

Comparison of Non-acid Wash and Acid Wash Preparation Methods for Zeolite A Synthesis from Coal Fly Ash

Sirirat Jangkorn^{1,2} and Pornsawai Praipipat^{1,2*}

¹ Department of Environmental Science, Khon Kaen University, Khon Kaen, Thailand

² Environmental Applications of Recycled and Natural Materials (EARN) Laboratory, Khon Kaen University, Khon Kaen, Thailand

*Corresponding Author: pornprai@kku.ac.th

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Abstract

Coal fly ash is a byproduct from power plants and causes environmental problems. Zeolite A was synthesized from coal fly ash by using the two-step method. The raw materials obtained without and with acid wash treatment were studied to determine whether acid wash treatment is an important pre-synthesis step to obtain complete zeolite A. The synthesized materials were characterized in terms of crystalline pattern, surface area, chemical composition, chemical functional groups, and cation exchange capacity through XRD, FESEM-FIB, EDX, XRF, FTIR and the cobalt cation exchange process. The results confirmed the preparation of zeolite A from the process with acid wash treatment and strongly indicated the quality of zeolite A with a cation exchange capacity of 2.38 meq/g. In contrast, the material synthesized without wash treatment corresponded to sodalite with a cation exchange capacity of 0.99 meq/g. Therefore, acid wash treatment is an important step before zeolite A synthesis, and the synthesized material has the potential to be utilized as a catalyst, adsorbent, and ion exchanger in the future.

Keywords: Electric power plant waste; Zeolite; Two-step method; Acid treatment; Adsorbent

1. Introduction

Zeolite is an aluminosilicate with a crystalline structure consisting of a framework of $[\text{SiO}_4]^{4-}$ and $[\text{AlO}_4]^{5-}$ tetrahedral linkages with three-dimensional networks and numerous pores. Zeolites are categorized as natural or synthesized. Natural zeolite is built from environmental minerals (Scott *et al.*, 2002), whereas synthesized zeolite is produced from raw materials containing high amounts of silicon (Si) and aluminum (Al): clay, rice husks, and coal fly ash (CFA) (Gougazeh and Buhl, 2014). Among these materials, CFA has been widely used for zeolite synthesis because of its high aluminosilicate content for zeolite formation (Scott *et al.*, 2002).

Zeolites can be utilized for molecular sieving, ion exchange, and water adsorption (Gougazeh and Buhl, 2014). They are also used as adsorbents for toxic pollutant removal

(Scott *et al.*, 2002). There are more than 200 types of zeolites, defined as A, P, X and Y, which differ in surface area, pore size, and ratio of Si/Al components (E Mgbemere *et al.*, 2017). As a heavy metal adsorbent, zeolite type A is widely used because of its high ion-exchange capacity (Chang and Shih, 2000).

CFA is a solid waste from power plant combustion that may create toxic wastes that are difficult to remove, making waste management a concern. Generally, this solid waste is managed by disposal in a landfill, but this method is expensive and creates environmental contamination. Since CFA has a high content of Si, which is the main element for zeolite synthesis (Tauanov *et al.*, 2020), using CFA for zeolite synthesis may also reduce toxic waste (Zhao *et al.*, 2010a). However, the contaminants in CFA can inhibit the synthesis process.

Thus, it is necessary to remove them by using an acid washing process to increase the amount of Si (Lee *et al.*, 2017). According to previous studies, zeolite A synthesized from CFA with acid was successfully removed the heavy metals such as Cu, Zn, and Cr from wastewater (Wang *et al.*, 2009; Hani *et al.*, 2010). However, no comparison has been made between materials synthesized from CFA with and without acid washing.

Among different techniques for zeolite A synthesis, i.e., hydrothermal, fusion, and two-step methods (Jha and Singh, 2012), the latter is the most suitable technique because of its higher silica extraction (Wang *et al.*, 2008). The present study, therefore, compares the results from using CFA with and without acid washing to prepare raw materials for zeolite synthesis using a two-step method to confirm whether the acid washing step is important for the synthesis.

This study aimed to synthesize zeolite A from CFA with and without acid washing treatment to investigate the crystalline pattern, surface area, chemical composition, chemical functional groups, and cation exchange capacity (CEC) of the obtained samples by using X-ray powder diffraction (XRD), field emission scanning electron microscopy and focus ion beam (FESEM-FIB), energy dispersive X-ray spectrometry (EDX), X-ray fluorescence spectroscopy (XRF), Fourier transform infrared spectroscopy (FTIR), and the cobalt cation exchange process. The results were compared to confirm whether an acid wash step during raw material preparation is important for zeolite A synthesis. Furthermore, this study provides a body of knowledge on producing zeolite A to be used as an adsorbent of heavy metals in wastewater to solve water pollution.

2. Materials and methods

2.1 Reagent and zeolite synthesis

Coal was burned at 1000 °C, collected as CFA from Mae Moh Power Plant located in Lampang Province, Thailand, dried in a hot air oven (Binder) at 60 °C overnight, and sieved with a mesh size of 125 µm. For this study, two conditions were set, non-acid wash and

acid wash treatment, for comparison of the qualities of the synthesized zeolites.

For the acid wash sample (CFAA), 10 g of CFA was added to 250 mL of 10% HCl (RCI Labscan) and then boiled at 80 °C for 1 h with constant stirring at 300 rpm. A glass microfiber filter (GF/C, Whatman) with vacuum filtration was used to filter the mixed solution, which was washed with deionized (DI) water. The filtered material was dried in a hot air oven at 100 °C overnight (Wang *et al.*, 2008). For the non-acid wash condition, the sample was prepared with no purification.

For zeolite A synthesis, the two-step method, which included the initial gel and zeolite synthesis, was applied. For the initial gel, 9 g of CFA/CFAA was added to 90 mL of 1.67 M NaOH (RCI Labscan) in a 250 mL round-bottom flask. The closed reflux system was connected with 500 mL of liquid parafilm in a 1 L stainless steel container. Then, the mixed solution was heated at 80 °C in a water bath for 2 h with a constant stirring of 300 rpm and filtered by a GF/C filter with vacuum filtration to collect 90 mL of solution 1. Next, solution 2, 30 mL of NaOH-NaAlO₂ solution was prepared by mixing 2.40 g of NaAlO₂ (Sigma–Aldrich) and 30 mL of 1.67 M NaOH. The two solutions were put into the same 100 mL nickel crucible, heated in a hot air oven for 340 min at 100 °C, filtered by a GF/C filter with vacuum filtration, washed with DI water, and finally dried in a hot air oven overnight at 100 °C. The resulting zeolites synthesized from CFA and CFAA were named ZCFA and ZCFAA, respectively. The process is demonstrated in Figure 1.

2.2 Characterization

The materials were characterized by XRD (PANalytical, EMPYREAN, United Kingdom) for crystalline phase identification. Two grams of each sample was analyzed by XRD with Cu K α radiation, a λ of 0.1514 nm, 40 mA, and 2 θ of 5 - 50° at 298 K. The surface area and chemical composition were analyzed by FESEM-FIB with EDX (FEI, Helios NanoLab G3 CX, USA). The samples were coated with a thin layer of gold by a gold sputter coater (Cressington Sputter, Coater 108 auto, USA) at 20 mA for 140 secs before analysis under

the conditions of 20 kV, 86 pA and 4 mm in secondary electron mode. The elemental components of the samples were analyzed through XRF (Rigaku ZSX Primus II, USA). The samples were ground by grinding balls before being put into a hydraulic press machine to produce sample pellets for XRF analysis

under the conditions of 50 kV and 2 mA. FTIR (Thermo Fisher Scientific, Nicolet 6700, USA) was used to analyze the chemical functional groups; 0.1 mg of sample was mixed with 1 mg of KBr, pressed under 5 ton/cm² and then analyzed by XRF in transmission mode with a range of 400 – 4,000 cm⁻¹.

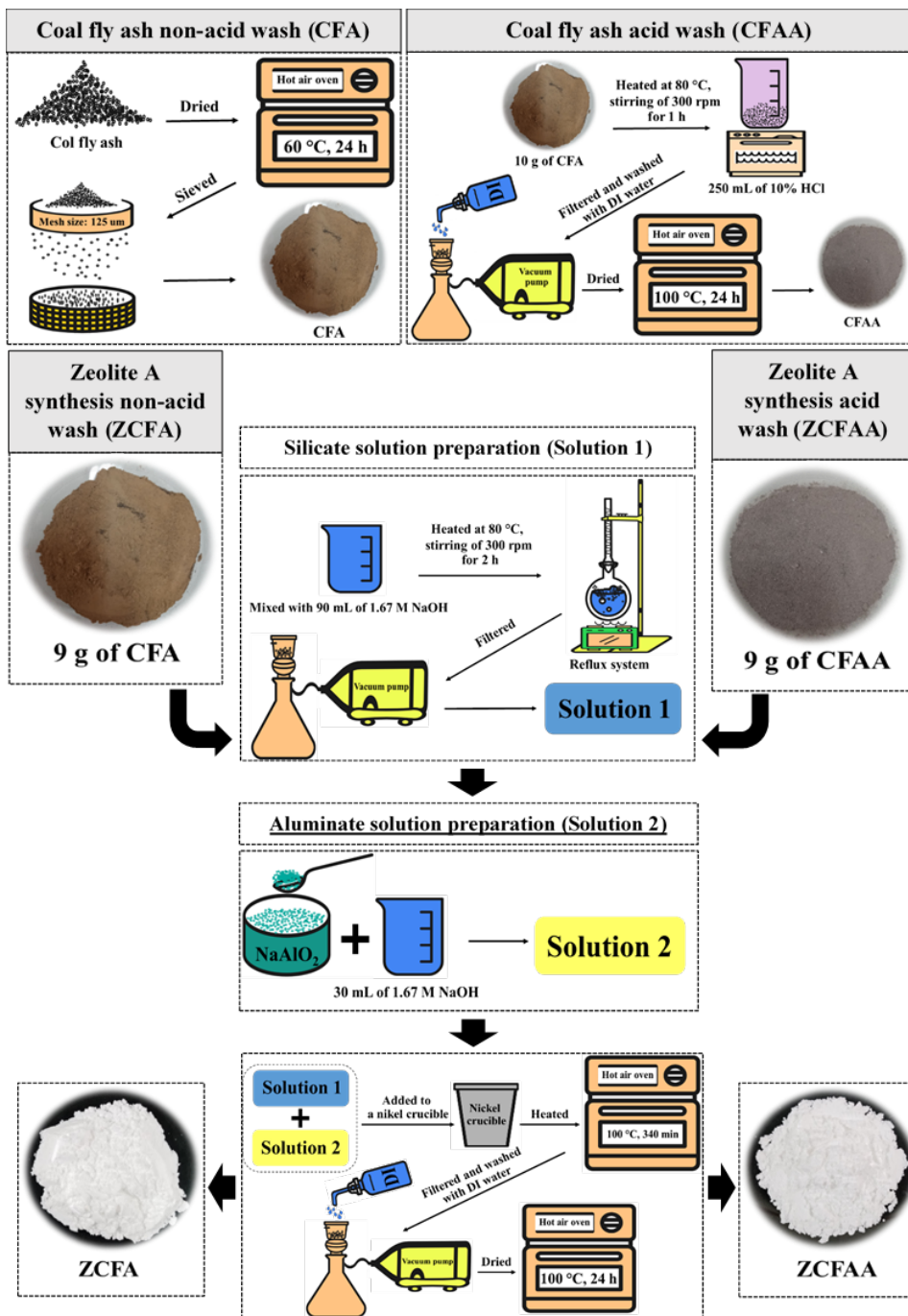


Figure 1. Flow diagrams of CFA, CFAA, ZCFA and ZCFAA preparation

2.3 CEC analysis

Sodium ions (Na^+) were analyzed to determine the CEC of each sample by using cobalt cation exchange. Two grams of each sample (ZCFA and ZCFAA) was mixed with 100 mL of 0.1 M $\text{Co}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$ in 250 mL polyethylene bottles at a shaking speed of 300 rpm at 30 °C for 30 h. Then, the solution was filtered by a GF/C filter with vacuum filtration and finally analyzed by atomic absorption spectrophotometry (Analytik Jena, Contra 800, USA).

3. Results and discussion

3.1 XRD results

The XRD results in Figure 2a-d show semicrystalline patterns for CFA and CFAA and crystalline patterns for ZCFA and ZCFAA. In Figure 2a, the peak of CFA at 2θ of 25.52° was quartz, whereas the peaks at 26.74° , 31.39° , 35.63° , 37.53° , 38.83° and 40.96° were mullite. In Figure 2c,

the CFAA peaks at 2θ of 20.98° and 26.63° were quartz, whereas those at 30.12° and 35.53° were mullite. These results corresponded to the observation of quartz and mullite in CFA reported by Musyoka *et al.* (2011). However, some mullite peaks disappeared after the CFA was treated with an acid wash process, which meant that this process helped to clean the CFA (Hani *et al.*, 2010). Figure 2d shows the results for ZCFAA, which had peaks similar to the zeolite A pattern at 2θ of 7.02° , 10.03° , 12.06° , 16.02° , 21.08° , 24.00° , 26.02° , 27.02° , 30.00° , 30.09° , 31.01° , 32.06° , 33.04° and 34.03° , corresponding to the zeolite A pattern of JCPDS 39 - 222 in the study by Jangkorn *et al.* (2022). In contrast, the pattern for ZCFA in Figure 2b was consistent with sodalite with peaks at 2θ of 14.00° , 24.43° , 31.79° , 34.86° and 43.01° , similar to the results of Günther *et al.* (2015), and corresponded to the sodalite pattern of JCPDS 00-037-0196 reported by Luo *et al.* (2016). Therefore, it is recommended to clean impurities in CFA before zeolite A synthesis, which is consistent with Wulandari *et al.* (2019).

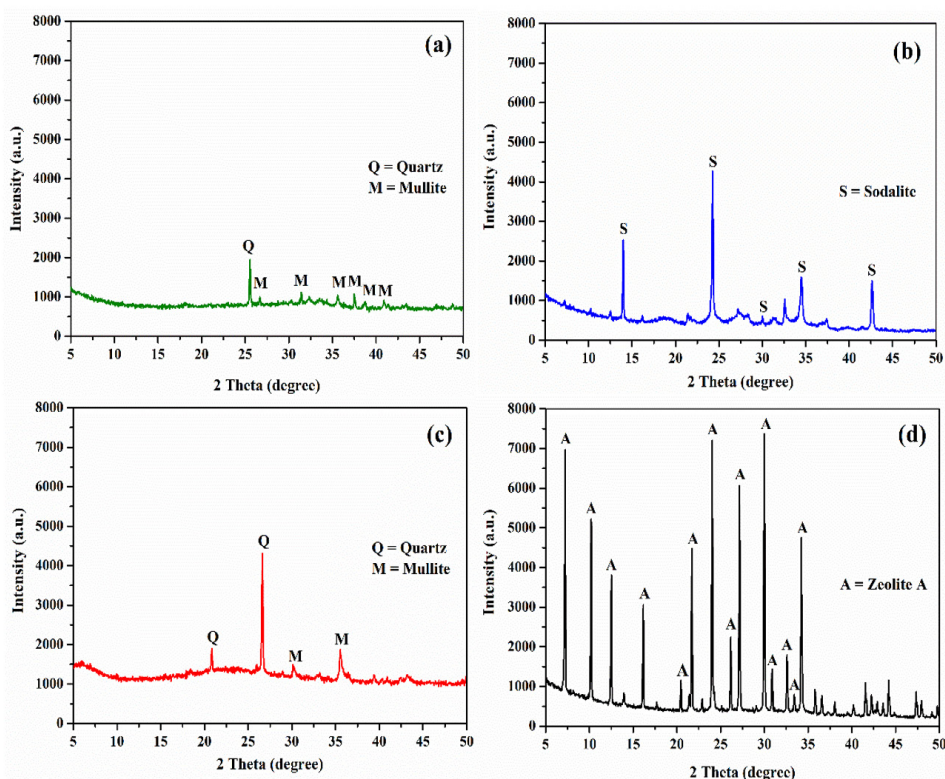


Figure 2. XRD pattern analysis of (a) CFA, (b) ZCFA, (c) CFAA and (d) ZCFAA (Q: quartz, M: mullite, S: sodalite and A: zeolite A)

3.2 FESEM-FIB results

Figure 3a-d shows SEM images of the samples with a magnification of 1,500X, indicating 50 μm particles. CFA had a spherical and smooth surface, whereas CFAA had a cracked and rugged surface, as shown in Figure 3a and c, respectively, which corresponded to a previous study by Wulandari *et al.* (2019) reporting erosion of the surface of CFA after acid washing. ZCFA (Figure 3b) was in a wool ball form similar to sodalite, corresponding to the study by Günther *et al.* (2015); in Figure 3d, ZCFAA showed a cubic structure similar to the zeolite A structure, in line with the results of Tauanov *et al.* (2020). Therefore, an acid washing was important before zeolite A synthesis. The FESEM-FIB results also corresponded to the XRD results.

Four main elemental components of oxygen (O), silica (Si), aluminum (Al), and sodium (Na) were investigated in EDX

analysis to determine their mass percentage by weight (%wt), as shown in Figure 4a-d. CFA and ZCFA contained 55.70 and 46.40 wt% O, 27.60 and 21.60 wt% Si, 13.70 and 19.10 wt% Al, and 3.00 and 13.00 wt% Na, respectively, as shown in Figure 4a and b. Moreover, the Si/Al ratio of ZCFA was close to 1, which confirmed the occurrence of sodalite (Luo *et al.*, 2016). The elemental compositions of CFAA and ZCFAA included 40.90 and 38.90 wt% O, 56.70 and 26.40 wt% Si, 1.10 and 20.20 wt% Al, and 1.30 and 14.50 wt% Na, respectively, as shown in Figure 4c and d. The Si content of CFAA was higher than that of CFA, indicating that acid washing helped reduce impurities, resulting in a higher amount of Si, the main element for zeolite A synthesis (Kuwahara *et al.*, 2008). Moreover, the Si/Al ratio of ZCFAA was close to 1, which confirmed the occurrence of zeolite A (Figure 4d) (Gougazeh and Buhl, 2014), indicating the need for acid washing for synthesis.

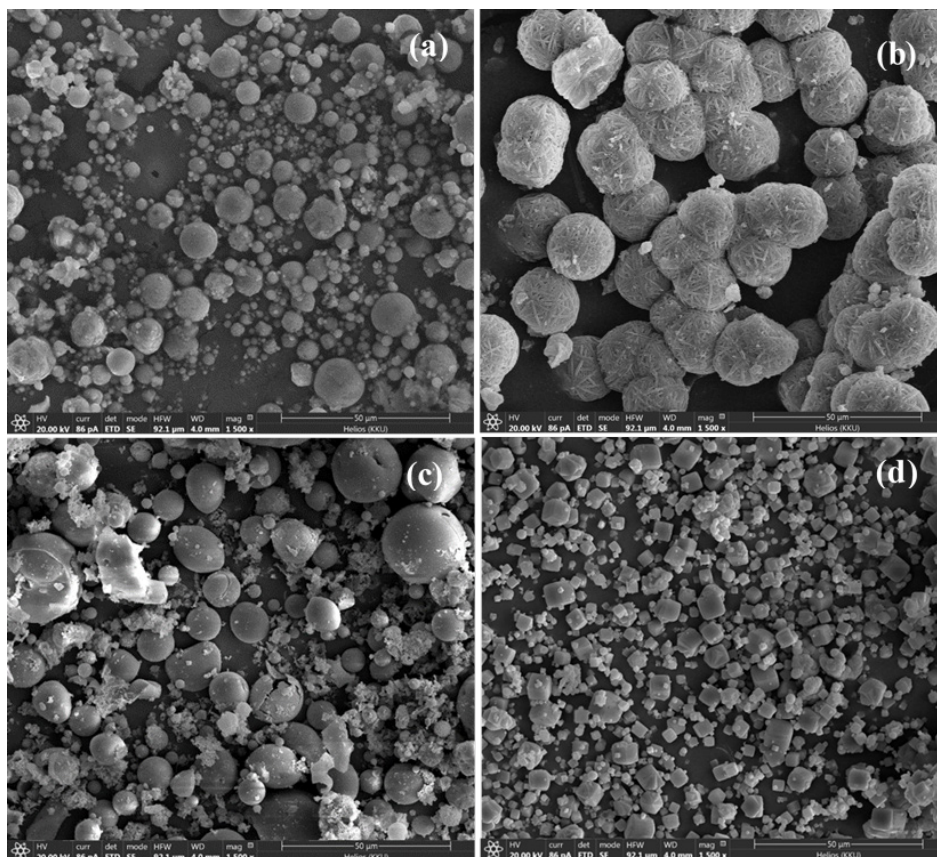


Figure 3. FESEM-FIB images of (a) CFA, (b) ZCFA, (c) CFAA and (d) ZCFAA

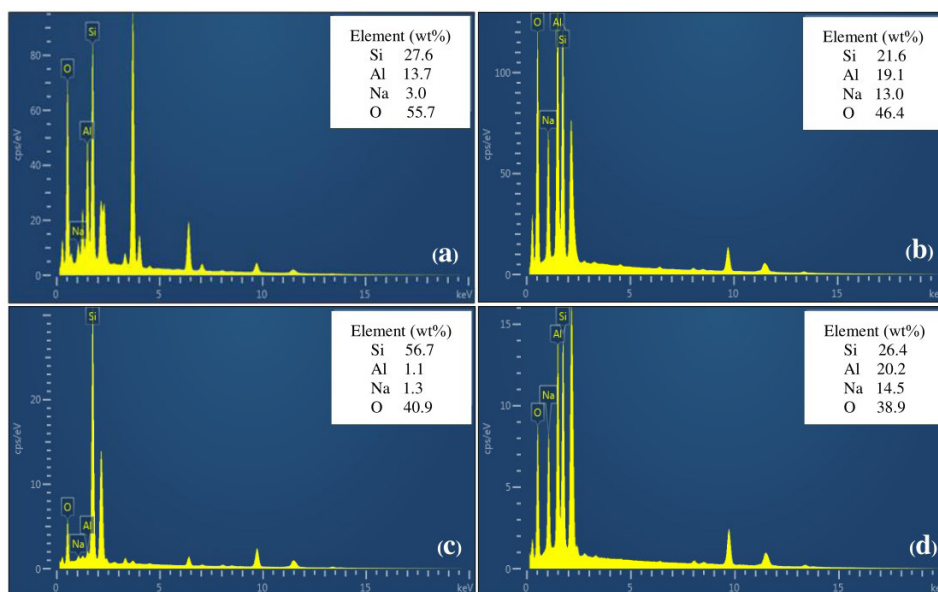


Figure 4. The chemical compositions of (a) CFA, (b) ZCFA, (c) CFAA and (d) ZCFAA by EDX analysis

3.3 XRF results

The chemical compositions in weight percentage (wt%) of the samples are shown in Table 1. The contents of Si, Al, Fe, Ca, Mg, Na and other elements in CFA were 15.20, 8.95, 21.00, 40.00, 1.64, 1.80, and 11.41 wt%, respectively, while an increase in Si from 15.20 to 55.40 wt% and a decrease in other components were found in CFAA. Therefore, an acid washing helped to increase the Si content, which further supported the need for the removal of impurities for the synthesis of zeolite A, similar to the studies by Molina and Poole (2004), Lieberman *et al.* (2014), and Cardenas (2019). The wt% contents of Si, Al, Fe, Ca, Mg, Na and other elements in ZCFA were 16.30, 11.50, 0.41, 0.06, 0.04, 12.20, and 40.51 wt%, respectively, while those in ZCFAA were 56.30, 41.60, 0.29, 0.21, 0, 11.10, and 2.00 wt%. Both samples exhibited the same change pattern: an increase in Si, Al, and Na and a decrease in Fe, Ca, and Mg. For zeolite A synthesis, the Si/Al ratio is recommended to be close to 1 (Gougazeh and Buhl, 2014), which corresponded to the EDX results for ZCFAA in the present study. The acid washing process was therefore important for treating CFA to obtain zeolite A.

The XRF analysis showed Si and Al in CFA and CFAA; the XRD results showed the semicrystalline phases of quartz and mullite (Kashiwakura *et al.*, 2009), which contain Si and Al with the chemical formulas of SiO_2 and $3\text{Al}_2\text{O}_3$. SiO_2 , respectively (Cavalcanti Gomes *et al.*, 2018; Musyoka *et al.*, 2011). Thus, the results from XRF and XRD analysis were related. The crystalline patterns obtained through XRD analysis confirmed the synthesis of ZCFA and ZCFAA, which were identified as sodalite and zeolite A, respectively. The chemical formulas of ZCFA and ZCFAA obtained through XRF analysis were $\text{Na}_{0.53}\text{Al}_{0.42}\text{Si}_{0.58}\text{O}_{2.53}$ and $\text{Na}_{0.48}\text{Al}_{1.54}\text{Si}_2\text{O}_{0.13}$, respectively. The formulas also confirmed the occurrence of sodalite and zeolite A.

3.4 FTIR results

The FTIR results for CFA (Figure 5a) and CFAA (Figure 5c) show T-O (T = Si or Al) groups at 987, 1112, and 1151 cm^{-1} and at 952 and 1083 cm^{-1} , respectively, C = O groups (carboxylate groups in organic matter) at 1629 and 1640 cm^{-1} , and broad O-H stretching and H-O-H bending of water molecules at 3643 and 1629 cm^{-1} and at 3449 and 1640 cm^{-1} , corresponding to the results

of Gougazeh and Buhl (2014). The main functional groups of ZCFA (Figure 5b) and ZCFAA (Figure 5d) were T-O at 991 and 1000 cm^{-1} , respectively, C = O at 1656 and 1633 cm^{-1} , and O-H at 1560 to 3466 cm^{-1} and 1633 to 3452 cm^{-1} . Moreover, the sodalite framework (T-O-T) was found at 434, 462, and 686 cm^{-1} (Figure 5b), while the characteristic zeolite A background of

TO_4 (T= Si or Al) was observed at 463, 554, and 667 cm^{-1} (Figure 5d), in line with the results of Gougazeh and Buhl (2014). These results confirmed the occurrence of sodalite and zeolite A, which corresponded to the XRD, FESEM-FIB, EDX, XRF, and FTIR results. Therefore, an acid washing step was recommended as an important step before zeolite A synthesis.

Table 1. Chemical compositions of CFA, CFAA, ZCFA and ZCFAA in weight percentage by XRF analysis

Sample	Chemical composition (wt%)						
	Si	Al	Fe	Ca	Mg	Na	Others
CFA	15.20	8.95	21.00	40.00	1.64	1.80	11.41
CFAA	55.40	5.05	28.90	3.34	1.54	0	5.77
ZCFA	16.30	11.50	0.41	0.06	0.04	12.20	40.51
ZCFAA	56.30	41.60	0.29	0.21	0	11.10	2.00

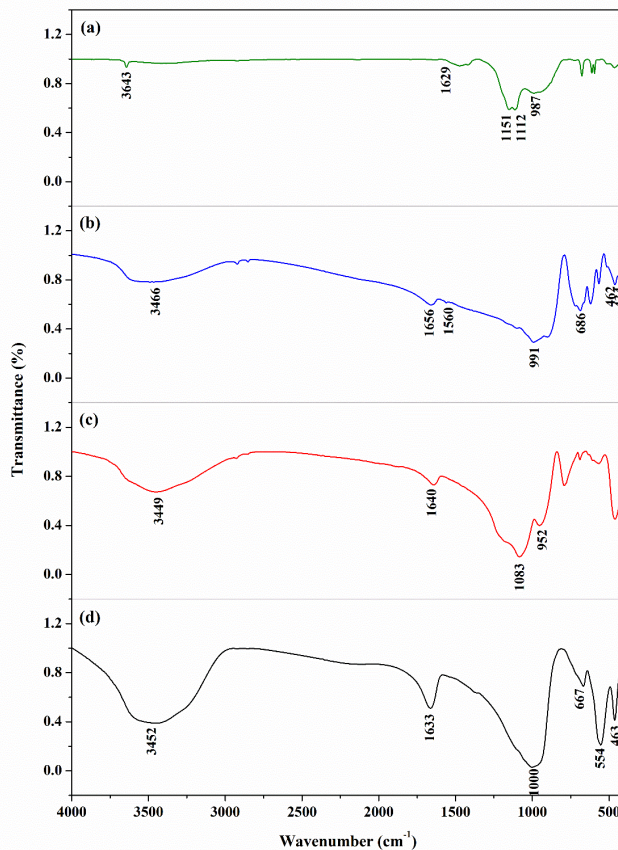


Figure 5. FTIR spectra of (a) CFA, (b) ZCFA, (c) CFAA and (d) ZCFAA

Table 2. Comparison of CEC values between previous studies and this study

Sample	CEC (meq/g)	Reference
Sodalite	0.56	(Franus <i>et al.</i> , 2014)
Commercial zeolite A	2.50	(Ren <i>et al.</i> , 2018)
Zeolite A	2.32	''
ZCFA	0.99	This study
ZCFAA	2.38	This study

3.5 CEC results

The CEC values of ZCFA and ZCFAA in this study are reported in milliequivalents/gram (meq/g). The CEC of ZCFA was 0.99 meq/g, which was lower than the value of 2.38 meq/g for ZCFAA (Table 2). The higher CEC of ZCFAA was the result of the removal of metal oxides caused by acid washing and was supportive of zeolite A synthesis. The results were in line with the previous studies of Molina and Poole (2004) and Kuwahara *et al.* (2008). A high CEC value was found to promote Na ion exchange in wastewater treatment (Fotovat *et al.*, 2009b). Acid washing before synthesis helped increase the CEC value, an indicator of success in producing zeolite A (Wang *et al.*, 2008). In previous studies, the CEC value of ZCFA was higher than that of sodalite (Franus *et al.* 2014), and the CEC value of ZCFAA was close to that of commercial zeolite A and slightly higher than that of synthesized zeolite A (Ren *et al.* 2018). Thus, the acid-washed sample was qualified for zeolite A synthesis. The produced zeolite A had a high CEC value appropriate for effective application for wastewater treatment with lower cost than commercial zeolite A.

4. Conclusion

CFA was used for zeolite A synthesis with and without acid washing by the two-step method. XRD, FESEM-FIB, EDX, XRF, FTIR, and CEC analysis were used to identify the crystalline phase patterns, surface areas, chemical compositions, chemical functional groups, and cation exchange capacities of the samples. CFA had a semicrystalline pattern with a spherical shape and smooth surface, and CFAA also had a semicrystalline pattern but had a cracked

and rugged surface. The crystalline pattern of ZCFA corresponded to the sodalite standard of JCPDS 00-037-0196 with a wool ball structure, while the pattern of ZCFAA was similar to the zeolite A standard of JCPDS 39 - 222 with a cubic structure. The EDX and XRF results showed that the Si and Al contents of CFA and CFAA were different: an increase in Si was observed in CFAA, while the contents of other elements decreased after acid washing. The EDX and XRF analyses showed that the Si/Al ratios in both ZCFA and ZCFAA were close to 1. Regarding the FTIR results, CFA and CFAA showed T-O (T = Si or Al) groups, C = O groups, O-H stretching and H-O-H bending. ZCFA and ZCFAA showed T-O-T and TO₄, which indicated sodalite and zeolite A, respectively. The CEC values of ZCFA and ZCFAA were 0.99 and 2.38 meq/g, respectively. Thus, the acid-washed sample was successfully synthesized, and all characterizations confirmed the occurrence of zeolite A, whereas the non-acid-washed sample displayed the characteristics of sodalite. Consequently, acid wash treatment is recommended for zeolite A synthesis from CFAA for utilization as a pollutant adsorbent for environmental remediation.

Declaration of competing interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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