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Chelating Zinc(II) in Crude Pheophorbide Gives It CO_2-Reducing Capability

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Abstract

A pheophorbide-based photocatalyst prepared from water hyacinth (Pontederia crassipes) was developed for photocatalytic CO_2 reduction. Crude pheophorbide was prepared from the chlorophyll extract of fresh water hyacinth leaves by a sequential acid–base treatment. The successful conversion of chlorophyll to pheophorbide was indicated by the decreased absorbance intensity and slight red shift of the Qy band from around 662 nm to approximately 667 nm, indicating demetallation at the chlorophyll core as well as dephytylation. Preliminary photocatalytic investigation proved that the presence of a Zn center in pheophorbide resulted in its ability to convert CO_2 into CO and other organic compounds in water as the solvent under light irradiation. CO and organic molecules within a closed reaction system were detected and confirmed photocatalytic reduction of CO_2. However, bubbles formed during the process, probably owing to residual impurities carried in the crude chlorophyll precursor, degraded reactor stability. Additional purification and optimization therefore are required for further stability and effectiveness of the catalyst. Although this research is at the initial stage, this study demonstrates the feasibility of utilizing water hyacinth–derived pheophorbide–Zn complexes as potential organometallic photocatalysts for CO_2 reduction in support of green chemistry principles.

Keywords: CO_2; Chlorophyll; Pheophorbide; Photocatalysis; Zinc

Cite This Article

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INTRODUCTION

Carbon dioxide (CO_2) is the main greenhouse gas causing global warming. Since the industrial revolution, humans are pumping out CO_2 into the atmosphere, raising its level at a speeding rate. The impact of the rising level of CO_2 in the atmosphere is mostly related to its aftermath of rising the average concentration of earth's surface [1].

Efforts to reduce CO_2 concentration in the atmosphere are a direct solution to the cause of the current global warming phenomenon [2]. Not only reducing it, but also converting it to useful chemicals will endorse the capture of the CO_2 from the atmosphere [3]. This capture and converting workflow is not only useful to mitigate global warming, but also potentially opens a new economic source for the community as well as offering a long term solution for CO_2 atmospheric level control, which is better compared to some short term solution suggested such as injecting heat absorbent gases e.g. sulfuric acid gas.

However, the main challenge of CO_2 chemical reduction is that it is an unfavorable reaction thermodynamically. This challenge including the need of better solutions for the energy source to run the reaction and better catalyst to make the reaction achievable [4].

One of the strategies to tackle these problems is by using photocatalysts. Photocatalysts offer the use of energy from sunlight, a free source of energy. Besides that, a natural reaction that already evolved for billions of years for CO_2 reduction is indeed based on photocatalysis known as photosynthesis. The success of photosynthesis lies on the special structure of chlorophyll molecule which can harvest the photon energy from the sun and use it to reduce CO_2 [5].

Many catalysts are already designed by incorporating chlorophyll-like moieties, especially containing the core strucutre of porphyrin [6]. However, there are still limited literature investigating about the ability of chlorphyll derived molecules, such as pheophorbide to reduce CO_2 outside a biological system.

This fact triggers us to investigate the ability of pheophorbide to reduce CO_2 in vitro. We also complexed the pheophorbide with Zn since Zn is repeatedly reported to have better CO_2-reducing catalytic ability [7] compared to Mg, the natural metal core in chlorophyll.

METHODS

Materials

Materials we used in the study were ethanol, Pontederia crassipes leaves, aquadest, ethyl acetate, HCl, NaOH, and Zinc acetate.

Equipments

Equipment used in the experiment includes lab glassware, a pestle and mortar, a UV-Vis spectrophotometer, a transparent reactor box, an 18 W LED lamp, and an air quality sensor.

Crude Chlorphyll Extraction

Pontederia crassipes leaves were harvested from wild-grown plants in Lake Sipin, Jambi City, Indonesia. The leaves were ground with sand until forming a paste texture. The paste obtained was then macerated with ethanol for 30 minutes and was extracted to obtain a green crude extract.

Synthesis of Crude Pheophorbide

As much as 150 ml of crude chlorophyll extract was acidified so the pH was 2. The acidified mixture was stirred for 2 hours. The mixture was then extracted with ethyl acetate. NaCl was added to break the ethyl acetate layer from the colloidal mixture. The ethyl acetate layer was collected and basified with 1 M NaOH solution until the pH was 14. The basified mixture was stirred for 2 hours. After that, aquadest was added multiple times to extract the pheophorbide sodium salt formed. The water layer was then acidified until the pH was 4 and was followed with extraction with ethyl acetate. The ethyl acetate layers were collected, dried over $MgSO_4$, and evaporated to yield crude pheophorbide.

Complexization of Crude Pheophorbide with Zn

Crude pheophorbide was reacted with Zn acetate with the equivalent molar ratio of 1:1. The solvent used was aquadest and the reaction mixture was stirred for 1 hour. No further purification was performed.

UV-Vis Characterization

Crude chlorophyl, pheophytin, and pheophorbide were characterized by UV-Vis spectrophotometer.

CO2 Reduction Catalytic Performance Test

The complexed Zn with crude pheophorbide obtained from previous treatment was placed into a 250 ml Erlenmeyer glass. The glass was then placed in an air-tight transparent box that was equipped with an air quality sensor and an aeration input which still allowed for limited air exchange. The air was pumped into the Erlenmeyer and the readings from the air quality sensor, namely CO, HCOC, and TVOC levels, were recorded. The observation of changes in CO, HCOC, and TVOC levels while the air was being pumped was conducted for 90 minutes under LED lamp light irradiation. For the negative control, a sample of aquadest was treated the same as the Zn–crude pheophorbide sample.

RESULTS AND DISCUSSION

Crude chlorphyll was succesfully extracted from Pontederia crassipes, showing that P. crasspies is a robust and easy-to-obtain source of chlorophyll in tropical countries where it grows. This result also support the finding from Kumar et al. [8] that P. Crasspies has higher chlrophyll content compared to more common chlorophyll souce for research such as spinach.

The success of the detachment of the Mg^{2+} core from extracted chlorophyll was observed from the change of color of the extract from green to olive green when it was reacted with 1 M HCl. When we tried to extract the pheophytin with ethyl acetate, the ethyl acetate would not separate from the mixture. This observation may be due to the content of ethanol in the mixture as well as other organic molecules in the mixture, which consequently made the mixture look homogeneous. After the addition of NaCl and more aquadest, the ethyl acetate layer started to separate.

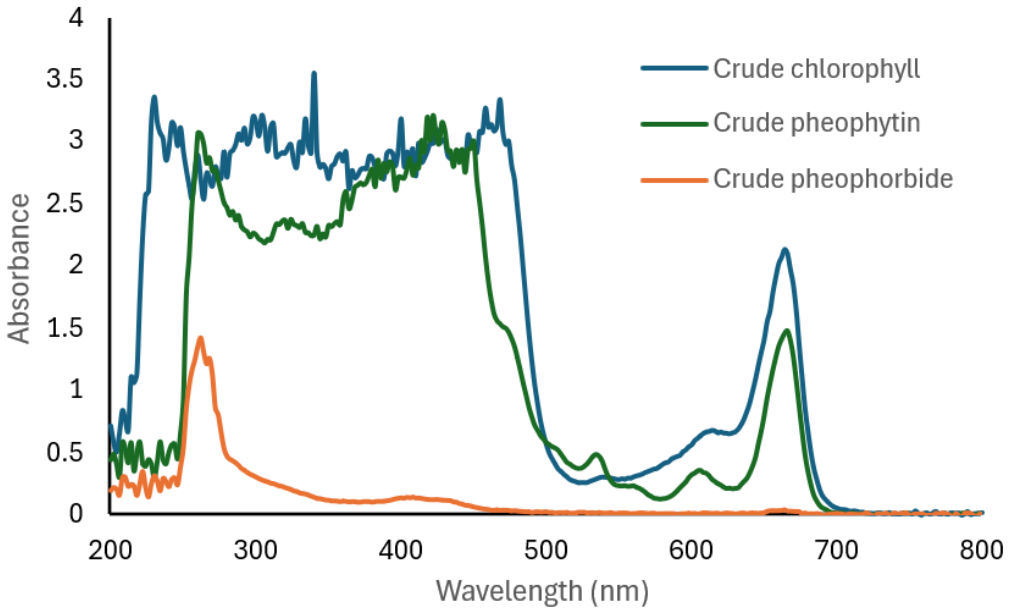
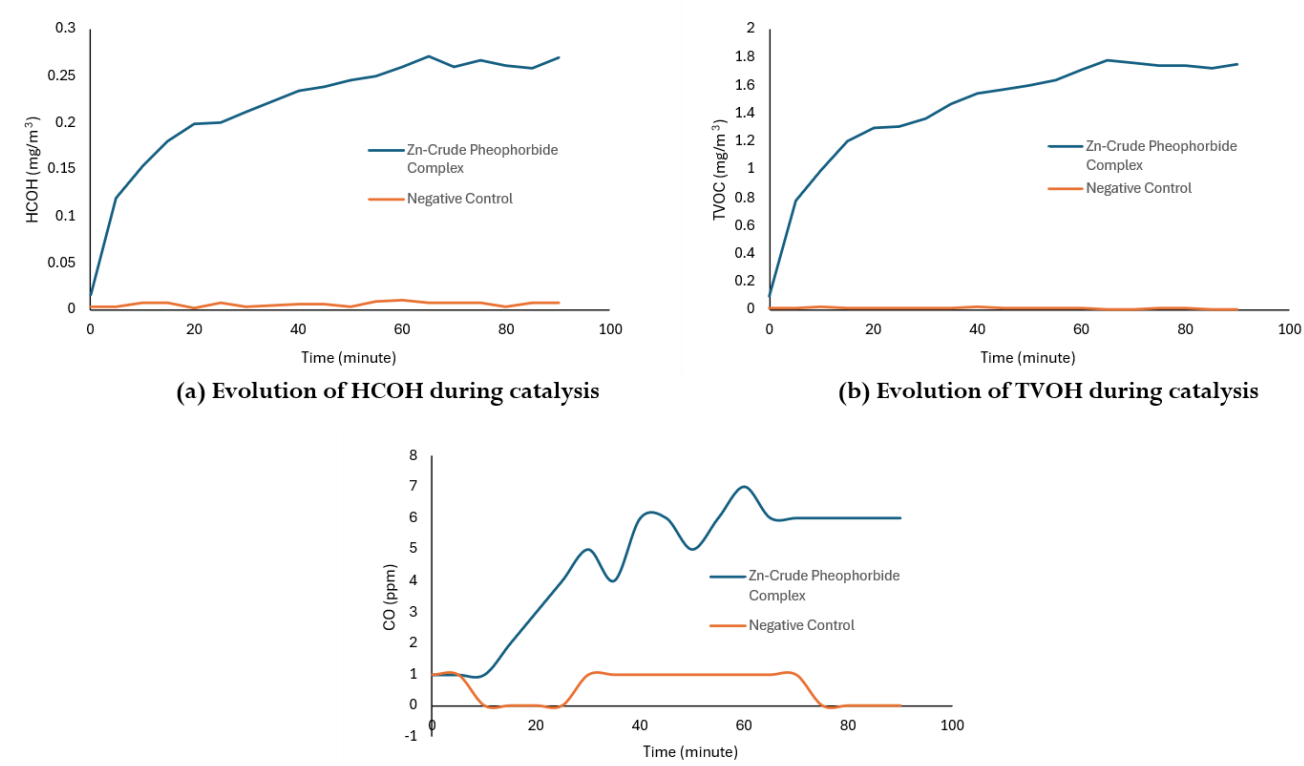


Figure 1. UV-Vis spectra of crude chlorophyll and its derivatives

Basification using 1 M NaOH was inteded to hydrolyze the phytol tail from pheophytine to form pheophorbide. The success of the reaction was observed from the change of the color from olive oil to dark brown. Besides of the change of color, the formation of pheophorbide was also confirmed using UV-Vis characterization. The UV-Vis spectra for crude chlorophyl, pheophytine, pheophorbide, are presented in Figure 1. The peak value of the UV-Vis spectrum for the chlorophyll extract was observed at 664 nm, while the peak value for the pheophytin extract was at 666 nm. The peak shift shows the detachement of Mg2+ from chlorophyll to form pheophytine. On the other hand, another peak spectrum for the pheophorbide extract was recorded at about 410 nm. This comparison is important to evaluate the success of modificaiton conducted Based on previous reports, chlorophyll extracts usually show a UV-Vis absorption peak at a wavelength of around 665 nm, which is comparable to the results obtained in this study, which is at 664 nm. This peak is characteristic of the dominant chlorophyll compound in the photosynthesis process. For the pheophytin extract, the literature indicates an absorption peak at around 665-667 nm, which is also in accordance with the results of this study which shows a peak at 666 nm. Meanwhile, for pheophorbide extract, the literature generally records an absorption peak at 390-412 nm, and the results of this study show a peak at about 410 nm.



(a) Evolution of HCOH during catalysis

(b) Evolution of TVOH during catalysis

(c) Evolution of CO during catalysis

Figure 2. Evolution of reduced-products formation

The formation and evolution of CO2 reduction species from the catalysis using Zn–crude pheophorbide was observed using an air quality sensor. The detected species were CO, HCOH, and TVOC. It is observed that the presence of Zn–crude pheophorbide influenced the formation of CO, HCOH, and TVOC, while none of those products were present in the absence of the catalyst (Figure 2). This preliminary test suggests that the complex of Zn2+ with pheophorbide can activate CO2 to form various reduced products. However, during the catalysis reaction, foaming formed, interrupting the catalysis process because the foam was flooding the transparent chamber. This problem might have occurred due to the use of crude pheophorbide. Other organic molecules from P. crassipes might be carried out along the process, making a stable foaming during the aeration. Although the problem occurred, the catalysis process could still be run to obtain the data.

CONCLUSIONS

Our brief experiment showed that Zn–crude pheophorbide has the ability to activate CO2 to make it able to undergo a reduction reaction. In this experiment, we also preliminarily proved the concept that a semi-synthetic metal complex molecule based on a natural product, chlorophyll, can still have the ability to harvest photon energy to reduce CO2. For future experiments, it is important to do further purification of the synthesized pheophorbide to eliminate potential problems such as foaming, interference of impurities in the reaction, as well as interference in the products' evolution readings.

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