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Bulletin of Chemical Reaction Engineering & Catalysis, 20 (3) 2025, 553-559

Original Research Article

Preparation and Characterization of Zeolite A Synthesized from Narathiwat White Clay

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Received: 16th July 2025; Revised: 18th August 2025; Accepted: 19th August 2025
Available online: 25th August 2025; Published regularly: October 2025

Abstract

Zeolite A is a widely used synthetic zeolite known for its high ion-exchange capacity and molecular sieving properties. This study explored the synthesis of zeolite A using Narathiwat white clay as a raw material. White clay, primarily composed of kaolinite ($Al_2Si_2O_5(OH)_4$), is considered a cost-effective source of aluminum and silicon for zeolite synthesis. The process involves the transformation of kaolinite into metakaolin via calcination, followed by hydrothermal crystallization under controlled conditions from natural white clay without conventional high-temperature calcination. Synthesized zeolite was characterized using X-ray diffraction (XRD), Differential Thermal Analysis (DTA), scanning electron microscopy (SEM), and X-ray fluorescence (XRF) spectroscopy to determine the elemental composition of the raw white clay and the synthesized zeolite A, while Fourier-transform infrared spectroscopy (FTIR) was used to confirm its structure and purity. Furthermore, the influence of the NaOH solution and the stability temperature of zeolite A are shown in this research. The optimum conditions for achieving zeolite A were calcined at 600 °C for 3 h in the first step, followed by using autoclaves at 200 °C for 24 h with a 3 M NaOH solution. The SEM results indicated that the Narathiwat white clay could be used to synthesize zeolite A, which exhibited a cubic morphology consisting primarily of silicon and aluminum. Notably, the crystallinity was influenced by the concentration of the NaOH solution employed. Moreover, the XRD and FTIR results demonstrate the feasibility of synthesizing high-quality zeolite A.

Keywords: Kaolinite; Hydrothermal synthesis; Autoclaves; Zeolite A

How to Cite: Sinchangreed, A., Watcharamaisakul, S., Janphuang, P. (2025). Preparation and Characterization of Zeolite A Synthesized from Narathiwat White Clay. *Bulletin of Chemical Reaction Engineering & Catalysis*, 20 (3), 553-559. (doi: 10.9767/bcrec.20445)

Permalink/DOI: https://doi.org/10.9767/bcrec.20445

1. Introduction

Zeolites are crystalline aluminosilicates with a three-dimensional porous structure possessing high value in various industrial applications [1]. Their unique framework includes silicon (Si), aluminum (Al), and oxygen (O) tetrahedra, forming channels and cavities that can selectively trap and release molecules [2,3]. Due to their molecular sieving, ion-exchange, and adsorption properties, zeolites are widely used in catalysis [4], water purification [5], gas separation [6], and detergent formulations [7]. Zeolite A has gained significant attention among different zeolite types due to its well-defined pore structure, high surface area, and excellent cation-exchange capacity. This makes it particularly useful in water softening, adsorption, and environmental remediation. While synthetic zeolites are commonly produced from pure chemical reagents such as sodium silicate and sodium aluminate [8,9], natural raw materials such as white clay are considered a cost-effective and eco-friendly alternative [10,11]. White clay, primarily composed of kaolinite ($Al_2Si_2O_5(OH)_4$), is a natural source of silicon and aluminum, which are essential for zeolite formation [12].

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Conventionally, synthetic zeolites are produced using pure chemical sources in silica and alumina, such as sodium silicate and sodium aluminate [13,14]. However, the production of these materials can be expensive and environmentally taxing. As a result, increasing attention has been directed toward using natural and low-cost raw materials such as clays, fly ash, and agricultural residues for zeolite synthesis [15,16]. In recent years, attention has shifted toward more sustainable synthesis approaches, driven by the need to reduce energy consumption, lower production costs, and minimize environmental impact. Clays are rich in silica and alumina, the essential components of zeolite frameworks, making them suitable and cost-effective precursors. Furthermore, clay-based synthesis often requires fewer chemical additives and can be conducted under hydrothermal conditions compared to conventional routes. Recent developments in green chemistry and materials science have enabled the optimization of clay activation processes, including thermal treatment and alkali fusion, to enhance their reactivity. These pathways to sustainable synthesis not only contribute to the circular economy by utilizing natural or industrial waste-derived clays but also align with environmental goals by reducing the carbon footprint associated with zeolite production. White clay consists of kaolinite and represents a promising alternative due to its abundance, low cost, and appropriate Si/Al ratio suitable for zeolite A formation [17]. The traditional method for producing zeolite A from natural kaolin begins with calcination, which decreases its resistance to chemical attack and converts it into an amorphous substance. This process yields metakaolin, which is then synthesized into zeolites through a hydrothermal reaction in an alkaline environment.

This research focuses on the synthesis of zeolite A from Narathiwat white clay, acquired from southern Thailand, through a hydrothermal process. The study aims to evaluate the feasibility of using white clay as a raw material by optimizing the synthesis conditions and characterizing the produced zeolite, thus developing a sustainable synthesis route. This study contributes to the advancement of environmentally friendly materials for industrial and environmental applications.

2. Materials and Methods

2.1 Preparation of Metakaolin and Zeolite A

The synthesis of zeolite A from white clay involves 2 steps as follows. The first step is metakaolinization, which involves the thermal treatment of raw white clay at a temperature of 600 °C for 3 h. The second step is the chemical treatment of the prepared metakaolin using sodium hydroxide (NaOH) solutions at concentrations of 1, 2, 3, and 4 M.

In the conventional hydrothermal synthesis technique, white clay is ground and screened through a sieve No.100 (aperture of 150 μm). Then, 200 grams of white clay powder in the particle range of 1-50 μm is calcined in a furnace at 600 °C for 3 h. This process converts kaolinite ($Al_2Si_2O_5(OH)_4$) into metakaolin ($Al_2Si_2O_7$). During this transformation, the structure of the kaolin changes, and volatile matter is released, leading to the formation of metakaolin, as illustrated in the following equation:

$$Al_2Si_2O_5\,(OH)_4\,\text{(kaolin)} \rightarrow Al_2Si_2O_7\,\text{(metakaolin)} + 2H_2O \qquad (1)$$

A total of 20 g of metakaolin was treated with 100 mL of NaOH solutions at concentrations of 1, 2, 3, and 4 M. The mixture was placed in stainless steel autoclaves lined with Teflon and heated at 200 °C for 24 h to facilitate the exchange of sodium ions within the metakaolin structure. Following this treatment, the sample was washed with deionized water to eliminate unreacted NaOH until the pH reached about 7. After that, it was filtered and dried in an oven at 100 °C overnight. Different samples with different NaOH concentrations were prepared to determine the optimal concentration for synthesizing zeolite A.

2.2. Characterizations

The material characterization of synthesized zeolite A and Narathiwat white clay was analyzed on a powdered sample by Energy Dispersive Spectrometer X-ray Fluorescence (ED-XRF) (Horiba/XGT-5200) to determine the composition. Simultaneous Thermal Analysis (NETZSCH, STA449F3 / QMS403C) was used to analyze the thermal behavior of Narathiwat white clay. XRD analyzed the crystalline phases present in Narathiwat white clay, metakaolin, and synthesized zeolite A. An X-ray Diffractometer (Bruker D2 Phaser) with Cu kα radiation was operated at 40 kV and 30 mA. Data were collected in the 2θ range of 5-70°. The morphology of Narathiwat white clay and synthesized zeolite A was observed using a Scanning Electron Microscope (SEM-EDS - JEOL/JSM-6010LV). SEM micrographs were obtained at an operating voltage of U = 15 kV and a magnification of 1000X. FTIR spectra were recorded by a Fourier Transform Infrared Microscope Spectrophotometry (FT-IR Microscope/Bruker, TENSOR 27-Hyperion-2000) for the identification and characterization of the chemical composition of Narathiwat white clay and zeolite A.

3. Results and Discussion

3.1. XRF Analysis

X-ray fluorescence (XRF) spectroscopy was employed to determine the composition of the raw white clay and the synthesized zeolite A. This analysis aimed to evaluate the transformation of the clay material during the synthesis process and confirm the formation of a zeolitic structure based on compositional changes, particularly in the Si/Al ratio and sodium content.

Table 1 presents the comparative XRF results of various white clays and synthesized zeolite materials. The raw white clay was primarily composed of silica (SiO_2) and alumina (Al_2O_3), with minor quantities of iron oxide (Fe_2O_3), potassium oxide (K_2O), calcium oxide (CaO), and magnesium oxide (MgO). After synthesis, a marked increase in Na_2O content was observed in the zeolite A sample, indicating the successful incorporation of sodium into the crystal framework during hydrothermal treatment. The significant increase in Na_2O content observed during the synthesis process demonstrates a strong correlation with the formation mechanism and stabilization of the zeolite framework. Sodium ions (Na^+), derived from Na_2O, serve as essential structure-directing and charge-compensating agents, particularly in low-silica zeolites such as zeolite A (LTA-type). Their presence facilitates the development of a well-ordered aluminosilicate framework by balancing the negative charges resulting from isomorphic substitution of Si^{4+} by Al^{3+} within the tetrahedral network. Increased Na_2O concentration contributes to elevated alkalinity in the reaction medium, which plays a crucial role in promoting the dissolution of silica and alumina sources. This enhanced solubility accelerates the nucleation and subsequent crystallization of zeolite structures through a dissolution - reprecipitation mechanism. Moreover, the Na_2O content directly influences phase selectivity during hydrothermal synthesis. Lower sodium concentrations tend to yield amorphous or poorly crystalline phases, whereas elevated Na_2O levels favor the formation of sodium-rich crystalline zeolite phases such as zeolite A. This is attributed to the role of Na^+ in stabilizing the intermediate aluminosilicate species and promoting the assembly of the targeted microporous framework.

The Si/Al ratio of Narathiwat white clay was approximately 1.27, closely matching the theoretical ratio for zeolite A (NaA-type zeolite) synthesis as confirmed by the calculation below Table 1 [18], confirming successful structural transformation. The significant reduction in Fe_2O_3 and other impurities also suggests the improved purity and crystallinity of the final product. These results demonstrate the effectiveness of using white clay as a raw material for synthesizing zeolite A and achieving the elemental composition through the applied synthesis process.

3.2. Differential Thermal Analysis (DTA) of Narathiwat white clay

Figure 1 illustrates the DTA curve of Narathiwat white clay. DTA thermograms exhibit endothermic peaks with the maximum at 105 and 570 °C, which can be attributed to the removal of adsorbed water, dihydroxylation of clay minerals, and release of structural hydroxyl groups where kaolinite turns to metakaolin. Therefore, the calcined temperatures used in this experiment were around 600 °C according to the DTA results.

Table 1. The chemical XRF composition of white clays and zeolite A.

Element	Narathiwat white clay (wt%)	Lampang white clay (wt%)	Ranong white clay (wt%)	Zeolite (wt%)
SiO_2	54.222	64.478	73.519	43.841
Al_2O_3	36.285	24.763	13.223	35.654
Fe_2O_3	1.431	1.268	2.209	1.287
K_2O	2.145	3.973	3.285	2.023
TiO_2	0.078	0.046	0.089	0.033
CaO	0.184	0.212	0.104	0.121
MnO_2	0.045	0.246	0.071	0.018
Na_2O	4.223	4.196	5.232	16.456
MgO	1.387	0.818	2.268	0.567

(1). Si/Al calculation of Narathiwat white clay : $SiO_2 = 54.222 \times 258.18 / (100 \times 60.08) = 2.33$ mol; $Al_2O_3 = 36.285 \times 258.18 / (100 \times 101.96) = 0.917$ mol; $Si/Al = 2.33 / (0.917 \times 2) = 1.27$

(2). Si/Al calculation of Zeolite A: $SiO_2 = 43.841 \times 1807.26 / (100 \times 60.08) = 13.19$ mol; $Al_2O_3 = 35.654 \times 1807.26 / (100 \times 101.96) = 6.32$ mol; $Si/Al = 13.19 / (6.32 \times 2) = 1.04$

3.3. X-ray Diffraction (XRD)

Figure 2 illustrates the X-ray diffraction (XRD) pattern of Narathiwat white clay. The significant phases of Narathiwat white clay are kaolinite, illite, and quartz, which are impurities. Narathiwat white clay metakaolin, calcined at 600 °C, when compared with the XRD pattern of Narathiwat white clay, was found to have the kaolinite phase disappear after thermal treatment due to the dihydroxylation in molecules of white clay, resulting in the change of white clay structure from crystal to amorphous. According to the Differential Thermal Analysis (DTA) results, as shown in Figure 1, the Narathiwat white clay loses water by dihydroxylation at calcined temperature. Moreover, the X-ray diffraction (XRD) pattern of the synthesized zeolite A using NaOH concentrations of 3M displayed sharp and intense peaks at characteristic 2θ values. The synthesized products matched the characteristic peaks of zeolite A at 2θ values of 7.1, 10.1, 12.4, 16.2, 21.6, 24, 26, 27.2, 30, 30.9, 32.6, and 34.2 [19]. The presence of these well-defined peaks confirms the successful formation of pure zeolite A, with no detectable impurities or secondary phases such as sodalite or amorphous aluminosilicates. The intensity and sharpness of the peaks imply a high degree of crystallinity, which can ensure the microporous structure and uniform pore size distribution typical of zeolite A. These results align with previous studies, such as the research by Smaihi [19] and other contemporary works, which reported similar XRD patterns for high-purity synthetic zeolite A [20]. Besides, no significant broadening of peaks was observed, suggesting that the crystal domains are sufficiently large and well-ordered. This high crystallinity is critical for the ion-exchange capacity and molecular sieving performance of the material. Combined with the SEM and FTIR results, the XRD findings strongly support the successful synthesis of high-quality zeolite A. In addition, Figure 3 demonstrates the XRD patterns of zeolite A at different concentrations of NaOH solution. At 1 and 2M NaOH, the zeolite A pattern was not observed. The optimum conditions for synthesizing zeolite A were obtained when using NaOH concentrations of 3 and 4M, with the mixture heated to 200 °C for a 24 h reaction time.

3.4. SEM Micrographs

The micrographs in Figure 4 show that the crystallites of Narathiwat white clay are in a random orientation before treatment. In contrast, the SEM micrographs of the synthesized zeolite A in Figure 5 revealed uniform, well-defined cubic crystals, which are characteristic of the LTA-type framework. The particles appeared relatively homogeneous in terms of size and morphology, consistent with the values reported in the literature for conventionally synthesized zeolite A [21]. This regular cubic morphology indicates successful crystallization and effective control over the nucleation and growth phases during synthesis. The surfaces of the crystals appeared smooth and free of significant defects, with minimal signs of amorphous material or secondary phases, suggesting a high degree of purity. Slight agglomeration was observed in some regions, which is commonly reported in zeolite systems and may result from drying or interparticle interactions during sample preparation. However, the overall structural integrity of the crystals was not impacted. The well-defined crystal shape and absence of irregular structures support the high crystallinity observed in the XRD results, further confirming the successful formation of zeolite A.

3.5. Fourier Transform Infrared Analysis of Zeolite A from Narathiwat White Clay

The FTIR spectra for zeolite A and Narathiwat white clay are presented in Figure 6,

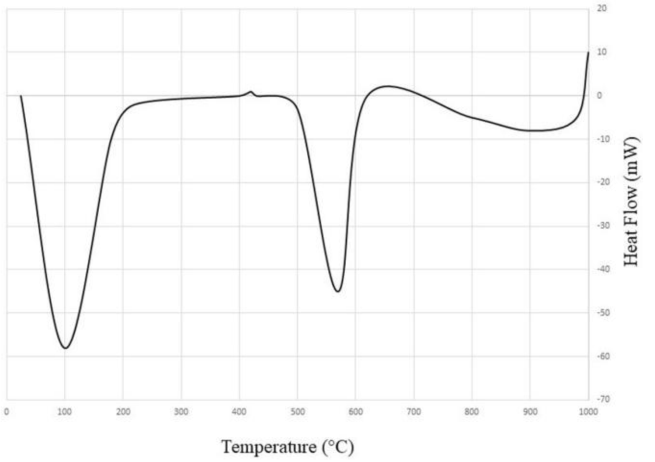

Figure 1. DTA curve of Narathiwat white clay.

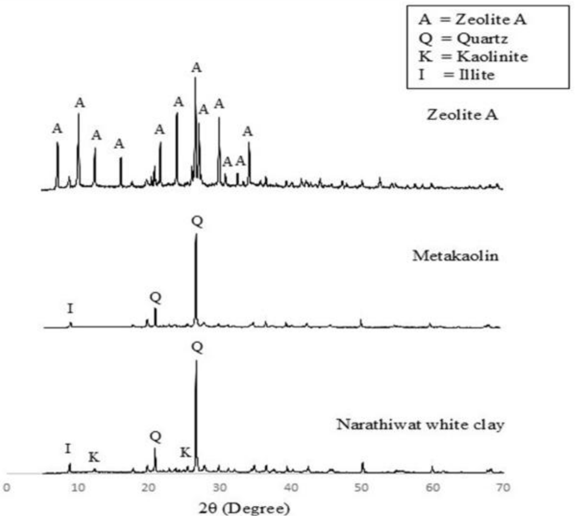

Figure 2. X-Ray diffraction pattern of Narathiwat white clay, metakaolin, and zeolite A.

along with the FTIR spectra of Narathiwat kaolin. The peaks observed at 3693 and 3622 cm^{-1} are linked to the stretching vibrations of hydroxyl groups within the structure of kaolin [22]. The peak at 3622 cm^{-1} specifically corresponds to the stretching vibration modes of the inner hydroxyl groups, mainly found in the octahedral and tetrahedral sheets. The peaks at 3693 cm^{-1} are associated with the stretching vibrations of the hydroxyl (OH) groups located on the inner surface, which are found on the surfaces of the octahedral sheets in the neighboring kaolin layer [23]. The peak at 1107 cm^{-1} is linked to the stretching vibrations of the Si-O bonds within the kaolin structure. In contrast, the peaks at 1029 cm^{-1} and 1002 cm^{-1} arise from the lattice vibrations of both Si-O-Si and Si-O-Al bonds. Moreover, the bending vibrations of the OH groups to the peaks at 910 cm^{-1} and 942 cm^{-1} correspond to the "surface OH bends" and the "inner OH bends", respectively. These vibrations of the OH groups are typically caused by the bonds in the Al-OH groups. The peaks at 790 cm^{-1} and 748 cm^{-1} were assigned to the Si-O-Si bonds, and finally, the 524 cm^{-1} peaks were assigned to the deformation of the vibration of the Si–O bonds [24]. The FTIR spectra of zeolite A illustrate broad bands around 466 cm^{-1}, which are related to the internal bending modes of the TO_4 (T = Si, Al). Additionally, bands at 550 cm^{-1} correspond to the vibrational modes of the double rings present in zeolite A. The prominent bands observed at 1002 cm^{-1} are linked to the T-O asymmetric stretching modes, while the band at 3446 cm^{-1}, along with the weaker band at 1650 cm^{-1}, is associated with water molecules and OH groups in zeolite A [25].

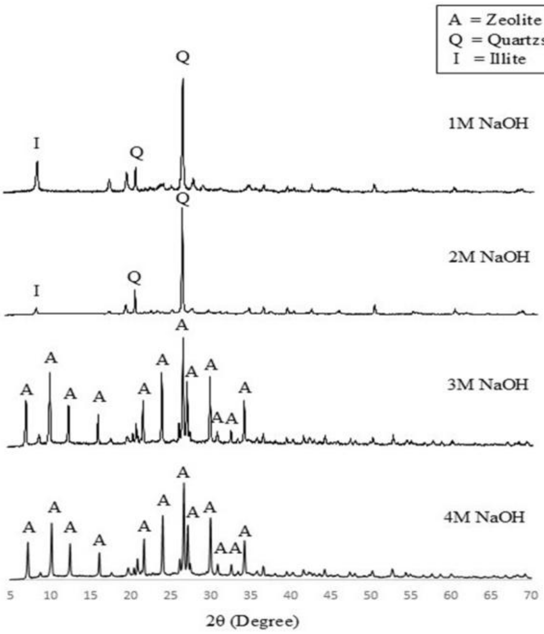

Figure 3. The XRD patterns of zeolite A prepared by heating at 200 °C in the NaOH solution at different concentrations (1, 2, 3, and 4 M).

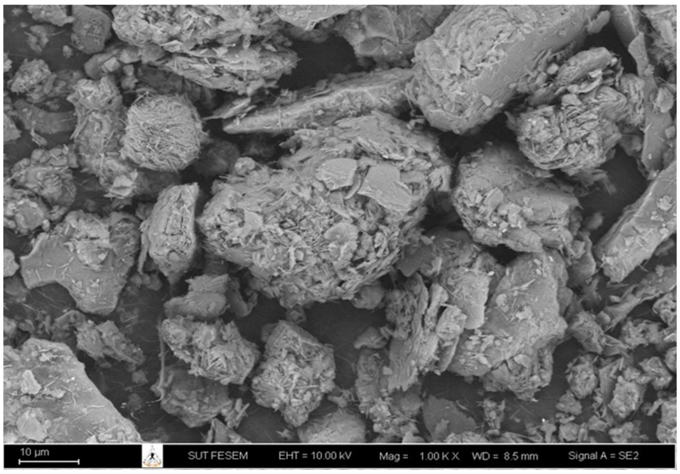

Figure 4. SEM micrograph of Narathiwat white clay.

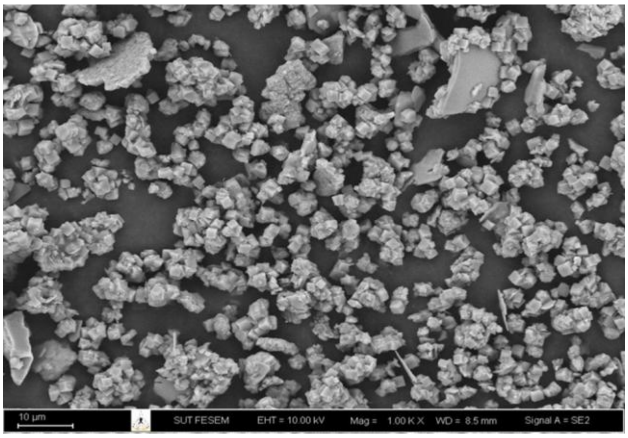

Figure 5. SEM micrograph of zeolite A.

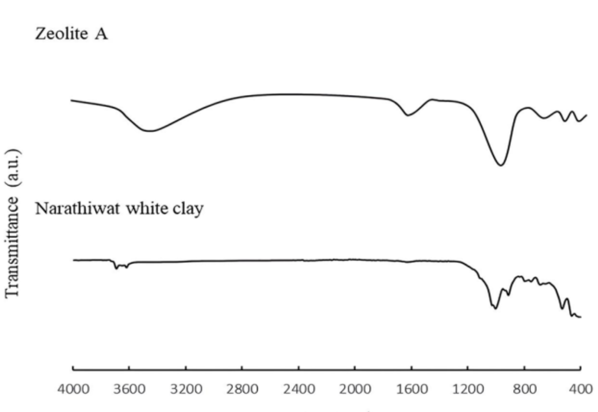

Figure 6. FTIR spectra of Narathiwat white clay and zeolite A.

4. Conclusions

This study successfully demonstrated the synthesis of zeolite A using locally sourced white clay (Narathiwat white clay) as a low-cost and environmentally friendly raw material. The first step starts with Narathiwat white clay calcined at 600 °C for 3 h. The synthesis parameters for zeolite A derived from Narathiwat white clay were optimized for the first time. The optimized parameters include a base of 3M NaOH, a crystallization time of 24 h, a temperature of 200°C, and a 24 h. The transformation of the clay into a crystalline zeolite structure was confirmed utilizing various characterization techniques. Differential thermogravimetric analysis further verified the thermal stability of the Narathiwat white clay, and its typical water loss behavior associated with the metakaolin structure for zeolite synthesis. XRF analysis confirmed the successful incorporation of sodium and an appropriate Si/Al ratio (~1.49), consistent with the zeolite A framework. The SEM results showed that the cubic crystal form was achieved. Overall, the results confirm that white clay is a viable and effective alternative raw material for synthesizing zeolite A. The synthesized product possesses properties suitable for applications in ion exchange, adsorption, and catalysis. This approach reduces production costs and promotes the utilization of abundant natural resources, contributing to sustainable material development.

Acknowledgments

The authors would like to acknowledge the Faculty of Materials Engineering, Suranaree University of Technology, and Synchrotron Light Research Institute for supporting the research fund.

CRedit Author Statement

Author Contributions: Sukasem Watcharamaisakul conceived the conceptualization, methodology, review, and editing of the manuscript. Abhirak Sinchangreed, author, performed methodology, formal analysis, investigation, validation, and writing-original draft. Pattanaphong Janphuang contributed to the experimental plan, measurement, data analysis, and revised the manuscript. All authors have read and agreed to the published version of the manuscript.

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