Synthesis of Superabsorbent Polymer via Inverse Suspension Method: Effect of Carbon Filler

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Abstract. This paper studies on the effect of the addition of carbon filler towards the performance of superabsorbent polymer composite (SAPc). In this work, the SAPc was synthesized using inverse suspension polymerization method. The process involved two different solutions; dispersed phase which contains partially neutralized acrylic acid, acrylamide, APS and NN-Methylenebisacrylamide, and continuous phase which contains cyclohexane, span-80 and carbon filler (at different weight percent). The optimum SAPs and filler ratio was measured in terms of water retention in soil and characterized by Mastersizer, FTIR and SEM. Biodegradability of the polymer was determined by soil burial test and SAPc with 0.02% carbon has highest biodegradability rate. SAPc with 0.04 wt% carbon showed the optimal water retention percentage among all the samples. The synthesized SAPc producing spherical shapes with parallel alignment due to the addition of carbon fiber. It can be concluded that the addition of carbon fiber able to enhance the performance of the SAP composite (SAPc).

1. Introduction
Superabsorbent polymers (SAP) are a type of three-dimensional matrix constituted by branched or linear hydrophilic polymers that are chemically or physically crosslinked which possess the ability to absorb large quantities of water or biological fluids [1,2]. The crosslinked structure of this polymer allows it to maintain a stable network even in its swollen state[2]. Acrylic monomers or polymers, polyacrylamide and other polyacrylates are the most widely chosen as the raw materials to form high performance superabsorbent materials[3-9]. SAP can also be associated with another class of compound or substances resulting in a composite superabsorbent polymers. In this study, superabsorbent polymer has been incorporated with fibril substance which is carbon fiber. Interaction between polymer matrix and carbon fiber may lead to the enhancement of the polymer properties. Recently, demand for biodegradable polymer material increase dramatically as people worldwide...
starting to pays attention on saving the environment. Carbon fiber associated in superabsorbent polymer as filler not only can enhance the performance of the material when this polymer used as soil enhancer in agricultural field but also increase its biodegradability[6]. Oil palm or *Elaeis guineensis* is a commercial crop that are widely planted in Malaysia. Extracting crude oil from fresh oil palm fruit leaving abundant of fibrous empty fruit bunches (EFB). EFB is a natural fiber that typically consist of 24-65% cellulose, 21-34% hemicellulose and 14-31% lignin[11-16]. As it is very abundant and can widely be found in Malaysia, this fiber is the most suitable fiber used and converted into carbon fiber through hydrothermal carbonization process in this study. This study is carried out to investigate the effect of carbon filler towards superabsorbent polymer.

2. Experimental

2.1 Materials.
Oil Palm empty fruit bunch (EFB) was obtained from Lepar Oil Palm Mill, Malaysia. Chemicals used in this experiment were acrylic acid, N,N’-methylenebisacrylamide (NNMBA), ammonium persulfate(APS), sorbite anhydride monostearic acid ester (span-80), acrylamide, and cyclohexane which were supplied by Merck. Carbon fiber is obtained by processing OPEFB fiber through hydrothermal carbonization process.

2.2 Methods

2.2.1 Synthesis of Superabsorbent Polymer Composites (SAPc) Via Inverse Suspension Polymerization.
Superabsorbent polymer composites was prepared by mixing cyclohexane and span-80 in a flask then heated to 55°C with nitrogen purged for 15 minutes. Carbon fiber (0-0.04wt%) was then added into the solution and left stirred for 10 minutes. Partially neutralized acrylic acid, acrylamide, initiator APS and cross linker NNMBA was mixed in another beaker. The mixture was then added slowly into the flask and the reaction held for 3h at 65 °C with 300 rpm agitation speed. The resulted polymer was filtered, washed and dried in the oven.

2.2.2 Characterization.
The superabsorbent polymer composite (SAPc) were characterized by recording the spectrum on a Fourier Transformed Infrared Spectroscopy (FTIR) by using the attenuated total reflectance technique( ATR). The morphologies of this polymer were analyzed using Scanning Electron Microscope

2.2.3 Measurement of Particle Size.
The measurement of the particle size of the SAP and SAPc beads was determined using a particle analyzer, MastersizerSrirocco 3000 model.

2.2.4 Water Retention in Soil.
The soil was sieved by using sieve shake(2mm) and dried in the oven at 60°C. Then, 50g of soil was inserted into the container and mix with 0.5wt%, 1.5wt% and 2.5wt% of SAP and SAPc. The water was added until all the soil in the container is wet. The container was then weighed using weighing
balance to determine its initial weight, Mo. The final weight, Mi was recorded each day for the following 30 days and water retention is calculated as ratio of final and initial weight; Mi / Mo.

2.2.5 Soil Burial Test (Biodegradability).
The soil was sieved using sieve shaker (2mm) and is dried in the oven at 60°C. 0.08g of SAP and SAPc inserted into a teabag and placed in containers in which the soil covered the SAP and SAPc wholly. The container was covered with aluminium foil and kept at room temperature. All samples were weighted with consistent time interval (5 days) and weight loss percentage was calculated as (initial weight at the beginning – final weight after ten days)/ initial weight at the beginning x 100%.

3. Result and Discussion

3.1 Water Retention in Soil.
Figure 1 and 2 shows water retention percentage of SAP and all SAPc samples varies in different sets of weight percentage (0.5wt%, 1.5wt% and 2.5wt%). SAPc sample that contains 0.04wt% of carbon shows the highest water retention percentage from all sets of weight percentage which were 70.06% for 0.5wt% set, 70.07% for 1.5wt% set and 70.35% for 2.5wt% set.

![Figure 1. Water Retention of SAP and SAPc samples with 0.5wt% in soil.](image1)

![Figure 2. Water Retention of SAP and SAPc samples with 1.5wt% in soil.](image2)
3.2 Characterization.

Figure 4 represents the FTIR spectrum of SAP and SAPc samples. A strong and broad OH− stretch with H− bonded at the band of 3250 cm⁻¹ indicates that there is presence of alcohols or phenols group. Medium peak of 2835 cm⁻¹ and 1450 cm⁻¹ indicates C−H with alkanes group are formed whereas at the peak of 1650 cm⁻¹, there is medium N−H bond which signifies the presence of primary amines. Then, medium C−N stretch from aliphatic amines detected at 1055 cm⁻¹ peak. A broad and strong peak of −C≡−C−H detected at 650 cm⁻¹.

Figure 5 shows Scanning Electron Microscope micrograph of SAPc that indicates that the SAPc produced via inverse suspension polymerization was in beads and spherical form.
3.3 Soil Burial Test (Biodegradability).
Soil burial test was the test used to determine the biodegradability of SAP and SAPc. In this test, the biodegradability was determined by the percentage of polymer weight loss over time. Figure 6 shows SAPc with 0.02wt% carbon possessed the highest percentage of weight loss over time. SAP sample records lower percentage of weight loss than SAPc with 0.02wt% but higher than other SAPc samples. Presence of carbon as filler does prove that carbon filler improved the biodegradability of SAP as shown by SAPc sample with 0.02wt%. However, the other SAPc samples shows lower degradability rate may be because too much carbon filler affect the polymerization of SAPc.

![Figure 6. Percentage of weight loss of SAP and SAPc](image)

3.4 Particle Size Analysis.
The size of SAP are relatively smaller than SAPc. SAP particle size range from 563.667 μm to 709.627 μm. Meanwhile, SAPc particle size range from 893.367 μm to 1124.683 μm. Thus, carbon filler added into the SAP polymer significantly affect the size of superabsorbent polymer.

4. Conclusion.
SAPc with 0.04wt% portrayed the highest water retention rate concludes that more carbon percentage helps SAPc to retain more water than original SAP. However, SAPc with 0.02% carbon possessed the highest degradability rate than other SAP and SAPc proves that carbon filler improved SAP biodegradability. Overall, SAPc possessed better performance than normal SAP. This polymer will be further studied and applied in agricultural field.

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