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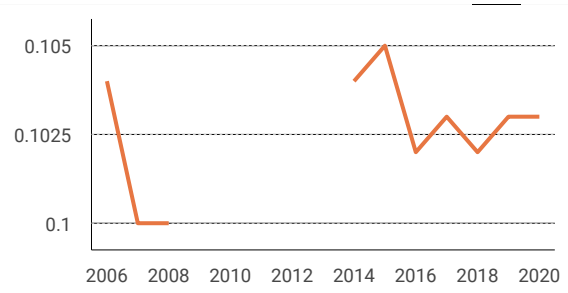

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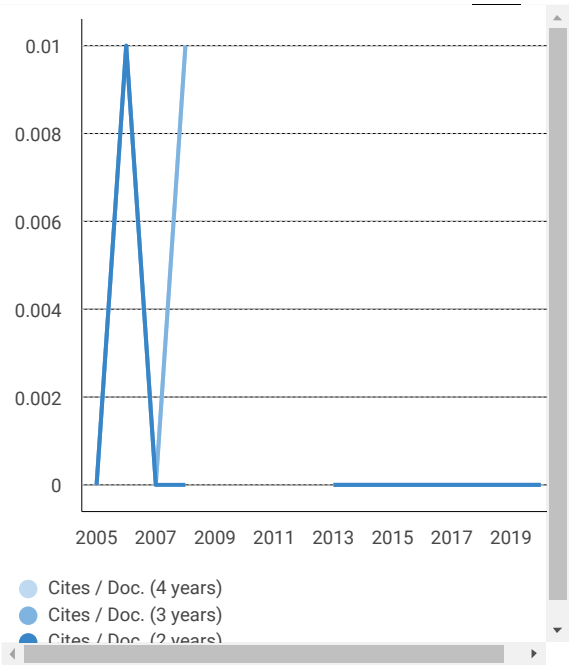

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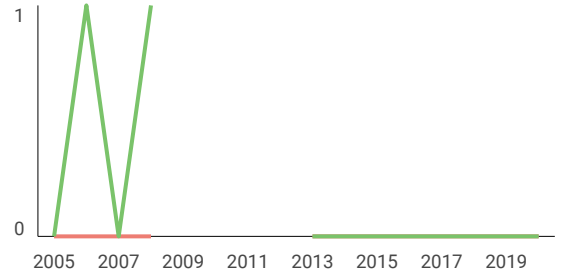

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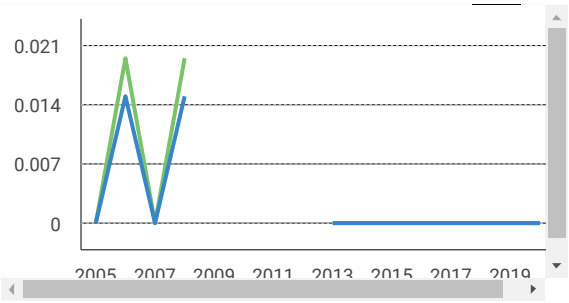

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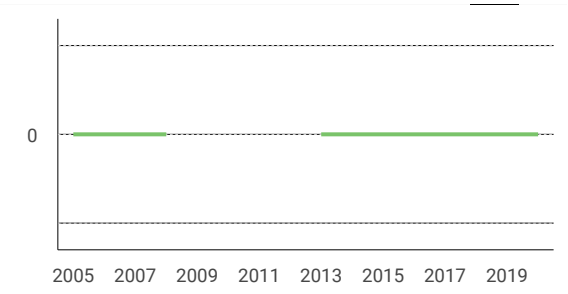

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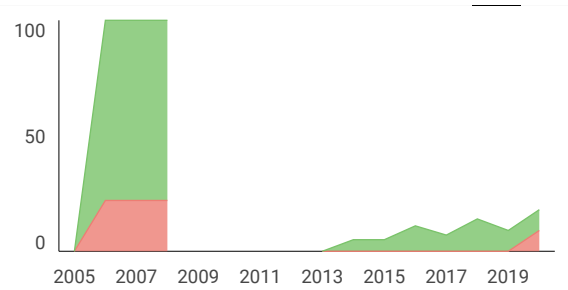

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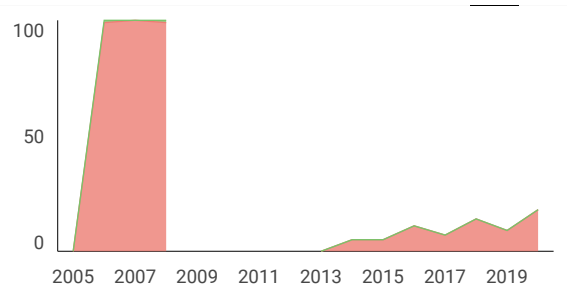

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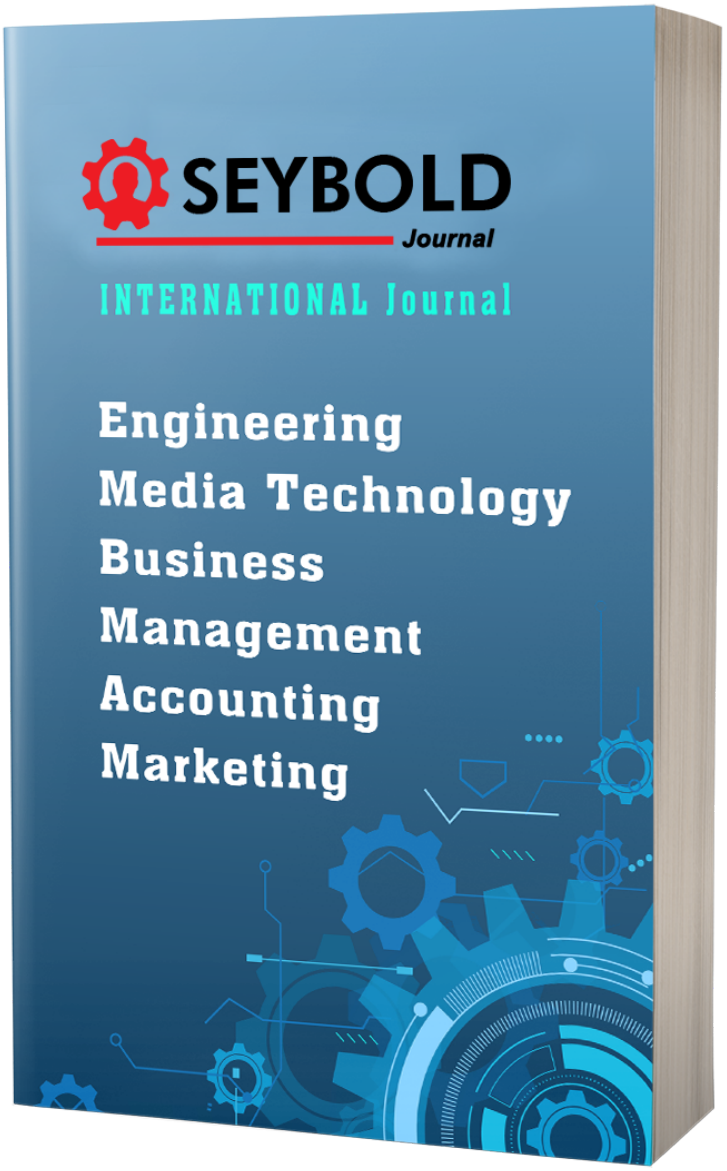

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SYNTHESIS AND CHARACTERIZATION OF NANOSILICA-CHITOSAN COMPOSITES FROM SUGARCANE BAGASSE AS Si SLOW-RELEASE SYSTEM

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Abstract

The nanosilica-chitosan composite is a combination of nanosilica and chitosan biopolymer synthesized from sugarcane and shrimp waste. This study aims to develop nanosilica-chitosan composite as fertilizers capable of releasing Si nutrients in slow-release mode and evaluating the physics, chemical and the composites quality characteristics as Si slow-release fertilizers on the nutrient uptake. The study involved nanosilica-chitosan composite synthesis and composites slow-release tests in a medium of citric acid solution of 0.33 M. The composites were characterized using an infrared spectrophotometer and X-ray diffractometer. The amount of Si test absorbed and released was analyzed using atomic absorption spectroscopy.

The characterization results using infrared spectrophotometer show that the Si composites functional group were a combination of chitosan and silica functional groups. X-ray diffraction shows that composites have amorphous properties. Composites with a large chitosan ratio have a faster Si release.

Keywords: Composites, slow-release, chitosan, nanosilica,

I. Introduction

The case of critical land has become a national issue since several decades. One source of the emergence of critical land management of forestland is not appropriate, as a result, the critical land in Indonesia is increasing every year. The real impact of the existence of critical land is environmental degradation and decline in socio-economic conditions. Critical land can lead to a decrease in land productivity and the main problems encountered in the critical land is the land of easy slopes, soil reacted sourly and poor nutrient elements.

Nutrient needs during the growth of the plant are one of the important things that determine the success of an agricultural production. Silica is one of much-needed nutrient elements of a plant. Benefits of silica among others to stimulate photosynthesis, fertilize plants, can overcome the dryness of the land, neutralizes the pH of the soil tends to be acidic urea fertilizer and granting due to pesticides that are not environmentally friendly and can strengthen the network of the plant making it more resistant to attack diseases (Suwardi, 2007).

Silica is used in the manufacture of nano silica in this research is synthesized from silica waste bark of sugar cane which is one of the alternatives to minimize waste that causes the sugar factory was forced to issue an additional cost leather waste disposal for a sugar cane. Leather waste generated some cane used as boiler fuel and the rest still as waste that is not utilized, whereas sugar cane husks skin contains a lot of nutrient elements such as Si, Fe, Al, and k. nutrient elements may be provided by way of furnace waste leather cane and synthesized for the characterization of materials (Fernandes, 2012). Purnomo and Prasetya (2007) examined the composition of BFA (Bagasse Fly ash that is kind of activated carbon derived from waste dregs of sugar cane burning results when sugar production) by using the XRF analysis, compound contained in the BFA, between other SiO2 (silica): 49.98%; Al2O3:2.20%; Cao: 2.78%; MgO: 1.65%; Natural occurring FEO: 1.22%; K2O: 3.97% and carbon: 36.50%.

According to Arizanova (2010) which analyses with XRF against the dregs of sugar cane sugar cane husks ash generated contains The most namely of 55.5%. With the presence of The compounds that were so great that in this study used the sugar cane by-product as a source of silica which is expected to produce composites that are off the slow-Si.

Fertilizer use slow-off is a new trend in saving fertilizer use and reduce the negative impact on the environment. Control or slow release fertilizers, fertilizer is that provides nutrients for the plant to the time the provision of longer than conventional fertilizer because the release of nutrients is controlled. This type of fertilizer can increase the efficient use of nutrients uptake by plants and reduce the loss of nutrients due to the dissolving (leaching) (Trenkel, 2010). During this research about a wide variety of upholstery at SRF had a lot to do. Some of the material used as a coating in the SRF, which is a material synthetic polymer. However, these materials have a shortage i.e. the non-biodegradable and will continue to accumulate so that potentially become new pollutants.

Chitosan is one of a biodegradable polymer, the availability of abundant in nature. Chitosan has an active cluster-NH2 and OH in a chain of Polymer that can be bound to a metal. In addition, it also has the properties of Chitosan is easily soluble in acid solution, so the release of micronutrient carried Chitosan corresponds to the solubility of chitosan in aqueous acid in the soil. Some research aims to obtain material that has a slow release kinetics, low solubility to water solubility and high organic acid. Therefore, this research using Chitosan as a silica coating, which is expected to release The better efficiency than not as Chitosan.

2. Research Methods

Bahan

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2.1. The synthesis of Nano silica-Chitosan Composite

As much as 0.5 grams of Chitosan acetic acid 20 ml added 2% and stirred until it formed hydrogel. Added by as much as 0.5-gram silica and stirred until homogenized. The mixture obtained is entered into the syringe and melted into the NaOH 1 M and shaped granule. Composite

washed with aquades. Dried in the oven at the temperature 50°C for 24 hours. Composites are characterized by XRD, FT-IR

2.2. analysis of Silica In Composite By Atomic Absorption Spectroscopy

Each as much as 0.1 gram of the mixed aqueous solution of HCl added composite 2.5 M and 0.5 M HF with a total volume of 10 ml for through a process of dissolution for 1 week. The total concentration of Si contained in the solution to be analyzed using atomic absorption spectroscopy (AAS) on long wave 213.86 nm. The making of the Si standard solution done by dissolving 0.1 grams of The metal in solution 20 ml HCl 37% and added aquades to the volume of 100 ml of The solution thus obtained 1000 ppm. The solution is diluted so that its concentration is obtained several times with variations 0.2; 0.4; 0.8; 1.2; 1.6 and 2.0 ppm.

2.3. Slow-Off Test Composite In Aqueous Media

Each as much as 0.1 gram composite silica composition and variation yield Chitosan incorporated into the different container that already contains a solution of citric acid 0.33 m. The solution then whipped with a shaker speed 170 rpm and taken a sample of it at the time be certain to observe the number Si are released. The ions in the solution are analyzed using the spektroskopy atomic absorption (AAS). The same procedure was done on testing with solvent citric acid 0.05 M.

3. Result and discussion

3.1. Synthesis of nanosilika-chitosan composite

3.1.I. Synthesis of Nanosilica-Chitosan Composite with XRD

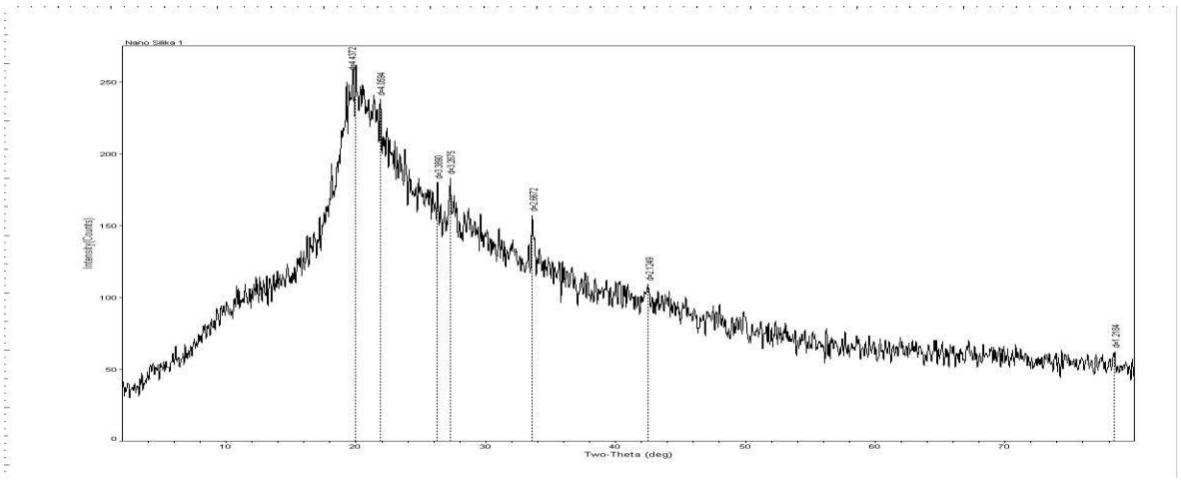


Figure 1a. Synthesis of Nanosilika-chitosan composite ratio with XRD of 1: 0.5

XRD analysis is done for to know what phase is contained in composite and change of crystallinity of base material after made composite. The basic ingredients contained in chitosan after added nanosilika. A qualitative comparison of chitosan nanosilica diffraction is compared

with chitisan. The results of chitosan and chitosan-nanosilica diffraction are used as a comparison to see the diffraction peaks or cristalinity of materials composite. Visible peak is quite sharp. The diffraction pattern in figure 1a shows the presence of a chitosan peak. Absorstion intensity d: 4,4372Å, observed peak crystalline chitosan with miller index (020) and (110) disappeared and turned into an amorphous peak of silica. The intensity absorption d:3,3890Å shows the quartz silica mineral composition d: 2,12 Å dan d :1,21Å.

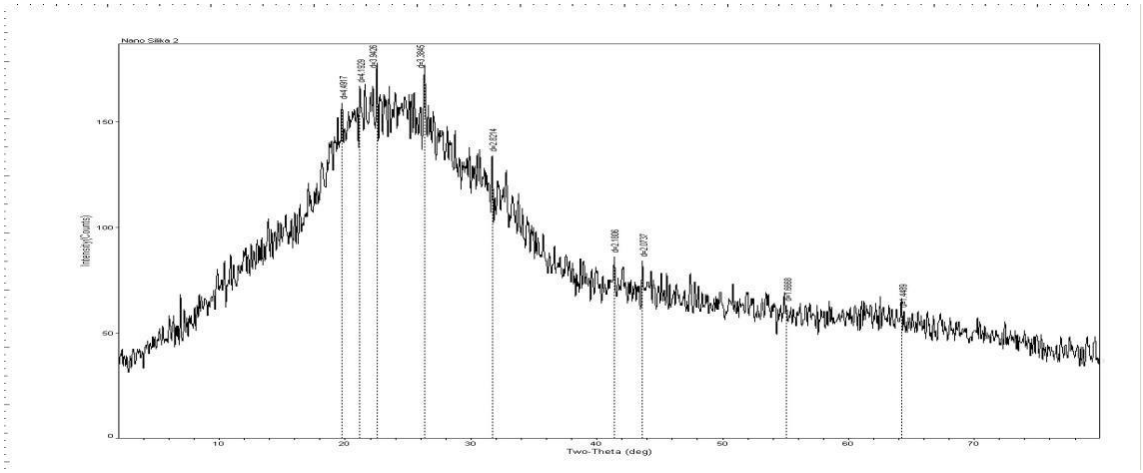


Figure 1b. Synthesis of Nanosilika-chitosan composite ratio with XRD of 1: 0.5

Figure 1b XRD nanosilica-chitosan composite with a ratio of 1:1 showing chitosan at intensity absorption intensitas d: 4,1929Å; d :4,4917Å with flat wider peak, d:4,1928Å indicating organic peak from chitosan, this is because the chitosan NH_2 group is protonated by H^+ form NH^{3+}. The nanosilica-chitosan diffractogram is not observed in sharp on amorphous peaks. Characterization of SiO_2 at :3,3845Å; d:2,1806Å; d: 2,0737Å; d: 1,4489Å. It was concluded that the results of the analysis by x-ray diffraction of the mineral types present in the nanosilica-chitosan of bagasse ash are quartz.

3.1.2. Characterization of nanosilika-chitosan composite with Infrared Spectroscopy (FTIR)

Characterization of nanosilica-chitosan composite using infrared spectroscopy to know the characteristic of functional groups in nanosilica-chitosan composite sample and to see the influence of variation of nanosilica-chitosan composite.

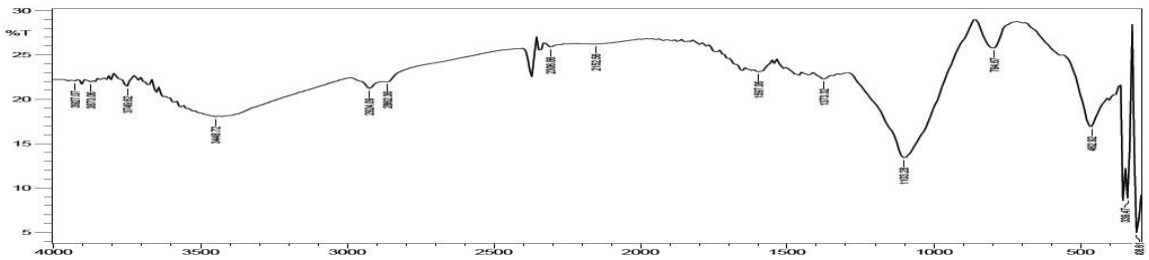


Figure 2a. Synthesis of Nanosilika-chitosan composite ratio with FTIR of 1: 0.5

Infrared spektra are presented in figure 2a and 2b. Based on Fig 2a there are characteristic peaks that appear in the area of about 3700, 3400, 2300, 1100, 794 cm^{-1}, indicated the presence of functional groups of chitosan and nanosilica-chitosan. The absorption at wave number indicating the vibration of O-H of Al(OH). The absorption at wave numbers 3448 cm^{-1} and 1597 cm^{-1} respectively states the vibration of the loose and bend OH of the H_2O molecule trapped in the semicrystal lattice.

The absorption at wave number 1373 cm^{-1} denotes vibration of Al (OH) at Si-O-Al, The absorption at wave number 1103 cm^{-1} states that the vibration of the Si-O and the absorption at the wave numbers 794 and 462 cm^{-1} shows the bend vibration Si-O (Paluszkiewicz dkk., 2011). Shift of wave numbers on the composite due to the addition of chitosan. The addition of chitosan only increases the intensity of chitosan peaks as in X-ray diffractogram.

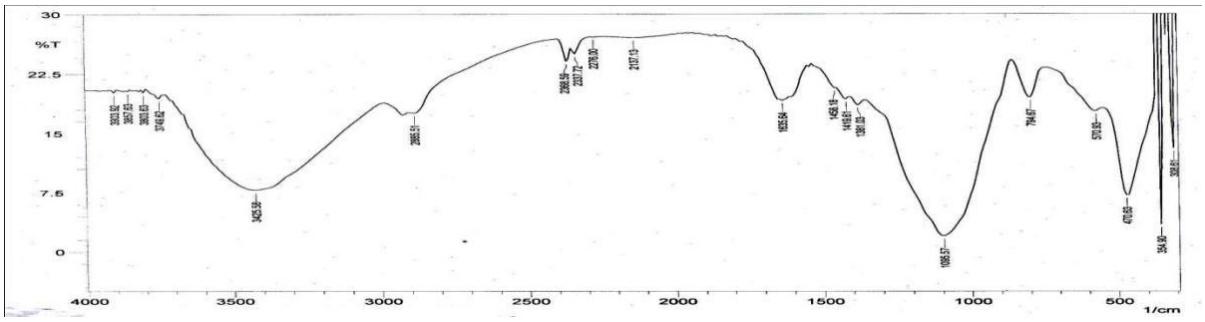
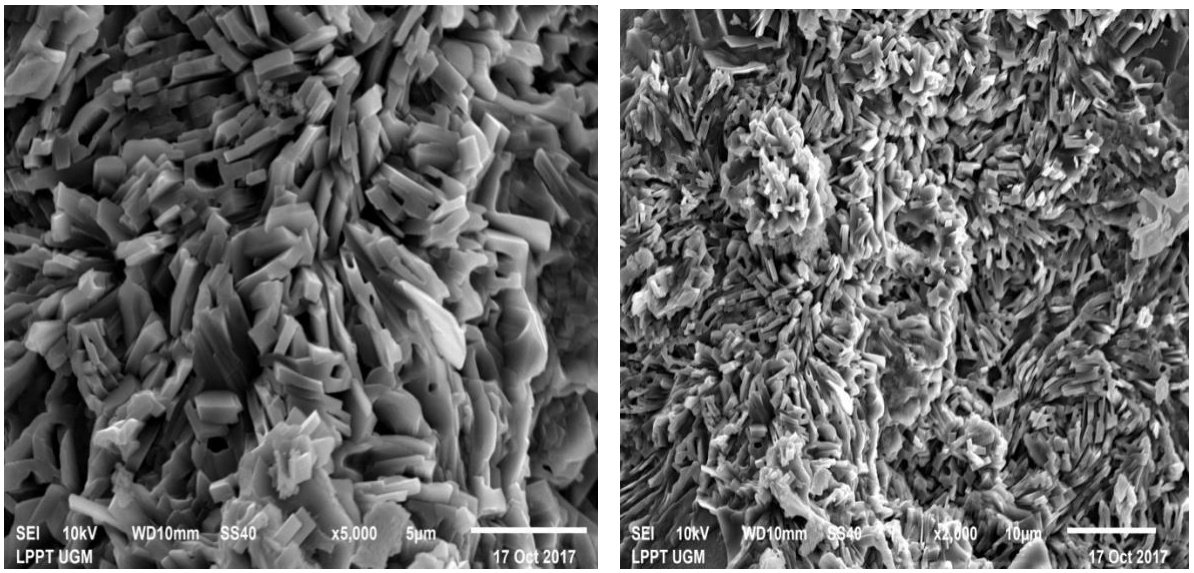


Figure 2b. Synthesis of Nanosilika-chitosan composite ratio with FTIR of 1: 0.5

FTIR spectra in figure 2b. Show an uptake at a wave number similar to the FTIR spectra in figure 2a, but there is a difference in the intensity of uptake. This change is thought to be due to reduced quartz minerals in silica. There is a characteristic peak at the region of the wave number about 3425; 2885; 1095; 470 cm^{-1} which is the absorption peak of the vibration C=O amide. –OH groups of silanol. Aluminol and water molecules that appear on wave numbers 1636 cm^{-1} and 3426 cm^{-1} and the presence of characteristic groups of chitosan ie the C-H group at the wave number 1381 cm^{-1}. Absorption at wave number 1103 cm^{-1} states the presence of vibration of Si-O and absorption at wave numbers 794 dan 462 cm^{-1} indicates the presence of vibration of Si-O buckling (Paluszkiewicz dkk., 2011).

3.1.3. Composite characterization by Scanning Electron Microscopy (SEM)



Characteristics of using SEM aim to know the morphology of composites in relation to support the prediction of Si release and the system formed by nanosilica-chitosan. By knowing the system in the composite, it will predict the release properties of Si in the nanosilica-chitosan composite. The morphology of the nanosilica-chitosan composite surface can be observed in Figure 3. The SEM photo of the composite nanosilica-chitosan (Fig. 3) shows the surface. Morphology of chitosan tissue that is porous and contains SiO particles. Chitosan tissue is represented by porous sheets, while SiO is represented by a cube-shaped material. It appears that composites with high nanosilical mass ratios exhibit rough surface morphology.

This indicates that chitosan tissue can not effectively close the silica and on composites with the same ratio of the same mass exhibit smooth surface morphology. This indicates that chitosan tissue can close the silica effectively. Composites with the same ratio are predicted to release faster Si while composites with high nanosilical ratios are predicted to be slower to release Si.

3.2. composite slow-release test in solution medium

The composites obtained were then tested for release of Si in an organic acid solution of 0,33 M citric acid. The reason for testing in citric acid because plant roots are able to secrete organic acids such as citric acid as chelates in helping the absorption of nutrients from the soil. Where as the use of citric acid concentrations of 0,33 was referred to earlier studies conducted by

Chandra *et al* (2009). The amount of Si released by the composite in citric acid was 0,33 M, then compared with the amount of Si released by the composite in 0,05 M citric acid solution.
The results of the Si release test of the composite with the silica mass variation in a 0.33 M citric acid solution were disclosed in Fig. 3.

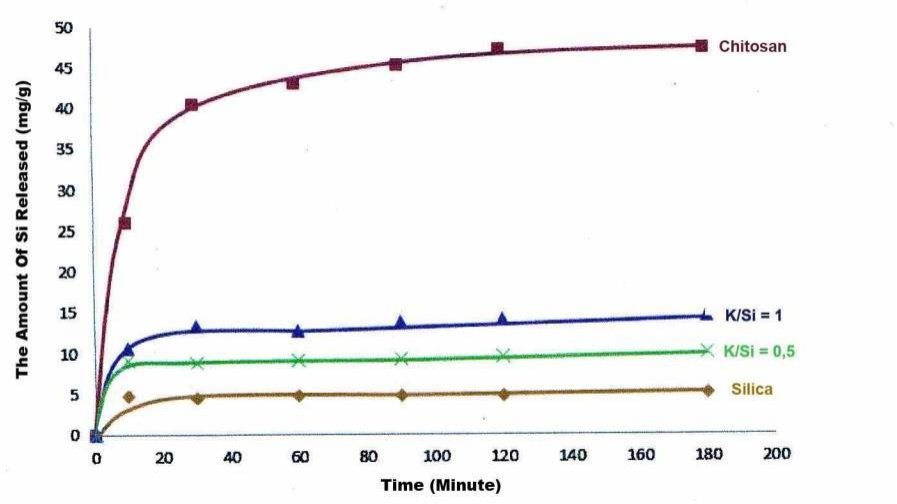


Figure 3a. Release of Si on a composite prepared with a silica mass variation in a solution of citric acid of 0.33 M

Table 1. Evaluation of Si quantities released from composites of silica mass variation in 0.33 M citric acid solution

Sample (K/Si rasio)	Si number in sample (mg/g)	Max number of Si detached (mg / g)	k (minute^{-1})
Kitosan-Si	46,96	46,96	0,0465
1	20,08	14,53	0,0339
0,5	11,42	9,96	0,0263

Based on Table 1. Viewed from the value of k (release rate constant) it can be seen that the addition of silica can decrease the value of k. This suggests that the addition of silica to the composite may decrease the release rate of Si in citric acid. Based on Figure 3. Large k values in Si-chitosan indicate Si ion is most rapidly released from chitosan-nanosilic, whereas a small k value in 0.5 variation signifies a slow Si ion release. Thus it can be said that the amount of Si released from the nanosilica-chitosan composite is greater than that released in the 0.5. The k value of the composite in the variation 1 is between the chitosan-nanosilika and the variation of 0.5. This indicates that the composite on variation 1 releases Si slower than chitosan-nanosilika, but faster than the composite at a variation of 0.5. The same data tendency was obtained in the Si release test of the composite in 0.05 M citric acid solution. The result of Si release test of composite with silica mass variation in 0.05 M citric acid solution is given in Fig. 4.

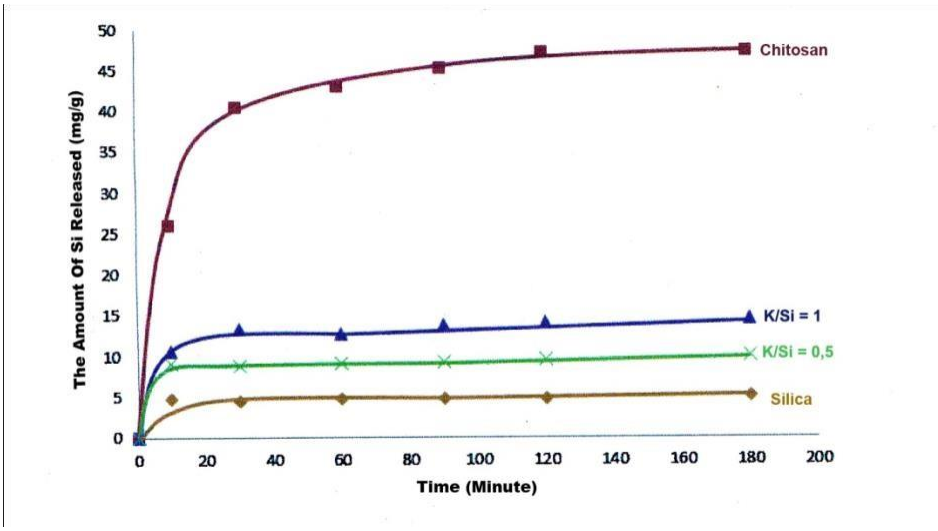


Figure 3b. Release of Si on a composite prepared with a silica mass variation in a solution of citric acid of 0.05 M

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4. Conclusion

Composites that have a high chitosan mass ratio can release Si quickly, whereas composites having a high Silica mass ratio can release Si more slowly. The k value (release rate constant) of the synthesized composite ranges from 0.0263 to 0.0465 min^{-1} for the test of 0.33 M citric acid, and ranged from 0.0228 to 0.0463 min^{-1} for the test at 0.05 M citric acid.

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