

## **Synthesis of superabsorbent carbonaceous kenaf fibre filled polymer using inverse suspension polymerisation**

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### **ABSTRACT**

This paper studies the synthesis of superabsorbent carbonaceous kenaf fibre filled polymer using inverse suspension polymerisation method. The kenaf fibre was prepared using the hydrothermal carbonisation process. Inverse suspension polymerisation process involved two different solution mixtures; a continuous phase containing cyclohexane, span-80, and kenaf fibre filler and a dispersed phase containing partially neutralised acrylic acid, acrylamide, initiator APS, and crosslinker NN-Methylenebisacrylamide. Kenaf fibre filler addition was varied with different weight percentages (0.01- 0.05 wt%). Water absorption testing using the teabag method showed sample containing 0.04 wt% carbon filler had the highest and optimal percentage of water absorbency, 55.27 g/g while the sample containing 0.01 wt% carbon filler displayed the lowest percentage of water absorbency, 45.27 g/g. All SPC samples showed a higher rate of water absorbency compared to SAP sample which had 40.61 g/g of average water absorbency. The samples were characterised by FTIR, FESEM - EDX, Mastersizer. All synthesised samples produced were in spherical beads form. It can be concluded that kenaf fibre affects the enhancement of superabsorbent polymer performance.

**Keywords:** Superabsorbent polymers; inverse suspension polymerisation; swelling properties; kenaf.

### **INTRODUCTION**

Superabsorbent polymer (SAP) is a type of lightly cross-linked hydrophilic polymer chains that possess the ability to absorb a high amount of water and swell up many times of their original weight and size. The ionic functional group which lies along the polymer chain promotes diffusion of water within the polymer network [1-3]. When exposed to an excess of water, superabsorbent polymer particles swell to their equilibrium volume and do not dissolve. SAP is known for its usage in the production of personnel disposable hygienic products mainly female napkins and diapers [2, 4-7]. This polymer also played part in forestry and agricultural field. The superabsorbent polymer used as soil enhancer played role in reducing water consumption for irrigation, increasing the rate of plant growth, lowering plant death rate, and also improving fertiliser retention [8]. SAP used in soils directly influences the structure, texture, density, permeability, evaporation, and infiltration rates of water [2].

Kenaf or *Hibiscus cannabinus L* is a herbaceous plant able to produce a high content of cellulose in an average of 44 to 63.5% and hemicellulose in average between

15 to 23% [9]. Kenaf fibre is a type of natural fibre that is abundant, cheap, and recyclable which makes it suitable to be used in this project. Kenaf is commercialised in paper production, polymer reinforcement, and bedding material for horses, cattle, poultry, and rodents [10-15]. A conversion process of natural fibrous material into a carbonaceous fibre is known as the hydrothermal carbonisation process. The interactions between fibrous materials and water in subcritical condition enhance the possibility for the carbonaceous fibre production at a low operating temperature compared to the conventional method [6, 16-19]. The modification of this material as a filler into the superabsorbent polymer is able to improve the characteristics of the superabsorbent polymer as well as contribute carbon in the soil environment. Inverse suspension polymerisation of water-soluble monomers is a proven method to produce powdered beads SAP form [4]. Powdered or bead SAP form is easier to control and manage especially if this polymer production is for hygienic products or soil conditioner. This paper studied the synthesis of SAP enhanced with carbon fibre known as superabsorbent carbonaceous fibre polymer (SPC) using the inverse suspension polymerisation method.

## **METHODS AND MATERIALS**

### **Materials**

Kenaf fibre was obtained from Lembaga Kenaf and Tembakau Negara (LKTN), Indera Mahkota, Pahang. Chemicals used in this experiment were acrylic acid, N,N'-methylenebisacrylamide (NNMBA), ammonium persulfate (APS), sorbitol anhydride monostearic acid ester (span-80), acrylamide, and cyclohexane supplied by Merck. Carbonaceous fibre was obtained by processing the kenaf fibre through hydrothermal carbonisation process.

### **Production of carbonaceous Kenaf Fibre**

Kenaf was cut into short length fibre ranging from 10 to 20 cm before grinding to fine pieces approximately 550 to 600  $\mu\text{m}$ . Hydrothermal carbonisation process (HTC) was used to synthesis carbon from kenaf fibre. The HTC process was conducted for 10h at a constant temperature, 225  $^{\circ}\text{C}$  in a supercritical unit Bulchiglauster model. 50g of ground Kenaf fibre was dispersed in 1L of distilled water and mixed homogeneously before inserted into the reactor. The reactor was sealed to avoid any pressure release and heated up to 225 $^{\circ}\text{C}$ . The reactor was then let cool to room temperature once reached 10h of residence time. Kenaf fibre from the process was filtered and dried in an oven at 105  $^{\circ}\text{C}$  until constant weight. The fibre was then sieved to a size ranging from 50 to 100  $\mu\text{m}$ .

### **Synthesis of Superabsorbent Carbonaceous Fibre Polymer**

Superabsorbent carbonaceous fibre polymer was prepared by mixing cyclohexane and span-80 in a flask then heated to 55 $^{\circ}\text{C}$  with nitrogen purged for 15 minutes. The carbonaceous fibre was then added to the solution and left stirred for 10 minutes. Partially neutralised acrylic acid, acrylamide, initiator APS, and cross linker NNMBA were mixed in another beaker. The mixture was then added slowly to the flask and the reaction was held for 3h at 65  $^{\circ}\text{C}$  with 300 rpm agitation speed. The resulted polymer was filtered, washed, and dried in the oven. Six samples were prepared including controlled sample SAP.

### Characterisation

The superabsorbent carbonaceous fibre polymer (SPC) was characterised by recording the spectrum on a Fourier transformed infrared spectroscopy (FTIR) by using the attenuated total reflectance technique (ATR). The morphology of this polymer was analysed using the field emission scanning electron microscope (FESEM) coupled with energy-dispersive X-Ray (EDX) using JEOL JSM-7800F model.

### Water Absorbency Measurement

The teabag method was used to test SPC water absorbency. 0.3g SPC was weighed and immersed in deionised water for 30 minutes for the first interval, then followed by 15 minutes each interval time until the bag reached a constant weight. Afterward, the teabag was allowed to drain for a minute to rinse off excess water and weighed.

### Measurement of Particle Size

The particle size of SAP and SPC beads was measured using a Mastersizer Sirocco 3000 model of particle analyser.

## RESULTS AND DISCUSSION

Superabsorbent carbonaceous fibre polymer obtained from inverse suspension polymerisation process appeared in bead solid form. The colour varied for each sample according to the percentage of carbon filler added into the polymerisation as they turned from yellow to brownish.

Table 1. Effects of carbon filler towards SPC water absorbency.

| Sample           | Carbon Filler Percentage (%) | Average water absorbency (g water/g sample) |
|------------------|------------------------------|---|
| SAP (controlled) | 0.00                         | 40.611                                      |
| SPC 0.01         | 0.01                         | 45.270                                      |
| SPC 0.02         | 0.02                         | 47.628                                      |
| SPC 0.03         | 0.03                         | 52.224                                      |
| SPC 0.04         | 0.04                         | 55.279                                      |
| SPC 0.05         | 0.05                         | 45.945                                      |

Table 1 shows the effect of carbon filler towards water absorbency in SPC. Generally, SPC absorbs more water than the SAP. This proves that carbon filler gives direct effect in enhancing the absorption of water in this polymer. The presence of carbon in the sample increases the hydrophilic characteristic of SPC that exhibits ionic group that helps SPC bonding with water molecules. There was an increment of water absorption in SPC sample between SPC 0.01 to SPC 0.04 but a slight decrement was recorded by SPC 0.05. The lowest water absorbency, which was 40.61 g/g, was recorded from the SAP, while SPC 0.04 held the highest quantity of water absorbed (55.279 g/g). Further increase in the carbon content in the polymer matrix resulted in a decrement of water absorbency. This was proven by the value obtained from the SPC 0.05. According to Wu and Liu (2008), water absorbency increases with an increase of diatomite filler until reaching a maximum at 8 wt% but when the amount of diatomite is higher than 8 wt%, water absorbency decreases. This can be related to the amount of carbon filler added in the SPC

produced in this study in which SPC added carbon filler at 0.04 wt% was the optimum amount of the SPC water absorbency.

Figure 1 represents the FTIR spectrum of the SAP and SPC samples. The SAP sample spectra recorded weaker peaks compared to SPC sample spectrum which indicates that carbon filler affects the overall chemical composition of superabsorbent polymers. As shown in the spectra, peaks appearing in the region of wavelength 3400-3100  $\text{cm}^{-1}$  were due to O-H stretching vibration. The absorption peaks at 2950 -2800  $\text{cm}^{-1}$  indicated the presence of C-H stretching. The C=O stretching vibration peak at 1730-1700  $\text{cm}^{-1}$  was due to the presence of the carboxylic group of methyl acrylic acid in the polymer. N-H bend peaks appeared at 1640-1500  $\text{cm}^{-1}$ . Meanwhile, the absorptions band at 1320-1049  $\text{cm}^{-1}$  showed the stretching vibrations of the C-O bond. The broad small peak at 770-710  $\text{cm}^{-1}$  represented the weak band of N-H bending from amide functional groups.

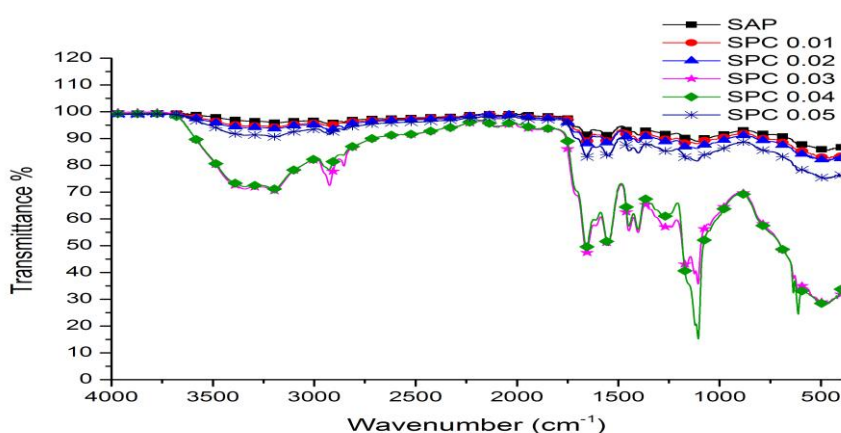


Figure 1. FTIR spectrum of SPC sample.

The distribution of particle size of SAP and SPC 0.04 are displayed in Figure 2 and 3. The highest percentage of SAP particle size ranged from 135.39  $\mu\text{m}$  to 689.716  $\mu\text{m}$  while the highest percentage of SPC particle size ranged from 93.108  $\mu\text{m}$  to 443.606  $\mu\text{m}$ . Thus, kenaf fibre added into SPC significantly affected the size of the superabsorbent polymer.

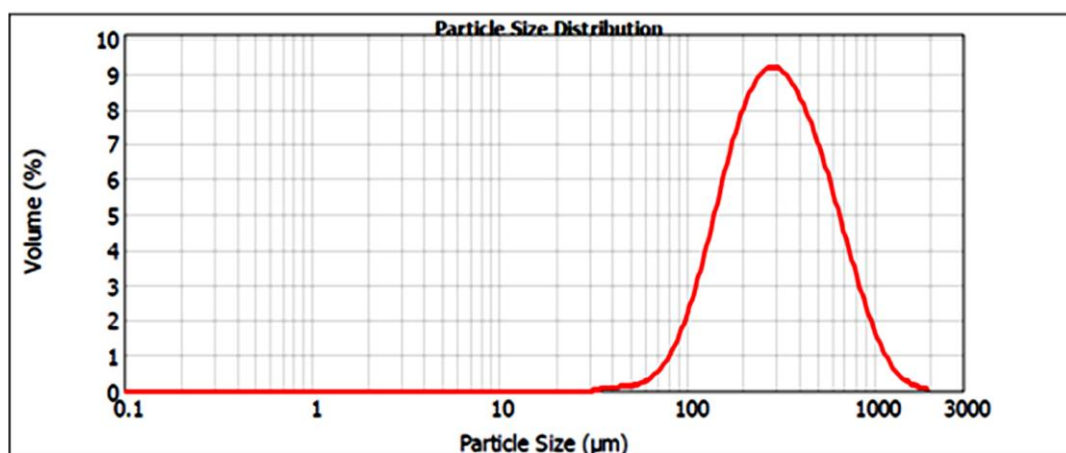


Figure 2. Particle size distribution for SAP.

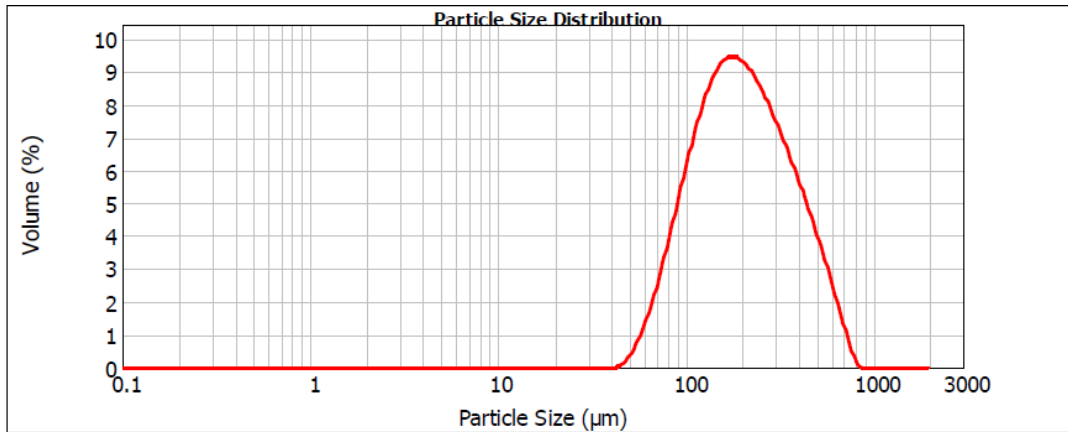


Figure 3. Particle size distribution for SPC.

Table 2. Percentage of carbon detected in SAP and SPC sample using EDX analysis.

| Sample   | Carbon Percentage Detected using EDX |            |
|----------|--------------------------------------|------------|
|          | Weight (%)                           | Atomic (%) |
| SAP      | 27.29                                | 36.52      |
| SPC 0.01 | 43.47                                | 53.52      |
| SPC 0.02 | 44.68                                | 54.18      |
| SPC 0.03 | 45.63                                | 55.29      |
| SPC 0.04 | 47.45                                | 56.69      |
| SPC 0.05 | 48.14                                | 59.02      |

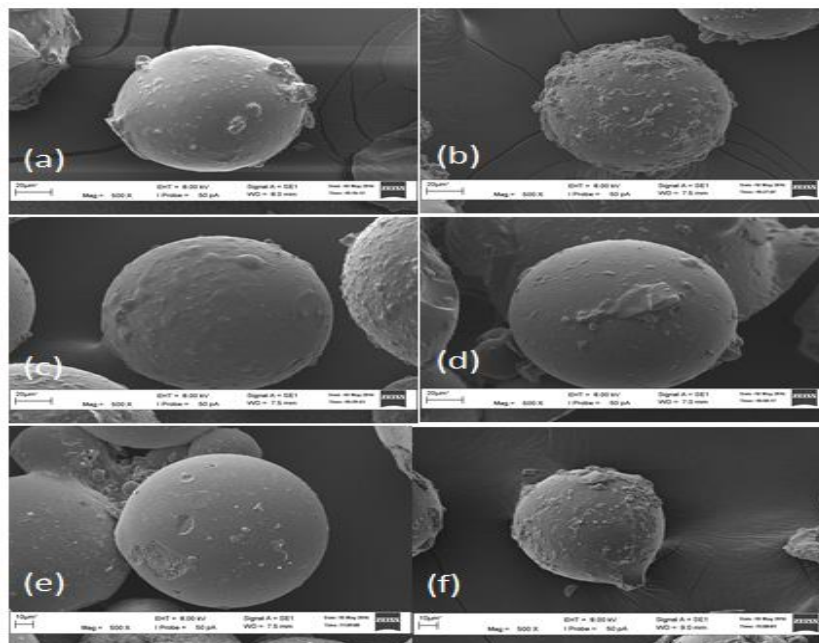


Figure 4. FESEM micrograph of SPC (a) SAP(controlled), (b) SPC 0.01, (c) SPC 0.02, (d) SPC 0.03, (e) SPC 0.04, (f) SPC 0.05.

Table 2 shows the percentage of carbon detected in SAP sample and all SPC samples using EDX analysis. The objective of this analysis is to detect the presence of carbon in SPC through quantitative analysis. It is also used to support the morphology results because FESEM alone is unable to differentiate between the carbon and polymer matrix. As expected, the SAP sample recorded the lowest percentage which was 27.29 wt% and 36.52 at%. Meanwhile, SPC 0.05 sample had the highest numbers which were 48.14 and 59.02. A slight increment was detected in SPC samples from SPC 0.01 sample until SPC 0.05 sample. This indicates that carbon filler increased in each SPC sample. Figure 4 shows the FESEM micrograph of SAP and SPC samples. As shown, it is obvious that all of the samples produced in beads sphere shaped form polymer were parallel with the theory of inverse suspension polymerisation technique. SAP and SPC bead forms are easier to handle and store for further utilisation [3-5].

## **CONCLUSIONS**

SPC sample containing 0.04 wt% kenaf fibre was portrayed to have the highest water absorbency percentage. This indicates that kenaf fibre filler affects the enhancement of SAP water absorbing potential. However, there was a slight decrement shown with 0.05 wt% SPC sample, which indicates that 0.04 wt% is the optimal amount of kenaf fibre filler needed. All samples produced were in spherical shapes. Overall, SPC samples possessed better performance than normal SAP sample. Further studies of this polymer will be conducted and optimised for future application.

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