



Research Article

Effect of Calcination Temperature on Performance of Photocatalytic Reactor System for Seawater Pretreatment

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Abstract

Conservative desalination technology including distillation requires high energy and cost to operate. Hence, pretreatment process can be done prior to desalination to overcome energy demand and cost reduction. Objective of this research is to study the effect of calcination temperature of hybrid catalyst in photocatalytic reactor system in the seawater desalination, i.e. salt removal in the seawater. The catalyst was synthesized via wet impregnation method with 1:1 weight ratio of TiO₂ and activated oil palm fiber ash (Ti:Ash). The catalyst was calcined at different temperature, i.e. 500 °C and 800 °C. The study was carried out in a one liter Borosilicate photoreactor equipped with mercury light of 365 nanometers for two hours with 400 rpm mixing and catalyst to seawater sample weight ratio of 1:400. The Chemical Oxygen Demand (COD), pH, dissolved oxygen (DO), turbidity and conductivity of the seawater were analyzed prior and after the testing. The fresh and spent catalysts were characterized via X-Ray Diffractogram (XRD and Nitrogen physisorption analysis. The calcination temperature significantly influenced the adsorption behaviour and photocatalytic activity. However, Ti:Ash which calcined at 800 °C has less photocatalytic activity. It might be because the surface of fiber ash was sintered after calcined at high temperature. The Ti:Ash catalyst that calcined at 500 °C was found to be the most effective catalyst in the desalination of seawater by reducing the salt concentration of more than 9 % compared to Ti:Ash calcined at 800 °C. It can be concluded that catalyst calcination at 500 °C has better character, performance and economically feasible catalyst for seawater desalination. Copyright © 2016 BCREC GROUP. All rights reserved

Keywords: Pre-treatment; Oil palm waste; photocatalyst; Seawater; Titanium Dioxide

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1. Introduction

The processing or desalination of natural seawater converted to drinking water by using various methods are increasing throughout the

world [1]. Seawater becomes the main water source worldwide. In fact, Middle East and North Africa have relied on desalted seawater as their viable solutions in restraining the water shortage [2]. Seawater contains high total dissolved solid and it is called salinity. The existing technology such as membrane processes and distillation processes required high energy consumption to convert the seawater become

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the fresh water [3]. Indeed, the innovative method for seawater desalination is needed to improve the quality and source of drinking water. Because of the high energy of seawater desalination processes, alternative pre-treatment processes of seawater needed to be developed to save some energy consumption in seawater desalination for example removing some of salts from the seawater before desalinating processes take over to produce fresh water.

The updated technologies, such as: photocatalyst for the seawater purification, have been studied to receive the sign of inadequate water in the future. During the last decade, photocatalytic reaction, a technique of advanced oxidation processes (AOP) has been an alternative to the water and wastewater treatment [4]. Photocatalyst are semiconductors that have capabilities to cause or accelerate the chemical reactions upon light absorption notably sunlight [5] can be one of the alternative ways to be used in seawater pre-treatment. Indeed, commercially available photocatalytic water treatment plant using artificial UV-light as the energy source is available and can be considered applied in the photoreaction system [6]. Currently, many researches on water purification using the semiconductor photocatalyst by TiO_2 catalyst are carried out. TiO_2 catalyst becomes well-known in the research field and industrial field. TiO_2 catalyst also becomes the important materials in many practical applications and is used as a desiccant, brightener or reactive radiator [6] and also can be applied in energy catalyst (in water splitting to produce hydrogen fuel), an environmental catalyst (in water and air purification) or an electron transport medium (in dye-sensitized solar cells) [7].

TiO_2 catalysts are naturally occurring oxide of titanium. TiO_2 is the one of catalyst that has competent photocatalytic activity, high stability and lowest cost [8]. TiO_2 has capability and efficiency in removing the wide range of pollutants. The removal of pollutant in a solution by TiO_2 significantly improves the efficiency of photocatalyst [9] and allows the complete degradation of organic pollutants into harmless species such H_2O and CO_2 [10]. However, the formations of CO_2 are not instantaneously but sometimes long-living intermediates and have been investigated as a limited extent [11]. The efficiency decomposition of high salt concentrations containing in seawater by adapting the application of TiO_2 photochemical reaction will be investigated in this studies. The advantages of TiO_2 are inexpensive, non-toxic, it has excellent in high production of hydroxyl radicals and

stable in physical and chemical properties [12, 13].

The application of TiO_2 catalyst in photoreaction is not well explored yet. Modified the surface of TiO_2 is one of the promising way to enhance the photocatalytic performance. Zeolite, carbon nanotube and active carbon are among porous substrate providing pore structures for dispersing TiO_2 photocatalyst [14]. Oil palm fiber ash contains an active carbon and can be a promising adsorbent for the removal of the pollutants in the water. Moreover, oil palm fiber ash also has some minerals like Alumina, Calcium oxide and silica which can also act as an adsorbent in purifying water [15]. Malaysia produced over 11.9 million tons of oil and biomass was produced approximately 100 million tons by 6 million hectares of plantations in Malaysia [16]. Combining TiO_2 with various nanocarbon materials enhances photocatalytic activities by controlling the band gap to photosensitization, mineralization of e^-/h^+ recombination, etc. [17].

In this study, the effect of calcination temperature on the hybrid catalyst Ti:Ash and its photocatalytic activity on seawater desalination were investigated. Biomass ash derived from oil palm fiber ash was used as a support for TiO_2 photocatalyst for seawater desalination. The hybrid TiO_2 and biomass ash catalyst was synthesized via wet impregnation technique with 500 °C and 800 °C calcination temperature.

2. Materials and Methods

2.1. Materials and catalyst preparation

Oil palm ash was obtained from Felda Lepar Hilir 3 Palm Oil Mill, Gambang, Kuantan, Pahang. TiO_2 was purchased from Sigma Aldrich with 99 % purity. The seawater was obtained from Teluk Cempedak, Kuantan Pahang (Latitude: 3.815, Longitude: 103.363). The seawater was kept at 4 °C.

The catalyst was prepared via wet impregnation. A percentage weight ratio of TiO_2 and oil palm ash (Ti:Ash) 50:50 was mixed with deionized water and stirred at 80 °C for four hours. It was then dried for overnight at 100 °C in the oven. The catalyst was calcined for either 500 °C or 800 °C in the furnace for four hours. The catalyst was crushed and sieved to the size of <100 μm to have homogenized catalyst size.

2.2. Catalyst characterization

X-ray diffraction (XRD) patterns of the samples were collected on an X-ray powder diffrac-

tometer. The samples of fresh and spent catalyst were scanned in the range from 10 to 80 of 2θ. The BET surface area (m²/g), pore volume (cm³/g) and pore size (nm) of each catalyst was obtained via nitrogen adsorption using Micromeritics ASAP2020. The catalyst was degassed up to 200 °C for 6 hours prior to analyze.

2.3. Photocatalytic reactor set up and catalyst testing

All the experiments were carried out in a photocatalytic reactor as shown in Figure 1. The seawater sample to photocatalyst (Ti:Ash with a weight ratio of 50:50) was tested in a one liter borosilicate glass cylinder photocatalytic reactor equipped with UV light of 365 nm wavelength. This system was operated in a black box to prevent the UV light from exposure to the surrounding. The mixture of seawater and catalyst with a weight ratio of catalyst to seawater sample of 1:400 was tested in the reactor for two hours. The magnetic stirrer was used with a speed rotation of 400 rpm to make sure that the solution was well mixed. Three regulator fans were installed to control the temperature of the black box in order to prolong the life span of the UV bulb.

After the investigation, the catalyst was filtered out from seawater. The quality of water was analyzed before and after the investigation. The pH, COD, conductivity, DO and turbidity of the water were determined. The measurement of seawater's acidity and alkalinity

was determined by using pH meter (Hach Sension+ 150 mm). Meanwhile, the conductivity was analyzed via Hach Sension+ 150 mm model. The amount of organic compound in the seawater was measured by using Hach (DRB 200) model. The COD Digestion Reagent Vial High Range (435 COD HR) was used to determine the COD. DO concentration for seawater was measured by using Dissolved Oxygen Meter (YSI 5100) model. Turbidity for seawater was measured by using Turbidity Meter Hach 2100 model. The collected gas was analyzed through gas chromatography (GC-TCD) model Agilent 19095p. The column oven held isothermally at 50 °C, while the injector oven and detector were kept at 150 °C.

3. Results and Discussion

3.1. Catalyst characteristic

The catalyst was synthesized via wet impregnation method. The fresh and spent catalysts were characterized their surface structure and crystal structure by using N₂ adsorption and XRD respectively.

The surface structure analysis result of Ti:Ash of 50:50 via N₂ adsorption is tabulated in Table 1. Both catalysts are mesoporous in which those pores falls in the range of 2-50 nm. The surface area of Ti:Ash catalyst that calcined at 500 °C was decreased from 9.15 m²/g to 6.67 m²/g as shown in Table 1. The same trend is also obtained for its pore size. The pore size

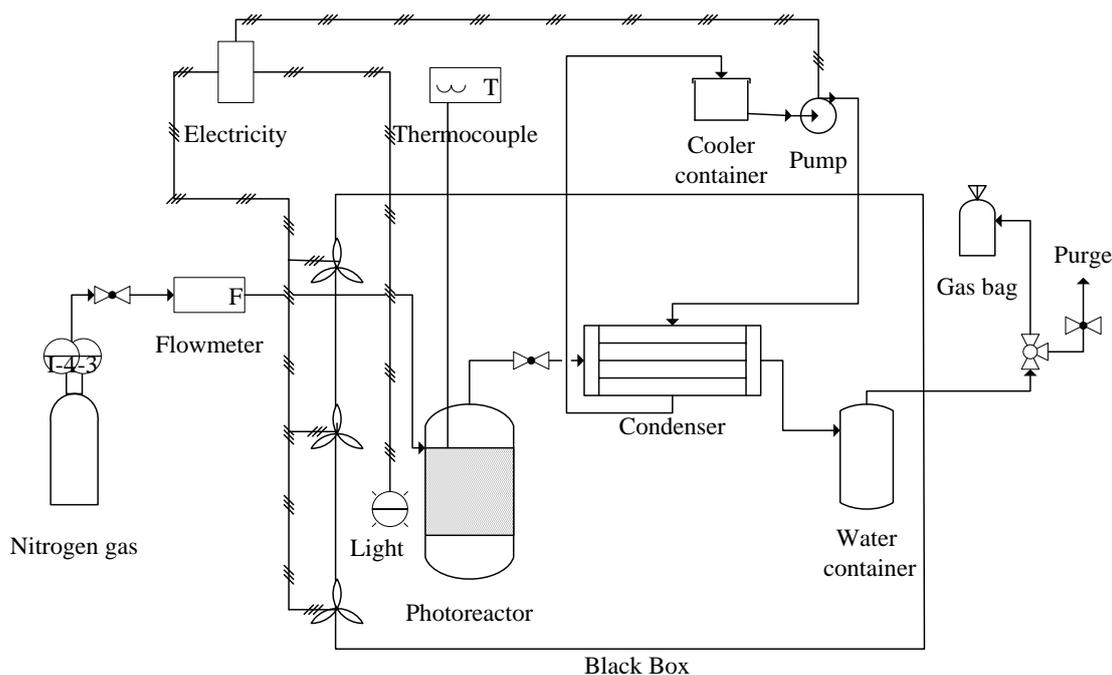


Figure 1. Schematic diagram of batch photocatalytic reactor set up

reduced significantly from 18.63 nm to 15.54 nm. In addition, pore volume also decreased from $3.49 \times 10^{-2} \text{ cm}^3/\text{g}$ to $2.29 \times 10^{-2} \text{ cm}^3/\text{g}$. This might be caused by the deposited crystal on the spent catalyst that leads to decrease the catalyst pore volume and pore size.

Meanwhile, Ti:Ash that calcined at 800 °C decreased from 5.98 m²/g to 5.47 m²/g. Conversely, the pore volume and pore size of Ti:Ash that calcined at 800 °C increased after the testify from $1.34 \times 10^{-2} \text{ cm}^3/\text{g}$ to $1.59 \times 10^{-2} \text{ cm}^3/\text{g}$ and 13.59 nm and 15.01 nm respectively. In comparison with Ti:Ash that calcined at 500 °C, the Ti:Ash that calcined at 800 °C has smaller surface area, pore volume and pore size. This may be due to the surface of Ti:Ash that calcined at 800 °C was breakage and damaged during calcination. High calcination temperature play the main role to destroy the surface of Ti:Ash catalyst. It is claimed that the crystalline structure of TiO₂ will be changed after calcination at high temperature and the specific surface area, particle size and other properties of TiO₂ can also be altered after calcinations at difference temperature [6].

Figure 2 illustrates that the XRD patterns for both Ti:Ash that calcined at 500 °C and 800 °C before and after the testing. It clearly can be seen that the presence of the salt at certain peak is detected on the both spent catalysts. The spent catalyst for Ti:Ash that calcined at 500 °C indicates that the present of NaCl phase peaks at 31.75°, 45.47° and 56.53°. The crystalline phase of spent Ti:Ash that calcined at 800 °C also shares the same peak profile. Yalçın and Mutlu [18] also reported that the salt peak did present around 30° to 60°. It can be seen that the salt peaks of Ti:Ash calcined at temperature of 500 °C after were bigger compared to Ti:Ash calcined at temperature 800 °C. It indicates that the catalyst at 500 °C has better efficiency in absorbance activity compared to Ti:Ash that calcined at 800 °C. The Ti:Ash catalyst that calcined at 800 °C adsorbed lesser amount of salt as it might be because of the changes of crystalline structure due to calcination temperature affect.

Other than that, it also can be observed from fresh catalyst that calcined at 800 °C has smaller peak intensity in XRD patterns than

Table 1. BET of Ti:Ash catalyst that calcined at different temperatures

Catalyst weight ratio, %	Specific surface area (m ² /g)		Pore volume ×10 ² (cm ³ /g)		Pore size (nm)	
	Before	After	Before	After	Before	After
500 °C	9.15	6.67	3.49	2.29	18.63	15.54
800 °C	5.98	5.47	1.34	1.59	13.59	15.01

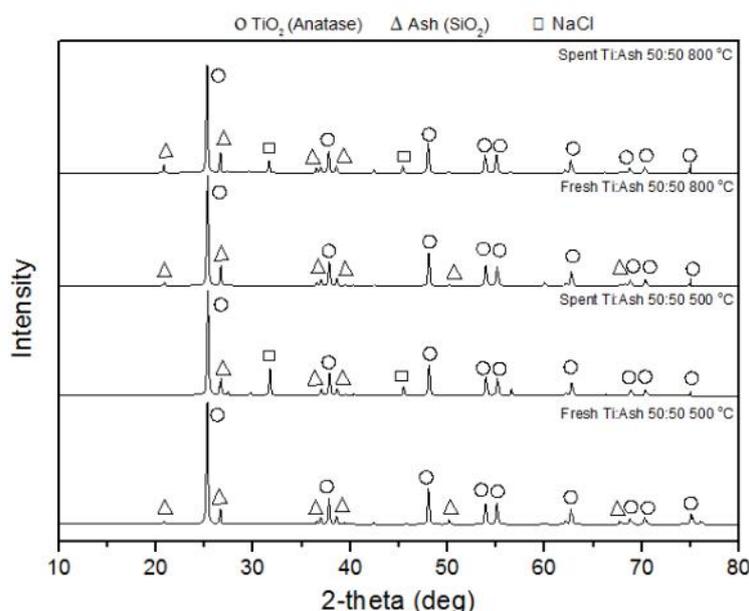


Figure 2. The XRD pattern of Ti:Ash before and after testify for different calcination temperature

catalyst that calcined at 500 °C. This might be caused by the effect of high calcination temperature (800 °C) which ruined the raw structure of fiber ash. The major component of fiber ash is silica. Hirano *et al.* [19] reported that the surface area decrease after high temperature (800 °C) affect that strike the surface area change due to sintering of the silica matrix but not to change the titania form.

According to Sun *et al.* [20], the improvement of the crystallinity of TiO₂ was related to the high calcination temperature. TiO₂ have a predominant crystal structure of anatase type crystal phase (Figure 2). No obvious diffraction peaks of TiO₂ was detected for the both calcined temperatures, as the fresh Ti:Ash remained to be dominated by anatase phase. No extra peaks detected, such as rutile or rutile-anatase mixed phase of TiO₂. This might be the TiO₂ particles have better thermal stability on the surface of fiber ash. Besides, the presence of fiber ash might suppress the diffusion between anatase crystallite and interface on the surface of the anatase crystallite. The effect created by promoter such as silica (SiO₂) led to the crystallite growth retarded and causes the suppression of the phase transformation [19].

Jamieson and Olinger [21] had reported that the transformation of anatase phase to rutile phase is not instantaneous; it is dependent on time because it is reconstructive and depends on the synthesis parameters, which in turn effect on the catalyst phase. The parameters that had been reported were impurities from raw materials and container, morphology of the catalyst, sample preparation technique, heating rate, surface area, particle size, volume of sample, supporting materials (dopants) and etc. [22]. It is claimed that the anatase was easily transformed to rutile phase when heated approximately or above at temperature of 600 °C to 800 °C [23, 24]. But, a study stated that the impurities present in anatase influence the transformation in the range of 600 °C to 1100

°C temperatures [25]. The investigation was done by Hirano *et al.* [19] also indicates that the anatase phase maintain after heating at 1000 °C and no trace of rutile up to 1300 °C. Anatase phase is necessary for photocatalytic reaction because the anatase phase is more photoactive than rutile [6, 26].

3.2. Effect of calcination to water desalination process

The investigation on the parameter of seawater which was pH, conductivity, Turbidity, COD and DO was carried out. Table 2 tabulates the result of the seawater desalination via photocatalytic reaction.

It can be observed that better water quality was obtained after using the Ti:Ash catalyst in treating the seawater. Table 2 indicates that the percentage reduction of turbidity after the testing was achieved more than 90 % for both catalysts. Moreover, the cloudiness of water also reduces significantly after filtration to separate the catalyst from seawater. In addition, it also can be observed that the COD of Ti:Ash that calcined at 500 °C reduced from 770 mg/L to 580 mg/L which signifies of up to 24 % reduction compared to Ti:Ash that calcined at 800 °C. The Ti:Ash catalyst that calcined at 800 °C only managed to decline from 770 mg/L to 655 mg/L which was only up to 14 % reduction. It clearly proves that the Ti:Ash that calcined at temperature 500 °C shows better performance in purifying the seawater compared to Ti:Ash that calcined at temperature 800 °C.

The conductivity of seawater via Ti:Ash that calcined at 500 °C managed to reduce up from 48.7 mS/cm to 44.3 mS/cm which made up about 9 % reduction. Meanwhile, Ti:Ash calcined at 800 °C reduced from 48.7 mS/cm to 47.3 mS/cm (2 % reduction) is also can be seen in Table 2. The photoreduction activity that occurred on the catalyst surface active site and

Table 2. The data analysis of seawater desalination

Seawater Sample	Seawater sample (Initial)	Ti:Ash, 500 °C	Ti:Ash, 800 °C
Analytical test			
Conductivity (mS/cm)	48.7	44.3	47.3
COD (mg/L)	770	580	655
Turbidity (NTU)	2.78	0.11	0.08
pH	8.17	7.57	7.65
DO (mg/L)	5.44	5.59	5.50

adsorption of salt in seawater is the reason why the conductivity of seawater declined after the testing. The Ti:Ash catalyst that calcined at 500 °C are able to adsorb more salt in seawater compared to Ti:Ash that calcined at 800 °C. The results demonstrate that the adsorption capacity of samples was well agreed with XRD analysis (Figure 2).

The adsorption of salt on Ti:Ash surface is a prerequisite for the photocatalytic reduction. It also can be observed that the Ti:Ash that calcined at 800 °C has lower surface area and has smaller pore size than Ti:Ash that calcined at 500 °C. The Ti:Ash that calcined at 800 °C does not change much after the test. This effect might be reason why Ti:Ash that calcined at 800 °C has lower activity than Ti:Ash that calcined at 500 °C. It is because calcination temperature at 500 °C led to higher surface area of Ti:Ash (as tabulated in Table 1) and be the responsible to improve the adsorption capacity and photocatalytic activity. In addition, high calcination temperature leads to gasify carbon in the oil palm fiber ash. Carbon is fully gasified at 600 °C [27]. Activated carbon is considered as an adsorbent for effluent treatment and is commonly utilized for the eliminating of various pollutants [28]. As higher calcination temperature was applied, less activated carbon remained in the catalyst. This might also be the reason of less adsorption capacity of Ti:Ash catalyst that calcined at 800 °C. Rubio *et al.*, [29] also reported that the adsorption of inorganic materials on the TiO₂ can form a thick layer making it less penetrable by light resulting in the TiO₂ less effective in terms of promoting the formation of oxygen radicals and bacterial inactivation. In addition, this factor that might happen during photocatalysis resulted to block the active sites of catalyst by adsorption of ions on the surface of TiO₂ [1].

The value of DO increased in the seawater after the testing as shown in Table 2. The higher oxygen present in water might be because of the water splitting reaction during photocatalysis process. Fresh water holds more oxygen compared to saltwater. As the amount of salt in water decreases, the amount of dissolved oxygen increases. The DO value of seawater treated by Ti:Ash catalyst that calcined at 800 °C is lower than seawater treated by Ti:Ash that calcined at 500 °C. The DO value in the seawater that treated with the Ti:Ash catalyst that calcined at 500 °C increased to 5.59 mg/L. Meanwhile, the DO value of seawater treated by Ti:Ash catalyst that calcined at 800 °C increased to 5.50 mg/L. Higher calcination temperature might result to decomposition of

anatase phase in TiO₂ [6, 26]. It is also a prove that anatase phase of TiO₂ (Figure 2) significantly influence the efficiency of the catalyst.

The effect of Ti:Ash catalyst on seawater by utilizing the photocatalytic reactor system seems satisfied because it could reduce the pH value in the seawater after treatment. The result in Table 2 signifies that the Ti:Ash that calcined at 500 °C and 800 °C reduced the pH of seawater to 7.57 and 7.65 respectively. Ti:Ash that calcined at 500 °C managed to reduce the pH value up to 7 % reduction, otherwise Ti:Ash that calcined at 800 °C managed to reduce the pH value up to 6 % reduction. pH plays an important role in photocatalytic degradation of organics content in the seawater. The slight reduction in pH value might be because due to the multitude intermediate formation by products that might cause different chemical functional groups and will affect the value of pH indifferently [30]. Surface charge property of TiO₂, organic molecule charge, the adsorption of organic molecule on to TiO₂ surface a hydroxyl radical concentration are the effects of pH solution [31].

The gas collected was analyzed via gas chromatography with thermal conductivity detector (GC-TCD) tested on both Ti:Ash catalyst that calcined at temperature 500 °C and 800 °C. This investigation is to verify that the reaction also occur in the photocatalyst system by using Ti:Ash catalyst. The presence of small amount of CO₂ for both catalyst conditions was detected by GC-TCD. Seawater alone was tested in the photocatalytic system to investigate the gas contained in the seawater itself. The molar percentage of CO₂ contain in the seawater was 0.0168 %. The reaction occur by using the Ti:Ash catalyst that calcined at 500 °C produced 0.022 % amount of CO₂, while Ti:Ash catalyst that calcined at 800 °C generated 0.028 % amount of CO₂. However, the CO₂ gas produced from the reaction is very minute. The CO₂ was produced due to the reaction of organic matter with OH⁻. Hydroxyl production generated from water splitting during photocatalytic reaction. Yoneyama and Torimoto [32] also claimed that adsorbed substances in the adsorbent support are oxidized that produced CO₂, It is a proof that the catalyst has dual function which is as an adsorbent and catalyst in light wavelength of 365 nm exposure.

4. Conclusions

In this paper, the investigation on the effect of catalyst calcination temperature towards TiO₂ supported by oil palm fiber ash was stud-

ied. The catalyst was prepared via wet impregnation technique and tested in the photocatalyst reactor scheme. The phase of anatase in TiO₂ appeared at both Ti:Ash catalyst at 500 °C and 800 °C and does not strongly influenced by calcination temperature. The XRD shows that the both catalyst are able to adsorb salt (NaCl). It is found that the adsorption and photocatalyst ability of hybrid catalyst Ti:Ash significantly depended on the calcination temperature. The result indicates that the Ti:Ash calcined at temperature 500 °C performed better adsorption capacity and reactivity compared to 800 °C catalyst calcination. It is shown that the oil palm fiber ash could be the supporter in purifying the seawater. Better pH, conductivity, COD, DO and turbidity values and amount of gas generated specially CO₂ were achieved and indicated that the catalyst has dual functions; adsorbent and catalyst.

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