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Calcium Silicate As A Media For Enhancement of Vitamin E Concentration from Candlenut Oil (*Aleurites moluccana*)

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Abstract. Vitamin E consists of two parts, namely tocopherol and tocotrienol which are found as minor components in candlenut oil. The process of increasing vitamin E concentrate in candlenut oil aims to obtain higher vitamin E levels from candlenut oil through a major and minor component separation with adsorption and desorption methods with three types of calcium silicate hydrate adsorbent namely calcium silicate with 90% absolute ethanol (Ca-S 90), calcium silicate with 75% absolute ethanol (Ca-S 75) and calcium silicate synthesized from CaO (Ca-S). The three adsorbents were synthesized by hydrothermal method at 170ºC for 24 hours accompanied by stirring and characterized by XRD, SEM-EDX, and BET. Furthermore, the adsorbent was applied to enrich vitamin E concentrate in candlenut oil through the adsorption process using a glass column with a mass ratio between candlenut oil and adsorbent that was 1:1, then the vitamin E desorption process was carried out by adding n-hexane solvent. Vitamin E levels obtained after the adsorption and desorption process were for Ca-S 75 adsorbent was 764.41 ppm (1. 82 times enriched), for Ca-S 90 adsorbent was 1011.29 ppm (2. 40 times enriched) and for Ca-S adsorbent was 1029, 38 ppm (2.4 5 times enriched) from the initial vitamin E level in candle nut oil was 420.66 ppm. Ca-S adsorbent showed the best vitamin E enrichment.

Keywords: Adsorbent, Antioxidant, Calcium Silicate, Candlenut Oil, Vitamin E.

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1 Introduction

Aleurites moluccana (L.) Willd or the so-called candlenut plant is a plant originating from Indo-Malaysia and distributed in almost all islands in the Indonesian archipelago (Krisnawati et al, 2011). Candlenut oil can be used as an ingredient for lighting, cooking spices, resins, pharmaceuticals (drugs), and cosmetic production (Silitonga et al., 2013). The advantages contained in candlenut oil are the content of vitamin E as a minor component consisting of tocopherol 59.9 ppm and tocotrienols 129.3 ppm (Sebayang et al., 2016).

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Pizziano et al. (2017) and Beytut et al. (2018) reported that vitamin E can act as an antioxidant that protects cells and tissues from oxidative damage and suppresses oxidative stress. In addition, vitamin E has been shown to be effective in suppressing the growth of cancer cells (Duhem et al., 2014). T Do et al. (2015) also reported that the antioxidant properties of vitamin E, in addition to reducing the risk of cancer, can also suppress the occurrence of cardiovascular disease, especially in smokers. And also vitamin E can function as a membrane stabilizer (Magdy et al., 2016). Reveny et al. (2017), Li (2015), and Uwa (2017) reported that vitamin E can prevent aging.

JECFA (Joint FAO/WHO Expert Committee on Food Additives) has defined an acceptable daily intake of vitamin E (Acceptable Daily Intake/ADI) of 0.15-2 mg/kg bw/day calculated as alpha-tocopherol (Aguilar et al. al., 2008). So in this case, a person weighing 60 kg needs to consume 9 mg to 120 mg of vitamin E per day.

The research that has been done previously in connection with the concentration of vitamin E concentrate is the process of adsorption of vitamin E from coconut oil distillate palm oil using four types of adsorbents, namely silica gel (SG), aluminum oxide (AO), synthetic brominated polyaromatic SP 207 (SP), and optipore dowex functionalized polyaromatic L-285 (DO), indicating that SG is the most appropriate adsorbent for adsorbing vitamin E with a higher adsorption capacity and the fastest desorption process (Chu et al., 2004).

Sebayang et al., (2016) have researched vitamin E enrichment using 4 types of adsorbents, namely calcium polystyrene sulfonate (Ca-PSS), aluminum polystyrene sulfonate (Al-PPS), calcium methyl ester sulfonate (Ca-MESP) and calcium silicate (Ca-S). The Ca-PSS adsorbent showed the highest enrichment of tocopherols and tocotrienols.

Seeing the great potential of vitamin E, researchers are interested to make vitamin E concentrates from candlenut oil but the vitamin E content has not been utilized significantly. This process is carried out by adsorption and desorption which aims to increase the concentration of vitamin E in candlenut oil so that it can produce food products that are equivalent to the body's daily intake of vitamin E.

The calcium silicate synthesis process in this study will be carried out using the hydrothermal method by heating at 1700ºC for 24 hours using aquabides, absolute ethanol, and 96% of ethanol following the procedure performed by Huang et al., (2017). Where this mixture is expected to produce a calcium silicate adsorbent that has good adsorption and desorption activity so that it can be used as a medium for increasing vitamin E concentrate from candlenut oil.

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2 Materials and Methods

2.1 Equipments

In this study, the equipment used were glassware, analytical balance, vacuum apparatus, hydrothermal autoclave, rotary evaporator, a set of XRD, SEM-EDX, and BET quantachrome.

2.2 Materials

The materials used were candlenut oil, $CaCl_2.2H_2O$, Na_2SiO_3 . $9H_2O$, CaO, absolute ethanol, 96% of ethanol, aquabidest, distilled water, n-hexane, and Whatman filter paper no.42.

2.3 Preparation of Calcium Silicate with 75% Absolute Ethanol (Ca-S 75)

A total of 1.20 g of CaCl 2. $2H_2O$ was put into a beaker glass then dissolved with 27.5 mL of distilled water and added 82.5 mL of absolute ethanol. Then the mixture was stirred, hereinafter referred to as solution A. Next, 2.30 g of Na 2 SiO 3 . $9H_2O$ was put into a beaker glass and then 10 mL of distilled water was added. The mixture was stirred, hereinafter referred to as solution B. Then solutions A and B were mixed rapidly with vigorous stirring to produce a white suspension and continued to be stirred for 5 minutes. The suspension obtained was put into a hydrothermal autoclave and heated at 170ºC for 24 hours with vigorous stirring. After the reaction was complete, it was then cooled in an autoclave to room temperature and the precipitate obtained was filtered with Whatman filter paper No. 42. The precipitate was washed using distilled water to pH 7 (neutral). Then it was dried in an oven at 60ºC for 24 hours to obtain solid calcium silicate (Ca-S 75) which was then weighed and characterized by XRD, SEM-EDX, and BET.

2.4 Preparation of Calcium Silicate with 90% Absolute Ethanol (Ca-S 90)

A total of 1.20 g of $CaCl_2.2H_2O$ was put into a beaker glass then dissolved with 11 mL of distilled water and added 90 mL of absolute ethanol. Then, the mixture was stirred, hereinafter referred to as solution A. Next, 2.30 g of $Na_2SiO_3.9H_2O$ was put into a beaker glass and then 10 mL of distilled water was added. The mixture was stirred, hereinafter referred to as solution B. Then solutions A and B were mixed rapidly with vigorous stirring to produce a white suspension and continued to be stirred for 5 minutes. The suspension obtained was put into a hydrothermal autoclave and heated at 170ºC for 24 hours with vigorous stirring. After the reaction was complete, it was then cooled in an autoclave to room temperature and the precipitate obtained was filtered with Whatman filter paper No. 42. The precipitate was washed with distilled water to pH 7 (neutral). Then it was dried in an oven at 60ºC for 24 hours to obtain solid calcium silicate (Ca-S 90) which was then weighed and characterized by XRD, SEM-EDX, and BET.

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2.5 Preparation of Calcium Silicate from CaO (Ca-S)

A total of 0.46 g of CaO was put into a beaker glass and then 110 mL of 96% ethanol was added. Then the mixture was stirred, hereinafter referred to as solution A. Next, 0.49 g of $SiO_2.xH_2O$ was put into a beaker glass and then 10 mL of distilled water was added. The mixture was stirred, hereinafter referred to as solution B. Then solutions A and B were mixed rapidly with vigorous stirring to produce a white suspension and continued to be stirred for 5 minutes. The suspension obtained was put into a hydrothermal autoclave and heated at 170ºC for 24 hours with vigorous stirring. After the reaction was complete, it was then cooled in an autoclave to room temperature and the precipitate obtained was filtered with Whatman filter paper No. 42. The precipitate was washed with distilled water to pH 7 (neutral). Then it was dried in an oven at 60ºC for 24 hours to obtain solid calcium silicate (Ca-S) which was then weighed and characterized by XRD, SEM-EDX, and BET.

2.6 Candlenut Seeds Maceration Extraction Process

A total of 250 g of candlenut seeds were put into a 1000 mL beaker glass, then 500 mL of n-hexane solvent was added and then mashed with a blender. Then the crushed candlenut seeds and the solvent were put into a plastic bottle and tightly closed. The bottles were then shaken in a shaker incubator for 48 hours. Then filtered candlenut seed oil extract. The oil was separated from the solvent using a rotary evaporator. Then the oil obtained was characterized by HPLC.

2.7 Adsorption and Desorption of Vitamin E from Candlenut Oil with a Mass Ratio of Adsorbent: Candlenut Oil (1:1)

A total of 0.5 g of calcium silicate adsorbent was put into a glass column. Passed 0.5 g of candlenut oil into the column and allowed to stand until the adsorbent absorbs the oil completely. Next, 1 mL of n-hexane solvent was passed and the liquid dripping from the column was accommodated and waited until there was no more liquid dripping. The obtained oil fraction was then removed by vacuum using vacuum to obtain a light yellow viscous liquid that was free of solvents. The mass of the liquid was weighed and characterized by HPLC.

3 RESULT AND DISCUSSION

3.1 XRD Analysis Results

The three-phase analysis of the adsorbent compared to the COD (crystallography open database) shown in figure 1.

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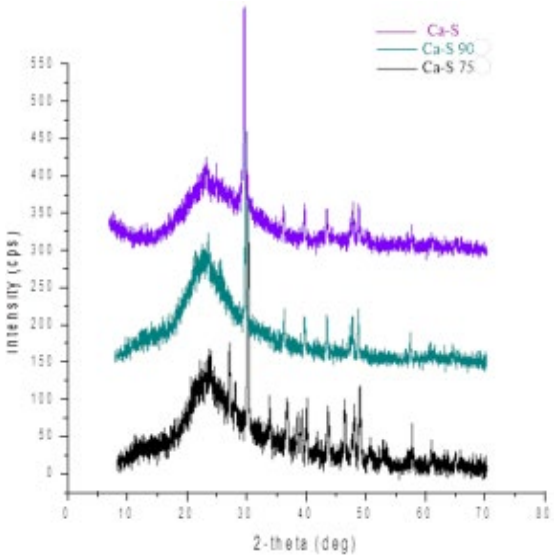

Figure 1. XRD diffractogram of Ca-S 75, Ca-S 90 and Ca-S

Tabel 1. The XRD analysis results of Ca-S 75, Ca-S 90, and Ca-S

No	Ca-S 75		Ca-S 90		Ca-S	
	2-Theta	**Compound**	**2-Theta**	**Compound**	**2-Theta**	**Compound**
1	23.10	SiO_2 _	22.90	Silica	23.02	Silica
2	26.20	SiO_2	24.42	Silica	24.08	Silica
3	27.25	SiO_2	29.21	C-S-H	29.42	C-S-H
4	29.42	C-S-H	35.75	C-S-H	31.52	C-S-H
5	31.10	C-S-H	38.41	C-S-H	35.89	C-S-H
6	33.30	C-S-H	38.58	C-S-H	39.36	C-S-H
7	35.94	C-S-H	42.91	C-S-H	42.09	C-S-H
8	38.50	C-S-H	46.92	C-S-H	43.12	C-S-H
9	39.37	C-S-H	47.30	C-S-H	43.32	C-S-H
10	41.30	C-S-H	48.30	C-S-H	47.56	C-S-H
11	43.02	C-S-H	49.87	C-S-H	48.47	C-S-H
12	45.79	C-S-H	57.14	C-S-H	57.31	C-S-H
13	47.51	C-S-H	60.42	C-S-H	60.64	C-S-H
14	48.67	C-S-H	64.34	C-S-H	64.93	C-S-H
15	50.00	C-S-H	-	-	-	-
16	52.72	C-S-H	-	-	-	-
17	57.42	C-S-H	-	-	-	-
18	60.99	C-S-H	-	-	-	-

Based on figure 1 and Table 1 show that there were 2 phases contained in the three adsorbent materials, namely C-S-H (calcium silicate hydrate) and amorphous silica. However, among the

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three adsorbents, the Ca-S 75 adsorbent has the highest peak position (strong peak) which was more numerous than the Ca-S and Ca-S 90 adsorbents. This indicates that the Ca-S 75 adsorbent has a higher compound phase.

3.2 SEM-EDX Analysis Results

The following were the surface shape of each three adsorbents presented in figure 2, 3, and 4.

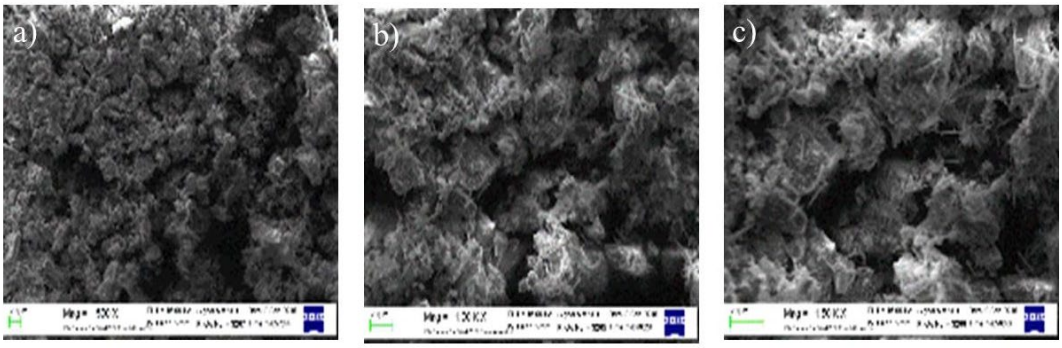

Figure 2. The SEM morphology of Ca-S 75 (a) magnification of 500X (b) magnification of 1000X (c) magnification of 1500 X

Tabel 2. The atomic composition of Ca-S 75, Ca-S 90, and Ca-S. Adsorbents

Element	Atomic Number	Ca-S 75		Ca-S 90		Ca-S	
		Wt.%	At.%	Wt.%	At.%	Wt.%	At.%
O	8	43.81	56.32	55.26	63.38	51.77	60.51
Ca	20	38.64	19.83	13.61	6.23	13.62	6.35
Si	14	6.35	4.65	19.32	12.62	22.74	15,14
C	6	11.20	19,20	11.43	17.47	11.22	17.47
Na	11	-	-	0.37	0.30	0.65	0.53
Total		100	100	100	100	100	100

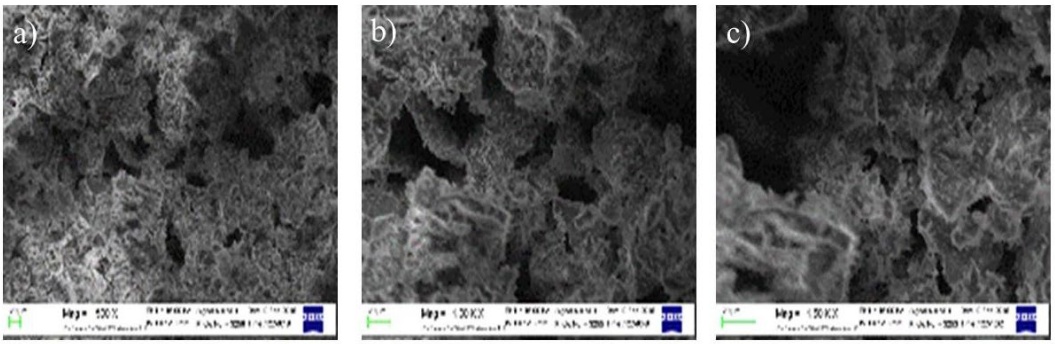

Figure 3. The SEM morphology of Ca-S 90 (a) magnification of 500X (b) magnification of 1000X (c) magnification of 1500X

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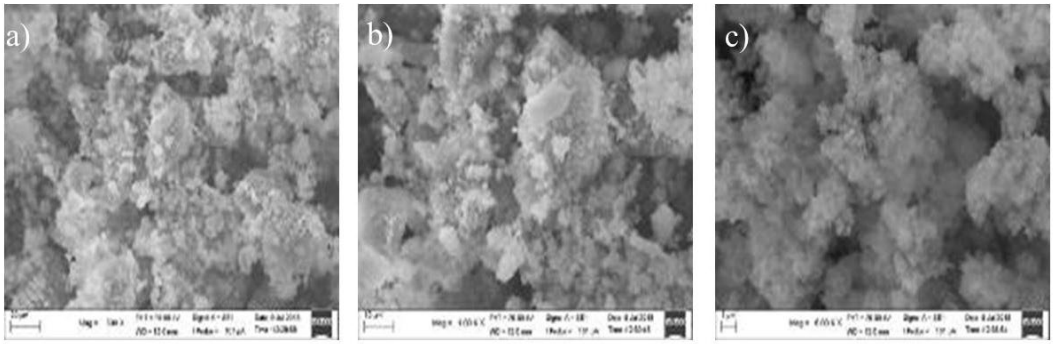

Figure 4.The SEM morphology of Ca-S Adsorbent (a) 1000X magnification (b) 1500X magnification (c) 5000X magnification

Based on figure 2 showed that in the SEM morphology the visible topographical form was like an aggregate-like structure and the presence of needle structures that were scattered in large quantities. Figure 3 showed a topographic form that looks like an aggregate-like structure and the presence of a needle structure that was scattered in fewer quantities, while figure 4 showed the topographical form looks only like an aggregate-like structure).

The following was a table of the atomic composition of the three adsorbents based on the results of the analysis by EDX

Calculation of the value of the Ca/Si ratio for the three adsorbents:

Ca/Si Ca-S ratio value 75 $= \frac{\%\ atom\ Ca}{\%\ atom\ Si} = \frac{19,83}{4,65} = 4.26$

Ca/Si Ca-S ratio value 90 $= \frac{6,23}{12,62} = 0.49$

Ca/Si Ca-S ratio value $= \frac{6,35}{15,14} = 0.41$

Table 2 showed that the adsorbent Ca-S 75 has the largest Ca/Si ratio value which according to Guan and Zhao (2016) an adsorbent was indicated to have the most Ca-OH bonds in its structure and can be theoretically referred to as an adsorbent having a structure. the most amorphous because it has a Ca/Si ratio greater than 1.5 (Yakub et al 2013). Gadde (2017) reperted that Ca/Si ratio was smaller than 0.66, this was due to the presence of silicate chains that were connected to form more polymerization in the silicate chain. The structure of the adsorbent Ca-S 90 according to Garbev et al. (2002) can be referred to theoretically as part of a class of phyllosilicate materials having a Ca/Si ratio between 0.44 to 0.66 which has a large number of polymerized silicate chains and for Ca-S adsorbents it can be referred to as a material having a polymerized chain. polymerized silicates in large quantities but are not included in the phyllosilicate class.

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3.3 BET Analysis Results

The adsorption and desorption isotherm curves of the three adsorbents indicated in figure 5.

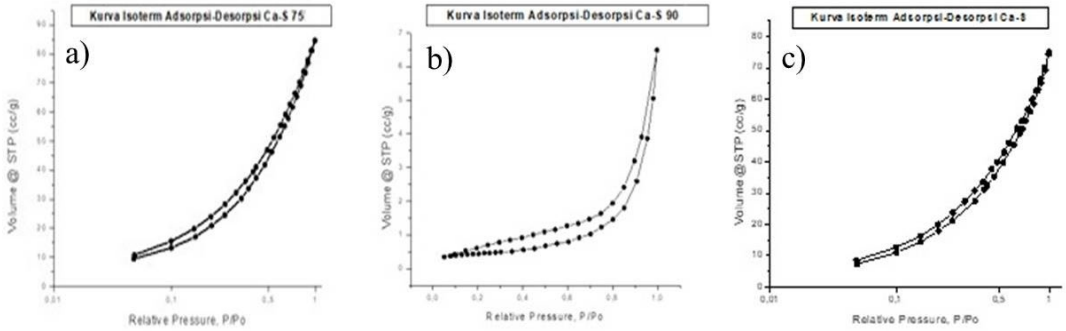

Figure 5. Adsorption-desorption isotherm curve (a) Ca-S 75 (b) Ca-S 90, and (c) Ca-S

Based on in figure 5 show that it can be classified into two classifications, namely the classification according to IUPAC and according to the Hysteresis Loop. According to IUPAC, the adsorption and desorption isotherm graphs of the three adsorbents can be classified into type V which states that the material is a mesoporous material with low adsorption energy and the formation of multilayers (Condon, 2006). Meanwhile, according to the Hysteresis Loop, the three graphs can be classified into type H3 which is a material that has slit-shaped pores with particles shaped like plates (Malherbe, 2007).

The characteristics of the adsorbent determined by the BJH (Barret-Joyner-Halende) adsorption method presented in Table 3.

Tabel 3. Characteristics of each adsorbent

Characteristics of Adsorbent (BJH Adsorption Method)	Ca-S 75	Ca-S 90	Ca-S
Pore Size (nm)	3.15	3.83	3.07
Surface Area (m^2/g)	74.814	1.909	68.225
Pore Volume (cc/g)	0.115	0.010	0.106

Table 3 show that of the three adsorbents synthesized, the Ca-S 90 adsorbent had the largest pore size since the synthesis process used more absolute ethanol as solvent. This is following the statement of Huang et al (2017) that the addition of more absolute ethanol will increase the pore size of the adsorbent but when the pore size becomes larger, the surface area size will decrease. Because of this, the surface area of the Ca-S 90 adsorbent was smaller than the surface area of the Ca-S 75 adsorbent. Meanwhile, the Ca-S adsorbent has the smallest pore size because the synthesis process only uses 96% ethanol, which was lower in purity than absolute ethanol

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3.4 HPLC Analysis Results

The following was a table of analysis results of vitamin E from candlenut oil enriched with adsorbents:

Tabel 4. The analysis results of vitamin E

Oil Mass (g)	Adsorbent	Adsorbent Mass (g)	Total Tocopherol (ppm)	Tocotrienol Total (ppm)	Total Vitamin E (ppm)	Oil Mass (g)
Initial Oil	-	-	210.74	209.92	420.66	Initial Oil
0.5	Ca-S 75	0.5	467.05	297.37	764.42	0.5
0.5	Ca-S 90	0.5	564.33	446.96	1011.29	0.5
0.5	Ca-S	0.5	676.20	353.18	1029.38	0.5

Table 3 show the analysis results of vitamin E enrichment can be observed that the highest increase in vitamin E concentrate was found in the enriched oil fraction from the Ca-S adsorbent, then followed by the enriched oil fraction from the Ca-S 90 adsorbent and the last oil fraction enriched from the Ca-S 75 adsorbent. It can be concluded that of the three adsorbents, the Ca-S adsorbent was the adsorbent that has the best level of enrichment. This was because of the Ca/Si ratio, the Ca-S 75 adsorbent has the highest value of 4.26, followed by the Ca-S 90 adsorbent which was 0.49, and the Ca-S adsorbent was 0.41, where the Ca/Si ratio of the adsorbent Ca-S 75 was much higher than the other two adsorbents. Where based on the results of EDX analysis, the higher the Ca/Si ratio value, the more Ca-OH bonds contained in the structure of the Ca-S 75 adsorbent where a large number of Ca-OH bonds will make the adsorbent more alkaline because of the Ca-OH group. it is alkaline. According to Riaz et al. (2009), vitamin E was unstable to bases. Meanwhile, according to Lambert and Leenheer (2000) in the presence of air, vitamin E will be oxidized more quickly by bases and metal ions.

4 Conclusion

Based on the data obtained in this study, it can be concluded that:

1. The mechanism of absorption of vitamin E by the calcium silicate adsorbent was through dipolar interactions, where there was an interaction between the negative pole of the adsorbent to the positive pole of vitamin E and vice versa there was an interaction between the positive pole of the adsorbent to the negative pole of vitamin E.

2. The effectiveness of calcium silicate adsorbent in increasing the concentration of vitamin E in candlenut oil was different, namely for Ca-S 75 it can increase the original vitamin E level from 420.66 ppm to 764.41 ppm (1.82 times enriched), while the adsorbent Ca-S 90 and Ca-S can increase the concentration of vitamin E from 420.66 to 1011.29 ppm (2.40 times

enriched) and 1029.38 ppm (2.45 times enriched) respectively. The highest enrichment results were shown by the Ca-S adsorbent.

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