

## Utilization Waste Materials to Synthesize Nano Al<sub>2</sub>O<sub>3</sub>-CaO Photocatalyst using Infused Red Guava Leaves (*Psidium guajava* L.)

Siti Rodiah<sup>1\*</sup>, Riana Aprilia<sup>1</sup>, Nina Ariesta<sup>2</sup>

<sup>1</sup>Chemistry, Faculty of Science and Technology, Universitas Islam Negeri Raden Fatah Palembang, Palembang 30267, Indonesia

<sup>2</sup>Chemistry, Faculty of Mathematics and Sciences, Nusa Bangsa University, Bogor 16166, Indonesia

\*Corresponding Author: [siti.rodiah\\_uin@radenfatah.ac.id](mailto:siti.rodiah_uin@radenfatah.ac.id)

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**Abstract:** Diazinon, a very toxic pesticide for animals and humans, is widely used in the agricultural sector, so this pollutant needs to be reduced or eliminated in the environment through photodegradation using photocatalysts derived from metal oxides. The catalytic activity of metal oxides is enhanced by adding a support material and increasing the surface area by reducing the particle size to nano size. In this study, kaolin as a source of Al<sub>2</sub>O<sub>3</sub> and golden snail shells as a source of CaO which used to synthesize Al<sub>2</sub>O<sub>3</sub>-CaO nanoparticles (NPs) with a simple and environmentally friendly method using plant infusions. Synthesis of Al<sub>2</sub>O<sub>3</sub>-CaO NPs using infused red guava leaves (*Psidium guajava* L.) had been done by utilized the secondary metabolites as reducing agents and stabilizers in the nanoparticle synthesis. FTIR analysis confirmed the presence of alumina (Al-O) groups at wave numbers 850 – 650 cm<sup>-1</sup> and CaO at 3642 cm<sup>-1</sup>. XRD analysis showed NPs Al<sub>2</sub>O<sub>3</sub>-CaO had cubic with the crystal size of 14.59 nm. The morphology of Al<sub>2</sub>O<sub>3</sub>-CaO NPs showed in the SEM pictures were agglomerated. Al<sub>2</sub>O<sub>3</sub>-CaO NPs degraded diazinon by 75% within 180 minutes under UV radiation. Thus, this research not only reduce golden snail shells as waste material, but also reduces water pollutant.

**Key word:** Al<sub>2</sub>O<sub>3</sub>-CaO nanoparticles; diazinon; infused red guava leaves; photocatalyst; waste.

### INTRODUCTION

Nanoparticles with a size of 1 – 100 nm have physical properties that are more characteristic than large particles. The main products made with these components have more surface area, smaller, lighter, stronger, more durable, and have unique physical and chemical qualities (Ramola, Joshi, Ramola, Chhabra, & Singh, 2019). The commonly used nanoparticles are derived from metal oxides which have high stability and good catalytic properties (Imtiaz, Farrukh, Khaleeq-Ur-Rahman, & Adnan, 2013). Nowadays, metal oxide nanoparticles were found to have many uses in industry, chemical, biological and catalytic fields because of their high catalytic properties, and relatively high oxide stability (Ghotekar, 2019). The metal oxide used in this study was Al<sub>2</sub>O<sub>3</sub> which had the advantage of being resistant to high temperatures, large surface area, relatively stable, and had a high melting point (Mohamad et al., 2019). Kaolin is one of the abundant sources of Al<sub>2</sub>O<sub>3</sub>, contains 30.51% of Al<sub>2</sub>O<sub>3</sub> after being calcined at 700 °C (Sri, Walmiki, G, & Kunci, 2015).

Kaolin is a mineral clay with the chemical formula Al<sub>2</sub>O<sub>3</sub>.2SiO<sub>2</sub>.2H<sub>2</sub>O which can be applied as a material because it has relatively large pores, non-toxic, stable at high temperatures and abundant in nature (Pasi, Bratadireja, & Chaerunnisa, 2020). Based on the Central Bureau of Statistics (2007), the potential for kaolin reserves in Indonesia is approximately 66.21 million tons spread across Indonesia. The management of kaolin

minerals in Indonesia, especially in Bangka Belitung Islands, had not carried out further development. Even today, kaolin mining in Bangka Belitung has long been abandoned. Kaolin samples used in this study were taken from abandoned mining ponds.

Based on government regulation number 25 of 2018 concerning increasing the added value of minerals, the management and development of kaolin minerals must be carried out so that they become one of the industrial raw materials and increase the added value of kaolin. The high Al<sub>2</sub>O<sub>3</sub> content allows kaolin to be used as a source of Al<sub>2</sub>O<sub>3</sub>. Previous studies reported that Al<sub>2</sub>O<sub>3</sub> NPs have been used in various fields such as an addition to ceramic structures, in the textile industry, in protein separation/purification, as biosensors, drug delivery, wastewater treatment, bio-filtration, and as catalysts (Ghotekar, 2019). Al<sub>2</sub>O<sub>3</sub> was used as a catalyst or solid catalyst support because its resistance to high temperatures, large surface area, high melting point and large pore structure (Riyadi & Mustikasari, 2017).

Catalytic activity of Al<sub>2</sub>O<sub>3</sub> was increased through the addition of CaO as supporting material. CaO has been used as Al<sub>2</sub>O<sub>3</sub> support for photodegradation 2,4,6-trinitrophenol, because CaO was semiconductor material that safe for humans and animals, had high catalytic activity and high adsorption (Imtiaz et al., 2013)(Anantharaman, Ramalakshmi, & George, 2016), so CaO was quite potential as a catalyst support material (Etuk, Etuk, & Asuquo, 2012). Golden snail shells are natural material that contains 61.95% of CaO after being calcined at 800 °C. In addition, the catalytic activity also can be increased by expanding the surface area through reducing the particle size to nano-sized.

There are several methods of synthesizing nanoparticles. The different method used to yield nanoparticles include microwave techniques, mechanical milling, homogeneous precipitation, spray pyrolysis, thermal evaporation, organometallic synthesis, and homogeneous precipitation. These techniques, however, are frequently pricy, labor-intensive, and hazardous to the environment. The presence of some toxic chemicals involved in the chemical methods, may have hazardous effects (Vijayakumar, Mahadevan, Arulmozhi, Subramanian, & P.K, 2018). Chemical reduction methods are widely chosen in synthesizing nanoparticles because they are easy, environmentally friendly, relatively inexpensive, and have the possibility to produce nanoparticles on a large scale. Most of nanoparticles were synthesized using toxic and hazardous solvents or reagents such as hydrogen, alcohol, sodium boron hydride, sodium citrate, etc., that requiring high energy and high pressure, so that not environmentally friendly (Abisharani, Kumar, Arthanareeswari, & Kamaraj, 2019; Rahmadani, Side, & Putri, 2020). Therefore, an environmentally friendly synthesis was developed by utilizing secondary metabolites of plants such as flavonoids, tannins, alkaloids, and terpenoids (El-Gendy, Nassar, & Speight, 2021) as reducing and stabilizing agents. The synthesis of nanoparticles with bio-reductants is called biosynthesis. Furthermore, the majority of bioprocesses take place at standard air pressure and temperature, which results in significant energy savings, high yield, and low cost (Abisharani et al., 2019).

Previous researches had utilized extracts Casuarinaceae (Ramola et al., 2019), *Cymbopogon citratus* (Ansari et al., 2015), *Pedalium murek* (Sivaprakash et al., 2019), *Psidium guajava* L. (Bose & Chatterjee, 2016)(Somchaidee & Tedsree, 2018) leaves to synthesize of CaO NPs, Al<sub>2</sub>O<sub>3</sub> NPs, Al<sub>2</sub>O<sub>3</sub>-CaO NPs, and Ag and Fe NPs (Bose & Chatterjee, 2016)(Somchaidee & Tedsree, 2018) respectively. Other studies had also reported that red guava leaves contain steroids, flavonoids, alkaloids, saponins, and polyphenols in the form of 7.82% tannins which have the potential as stabilizing and reducing agents (Sudira, Merdana, & Qurani, 2019)(Zheng, Zhao, Wu, Wang, & Zou, 2021)(Yana, 2018). Secondary metabolites used to synthesis NPs of previous research were

extracted using the maceration method with organic solvents, while in this study, the researchers offered a simple and environmentally friendly preparation method using an abundant solvent, namely water distilled and treated through the infusion method. The water-soluble heterocyclic constituents of the infusion samples were responsible for the formation and stabilization of NPs. The extraction of the constituents was carried out through heating so that more compounds were attracted and the process went faster.

The synthesized nanoparticles were used as a photocatalyst in the degradation of diazinon. Diazinon is widely used in the agricultural sector to remove pests on corn, sugarcane, coconut, rice and fruit crops. This pesticide is very toxic to animals, humans and the environment and can cause death, so the use of diazinon in the environment must be reduced (Khoiriah, Wellia, Gunlazuardi, & Safni, 2020). The synthesized nanoparticles were used to degrade pesticide pollutants of diazinon with mass variations of 5 mg, 10 mg, 15 mg, 20 mg, 25 mg. The degradation results were analyzed using UV-Vis spectrophotometry and GC-MS. Fourier Transform InfraRed (FTIR) analysis was used to ensure that the support material (CaO) has combined with Al<sub>2</sub>O<sub>3</sub>. Crystal structure, phase identification, and crystalline particles size was determined using X-ray Diffraction (XRD) analysis, while morphology of Al<sub>2</sub>O<sub>3</sub>-CaO NPs were examined by using scanning electron microscope (SEM) analysis.

## RESEARCH METHODS

### Materials and Tools

Samples used in this study such as red guava leaves, golden snail shells and kaolin. Red guava leaves and golden snail shells were collected from South Sumatera and kaolin was taken from an abandoned kaolin mine pond in Bangka Belitung, Indonesia. The chemicals HCl, NaOH were purchased from Merck, while aquadest and diazinon reagents were from Puduk Scientific.

### Methods

#### *Sample Preparation*

NPs Al<sub>2</sub>O<sub>3</sub>-CaO was synthesized using Al<sub>2</sub>O<sub>3</sub> from kaolin and CaO from golden snail shells by adding infused red guava leaves as reducing and stabilizing agents. Kaolin was used as Al<sub>2</sub>O<sub>3</sub> source. Al<sub>2</sub>O<sub>3</sub> was prepared by dissolving 1 gram of kaolin in 10 ml of 6 M HCl at 90 °C for 2 hours. Then added 6 M NaOH solution until alkaline to separate the impurities. After that, it was calcined at 750 °C for 4 hours to obtain alumina then characterized by FTIR (Ramdhani, Yanuar, Zulkifli, & Sarwana, 2018). CaO had been prepared from golden snail shells and used by previous research (Rodiah, Erviana, Rahman, & Budaya, 2020). Infused red guava leaves were prepared by washing the leaves in running water, then boiled 5 grams of clean red guava leaves in 100 ml of distilled water at 60 °C for 15 minutes, then cooled and filtered to obtain an infusion (Taba, Parmitha, & Kasim, 2019).

#### *Synthesis Al<sub>2</sub>O<sub>3</sub>-CaO NPs*

Al<sub>2</sub>O<sub>3</sub>-CaO nanoparticles were synthesized by precipitation method from Al<sub>2</sub>O<sub>3</sub> and CaO. Al<sub>2</sub>O<sub>3</sub> was prepared from kaolin, that activated by acid to remove the impurities and to produce a higher surface area and better adsorption properties (Irawati et al., 2013). Preparation of catalyst was form 2 g of Al<sub>2</sub>O<sub>3</sub> and 1 g of CaO were dissolved in 10 ml of infused red guava leaves for 1 hour. After that, it was calcined at 400 °C for 3 hours to obtained Al<sub>2</sub>O<sub>3</sub>-CaO NPs, then the nanoparticles were characterized using XRD, SEM, and FTIR.

### ***Photocatalytic Degradation of Diazinon***

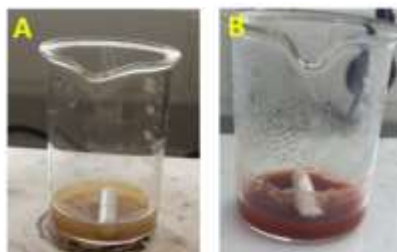
Al<sub>2</sub>O<sub>3</sub>-CaO NPs were added to 20 ml of 11 ppm diazinon solution. Al<sub>2</sub>O<sub>3</sub>-CaO NPs mass was varied of 5 mg, 10 mg, 15 mg, 20 mg, and 25 mg then irradiated under UV radiation for 180 minutes for each. The centrifuged mixture was analyzed by UV-Vis spectrophotometry at a wavelength of 247.5 nm (Irawati, Sunardi, & Suraida, 2013). Samples were analyzed by GC-MS to determine the fragmentation of diazinon.

## **RESULTS AND DISCUSSION**

### **Characteristics of Al<sub>2</sub>O<sub>3</sub>-CaO NPs**

The remaining impurities present in the sample such as water molecules, CO<sub>2</sub> and other volatile molecules were also removed by calcination at 750 °C and to activate the surface site of the catalyst. The water-soluble heterocyclic constituent in infused red guava leaves played an important role in the formation and stabilization of nanoparticles (Ghotekar, 2019). Red guava leaves contain polyphenolic metabolite compounds in the form of tannins which have the ability as reducing and stabilizing agents, where in the tannin structure there were adjacent hydroxyl groups so that they were able to donate electrons to metals by releasing hydrogen atoms from the hydroxyl group (Amin, Mahardika, & Fatimah, 2020; Widia, Zulhadjri, & Arief, 2018). These metabolite components converted metal compounds (metal ions) into metals (oxidation number of 0) with specific nanoparticle sizes (Vijayakumar et al., 2018).

The initial color of the solution of Al<sub>2</sub>O<sub>3</sub>-CaO nanoparticles using infused red guava leaves is yellow pale (Fig. 1A), after heating the solution becomes brownish red (Fig. 1B) as shown in Figure 1. The color change was due to the reduction process of Al and Ca metal ions by tannin compounds where the hydroxyl groups in tannins reduced metal ions to form nanoparticles (Rahmadani et al., 2020). The synthesized Al<sub>2</sub>O<sub>3</sub>-CaO NPs showed white crystals as shown in Figure 2.

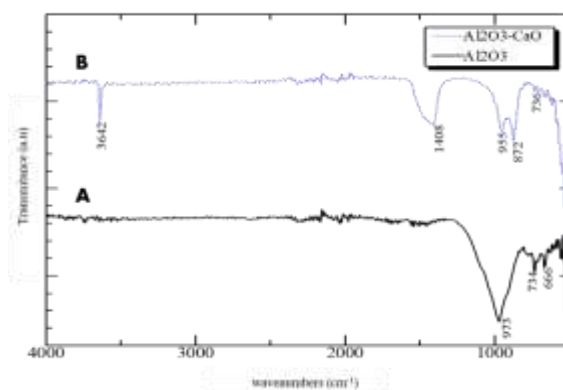


**Figure 1.** Color change in solution of Al<sub>2</sub>O<sub>3</sub>-CaO NPs



**Figure 2.** Synthesized Al<sub>2</sub>O<sub>3</sub>-CaO NPs

FTIR analysis of Al<sub>2</sub>O<sub>3</sub> of this study is shown in **Figure 3**. The absorption area of 1000 – 435 cm<sup>-1</sup> showed the presence of Al<sub>2</sub>O<sub>3</sub> peaks (spectra A), which the absorption of Al-O bond vibration appeared at 850 – 650 cm<sup>-1</sup> particularly at wavenumbers of 734 cm<sup>-1</sup> (Irawati et al., 2013; Widia et al., 2018). In the B spectra, the Al-O bond vibration appeared at 736 cm<sup>-1</sup>. That also reported in previous research that Al-O vibrations peaks area was 850-650 cm<sup>-1</sup> (Irawati et al., 2013). The presence of O-Ca-O, O-C, and OH bond only appeared in spectra B in area of 1410 cm<sup>-1</sup> and 872 cm<sup>-1</sup>, and 3642 cm<sup>-1</sup> respectively. This indicates that CaO has been dispersed in Al<sub>2</sub>O<sub>3</sub>. These peaks appeared in NPs on this study was also reported in previous research that characteristics of the O-Ca-O, O-C and -OH groups were located in the 1450 cm<sup>-1</sup>, 858 cm<sup>-1</sup>, and 3640 cm<sup>-1</sup> sequently. The O-H groups with sharp peaks were the characteristic of CaO standard (Ghazali, Kem, Jusoh, Abdullah, & Shariffuddin, 2019).



**Figure 3.** FTIR Spectra of Al<sub>2</sub>O<sub>3</sub>-CaO Nanoparticles

**Figure 4** shows the diffractogram of Al<sub>2</sub>O<sub>3</sub>-CaO. The presence of Al<sub>2</sub>O<sub>3</sub> at 2θ peaks at 5°, 35°, 37°, 46°, dan 57° with miller index (102), (104), (110), (202), and (116) respectively. The presence of CaO was shown with 2θ peaks at 32°, 64°, 67°, and 79° with miller index (111), (311), (222), and (400) sequently. The 2θ peak formed on the diffractogram corresponds to the standard database AMCSD No. 0009325 (Al<sub>2</sub>O<sub>3</sub>) and AMCSD No. 0008276 (CaO). The crystal size of NPs Al<sub>2</sub>O<sub>3</sub>-CaO was calculated by the formula Debye-Scherrer (Eq.1).

$$D = \frac{k\lambda}{\beta \cos \theta} \quad (1)$$

where D is the crystal size, β is the maximum half width (FWHM), λ is wavelength and θ is the Bragg angle at the diffraction peak. From these data, Al<sub>2</sub>O<sub>3</sub>-CaO nanoparticles had a cubic structure with a crystal size of 14.59 nm.

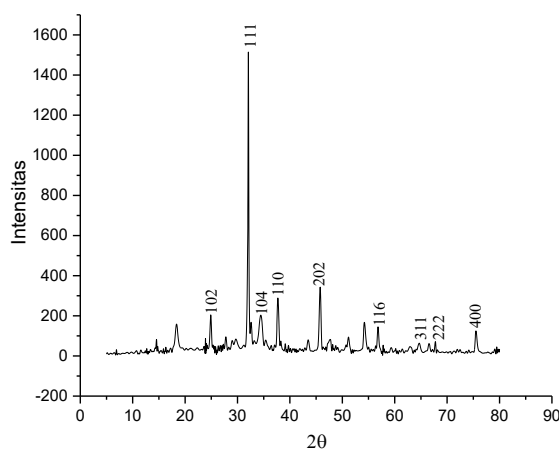


Figure 4. Diffractogram of Al<sub>2</sub>O<sub>3</sub>-CaO NPs

Morphology of the Al<sub>2</sub>O<sub>3</sub>-CaO NPs was determined by SEM analysis under 20 kV at a magnification of 10,000 and 20,000 as shown in **Figure 5A** and **5B**, respectively. At a magnification of 10,000 (Fig. 5A) showed that the particles were agglomerated and had an irregular shape. Agglomerated particles were seen more clearly at a magnification of 20,000 (Fig. 5B). Previous studies reported that the synthesis of Al<sub>2</sub>O<sub>3</sub>-CaO NPs using precursors and synthetic reagents resulted in agglomerated nanoparticles as reported in this study. Agglomeration occurred because of many chemical compounds contained in the infusion that played an important role in the synthesis. On the other hand, the influence of Nps electrostatic forces and energy when the synthesis take place and the collisions between particles caused the particles to be agglomerated (Sari, Nurhasni, & Yaqin, 2017)(Masakke et al., 2015).

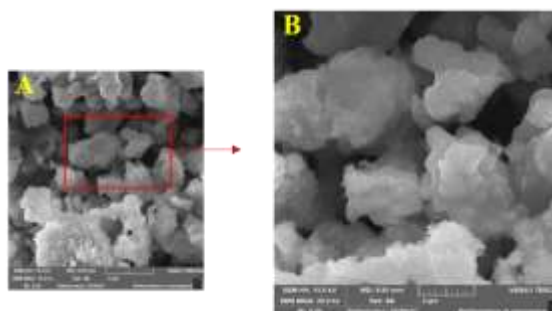


Figure 5. Morphology Al<sub>2</sub>O<sub>3</sub>-CaO NPs at (A) 10.000, (B) 20.000

#### Photocatalytic Degradation of Diazinon using Al<sub>2</sub>O<sub>3</sub>-CaO NPs

Activity of Al<sub>2</sub>O<sub>3</sub>-CaO NPs was observed through its performance to degrade diazinon. The maximum wavelength of diazinon analysis using UV-Vis spectrophotometry was 247,5 nm. The analysis data showed that the percent degradation increased with increasing catalyst mass from 0-0,21 %, then decreased at 0,28%-0,35% as shown in Figure 6. The highest amount of percent degradation was 75% of 15 mg catalyst. More increase the amount of catalyst, more increase the surface area of the catalyst so that increase the number of photon absorption on the surface that results in many •OH radicals that played an important role in the photocatalysis (Khoiriah et al., 2020). Decreasing of present

degradation due to the increase in turbidity of excess catalyst mass, thereby preventing light from reaching the active site of catalyst. The surface area of the catalyst is affected by the physical properties of the catalyst. Powdered solid catalysts have a larger surface area than agglomerated catalysts. Agglomerated Al<sub>2</sub>O<sub>3</sub>-CaO NPs had low surface area and may deactivate the active sites so that the photocatalytic performance of the NPs was decreased (Khoiriah et al., 2020)(Aprilianingrum & Fia, 2016)(Li et al., 2010).

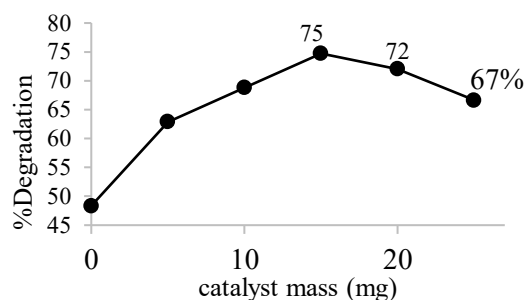


Figure 6. Line Graph of the Effect of Addition of Catalyst Mass

Diazinon decomposition was confirmed using Gas Chromatography and Mass Spectroscopy (GC-MS). Figure 7 peak intensity of diazinon before (RT of 7.27 minute) and after (RT of 7.25 minute) being degraded was decreased that proved diazinon had been successfully degraded.

Diazinon was degraded because it became fragments as shown in its mass spectra in Figure 8. Three main fragments of diazinon were diethyl fosfonat, 2-isopropil-6-metil-4-pyrimidinol (IMP), and diazoxon (Sakkas, Dimou, Pitarakis, Mantis, & Albanis, 2005) (Zhang et al., 2010). Diazoxone was derived from the rearrangement of the thiono-thiolo diazinon through the substitution of sulfur with oxygen in the formation of P = S (Muñoz et al., 2011). Then, IMP was generated from the hydrolysis of diazoxone which involved breaking the P – O bond in the pyrimidine group (Zhang et al., 2010). •OH attacked on the –O– functional group of diazinon produced two by-products, namely diethyl phosphonate and IMP (Wang, Wang, & Shih, 2016). Fragmentation of diazinon was shown in Figure 9.

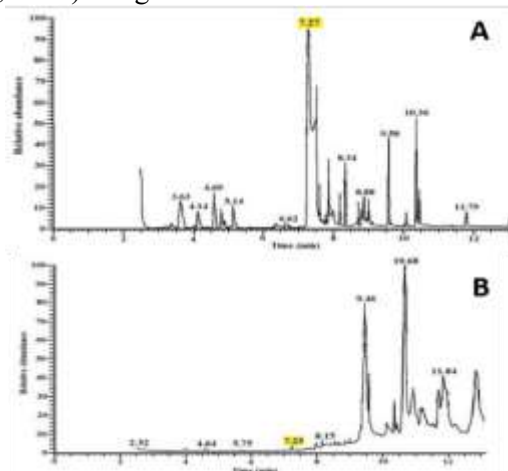


Figure 7. Spectrum GC Diazinon (A) Before (B) After Degradation

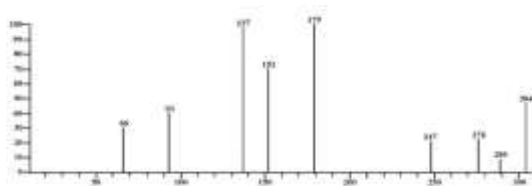


Figure 8. Spectrum Mass Diazinon

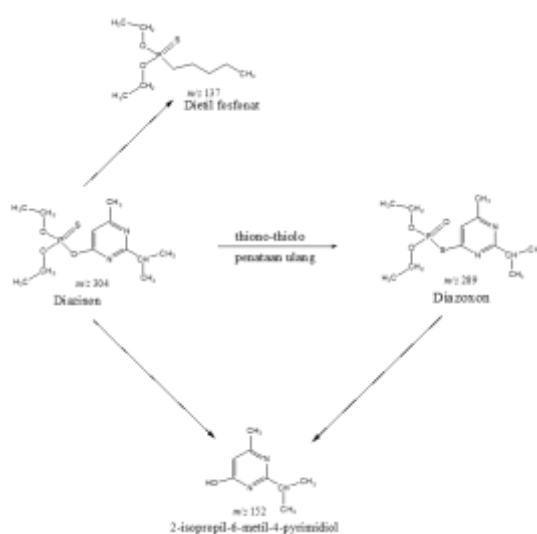


Figure 9. Fragmentation of Diazinon

## CONCLUSIONS

Infused red guava leaves played an active role as reducing and stabilizing agents for the synthesis of Al<sub>2</sub>O<sub>3</sub>-CaO nanoparticles. The resulting nanoparticles obtained was white with a crystal size of 14.59 nm and agglomerated. Therefore, infused red guava leaves has the potential to be used for large-scale nanoparticle biosynthesis applications, but appropriate techniques to increase crystallinity and reduce agglomeration are needed. Percent degradation of diazinon using Al<sub>2</sub>O<sub>3</sub>-CaO NPs was 75% at optimum catalyst mass of 0,21 % for 180 minutes. Thus, of Al<sub>2</sub>O<sub>3</sub>-CaO NPs derived from kaolin and golden snail shell waste can be an alternative solution for handling golden snail shell waste and handling diazinon pesticide in waters. In addition, the bio-synthetic method applied by using infused red guava leaves also reduces environmental pollution.

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