In pulverized conditions, the carbon was perfectly burned; thus, some mercuries evaporated due to the volatile form of the mercury. Mercury measurements were performed on ash left behind after burning, as it is considered an adsorbent. Volatile mercury can pollute the air and easily disburse and is very hard to capture by increasing of combustion temperature. The use of natural zeolite as an adsorbent is expected to control release to the air and control mercury content remain in bottom ash. The effective temperature of zeolite to capture mercury emission during combustion is 600 °C–700 °C. At temperature 800 °C, the effectivity of zeolite to adsorb mercury decreases for both case briquette and pulverized forms. The optimum ratio of adsorbent in mercury adsorption for briquette was achieved at 8% adsorbent ratio for both 600 °C and 700 °C combustion temperatures. While in pulverized conditions, the optimum condition was obtained at 10% adsorbent at temperatures of 600 °C and 700 °C.

### 3.3. Adsorption capacity of zeolite

SO<sub>2</sub> and mercury adsorption during coal combustion occurred simultaneously, requiring observation on the adsorption capacity. Adsorption capacity is the ability of one gram adsorbent to adsorb adsorbate by the following equation:

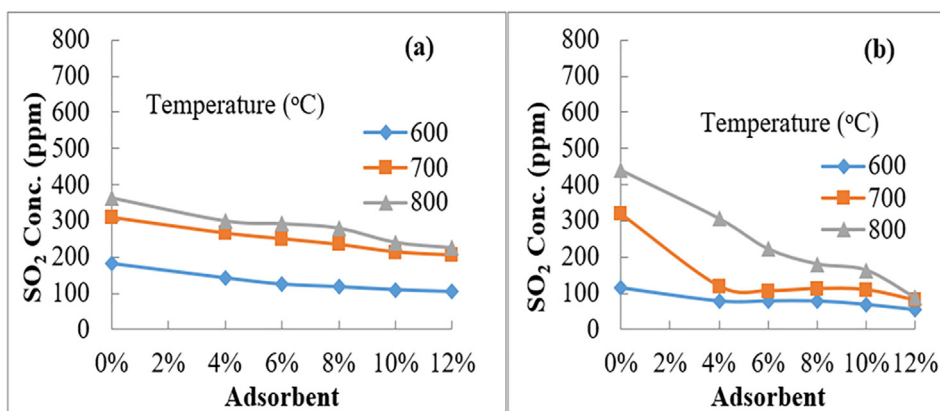


Figure 3. Effect of adsorbent on SO<sub>2</sub> adsorption during combustion of low-rank coal for (a) briquette and (b) pulverized cases at 4 min.

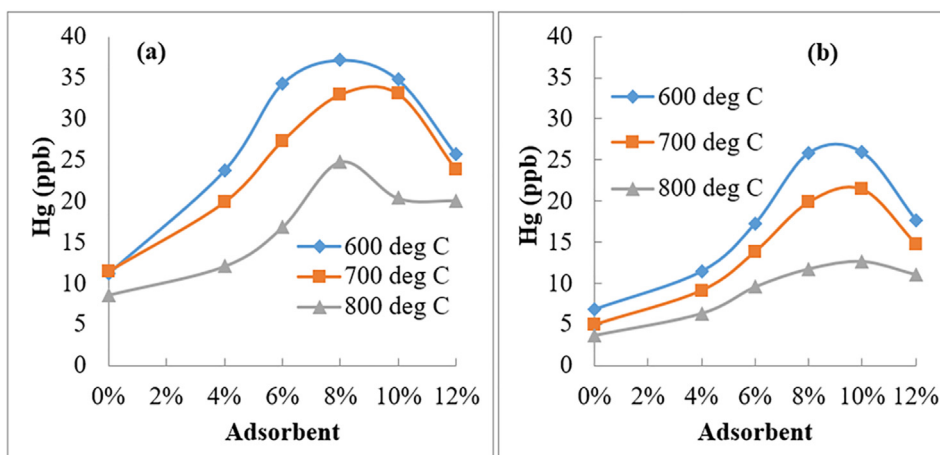


Figure 4. Effect of adsorbent on Mercury captured at bottom ash during combustion for (a) briquette and (b) pulverized cases.



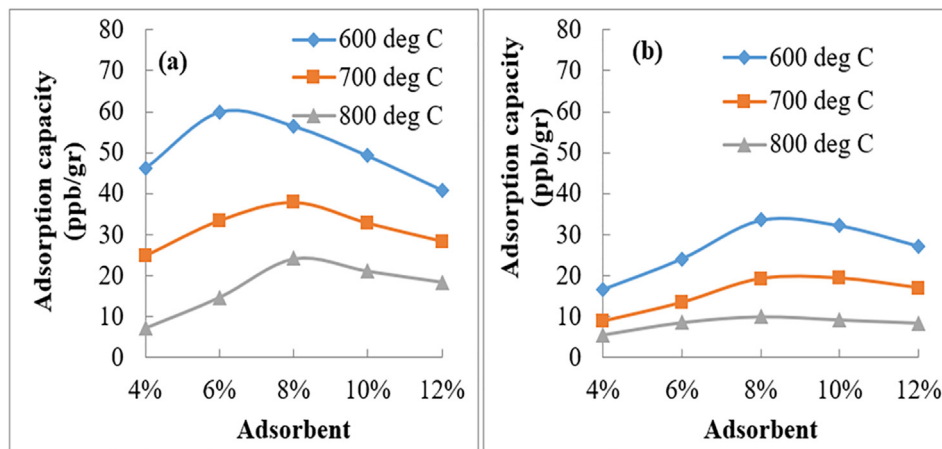


Figure 5. Adsorption capacity of mercury by zeolite for (a) briquette and (b) pulverized cases at different temperature.

$$Q = \frac{Ca - Ca_0}{M_{adsorben}} \quad (1)$$

The adsorption capacity of mercury by zeolite for briquette and pulverized cases at different temperatures is shown in Figure 5. The result shows adsorption capacity optimum at 6% and 8% for briquette and pulverized forms, respectively. In general, the adsorption capacity or efficiency will increase as the adsorbent ratio increases. The results show that adsorption capacity of mercury in briquette form was better than pulverized form. Adsorption efficiency of adsorbent showed significant effect of temperature change. The adsorption capacity decreased as the temperature increased. Since mercury is easily evolved with increasing temperature, adsorption capacity of zeolite decreases as the temperature increases. Figure 5 suggests that when combustion temperature was low, some mercuries did not volatilize but stayed in bottom ash, then were captured by the sorbent or deposited on it. As the result, adsorption capacity of zeolite was high at low combustion temperature. When some volatile mercuries existed in the gas phase, some formed particles were captured by sorbents. Another mechanism is that mercury particles were captured by sorbents through nucleation and coagulation.

#### 4. Conclusion

Simultaneous adsorption SO<sub>2</sub> and mercury during coal combustion using natural zeolite was observed. SO<sub>2</sub> emission during combustion tended to increase during at the earlier combustion process. The adsorption capacity of mercury was influenced by its high volatility and tended to decrease as the temperature increased. The optimum adsorption capacity was determined at 6% and 8% for briquette and pulverized forms, respectively. The adsorption capacity of zeolite to adsorb SO<sub>2</sub> and mercury was controlled by diffusion rate of the gases to zeolite particles. The performance of zeolite to adsorb SO<sub>2</sub> and mercury tended to decrease at 800 °C because it started to desorb.

#### Declarations

##### Author contribution statement

Asri Gani: Conceived and designed the experiments; Contributed reagents, materials, analysis tools or data; Wrote the paper.

Yuanda Wattimena & Medyan Riza: Conceived and designed the experiments; Performed the experiments.

Erdiwansyah: Conceived and designed the experiments; Analyzed and interpreted the data; Wrote the paper.

Mahidin: Contributed reagents, materials, analysis tools or data; Wrote the paper.

Muhibbuddin: Conceived and designed the experiments; Analyzed and interpreted the data.

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##### Data availability statement

Data included in article/supplementary material/referenced in article.

##### Declaration of interests statement

The authors declare no conflict of interest.

##### Additional information

No additional information is available for this paper.

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