Properties of Controlled-Release-Water-Retention Fertilizer Coated with Carbonaceous-*g*-Poly (acrylic acid-co-acrylamide) Superabsorbent Polymer

S. Ghazali, S. Jamari, N. Noordin, and K. M. Tan

***Abstract*—Fertilizer and water are very important in determining the production of agriculture nowadays. Apart of that, the excessive use of fertilizer in plantation somehow could leads to environmental pollution. The present study reported a preparation of controlled release water retention (CRWR) fertilizer coated with superabsorbent polymer (SAPs). The purpose of coating the fertilizer using SAPs is to enhance the utilization of fertilizer and reduce the environmental pollution. In this study, the synthesis of carbonaceous-SAPs was carried out via solution polymerization technique by using monomers of poly(acrylic acid) (AA) and acrylamide (AM), cross linker of methylene bisacrylamide (MBA) and initiator of ammonium peroxodisulfate (APS) that partially neutralized with sodium hydroxide (NaOH). The fertilizer granule then was coated with carbonaceous-SAPs which later known as CRWR fertilizer. The morphology and the bonding formation of the CRWR fertilizer were investigated by using Scanning Electron Microscopy (SEM) and Fourier Transform Infrared Spectrophotometer (FTIR), respectively. Moreover, the water absorbency and water retention in soil were conducted in order to investigate the efficiency of carbonaceous-SAPs on the properties of CRWR fertilizer. Based on the results, it was found that the CRWR fertilizer coated with carbonaceous-SAPs had higher water absorbency and water retention ability than the CRWR fertilizer without carbonaceous-SAPs. The addition of the carbonaceous filler in the formulation of SAP increase the water uptake compared to unfilled-SAP. Moreover, types of soil also play an important factor in water retention properties. CRWR fertilizer in organic soil results in higher water retention ability compared in top soil.**

***Index Terms*—Carbonaceous particles, superabsorbent polymer, coating material, water retention.**

I. Introduction

The agriculture sector plays an important role in economic development, uplifting rural incomes and help in ensuring national food security [1]. Internationally agriculture has become the centre of cutting-edge research and development (R&D) as the drive to feed and cope with global population. In general, the main factors that affect the growth of plants and their quality include the amount of water and fertilizers that can be absorbed by plants. Besides maintaining the water resources, the used of fertilizer is also crucial for plant growth as it function as one of the nutrients sources for plant in order to achieve maximum efficiency and highest quality product. In order to increase the production, excessive fertilizers are normally were practiced in agricultural activities in order to fulfill the required nutrients as it will subsequently affect the quality of yield [2]. The practice of excessive used of fertilizer however is not efficient as not all nutrients from these fertilizers can be absorbed by the plant as it will be washed or leached out during rain [3]. According to Al-Zahrani [4], only 30% of the fertilizers are effectively used and utilized by plants while the balance is lost to environment. Besides of the increasing of productive cost, this practice also causes a negative impact especially on the environment as some fertilizers does contains heavy metals such as cadmium, chromium and high concentrations of radionuclides [2].

As reported by Guo et al., (2005), the loss of fertilizer in agricultural activities can cause large economic losses and environmental pollution. One of the solutions to this problem is by applying controlled release fertilizer. The use of slow release fertilizer is considered as a new trend to save fertilizer consumption and to minimize environmental pollution [6]. Practically, there are a few factors that controlled the release of the fertilizers include the soil's temperature, microbial activity, soil moisture, pH and organic matter. The present study proposed a preparation method of controlled release water retention (CRWR) fertilizer coated with carbonaceous-superabsorbent polymer material. CRWR fertilizer is known as a basic type of controlled release fertilizer (CRFs) that implement superabsorbent polymer (SAPs) as a coating material which designed to control the nutrients release from fertilizer and enhance the efficiency of nutrients use for plant growth [7]. Meanwhile, according to Liu & Guo [8], SAPs can be defined as a long chains polymer which are slightly cross-linked that has ability to swell and maintain bulky of water and aqueous solutions after absorb. The presence of hydrophilic groups such as carboxylic, carboxamide, hydroxyl, amine and amide in the polymer backbone are the main cause of the hydrophilic nature of SAPs [9]. Due to their ionic nature and interconnected structure, they absorb large quantities of water via hydrogen bonds, increasing the entropy of the network to make the SAPs swell dramatically in size [10]. In agriculture, superabsorbent polymers can be used to preserve the water in the soil and carriers for nutrients [11]. Higher water-holding capacity and nutrient retention in soils is important as it can increase the soil's aeration and microbial activity, reduce the

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environmental impact from water-soluble fertilizers and lower frequency of irrigation [12]. High water absorption of these materials is mainly attributed to the interconnected superpore structures with diameters of several hundred microns, which creating open channels that allow for capillary action. However, the use of SAPs as coatings alone is not enough as polymeric materials are known to be less biodegradable. After nutrients consumption, there is still a considerable amount of useless polymer left in the soil, which estimated around 50 kg/ha per year [13]. The incorporations of SAPs with fillers such as biochar could help in enhancing properties of SAPs as well as helping it to decompose as the properties of carbon itself that are suitable for microbe's activity [14]. Moreover, the use of biochar for agricultural application had attracted many attentions and recent studies have focused on the application of biochar to soils for carbon storage and soil fertilization [15]. The application of biochar has a number of specific functions in the natural environment as it is beneficial to prevent global warming and increases the functionality of soils. Carbon from biochar have the ability to improve the physical properties; by increasing the cation exchange capacity (CEC) and water-holding capacity, of soil [16]. Besides for soil conditioning and fertilization purposes, this application can also gave advantage in reduction of toxic components. Biochar is also capable of absorbing metals such as lead, and organics that contaminate soils which harm people, plants and animals [17].

One of the best methods to produce biochar is by pyrolysis of biomass feedstock. This process is much environmental friendly and enables the biomass conversion to biochar that are more stabilized form during the application to soil [18]. This process also requires less of the supplied fuel than traditional combustion. This present study reported the pyrolysis process of biochar, solution polymerization process of SAPs and carbonaceous-SAPs as well as preparation of CRWR. The influence of these coating materials on the water absorbency, release behavior and water retention properties of the CRWR fertilizer in different types of soils were investigated and reported in this study.

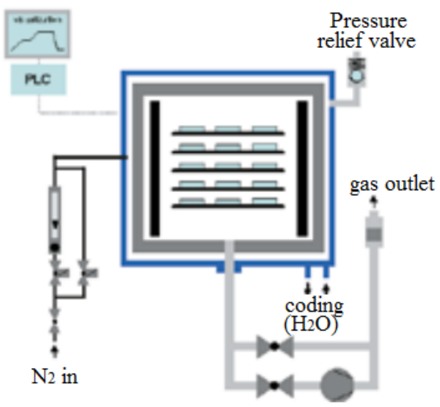

Fig. 1. Nabertherm Furnace attached with nitrogen tank.

II. Materials and Methods

A. Pyrolysis of Carbonaceous Particles

Pyrolysis of carbonaceous was carried out using biomass of empty fruit bunch (EFB) using Nabertherm furnace with nitrogen gas (Fig. 1). During the process, the nitrogen gas flow rate was set at 250 ml/min to purge out the presence of oxygen inside the furnace. The temperature rate was set constant at 10°C/min. The obtained carbonaceous particles were collected and characterized for moisture content, ash content, volatile content, and calorific value.

B. Synthesis of SAP and Carbonaceous-SAP

In this study, acrylic acid (AA) and acrylamide (AM) that acts as monomer and co-monomer, respectively were neutralized with NaOH and distilled water in 250 mL three neck flasks equipped with stirrer, condenser, nitrogen line and thermometer. MBA acts as a cross-linker agent was added in the monomer solution and was stirred at room temperature until the MBA was completely dissolved. The flask then was immersed in a water bath and heated up to 70 ℃ and then APS initiator was added to the solution that was magnetically stirred at 300 rpm [19]. The reaction mixture was left to stir for 2 hours until gel point before the resulting SAPs was removed from the reaction and dried overnight in oven at 60 ℃. The dried sample was then further crushed using ball mill and sieved. In preparation of carbonaceous-SAPs, similar procedure was repeated with addition of 0.5 wt% of carbonaceous particles as filler that obtained from pyrolysis process. Finally, the commercial fertilizer granule were dipped in starch solution and then immediately placed on the prepared SAPs and carbonaceous-SAPs powder to form CRWR fertilizer. The CRWR fertilizer then was dried in oven at 60 ℃ to obtain the final product of CRWR fertilizer.

C. Water Absorbency Properties

In order to determine the water absorbency properties, about 2 g of sample of SAPs and carbonaceous-SAPs were put in a tea bag (M_0) was immersed in 500 mL tap water and allowed to soak at room temperature up to 7h. Then, the immersed tea bags were taken out from water for every certain time and weighted (M_1). The water absorbency (WA) was calculated using the Equation (1) [20]:

$$WA = \frac{M_1 - M_0}{M_0} \tag{1}$$

D. Kinetic Diffusivity Study

Similar experiments were repeated for every 5 minutes to study the kinetic water diffusivity behavior of both samples where F stands for the diffusion mechanism of water in the SAPs, calculated by using Equation (2) [21]:

$$F = \frac{Mt}{M''} = kt^n \tag{2}$$

where Mt and M'' are the amounts of solvent diffused into the SAPs at time, t and at equilibrium, respectively; k is the diffusion rate constant and n is the diffusion exponent which determines the type of diffusion mechanism. k and n stands for intercept and slope of the line value which can be obtained from the graph of $\ln F$ versus $\ln t$.

E. Water Retention Properties of CRWR in Soil

The water retention of CRWR fertilizer in soil was determined by mixing the CRWR fertilizer in soil for a certain period of time. In this step, 2 g of CRWR fertilizer was well mixed with 200 g of soil (ratio 1:100) and kept in a beaker. Then 120 mL of tap water was slowly added into the beaker and weighed (W_1) and maintained at room temperature. In this step, the control (fertilizer without any coating material) was also being observed for comparison purposed. The beakers that contained fertilizer and soil were weighed every 2 days (W) over a period of 21 days. The water retention ratio (*WR%*) of soil was calculated using the Equation (3) [22]:

$$WR\% = \frac{W}{W_1} X100 \tag{3}$$

F. Fourier Transform Infrared (FTIR) Spectrophotometer

The chemical structure of the SAP samples was established using FTIR spectroscopy. FTIR spectra (thermo, Nicolet, NEXUS, TM) of the samples were taken in KBr pellets.

G. Morphological of SAP and CRWR

The surface morphology of the sample was observed by using Field Emission Scanning Microscope (JSM-5610 LV SEM). The sample is sputter-coated with a gold layer in a vacuum using an Emitech K550 Sputter Coater, prior to analysis.

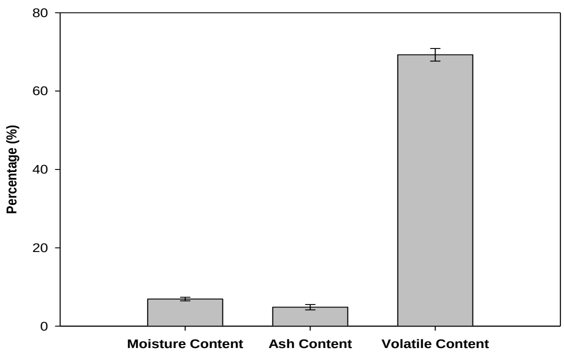

Fig. 2. Moisture content, ash content, volatile content and calorific value of carbonaceous particles.

III. Result and Discussions

A. Pyrolysis of Carbonaceous Particles

The efficiency of any future energy application process depends on the calorific value of the inlet, where high calorific value will increase the amount of energy released. In addition to calorific value, other important properties are moisture content, ash content and volatile content. Energy source that have high calorific value, low moisture content and low ash content can be burned without additional fuel, whereas materials with low calorific value, high moisture content and high ash required supplementary fuel. Analysis to determine the calorific value, moisture content, ash content and volatile content is called a proximate analysis. Fig. 2 depicted proximate analysis of carbonaceous particles that obtained from pyrolysis process. The decomposition mechanism of pyrolysis involves a significant dehydration as the changes in the H:C and O:C atomic ratios of biomass follows the dehydration pathway. The higher loss in oxygen and hydrogen compared to carbon is highly related to the increase in energy value of the biomass. Compared to oxygen and hydrogen, the bonds between carbon atoms are more stable. Therefore, more energy is needed to break the carbon bond, which will reduce the ability of the combustion.

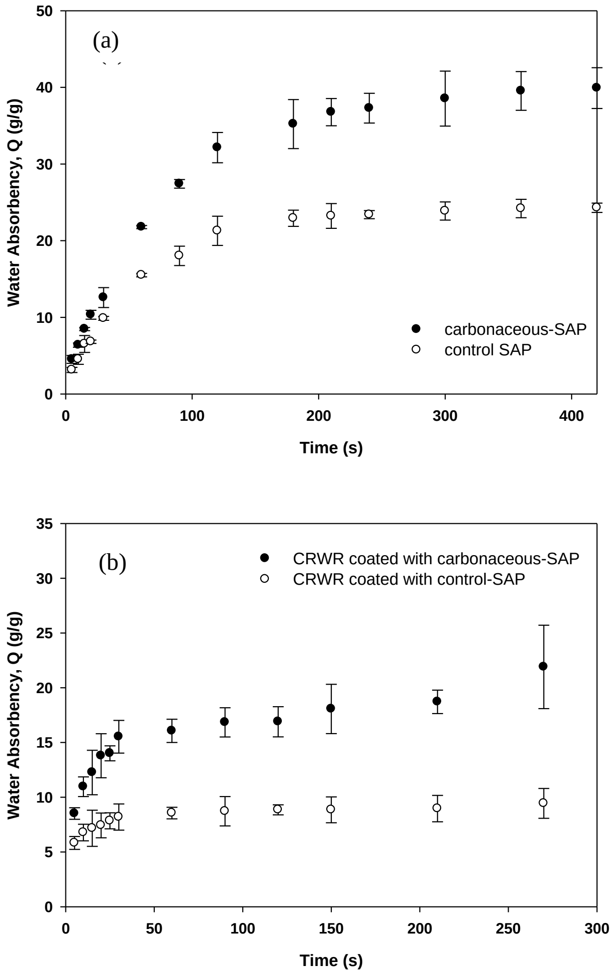

Fig. 3. Water absorbency of (a) SAP and carbonaceous-SAP, (b) CRWR coated with SAP and carbonaceous-SAP.

B. Water Absorbency Properties

The water absorbency of SAPs and carbonaceous-SAPs at various swelling time until 420 min is shown in Fig. 3(a). The absorption value of both samples (SAPs and carbonaceous-SAPs) were increased tremendously at the beginning of swelling time and reached a maximum value at certain period. From Fig. 3(a), it can be seen that the carbonaceous-SAPs had higher water absorbency value than the SAPs throughout the swelling time. The SAPs sample reached saturation or maximum absorbency value of 25 g/g at 180 min (3 h) whereas the carbonaceous-SAPs reached maximum absorbency value of 40 g/g at 300 min (5 h). From this observation, it can be stated that the addition of carbonaceous particles improved the water absorbency of the samples but somehow reduce the absorbency rate as it required longer swelling time to achieve maximum absorption compare with the SAPs sample. The presence of carbon could enhance the absorbency properties of SAP by influencing the formation of crosslinks, adding more hydrophilic groups and increasing the crosslinking density of the network which leads to higher absorbency. Apart from that, carbon itself has high porosity on its surface which enables it to hold and retain water molecules [23].

According to Zangi *et al.*, [24], fertilizer is easily dissolve in water and normally takes around 270 seconds to completely dissolve in water. Fig. 3(b) showed that water absorbency of CRWR fertilizer increased as the swelling time up to 4.5 h. It is clearly observed that CRWR coated with carbonaceous-SAPs show the higher water absorption value than CRWR coated with control-SAPs. For the CRWR coated with control-SAPs, it achieved maximum point faster than the fertilizer with carbonaceous filler which is about 8 g/g at 1 h. Meanwhile, CRWR coated with carbonaceous-SAPs approached maximum value about 18 g/g at 150 min or 2 h 30 min. The observation of water absorbency of these two types of CRWR fertilizer can be related to its morphological structure. The higher porosity governed by CRWR coated with carbonaceous-SAPs improve the ability of water uptake and increase water absorbency value. The weight gained of CRWR coated with carbonaceous-SAPs can be respected to pores that allow the water to diffuse in and occupy the space. The presence of this pores able to increase the water uptake by the fertilizer and converted it into swollen substance before diffusion taken place [19].

C. Kinetic Water Diffusivity Study of Carbonaceous SAP and Controlled SAP

As SAPs is contacted to water or a solvent, the water or solvents molecules will diffuses into the SAPs which later resulting an expansion. The diffusion process involves migration of water into pre-existing or dynamically formed species between hydrogel chains [25]. Swelling of hydrogel involves larger scale segmental motion resulting increase in spaces between polymer chains. Plots of *ln F* versus *ln t* for SAPs could be seen in Fig. 4. The data obtained for absorbency at equilibrium and at time intervals were fitted in the equation to determine the value of n and k.

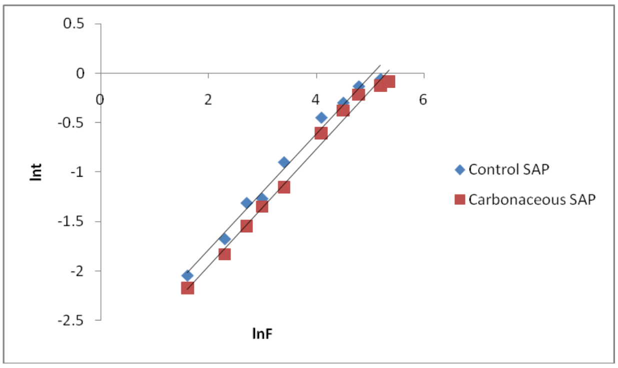

Fig. 4. Plot of ln F versus ln t for control SAP and carbonaceous SAP.

The diffusion is characterized Fickian if n <0.50 while in the case 0.50<n<1.00, the diffusion is non- Fickian (anomalous) type [26]. The n and k values depend on the absorbency behavior of the SAPs which were influenced by many factors. Based on Table I, it can be observed that both samples showed slightly non-Fickian behavior. Generally, when the diffusion is Fickian type, the water (solvents) absorbed by the SAP is relatively small and the possibility for polymer chain relaxation is seldom happened. However, when it relaxes quickly, the swelling of SAP is controlled by the diffusion of water molecule into the network. If the diffusion mode shows anomalous behavior (non-fickian), the relaxation and diffusion time are of the same order of magnitude. Therefore, as the solvent diffuses into the SAPs, there is delay in rearrangement (rupturing) within the polymer chains.

TABLE I: VARIATION OF *N* AND *K* PARAMETERS OF THE SUPERABSORBENT POLYMER COMPOSITE WITH AND WITHOUT CARBONACEOUS FILLER

Sample	*k*	*n*
Control SAP	0.0516	0.586
Carbonaceous SAP	0.0433	0.593

According to Zhao *et al.*, [27], the increasing phenomenon of n value was a result from the entanglement and thermodynamic forces. Firstly, when n value increased, chain entanglement was also increased, hence reducing the space between different polymer chains. Secondly, strong thermodynamic forces which caused from high crosslinking density, makes water to diffuse faster. When this occur, the time needed for water to diffuse in the whole network will also decrease hence resulting the increase in absorption rate. Meanwhile, the increase in k values suggests an increase in polymer-solvents interaction. The liquid flux rates through control-SAPs are faster compared to carbonaceous-SAPs. The presence of carbon in SAP forms crosslinking which increases mechanical strength and made the polymer "stiffer" compared to SAP [28]. Therefore SAP without carbonaceous fillers is considered to be more flexible which enhances permeability [25].

D. Water Retention Properties of CRWR in Soil

Some of the important soil physical conditions that influence the crop production are water capacity and water retention properties. The incorporation of carbonaceous particles could improve soil physical properties by increase the aggregate stability and reduce bulk density which eventually affects the water retention properties of the soil.

TABLE II: COMPARISON OF WATER RETENTION PROPERTIES OF 1 WT.% CRWR FERTILIZER COATED CARBONACEOUS-SAP IN ORGANIC AND TOP SOIL

Day	Water Retention (%)	
	CRWR in organic soil	CRWR in top soil
1	100.00	100.00
3	94.24	92.28
5	89.24	85.66
7	84.03	79.04
9	79.64	73.11
11	75.71	67.88
13	71.68	62.65
15	67.61	57.12
17	64.15	55.56
19	62.11	51.94
21	60.04	48.33

Organic soil normally used as potting soil for plantation. It is the organic matter component of soil, consisting of plant and animal residues at various stages of decomposition, cells and tissues of soil organisms, and substances synthesized by soil organisms [29]. Meanwhile, the top soil in general is the top layer of the earth's surface. It is in dark brown colour and high in organic matter, which makes it very easy to till and fertilize ground for growing plants. It is comprised of the three main groups of organic matter which are humus, clay particles and sand. In this study, the effect of 1 wt. % of CRWR fertilizer coated with carbonaceous-SAPs in two different soils on water retention properties were observed and summarized in Table II. The obtained result in Table II shows that CRWR coated with carbonaceous-SAPs has capability to absorb and stored water before slowly released the nutrients with soil moisture [6]. By comparing the results in Table II, it can also be seen that the water retention of CRWR coated with carbonaceous-SAPs in top soil decreased faster than the organic soil. In 7th day, the water retention of CRWR coated with carbonaceous-SAPs in organic soil and top soil is 84.03 % and 79.04 %, respectively. This results shows that the organic soil had higher water retention ability for CRWR fertilizer compared with top soil. According to El-Rahim [30], the water retention ability in soils is related to the way of soil in holding the water. There is two ways for soil holding water which are a film coating on soil particles and in the pore space between particles. Moreover, the soil porosity also could affect the water retention in soil. Large pore spaces between the soils could prevent water retention, where the water transpiration is quickly. This soil porosity are depends on soil texture and structure, where water can be held tighter in small pores than in large pores through capillary forces [31]. Therefore, organic soils with small pores can hold more water than top soil.

E. Fourier Transform Infrared (FTIR) Spectrophotometer

Fig. 5 shows the peak of O-H, C=C, C-H and C-O bonds for commercial fertilizer and CRWR fertilizers coated with SAPs and carbonaceous-SAPs. Based on Fig. 5, the commercial fertilizer had the highest wavelength peak at 3163 cm^{-1} which represents the carboxylic group, and the peaks at 1636, 1402 and 1107 cm^{-1} indicate the presence of alkene, alkane and alcohol, phenol or ester groups respectively. Meanwhile, the spectrum for the CRWR coated with SAP and carbonaceous-SAPs shows the highest wavelength at 3436-3437 cm^{-1} representing O-H bond, at 1638 cm^{-1} representing alkene group, and at 1384-1401 cm^{-1} representing alkane. Moreover, for the CRWR coated with carbonaceous-SAPs, there is three wavelengths are close to one another at 1151, 1104 and 1023 cm^{-1} that corresponding to the C-O group from the carbonaceous filler. In this study, there is an intermolecular hydrogen bond formation between the fertilizer core and SAP monomers. The broad stretching band of hydroxyl groups (3600-3300 cm^{-1}) is relating to the stretching O–H from the intermolecular and intramolecular hydrogen bonds.

F. Morphological Properties of CRWR

The surface and cross sectional image of CRWR coated with carbonaceous-SAPs that was observed under SEM at magnification of 50X are shown in Fig. 6 (a). According to Wu and Liu [15], CRWR with course and porous surface could help the sample to absorb water quickly in large amount. The cross-section for the both CRWR under SEM also reveals the presence of two layers as shown in Fig. 6 (a). However, in this study, the presence of these two layer are not uniform in thickness due to the variety of SAPs powder used during the coating process.

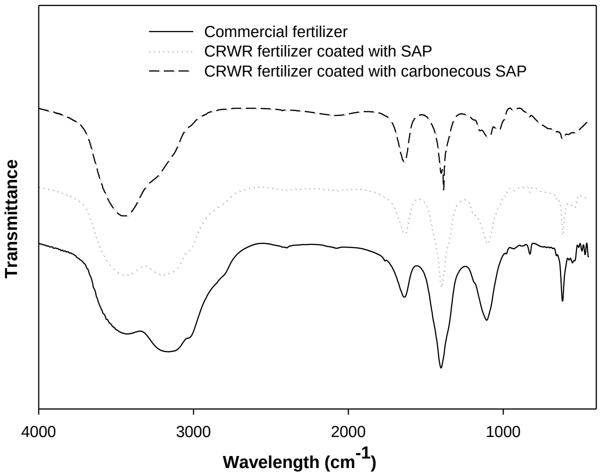

Fig. 5. The IR spectrum of commercial fertilizer, CRWR fertilizer coated with SAP and CRWR fertilizer coated with carbonaceous-SAP.

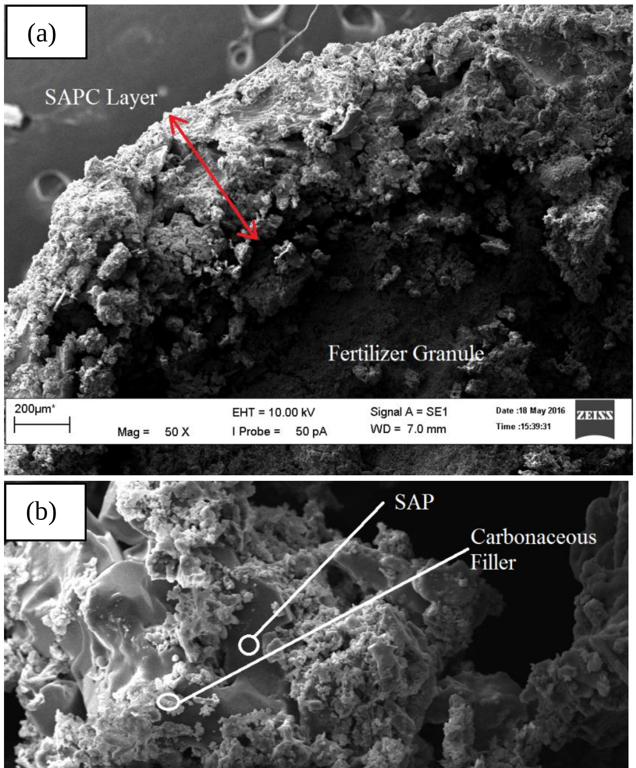

Fig. 6. SEM of the cross-section of CRWR fertilizer coated with (a) SAP (b) carbonaceous-SAP.

IV. Conclusion

In this study, the single coated of controlled release water retention (CRWR) fertilizer has been successfully prepared by using two types of SAPs coating layer. The presence of hydrophilic carbonaceous particles increased the water absorbency of the sample thus results in higher water absorbency of CRWR coated with carbonaceous-SAPs. The obtained CRWR coated with SAPs and carbonaceous-SAPs are loose and porous, with presence of holes that allowing the entry of water into the system and expands their size due to the storage of water at its outer layer. Result from FTIR analysis shows the presence of O-H, C=C, N-H, C-H, C-O, C-S and O=C=O bonds that results in high water absorbency of CRWR fertilizer. Moreover, 1 wt % CRWR fertilizer in organic soil had higher water retention ability compared with 1 wt % CRWR fertilizer in top soil. In conclusion, the presence of carbonaceous filler in CRWR gave a significant influence on the performance of water absorbency and function as water retention in soil.

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