The utilization of activated carbon as micronutrients carrier in slow release fertilizer formulation

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Abstract. In many plantations, fertilizers were often added in high dosage and applied several times to get maximum productivity. But processes in soil caused not all nutrients in fertilizer could be uptaken by plants. Besides, the high dosage can be toxic to plants. The use of slow release fertilizer can overcome this case. This research was aimed to obtain the data of adsorptive capacity of activated charcoal to the nutrients mixed and gathering the information about nutrients release rate of fertilizer produced. The study was conducted by converting materials into activated charcoal. SRF was formulated by soaking fine activated charcoal CuSO\textsubscript{4}, FeSO\textsubscript{4} or ZnSO\textsubscript{4} solution for 24 hours. Analysis conducted were the observation of surface topography, qualitative analysis using EDX, and determination of total Cu, Fe and Zn in SRF. The optimal treatment was chosen to determine its release rate by extracting fertilizer with distilled water and 2\% citric acid. The results showed the activated charcoal could be used to adsorb nutrients mixed as shown by SEM and EDX results. Nutrients in the charcoal could be released slowly and could not be easily leached out.

1. Introduction

Fertilization is an effort to meet the plant’s nutrient need in order to increase productivity. Sometimes, fertilizers were added in high dosage and several times to reach optimum productivity. However, processes in soil caused not all nutrients could be used optimally by plants. Fertilization in high dosage could also be toxic to the seed in its growth period.

Fertilization that carrier out several times during the planting period increase the production cost to pay the labor. This can be overcome with the use of slow release fertilizer. Slow release fertilizer is a fertilizer that contains nutrients in it as a chemical compound or has certain physical properties so that the availability is delayed shortly after the application until finally used by plants [1]. This delay can be done one of them by entering the required nutrients into a porous material that can act as a “home” for these nutrients.

This study tries to utilize activated charcoal as a nutrient carrier in producing slow release fertilizer. Alfianto's research[2] shows that activated charcoal has the potential to be used as a micro fertilizer carrier. Morphologically, activated charcoal has a pore that is very effective in binding and storing nutrients in the surrounding soil which is then released slowly according to the rate consumed by plants.

According to Sudrajat and Soeleh [3], activated charcoal raw material determines the active charcoal character including its ability to adsorb a material. The raw material used in this study is bamboo dominated by macropores and coconut shells which are dominated by micropores. Based on Nurhayati [4] research, bamboo is one of the raw materials that can be used to produce activated charcoal because it has a high yield of charcoal and adsorption capacity.

Several studies have shown that hard raw materials such as coconut shell, oil palm, and coal produce active charcoal with high density which is suitable for gas absorption[5]. Raw materials that have large amounts of pores with small diameters and hard textures have a broad active surface and can be made of activated charcoal with high adsorption capacity. One of the raw materials with such properties is the coconut shell.

Activated charcoal has the ability to adsorb liquids and gases of various sizes. The elements added
in this study were Cu, Fe, and Zn which are micronutrients. Micronutrients are nutrients needed by plants in small amounts. The addition of these elements in excess will be toxic to the plant. By "inserting" micro nutrients into activated charcoal, it is expected that the plant's needs for these elements can be fulfilled without worrying that the added dose will be excessive because nutrients will be released slowly according to the plants taken.

This study was aimed to identify the characteristics of activated charcoal from bamboo and coconut shell activated at temperatures of 600°C and 700°C and steamed for 90 minutes, obtain data on activated charcoal adsorption capacity on mixed nutrients, and obtain information on the nutrient release rate of the mixing activated charcoal with nutrients produced.

2. Research method

2.1. Conversion of raw material into charcoal
Charcoal production is done by using a drum furnace. Combustion is carried out at a temperature that increased gradually until it reaches a temperature of ± 500°C. After all the raw materials in the furnace are completely burned, the combustion process is stopped by closing all the paths that air passes into the furnace. The process of cooling charcoal in the furnace is carried out for ± 24 hours.

2.2. Charcoal activation and analysis of activated charcoal properties
The process of producing activated charcoal is done by storing charcoal into an electric retort and activating it with water vapor at a temperature of 600°C and 700°C for 90 minutes. The activated charcoal produced was then ground to pass the 100 mesh sieve and analyzed based on SNI 06-3730-1995[6] which included rendemen active charcoal, water content, volatile content, ash content, fixed carbon content, activated charcoal adsorption capacity against iodine solution, and adsorption capacity of activated charcoal against methylene blue solution.

2.3. Formulation of Slow Release Fertilizer
Fertilizer formulation is done by soaking fine activated charcoal powder (100 mesh) in "fertilizer solution" for ± 24 hours. The concentration of the solution used is 1N or 2N and the ratio of activated charcoal to the solution is 1: 3, 1: 5, or 1: 7. Then the immersion results are cleaned and dried. After obtaining dry powder fertilizer, the analysis was then carried out which included observation of surface topography using SEM and EDX, and analysis of total Cu, Fe and Zn content by the wet digestion method.

2.4. Testing of nutrient release rates in fertilizer
This experiment was aimed to determine the release of Cu^{2+}, Fe^{3+} and Zn^{2+} contained in fertilizers. The experiment was carried out by extracting fertilizer with distilled water and 2% citric acid for 0; 15; 30; 45 and 60 minutes, and by washing the fertilizer for 25 times, then a qualitative analysis using EDX was carried out.

3. Results and discussion

3.1. Results of characterization of active charcoal analysis
Results of characterization of charcoal analysis were presented in Table 1.
Table 1. Results of active charcoal characterization analysis

| Sample   | Rendemen (%) | Water content (%) | Volatile content (%) | Ash Content (%) | Fixed Carbon (%) | Iodin Adsorption Capacity (mg/g) | Methylene blue adsorption capacity (mg/g) |
|----------|--------------|-------------------|----------------------|-----------------|-----------------|----------------------------------|------------------------------------------|
| AB       | -            | 6.16              | 16.56                | 6.53            | 71.41           | 256                              | 3.89                                     |
| B1       | 79.04        | 1.59              | 10.90                | 6.86            | 80.65           | 446                              | 18.70                                    |
| B2       | 74.64        | 2.14              | 8.95                 | 5.67            | 83.24           | 734                              | 21.99                                    |
| ATK      | -            | 7.27              | 23.69                | 7.34            | 61.69           | 466                              | 23.26                                    |
| T1       | 85.22        | 2.12              | 14.26                | 2.04            | 81.58           | 648                              | 24.94                                    |
| T2       | 81.33        | 1.62              | 13.78                | 3.22            | 81.37           | 760                              | 49.63                                    |
| SNI 06-3730-1995 | Not required | Max. 5            | Max. 25              | Max. 10 | Min. 65 | Min. 750 | Min. 120 |

Note: AB = Bamboo charcoal; B1 = Activated bamboo charcoal in 600°C with 90 minutes steamed; B2 = Activated bamboo charcoal in 700°C with 90 minutes steamed; ATK = Coconut shell charcoal; T1 = Activated coconut shell charcoal produced with 90 minutes steamed; T2 = Activated coconut shell charcoal produced with 90 minutes steamed

Based on the data in Table 1, it is known that the activated charcoal produced meets SNI 06-3730-1995 standards except for adsorption power on iodine and methylene blue. The low adsorption capacity indicates that the activation treatment of the material is not enough to open the pores of the material.

3.2. Results of slow release fertilizer analysis

The optimum condition of activated charcoal in absorbing nutrients is unknown, therefore an experiment was conducted by combining the concentration of fertilizer solution and comparison of charcoal with the fertilizer solution. Furthermore, the analysis was carried out using SEM to determine the topography of the charcoal surface, activated charcoal and activated charcoal that had been given immersion treatment. The results of the observations can be seen in Figure 1 and Figure 2.

Figure 1(a) and Figure 2(a) show that the pore has formed in charcoal, but part of the surface is still covered with hydrocarbon compounds and ash. The activation process causes shrinkage in the charcoal because more volatile material is released. This is also seen in the volatile content in activated charcoal are lower than charcoal (Table 1). Activation causes the formation of new micropores and damage to the micropore wall so that the diameter becomes larger. Figures 2 (b) and 2 (c, d, e) show that the pores which were initially empty on the activated charcoal become filled after being immersed. The same results are found in Figures 3 (b) and 3 (c, d, e), only the pore size in coconut shell activated charcoal is smaller than that of bamboo activated charcoal.
Figure 1. Surface topography of bamboo charcoal (a), bamboo activated charcoal (b), bamboo activated charcoal + Cu (c), bamboo activated charcoal + Fe (d), and bamboo activated charcoal + Zn (e) with 1000x magnification

Figure 2. Surface topography of coconut shell charcoal (a), coconut shell activated charcoal (b), coconut shell activated charcoal + Cu (c), coconut shell activated charcoal + Fe (d), and coconut shell activated charcoal + Zn (e) with 1000x magnification

Although it is known that the activated charcoal pore has been filled after being given immersion treatment, further testing is needed to ensure the element that fills the charcoal. Tests are carried out qualitatively using EDX. The test results can be seen in Figure 3 and Figure 4.
Figure 3. Observation results of EDX on bamboo activated charcoal (a), bamboo activated charcoal+Cu (b) bamboo activated charcoal+Fe (c), and bamboo activated charcoal+Zn (d).

Based on Figure 3, it is known that Cu, Fe and Zn are found in bamboo activated charcoal which has been soaked with a CuSO₄, FeSO₄ and ZnSO₄ solution which is then washed and dried. Qualitative analysis was also carried out on coconut shell activated charcoal and coconut shell activated charcoal given immersion treatment. Figure 4 showed that Cu, Fe, and Zn are found in coconut shell activated charcoal which has been soaked with a CuSO₄, FeSO₄, and ZnSO₄.

Figure 4. Observation results of EDX on Coconut Shell Activated Charcoal (a), Coconut Shell Activated Charcoal + Cu (b) Coconut Shell Activated Charcoal +Fe (c), and Coconut Shell Activated Charcoal + Zn (d)

Furthermore, quantitative analysis of treated charcoal was carried out. The analysis was carried out by the wet digestion method using aqua regia. The extracts were then measured for total Cu, Fe, and Zn levels using AAS. The results of the analysis are listed in Table 2, 3, and 4.
Table 2. Results of Activated Charcoal Analysis after Soaking in CuSO₄ Solution

| Treatment (charcoal : solution (w/v)) | Water content (%) | Ash content (%) | Total Cu (ppm) |
|--------------------------------------|-------------------|----------------|---------------|
| Control                             | 1.59              | 6.86           | 96            |
| CuSO₄ 1N (1 : 3)                    | 0.38              | 6.41           | 11,443        |
| CuSO₄ 1N (1 : 5)                    | 0.85              | 6.45           | 8,749         |
| B1                                  |                   |                |               |
| CuSO₄ 1N (1 : 7)                    | 0.76              | 6.47           | 7,850         |
| CuSO₄ 2N (1 : 3)                    | 1.75              | 6.43           | 9,002         |
| CuSO₄ 2N (1 : 5)                    | 1.68              | 6.28           | 6,866         |
| CuSO₄ 2N (1 : 7)                    | 1.12              | 6.38           | 6,109         |
| Control                             | 2.14              | 5.67           | 104           |
| CuSO₄ 1N (1 : 3)                    | 0.80              | 6.59           | 9,501         |
| CuSO₄ 1N (1 : 5)                    | 0.93              | 6.80           | 8,530         |
| B2                                  |                   |                |               |
| CuSO₄ 1N (1 : 7)                    | 0.82              | 6.79           | 6,845         |
| CuSO₄ 2N (1 : 3)                    | 1.61              | 6.91           | 8,541         |
| CuSO₄ 2N (1 : 5)                    | 1.06              | 6.57           | 6,293         |
| CuSO₄ 2N (1 : 7)                    | 1.65              | 6.32           | 5,951         |
| Control                             | 0.51              | 2.04           | 36            |
| CuSO₄ 1N (1 : 3)                    | 2.33              | 2.61           | 4,260         |
| CuSO₄ 1N (1 : 5)                    | 521               | 2.41           | 3,206         |
| T1                                  |                   |                |               |
| CuSO₄ 1N (1 : 7)                    | 6.90              | 2.83           | 4,064         |
| CuSO₄ 2N (1 : 3)                    | 0.33              | 3.01           | 4,880         |
| CuSO₄ 2N (1 : 5)                    | 0.60              | 2.84           | 4,062         |
| CuSO₄ 2N (1 : 7)                    | 1.26              | 2.45           | 3,509         |
| Control                             | 4.49              | 3.22           | 91            |
| CuSO₄ 1N (1 : 3)                    | 4.10              | 2.95           | 7,435         |
| CuSO₄ 1N (1 : 5)                    | 0.85              | 3.01           | 7,741         |
| T2                                  |                   |                |               |
| CuSO₄ 1N (1 : 7)                    | 1.11              | 3.29           | 10,775        |
| CuSO₄ 2N (1 : 3)                    | 0.36              | 3.32           | 3,558         |
| CuSO₄ 2N (1 : 5)                    | 0.45              | 3.02           | 5,513         |
| CuSO₄ 2N (1 : 7)                    | 0.41              | 3.17           | 5,968         |

Note: B1 = bamboo activated charcoal 600°C steamed 90 minutes; B2 = bamboo activated charcoal 700°C steamed 90 minutes; T1 = coconut shell activated charcoal 600°C steamed 90 minutes; T2 = coconut shell activated charcoal 700°C steamed 90 minutes.

Based on the data in Table 2 it is known that in general, the total Cu content in B1 is higher than the Cu content in B2, while the Cu content in T2 is higher than the Cu content in T1. According to the data in Table 1, B2 adsorption on iodine is higher than B1, meaning B2 has more pores. However, because the bamboo activated charcoal is dominated by macropores, the Cu²⁺ which initially made it into the pore can be lost because it was washed when the charcoal was cleaned.

In coconut shell activated charcoal, the adsorption power of T2 is higher than T1, so that Cu is adsorbed more. The optimal combination of treatments for each raw material is immersion B1 in CuSO₄ 1N (1 : 3), hereinafter referred to as C1 fertilizer and T2 immersion in CuSO₄ 1N (1 : 7) which is hereinafter referred to as C2 fertilizer. In addition to Cu, in this experiment immersion was also carried out in FeSO₄ and ZnSO₄. The results of the analysis of activated charcoal soaked in FeSO₄ and ZnSO₄ are presented in Table 3 and Table 4.
### Table 3. Results of Activated Charcoal Analysis after Soaking in FeSO₄ Solution

| Treatment (charcoal : solution (w/v)) | Water content (%) | Ash content (%) | Total Fe (ppm) |
|--------------------------------------|------------------|----------------|----------------|
| B1 Control                           | 1.59             | 6.86           | 303            |
| FeSO₄ 1N (1 : 3)                     | 0.94             | 6.13           | 2,034          |
| FeSO₄ 1N (1 : 5)                     | 1.37             | 6.30           | 2,499          |
| FeSO₄ 1N (1 : 7)                     | 1.06             | 6.16           | 3,797          |
| FeSO₄ 2N (1 : 3)                     | 1.55             | 6.28           | 4,241          |
| FeSO₄ 2N (1 : 5)                     | 2.31             | 5.93           | 5,476          |
| FeSO₄ 2N (1 : 7)                     | 2.47             | 6.15           | 4,748          |
| T2 Control                           | 4.49             | 3.22           | 294            |
| FeSO₄ 1N (1 : 3)                     | 1.68             | 2.54           | 1,266          |
| FeSO₄ 1N (1 : 5)                     | 1.69             | 2.34           | 5,448          |
| FeSO₄ 1N (1 : 7)                     | 1.85             | 2.40           | 4,775          |
| FeSO₄ 2N (1 : 3)                     | 1.98             | 2.65           | 6,581          |
| FeSO₄ 2N (1 : 5)                     | 1.80             | 3.20           | 7,611          |
| FeSO₄ 2N (1 : 7)                     | 2.03             | 3.22           | 7,051          |

Note: B1 = bamboo activated charcoal 600°C steamed 90 minutes; T2 = coconut shell activated charcoal 700°C steamed 90 minutes

Based on the data in Table 3, the optimal treatment combination for each raw material is B1 immersion in FeSO₄ 2N (1 : 5), hereinafter referred to as F1 fertilizer and soaking T2 in FeSO₄ 2N (1 : 5) which is hereinafter referred to as F2 fertilizer. The amount of Fe adsorbed activated charcoal is relatively lower compared to Cu absorption, this is because the radius of the Fe atom is larger than the Cu atom.

### Table 4. Results of Active Charcoal Analysis after Soaking in ZnSO₄ solution

| Treatment (charcoal : solution (w/v)) | Water content (%) | Ash content (%) | Total Zn (ppm) |
|--------------------------------------|------------------|----------------|---------------|
| B1 Control                           | 1.59             | 6.86           | 23            |
| ZnSO₄ 1N (1 : 3)                     | 1.97             | 6.00           | 6,603         |
| ZnSO₄ 1N (1 : 5)                     | 1.74             | 5.94           | 4,648         |
| ZnSO₄ 1N (1 : 7)                     | 1.85             | 6.18           | 3,879         |
| ZnSO₄ 2N (1 : 3)                     | 2.36             | 6.09           | 4,290         |
| ZnSO₄ 2N (1 : 5)                     | 1.87             | 6.24           | 2,873         |
| ZnSO₄ 2N (1 : 7)                     | 2.14             | 6.15           | 2,856         |
| T2 Control                           | 4.49             | 3.22           | 8             |
| ZnSO₄ 1N (1 : 3)                     | 1.96             | 2.60           | 6,343         |
| ZnSO₄ 1N (1 : 5)                     | 1.79             | 2.81           | 4,443         |
| ZnSO₄ 1N (1 : 7)                     | 1.60             | 2.96           | 2,638         |
| ZnSO₄ 2N (1 : 3)                     | 1.78             | 3.07           | 4,441         |
| ZnSO₄ 2N (1 : 5)                     | 1.93             | 2.60           | 2,205         |
| ZnSO₄ 2N (1 : 7)                     | 1.75             | 2.77           | 1,900         |

Note: B1 = Bamboo activated charcoal 600°C steamed 90 minutes; T2 = coconut shell activated charcoal 700°C steamed 90 minutes

Based on the data in Table 4, the optimal treatment combination for each raw material is B1 immersion in ZnSO₄ 1N (1 : 3) which is then referred to as Z1 fertilizer and T2 immersion in ZnSO₄ 1N (1 : 3) which is then referred to as Z2 fertilizer. The amount of Zn which activated adsorbed charcoal is relatively lower compared to the absorption of Cu and Fe, this is because the radius of Zn atoms is smaller than Cu and Fe atoms, so even though Zn atoms can be adsorbed by activated charcoal, they can also be lost during the washing process.
3.3. Result of testing of nutrient release in products
This experiment was aimed to determine the release of Cu$^{2+}$, Fe$^{2+}$, and Zn$^{2+}$ in the slow release fertilizer produced. The Experiment was carried out by extracting fertilizers with distilled water and 2% citric acid at extraction time 0, 15, 30, 45, and 60 minutes. The test results are presented in Table 5 - Table 10.

**Table 5.** Results of Cu Fertilizer Extraction with distilled water

| Fertilizer | Cu content (ppm) in extraction time | Total Cu content (ppm) |
|------------|------------------------------------|------------------------|
| C1         | 0’ 15’ 30’ 45’ 60’                | 11,443                 |
| C2         | 33 41 36 46 76                    | 10,775                 |

The data in Table 5 shows that Cu$^{2+}$ has been dissolved in distilled water even without shaking. Distilled water extracted Cu$^{2+}$ at C1 were higher than C2, so Cu$^{2+}$ was more easily available in C1 fertilizer.

**Table 6.** Results of Fe Fertilizer Extraction with distilled water

| Fertilizer | Fe content (ppm) in extraction time | Total Fe content (ppm) |
|------------|------------------------------------|------------------------|
| F1         | 0’ 15’ 30’ 45’ 60’                | 5,476                  |
| F2         | 2 1 1 2 2                         | 7,611                  |

Based on the data in Table 6, extracted Fe$^{2+}$ content are very low. This is because during the drying process, Fe$^{2+}$ is oxidized to Fe$^{3+}$ which is more stable and difficult to dissolve in distilled water.

The results of extraction of fertilizers with distilled water indicate the amount of elements available on the soil with neutral conditions that can be immediately adsorbed by plants. The distilled water content of Cu$^{2+}$, Fe$^{2+}$, and Zn$^{2+}$ is much lower than the total elements in activated charcoal after immersion, this data shows that the release of nutrients occurs slowly.

**Table 7.** Results of Zn Fertilizer Extraction with distilled water

| Fertilizer | Zn content (ppm) in extraction time | Total Zn content (ppm) |
|------------|------------------------------------|------------------------|
| Z1         | 0’ 15’ 30’ 45’ 60’                | 6,603                  |
| Z2         | 45 63 55 52 74                    | 6,343                  |

**Table 8.** Results of Cu Fertilizer Extraction with 2% citric acid

| Fertilizer | Cu content (ppm) in extraction time | Total Cu content (ppm) |
|------------|------------------------------------|------------------------|
| C1         | 0’ 15’ 30’ 45’ 60’                | 11,443                 |
| C2         | 6,219 8,418 8,138 8,513 8,692     | 10,775                 |

Citric acid has the ability to chelate metal ions and keep them in solution in pH conditions where they should settle. The extraction results with citric acid indicate that the Cu$^{2+}$ will be available entirely after being shaken for 60 minutes.

**Table 9.** Results of Fe Fertilizer Extraction with 2% citric acid

| Fertilizer | Fe content (ppm) in extraction time | Total Fe content (ppm) |
|------------|------------------------------------|------------------------|
| F1         | 1,220 2,121 2,176 2,230 2,548      | 5,476                  |
| F2         | 1,653 2,283 2,476 2,476 2,450      | 7,611                  |
Citric acid forms a more stable chelate with Fe$^{3+}$. However, the amount of Fe extracted with citric acid tends to be much lower than the total Fe in fertilizer.

| Fertilizer | Zn content (ppm) in extraction time | Total Zn content |
|------------|-----------------------------------|------------------|
|            | 0’  | 15’ | 30’ | 45’ | 60’ |               |
| Z1         | 5,699 | 6,058 | 6,195 | 6,162 | 6,330 | 6,603 |
| Z2         | 5,964 | 5,911 | 5,960 | 6,221 | 6,483 | 6,343 |

The results of Zn fertilizer extract with citric acid showed that without any shaking Zn$^{2+}$ extracted approached the total Zn level in fertilizer. The results of this test showed that in the fertilizer made in this study, Fe was adsorbed more strongly by activated charcoal compared to Cu and Zn. Some of the properties that affect activated charcoal adsorption are the chemical-physical properties of adsorbents such as pore size, and chemical composition of activated charcoal, chemical-physical properties of adsorbates such as molecular size and polarity, liquid phase properties such as pH and temperature and length of the adsorption process.

Further tests were also carried out to determine whether or not the nutrients in activated charcoal were lost due to washing. The test was carried out by washing 25x coconut shell activated charcoal + Cu (C2), then the fertilizer was dried and observed with EDX (Figure 6). From Figure 6 it is known that Cu elements are still found in activated charcoal even though it has been washed as much as 25x. This illustrates that Cu is strongly adsorbed in activated charcoal and is not easily released.

![Figure 5. Observation results of EDX on C2 washed 25x](image)

**4. Conclusions**

According to the results of the study, bamboo and coconut shell activated charcoal made by the activation process at temperatures of 600°C and 700°C and steamed for 90 minutes fulfills SNI 06-3730-1995, except for adsorption capacity on iodine and methylene blue. Only coconut shell activated charcoal is activated at a temperature of 700°C and steamed for 90 minutes which has adsorption capacity on iodine according to SNI 06-3730-1995. This shows that the activation process carried out is not enough to open the charcoal pores. However, activated charcoal can be used to adsorb added micronutrients. The adsorption capacity of activated charcoal on nutrients added is different, with the total adsorbed Cu content higher than total Fe and Zn. This is influenced by the pore size of each activated charcoal and the size of Cu, Fe and Zn atoms.

Based on the testing of nutrient release rate, it is known that the fertilizer made in this study is slowly released, with a different release rate, ie Zn is more easily released compared to Cu and Fe. In addition, it is also known that the elements in fertilizer are not easily lost due to washing experiment because they are adsorbed strongly enough by activated charcoal.

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