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Development of Magnesium Fertilizer Replacing Kieserite from Bio-Physico-Chemical Activated Dolomite

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Abstract

All plants, especially cocoa and oil palm, require magnesium (Mg) along with other macro- and micro-nutrients. Kieserite as the most common Mg-supplying fertilizer for crops is still imported for Indonesia due to lack of natural deposits in the country. On the other hand, dolomites ($CaMgCO_3$) are found abundantly and mostly used as liming material due to low MgO content and solubility as well. Many efforts have been carried out since a century ago to convert dolomite into Mg fertilizer substituting kieserite. However, there are no efficient processes available yet and therefore it is the objective of this study to develop a potentially efficient process by employing bio-physico-chemical activation approach in converting dolomite into kieserite-like product. A series of experiments were conducted by using a calcinated dolomite from Sidayu, Gresik, East Java, reacting with H_2SO_4, and inoculating with Mg-solubilizing microbes. An 80-mesh calcined dolomite powder was reacted with water at 1:3 ratio (% w/v) and after one hour stirring added with technical grade H_2SO_4 improving 27.7% MgO content from 18% to 23%. The formula was then tested its effectiveness for cocoa and oil palm seedlings in the greenhouse for three and six months, respectively. By combining NPK fertilizer at standard dosage with the formula of Mg-fertilizer at 75% of kieserite rate yielded the highest performances by six-month-old oil palm seedlings in a greenhouse experiment. To obtain conclusive results under field conditions, the formula was tested at field experiments using immature and mature oil palm trees. At 0.9 kg/tree/year dosage, the Mg-fertilizer formula was superior to conventional kieserite at 1.6 kg/tree/year rate in supporting vegetative growth of immature oil palm. From the mature oil palm trial, it can be concluded that the use of the new formulated Mg-fertilizer at 50% rate of conventional kieserite (2 kg/tree/year) resulted in an insignificant different yield of fresh fruit bunches (FFB) compared to standard kieserite dosage.

Keywords

Sidayu Dolomite, Calcination, Acidification, Mg-Solubilizing Microbe

1. Introduction

Kieserite ($MgSO_4$) is a well-known magnesium (Mg) fertilizer in the world that originated from natural deposits especially in sub-tropical regions containing > 26% MgO and 50% SO_3 [1]. In contrast, most of agricultural countries in tropical regions have no natural resources so that almost all Mg fertilizer supplied by Kieserite is highly dependent on imported products. As consequence, the price is considerably high since very limited, if any, domestic resources are readily available to substitute kieserite as Mg-fertilizer for agricultural crops. There are some others Mg-bearing mineral present in tropical region, such as dolomite ($CaMgCO_3$), serpentine ($MgSiO_4$), and feldspars ($KCaMgSiO_4$) [2]. However, these minerals neither have high content nor high solubility of Mg compared to Kieserite. Therefore, there were many interests by researchers to develop an efficient process in conversion such minerals to be compatible with Kieserite as Mg fertilizer. Many researchers have reported the application of this fertilizer [3] [4], especially for oil palm [5] [6] [7] in which some of them were interested in finding the most effective mineral to substitute kieserite.

Dolomite is a calcium-magnesium carbonate ($CaMgCO_3$) mineral that has been thoroughly reviewed by [8] and found abundantly in sub- and tropical regions including China [8] [9] [10] [11], Hungary [12], United States of America [13], and Indonesia [14]. The use of dolomite for crop has also been widely known [15] [16]. However, due to its low content and solubility of Mg many efforts were carried out to find most efficient process in improving these characteristics so that the processed dolomite could be used to substitute kieserite. Many processes have been proposed mainly based on calcination and/or acidification [17] [18] [19]. However, all of these results were still failed to achieve a high MgO content. Therefore, it is time to develop other alternative for processing dolomite to be a kieserite substitute.

Nutrient solubility in soils to some extend is almost always facilitated by microbial activities, especially when dealing with the solubilization of hardly soluble nutrients from natural rock materials. Recent studies indicated the incorporation of silica-solubilizing microbe improved significantly soluble Si in a Si fertilizer formulation [20] [21] [22]. Such phenomenon was also reported earlier by [23] [24] for potassium from feldspar and phosphate from selected natural phosphate rocks, respectively. The current study reports the development of bio-physico-chemical activation of a domestic dolomite to produce alternative Mg fertilizer for conventional kieserite. Considering the highly abundant dolomite deposits in Indonesia, this technology will enjoy profitable commercial application to diversify the more affordable Mg fertilizer, especially for oil palm smallholders.

2. Materials and Methods

2.1. Chemical Properties

A 100-mesh dolomite originating from Sidayu, Gresik, East Java, Indonesia was used in this experiment as raw material for formula development. Prior to and after physical-chemical treatment it was subjected to selected chemical analyses. Selected chemical analyses performed were including pH, calcium (Ca), total and water-soluble magnesium (Mg), aluminum (Al)-, iron (Fe)-, and silicon (Si)-oxides, neutralizing capacity, and water content determination by using standard laboratory analyses outlined by SNI 13-3494-1994 (ICS 73.080).

2.2. Physico-Chemical-Biological Treatments

The physical treatment was carried out by calcining a 100-mesh dolomite powder at 400˚C in a muffle furnish for one hour [25] [26]. The calcination was continued at 800˚C for another one hour. After cooling at room temperature, the calcinated dolomite was added with distilled water with 1:3 ratio (w/v) stirred for 30 minutes. Finally, a technical grade of sulfuric acid (H_2SO_4) solution (96% in purity, Shijiazhuang Xinlongwei Chemical Co., LTD) was added to dolomite milk suspension at 14% (v/v) and stirred further for another 30 minutes. The treated sample was then dried oven at 110˚C for overnight. Inoculation of the activated sample was performed by spraying a suspension containing 10^9 cfu of phosphate-magnesium-solubilizing *Pseudomonas sp* at 5% (v/w) rate. The isolate was obtained from the IRIBB Culture Collection and precultured on a Pikovskaya agar medium containing (per L medium): 5 g $Ca_3PO_4 \cdot 3OH$, 0.2 g NaCl, 0.2 g KCl, 0.1 g $MgSO_4 \cdot 7H_2O$, 2.5 mg $MnSO_4 \cdot H_2O$, 2.5 mg $FeSO_4 \cdot 7H_2O$, 0.5 g $(NH_4)_2SO_4$, 10 g glucose, 0.5 g yeast extract, and 15 g bacto agar. The formulated kieserite (FK) was in the granular form with 3 - 5 mm diameter average after mixing with non-activated dolomite [25%:75% (w/w)] and adding 1% (w/w) humic substance. The final formula of FK contained 28% MgO (minimum).

2.3. Mineralogical Characteristics

An X-ray diffraction (XRD) analysis was performed using random-oriented samples analyzed by XRD using Cu Ka radiation at 40 kV and 30.0 mA from XRD—6000 Shimadzu equipped with low divergence and receiving slits and a graphite monochromator on the 800˚C—one hour calcined and uncalcined dolomite samples to determine mineralogical composition by running at 0˚ - 30˚ 2-theta with Cu Ka at the Research Institute for Mineral and Coal (Tekmira) Bandung. Identification of the mineral present was carried out by using major intensive peak characterizing the mineral according to [27]. Differences in XRD patterns between the two sample will be used as supporting evidences for evaluating the effects of the physical treatment on the mineralogical characteristic.

2.4. Experimental Designs

A greenhouse experiment was conducted to evaluate the effectiveness of formulated dolomite as Mg fertilizer for oil palm seedlings in comparison with kieserite. Two-month old Dura x Psifera oil palm seedlings were grown for six months in a pot containing Ultisols soil media. The treatments applied were eight combinations of conventional NPK fertilizer and selected dosages of formulated kieserite (FK) in comparison to conventional kieserite and dolomite as follows: 1) NPK (A) + kieserite (B), 2) A + FK at 25% B, 3) A + FK at 50% B, 4) A + FK at 75% B, 5) A + FK at 100% B, 6) A + FK at 125% B, 7) A + FK at 150% B, and 8) A + dolomite. Arranged in complete random design the treatments were replicated three times. Parameters observed include leaf, roots, and stem dry weight and the data were subjected to analysis of variance and Duncan's Multiple Range Test ($p < 0.05$) to determine the different effect among treatments. A field experiment was conducted both with immature and mature crops in Tinjowan Estate, North Sumatera, Indonesia, on an Ultisols soil. Using three-year old immature and a five-year-old mature crops the experiments were set in randomized block design with three replicates and the plot size was two hectares for immature and five hectares for mature crops. The treatments for immature crops consisted of some combinations of NPK fertilizer with: 1) kieserite 1.6 kg/tree/year, 2) FK 0.6 kg/tree/year, 3) FK 0.9 kg/tree/year, 4) FK 1.2 kg/tree/year, 5) FK 1.5 kg/tree/year, and 6) FK 1.8 kg/tree/year, whereas those for mature crops were similar but different dosages, *i.e.*, 1) kieserite 2.0 kg/tree/year, 2) FK 1.0 kg/tree/year, 3) FK 1.5 kg/tree/year, 4) FK 2.0 kg/tree/year, 5) FK 2.5 kg/tree/year, and 6) FK 3.0 kg/tree/year. Vegetative and generative observations were made for one year after treatments and the data were subjected to statistical analyses. The vegetative parameters were number of fronds and number, length, and width of petioles, whereas the generative ones were number, weight, and average weight of fresh fruit bunches (FFB).

3. Results and Discussion

3.1. Properties of Sidayu Dolomite

Dolomite ores in Indonesia are found in several locations spreading from Aceh in the North up to East Java in the East. Among dolomite sites, the highest deposits and quality is the one in Sidayu, Gresik, East Java. Its quality for agricultural use has been set by the Industrial National Standard (SNI) # 02-2804-2005 especially for Mg fertilizer (**Table 1**). Compared with chemical characteristics of kieserite, this dolomite consists less MgO, both for total and water-soluble one, dominated by Ca, less soluble, and alkaline in reaction. According to the SNI, a kieserite as Mg fertilizer must contain 27% - 29% MgO (17.56% Mg), whereas in dolomite from Sidayu the MgO was only 18% - 22% (13.18% Mg). Therefore, the process intended to process dolomite to meet kieserite standard is to reduce CaO and/or CO_2 resulting ini the increase of relative composition of the MgO.

Due to its lower MgO contents compared to kieserite, therefore some additional process was performed. Several studies have indicated that calcination will improve the MgO content of dolomite while in the same time reduce the CaO content [Pustaka]. Our data in Table 2 show that after calcination at 600˚C for one hour, the characteristics of dolomite were improved approaching those of kieserite, especially in those of total and water-soluble MgO.

3.2. Mineralogical Characteristics

Data shown in Figure 1 indicate the present of several minerals based on XRD analysis both in non-calcined and calcined dolomites. The present of dolomite mineral ($CaMg(CO_3)_2$) in both samples are shown by the peak with 2 theta symbol of 2.86 Å. Original dolomite was dominated by dolomite (99.8%) and only 0.2% cristobalite (SiO_2) present (Table 3). The latter is shown by a 2.33 Å peak at 30˚ 2-theta. In calcined dolomite, a layer is formed that covers the surface pores of the original dolomite, resulting in the inhibition of CO_2 gas product from exiting the interface reaction. It is expected that there will be an increase in the levels of CaO and MgO when compared to the non-calcined dolomite (Figure 1). Calcined dolomite is dominated by calcite ($CaCO_3$) about 80.2% with 19.4% of an MgO mineral periclase and a few dolomite (0.4%) (Table 3). These data confirmed that thermal treatment promotes decomposition of $CaMg(CO_3)_2$ at certain temperature [28] [29]. It has been reported by others [30] that decomposition of this carbonate mineral will first release magnesium and later at a higher temperature followed by calcium. As reported by [31] the conversion reaction is most likely following this path:

$$CaMgCO_3 + O_2 \rightarrow CaCO_3 + MgO + CaMg(CO_3)_2$$

Table 1. Physico-chemical characteristics of standar Kieserite and the Sidayu dolomite.

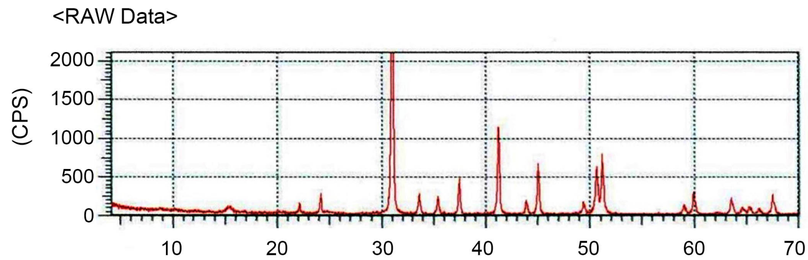
Characteristics	Kieserite	Dolomite
Total MgO (%)	27 - 29	18 - 22
MgO (%)	17.56	13.18
Other Nutrient (%)	S = 22	CaO = 30
Water solubility	Fairly	Hardly
Reaction	Slightly acid	Alkaline

Table 2. Effect of calcination on dolomite characteristics.

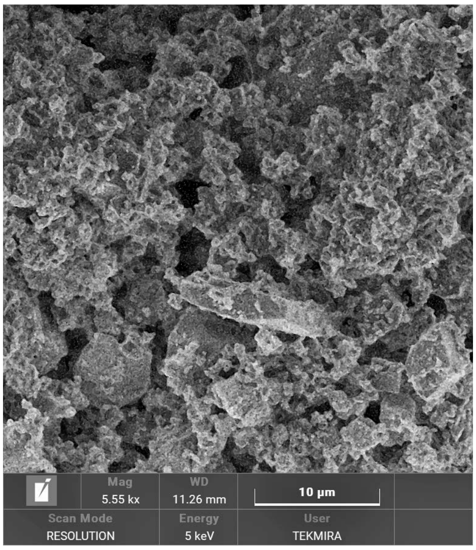
Characteristics	Untreated Dolomite	Calcined Dolomite
Total MgO (%)	16.10	26.70
Water soluble MgO (%)	4.50	9.20
CaO (%)	29.10	30.04
Al_2O_3 (%)	0.02	0.03
Fe_2O_3 (%)	0.27	0.25
SiO_2 (%)	0.03	0.02
pH	8.5	9.10
Neutralizing capacity (%)	106.2	100.10
Water Content (%)	1.15	1.02

Table 3. Quantitative analysis of minerals presence in non- and calcined dolomite by using XRD.

Sample	Mineral Species	Amounts (%)
Non-calcined	Dolomite	99.8
	Cristobalite	0.2
Calcined	Calcite	80.2
	Periclase	19.4
	Dolomite	0.4



(a)



(b)

Figure 1. X-Ray Diffractogram: non-calcined (upper) and calcined (bottom) (A) and Scanning Electron Microscopes: non calcined (left) and calcined (right) dolomites (B), indicating decomposition of the mineral after 800 ˚C heating for one hour.

The data in **Table 3** also show that cristobalite (SiO_2) was totally decomposed at 800˚C temperature as reported by [29]. The evidences of calcite ($CaCO_3$) and periclase (MgO) minerals in **Figure 1(a)** are shown by the peaks at 2.71 and 3.59 Å, respectively. These evidences were not conclusively supported by SEM images which indicate that calcination at 800˚C decomposes dolomite mineral into a smaller particle. However, the SEM images of dolomite obtained in this study are similar to those reported by [32].

3.3. Effect of Formulated Kieserite on Oil Palm

Results on greenhouse experiment indicated that the newly formulated kieserite fertilizer could replace conventional kieserite (Table 4). The replacement of Mg-fertilizer from kieserite to FK at reduced dosage (75%) produced seedlings with dry weights of leaves, stems, and roots similar to those of conventional kieserite. Vegetative growth improvement obtained was presumably due to the present of Ca in the FK compared to none in the conventional kieserite. Other implication of this result is that the cost of fertilization by using FK would be less than conventional kieserite assuming that the price of the former is less than the later.

Data shown in Figure 2 and Figure 3 indicate the effect of new formulated Mg fertilizer on the growth of a three-year-old immature oil palm represented by length and number of fronds, respectively, in comparison with the standard application of Mg fertilizer (*i.e.*, kieserite). It is indicative that the use of 0.9 kg FK was producing significantly longer fronds and similar number of fronds compared to that of control treatment (1.2 Kg Kieserite/tree). This means that the newly formulated Mg fertilizer can reduce the dosage up to 25% compared to kieserite dosage. Figure 4 and Figure 5 present the effect of FK dosage on the yield components of mature oil palm, *i.e.*, on the fresh fruit bunches (FFB) number and average weight of FFB, respectively. The application of newly formulated kieserite at 1.0 kg/tree rate provided similar to or higher than the conventional kieserite applied at 2.0 kg/tree. A fifty percent reduction on fertilizer rate without significant effect on the crop performances especially yield will in turn provide a better economic value to the farmers.

Table 4. Dry weight average of six-month old oil palm seedlings grown on an acid soil under greenhouse condition affected by various dosages of newly formulated kieserite (FK).

No	Treatments	Dry Weight of (g)		
		Leave	Stem	Roots
1	100% dosage of NPKMg-kieserite	18.5 a*)	17.8 a	5.6 abc
2	100% dosage of NPK + 25% FK	15.6 bc	15.5 ab	5.2 bcd
3	100% dosage of NPK + 50% FK	14.3 cd	12.8 b	3.9 d
4	100% dosage of NPK + 75% FK	19.8 a	18.1 a	6.9 a
5	100% dosage of NPK + 100% FK**)	13.9 cd	13.1 b	4.6 cd
6	100% dosage of NPK + 125%	17.7 ab	17.9 a	5.9 ab
7	100% dosage of NPK + 150% FK	15.9 bc	15.2 ab	4.8 bcd
8	100% dosage of NPKMg-dolomite	15.4 bc	15.2 ab	4.9 bcd
	CV (%)	8.3	10.7	13.1

*) Figures in the same column followed by the letter(a) were not significantly different according to Duncan Multiple Ranget Test ($P < 0.05$). **) 100% dosage of Mg-FK equals to optimum dosage of Mg-Kieserite, *i.e.*, 0.8 gram/seedling (Kasno & Nurjaya, 2011).

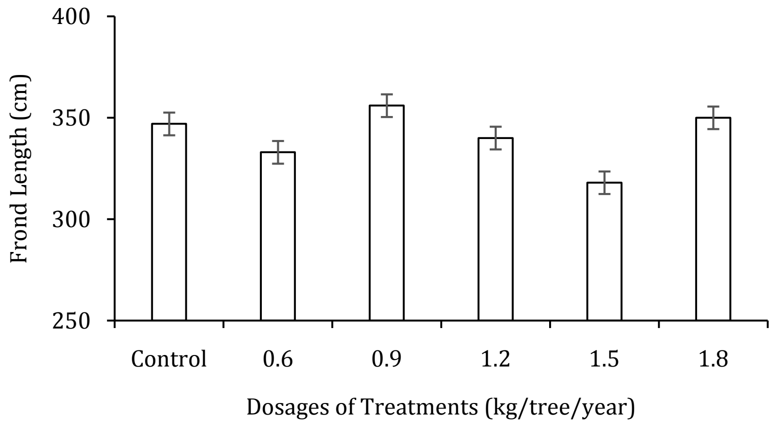


Figure 2. Effect of newly-formulated Mg fertilizer on the frond length of a three years old immature oil palm in comparison with conventional kieserite (Control, 1.2 Kg kieserite/tree/year).

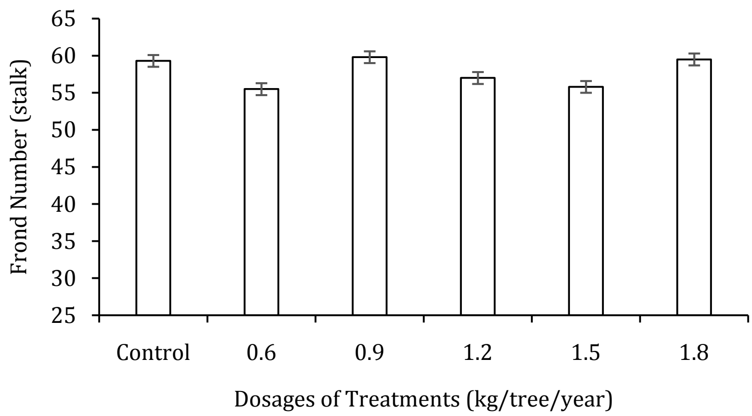


Figure 3. Effect of newly-formulated Mg fertilizer on the frond number of a three years old immature oil palm in comparison with conventional kieserite (Control, 1.2 kg kieserite/tree/year).

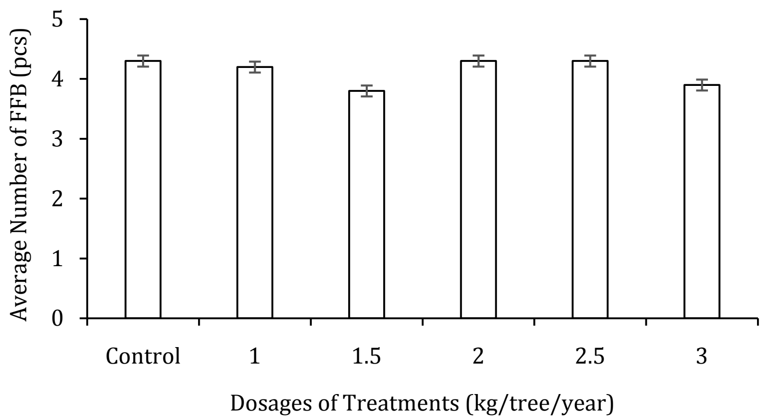


Figure 4. Effect of newly-formulated Mg fertilizer on the FFB number of a five years old mature oil palm in comparison with conventional kieserite (Control, *i.e.*, 2 kg/tree/year).

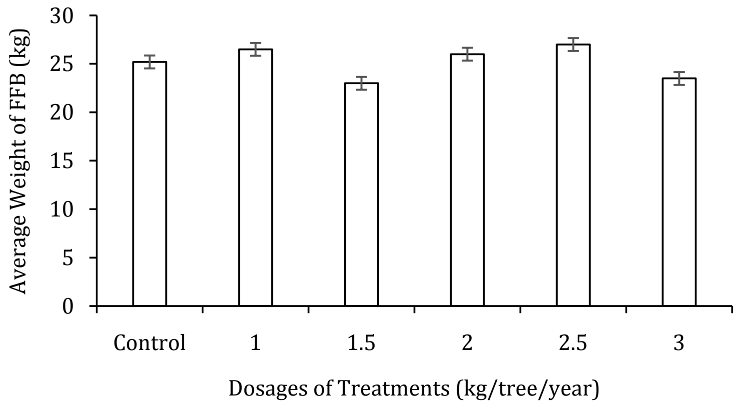


Figure 5. Effect of newly-formulated Mg fertilizer on the FFB average weight of a five years old mature oil palm in comparison with conventional kieserite (Control, *i.e.*, 2 kg/tree/year).

Based on the above findings, the reduction of Mg fertilizer up to 50% compared to that of kieserite under current prices of fertilizers locally yields a cost efficiency of fertilization up to 14.4%. Under current prices condition in which crude palm oil reaching high price level ever and the significant increase in price of all fertilizers, this newly formulated Mg fertilizer will in turn provide a better profit margin to the farmers.

4. Conclusion

To relieve the pressure on profitability of oil palm farmers in oil palm producing countries due to fertilizer price hikes, this technology has shown a highly prospective as alternative for a new Mg fertilizer replacing conventional kieserite. By applying bio-phisico-chemical treatment on a Sidayu dolomite, an alternative kieserite was developed with a superior performance compared to that on conventional kieserite for oil pam both in nursery and in the field (immature and mature trees).

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Conflicts of Interest

The authors declare no conflicts of interest regarding the publication of this paper.

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