



## Preparation of alginate/2-hydroxyethyl methacrylate composite beads: Role of fillers and characteristics of bead formation

Somkiet URUSETTHANON, Somchai UDON, and Boonsri KUSUKTHAM\*

Rajamangala University of Technology Krungthep, Nanglinchee Road, Sathorn, Bangkok, 10120, Thailand

\*Corresponding author e-mail: boonsri.k@mail.rmutk.ac.th

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### Abstract

In this research, alginate composite beads were prepared. The objective was to study the effect of particle size of fillers on the viscosity of alginate solution and bead formation. The sodium alginate solution with and without 2-hydroxyethyl methacrylate was dropped into calcium chloride to form the beads. The polymerization of the 2-hydroxyethyl methacrylate in the alginate matrix was occurred by using potassium persulphate as an initiator and *N,N'*-methylenebisacrylamide as a crosslinking agent and *N,N,N',N'*-tetramethylethylenediamine in the calcium chloride solution as an accelerator. For the preparation of the composite beads, the cotton dust, and sawdust at various ratios were mixed in the matrix. The results showed that the particle size and content of the fillers, and 2-hydroxyethyl methacrylate had an effect on the viscosity of the alginate solution. The particle size and shape of the beads depended on the particle size of fillers and viscosity of the alginate solution. Also, the addition of the cotton dust, sawdust, and 2-hydroxyethyl methacrylate increased the swelling property of the beads.

## 1. Introduction

In the cotton spinning step, it is a process to produce the cotton fibres into yarn. In this process, there is a lot of waste such as cotton dust. Thus the spinning factories need to hire a company to get rid of the waste disposed. This makes the expense of elimination. Many researchers have reported the use of the cellulose fibres in various fields such as preparation of cellulose triacetate [1], using as the substance filled with poly (lactic acid) [2,3] and used as reinforcement material of the alginate film [4].

Hydrogels are three-dimensional networks of polymers. These materials were used in different fields such as drug delivery [5], water treatment [6] and tissue engineering [7]. Hydrogels from the natural origin are polysaccharides such as alginate and chitosan. Also, the synthetic polymers are synthesized from monomers like 2-hydroxyethylmethacrylate, acrylamide and vinyl acetate. An interpenetrating polymer network (IPN) hydrogel composes of two components of polymers with distinct characteristics. The IPN is prepared by polymerization of at least one crosslinked polymer in the presence of the other. The examples of IPN are simultaneous IPN and semi-IPN. In the simultaneous IPN, both of the crosslinked polymers are polymerized concurrently. For semi-IPN, one type of polymer is crosslinked, but the other one is a liner structure.

In polymer composite, the matrix is the bulky part of a composite. While the discontinuous phase is the minor part of the materials. The disperse phase might be organic or inorganic material, e.g., paraffin [8], and

cellulose nanofibres [9,10]. For the preparation of the hydrogels and composite hydrogels, the alginate was widely used in this field. The hydrogels were prepared in the forms of microsphere [11] and film [12].

Mixing of the alginate with other substances changes the viscosity of the solution. For the preparation of the composite beads, few papers reported the relation between particle size of filler, the viscosity of the alginate solution, and bead formation. It is important to investigate the role of particle size of filler on the viscosity of the alginate solution and the formation of composite beads.

The cotton dust is an industrial waste in the textile industry. Also, the sawdust is a waste from the wood industry. Both of them should be used for a benefit substance such as a filler in polymer composite. In general, cotton dust and sawdust like to absorb water. Hence, in this research, they were chosen as the fillers in the polymer matrix to prepare the water absorption composite beads.

The alginate is a polysaccharide polymer from seaweed which has a high swelling ratio in water. In addition, 2-hydroxyethyl methacrylate is a hydrophilic polymer with polymerized easily. Thus, in this study, the mixing of the two polymers would improve the water absorption of the product. Therefore, this study was carried out to study the role of fillers on the viscosity of alginate solution and bead formation. The composite beads were prepared from the alginate/2-hydroxyethyl methacrylate mixed with the cotton dust and sawdust. Calcium chloride was used as the crosslinking agent for sodium alginate. Also, the polymerization of the 2-hydroxyethyl methacrylate

was occurred by using potassium persulphate as an initiator, *N,N,N,N*-tetramethylethylenediamine as an accelerator. The morphologies and swelling property of the beads were examined.

## 2. Experimental

### 2.1 Materials

Sodium alginate (ALG) and calcium chloride were received from Fluka (Singapore). 2-Hydroxyethyl methacrylate (HEMA), potassium persulphate, *N,N*-methylenebisacrylamide and *N,N,N,N*-tetramethylethylenediamine (TMEDA) were purchased from Aldrich (Singapore). Cotton dust (CD) was supplied by the Thai Alliance Textile Co., Ltd (Thailand). Sawdust (SD; rubberwood) was supplied by a local vendor. Nonionic surfactant (aliphatic esters and fatty alcohol) was received from Star Tech Chemical Industrial Co., Ltd (Thailand).

### 2.2 Preparation of alginate beads

Sodium alginate solution (2-20%w/v) in the burette was dropped into the calcium chloride solution at a concentration of  $0.3 \text{ mol}\cdot\text{l}^{-1}$ . Then aqueous alginate formed the pellets and left them for 1 h. They were washed with distilled water three times and dried.

### 2.3 Preparation of ALG composite beads

The CD and SD were mixed into the ALG solution at a concentration of 10%w/v. The surfactant (0.08%v/v) was added in the mixture for dispersion of the CD and SD. Then the composite beads were occurred by dropping the mixture into the calcium chloride solution and stood at room temperature for 1 h. Finally, the polymer composite beads were washed with water as described above.

### 2.4 Preparation of ALG/HEMA composite beads

The HEMA (5%v/v) was added into the ALG solution mixed with the CD and SD at various ratios. Then potassium persulfate (2 wt% of HEMA) was added, stirred and mixed them together. The composite beads were occurred by dropping the mixture into the calcium chloride solution mixed with the TMEDA (1 ml) for crosslinking of the alginate and polymerization of the HEMA.

### 2.5 Particle size measurement

The means the particle size of the cotton dust (10 g) and sawdust (10 g) were measured by Laser Particle Size Distribution Mastersizer S long bed Version 2.19, Malvern Instrument Ltd., England.

### 2.6 Viscosity of alginate solution

The alginate solution viscosity was measured by dial reading viscometer (RVT, Brookfield, USA) at a temperature of 25°C. The spindle type used was RV-3 at speed 20, and the amount of the sample used was 100 ml. The three samples were measured and calculated the mean value.

### 2.7 Particle size of bead

The particle size of the beads (10 beads) was measured by vernier caliper (Mitutoyo Corporation, Japan) and calculated the mean value.

### 2.8 FTIR measurement

The FTIR spectra of materials were taken using Perkin-Elmer FTIR Spectrophotometer System 2000, USA. The samples were pelleted with KBr.

### 2.9 Microscopic studies

The samples were coated with gold in Hitachi E 102 ion sputter (Japan). Then the SEM micrographs were taken with JEOL JSM-5410LV Scanning Electron Microscope, USA.

### 2.10 Swelling studies

The dried beads were immersed in water at room temperature for 24 h. The swollen weight of the beads was determined by blotting it with a filler paper and weighted. The percent swelling was calculated by Equation 1.

$$\% \text{ Swelling} = [(W - W_0) / W_0] \times 100 \quad (1)$$

where  $W_0$  is the initial weight and the  $W$  is the final weight of the sample. The mean value was calculated from the three samples.

## 3. Results and discussion

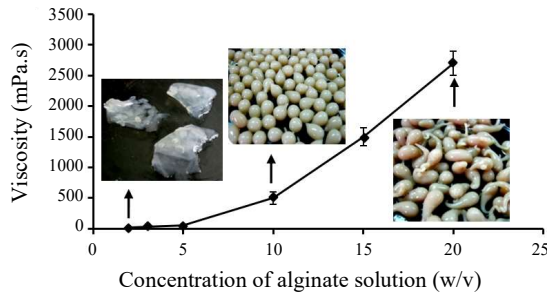
### 3.1 Alginate beads formation

For the preparation of alginate beads, the alginate solution was dropped into the  $\text{CaCl}_2$ . The gel beads appeared suddenly when the solution was contracted with the  $\text{CaCl}_2$  solution to form the alginate beads. The effect of the alginate concentration on beads formation was investigated.

Figure 1 shows the solution viscosity and the ALG bead products at various concentrations of the ALG solutions. At the concentration below 5%w/v, the beads were not formed. This resulted in not enough the carbonyl functional groups to form the beads and led to the flat of the beads [13]. The spherical ALG beads were formed completely at the concentration of

5%w/v. In comparison with 5%w/v, the fresh beads at 10%w/v were strong with mean particle size 4.4 mm. Also, the long tail beads were observed at the concentration of 20%w/v.

The flow of the ALG solution was similar to the flow of a fluid in a tube, consisting of pressure-flow (from pumping) and shear flow from the wall of the tube. When the ALG solution emerged from the orifice into the air, the broke-up of viscous liquid into drops was caused by gravity. At a low concentration of the ALG solution, the flow of solution liked a liquid. For medium viscosity, the droplet was spherical. Also, the high viscosity of the ALG solution formed the long tail at the end of the droplet. The shapes of the ALG beads were changed at high concentrations, e.g., 10-20%w/v due to the viscous solution of the ALG. This caused the elongational flow of the ALG solution from the orifice. Thus, the concentration of the ALG solution produced the different shapes of beads. In the next step, the ALG solution at a concentration of 10%(w/v) was used to prepare the beads.



**Figure 1.** Viscosity of the alginate solutions and alginate bead products.

### 3.2 Alginate composite beads formation

#### 3.2.1 Effect of CD and SD content

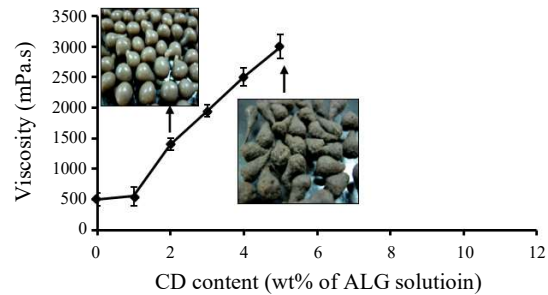
The mean particle size of the CD and SD is shown in Table 1. The result showed that the SD exhibited a large mean particle size in comparison with the CD. However, these materials were used as the fillers in the ALG solution for the ALG composites beads preparation.

The effect of the CD and SD content on the characteristic of the beads was investigated. Figure 2-3 show the relationship between the fillers content and viscosity of the solutions. The result showed that an increase in the CD content increased the viscosity of the ALG solutions (see Figure 2). Also, the ALG/CD had a high viscosity in comparison with the ALG/SD (see Figure 3). The addition of the CD in the ALG solutions changed the products from spherical beads to non-spherical form (see Figure 2). However, an increasing in the amount of the CD higher than 5%, the bead could not be prepared due to the CD clogged at the orifice. This resulted from the small particle size and high surface area of the CD particles. In contrast

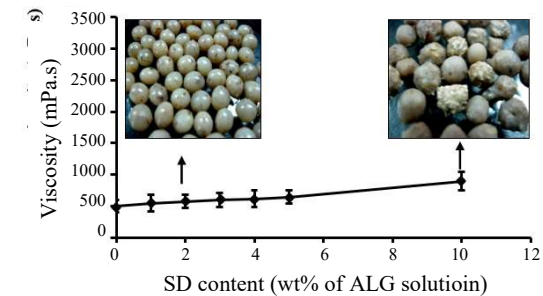
with the CD, the mixing of the SD in the ALG solutions, the particles showed a round shape (see Figure 3). This may be due to the large particle size of the SD (see Table1).

**Table 1.** Mean particle size of the CD and SD.

Materials	Code	Mean particle size (micron)
Cotton dust	CD	113.67
Sawdust	SD	168.51



**Figure 2.** Viscosity of the alginate solution at various CD content.



**Figure 3.** Viscosity of the alginate solution at various SD content.

#### 3.2.2 Effect of fillers and HEMA on viscosity of slurries

The viscosity of the ALG solutions with and without the CD and SD is shown in Figure 4. In the case of the CD (see Figure 4 at CD: SD = 1:0), the viscosity of the ALG solution increased with the addition of the CD. The fuzzy structure and ribbon-like on the CD are shown in Figure 5. In the wet state, the entanglement of the CD fibres increased friction during application shear stress and led to increasing the viscosity of the slurries [14].

In the case of the SD, the sample showed a rigid particle unlike the CD (see Figure 6). Hence, the viscosity of the slurries mixed with the SD was lower than mixed with the CD (see Figure 4 at CD: SD = 0:1). In addition, the small particle size of the CD (with mean particle size 113.67 micron) compared with the SD (with mean particle size 168.51 micron) led to increase in the friction and caused the viscosity of the slurries higher than mixed with the SD. It

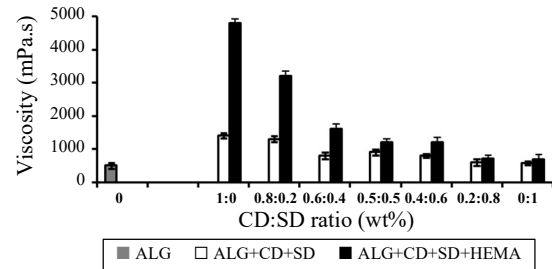
indicated that the small particles induced more viscous than the large one. For the mixing of the CD and SD at various ratios (see Figure 4), the viscosity of the slurries decreased with an increase in the SD ratio. Hence, the viscosity of the ALG depended on the type of fillers and particle size.

In the polymerization system of the HEMA and the ALG, the poly(HEMA) was crosslinked, also, the alginate was in a crosslinked form. This system was termed as a simultaneous interpenetrating polymer network (simultaneous IPN). It means both of them were crosslinked at once. The simultaneous IPN bead formation between the HEMA and the ALG is shown in Scheme I. The poly(HEMA) was crosslinked with the MBAM while the  $\text{CaCl}_2$  was used as the crosslinking agent for the ALG.

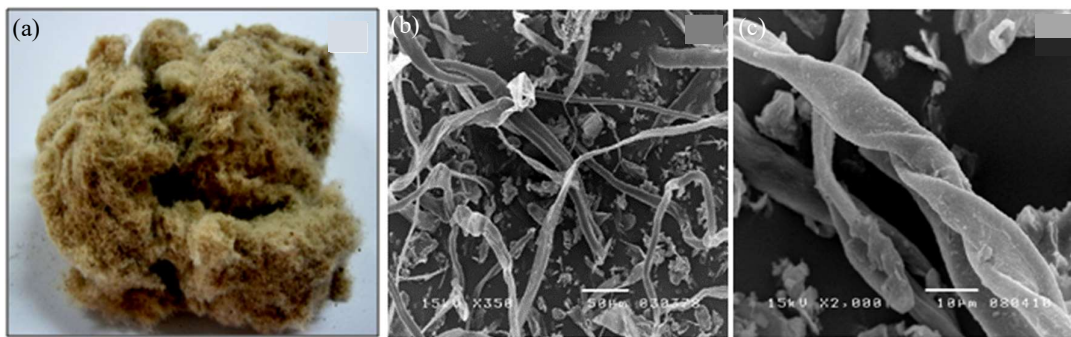
The effect of HEMA on the viscosity of the ALG slurries is shown in Figure 4. The result showed that the addition of the HEMA into the ALG solution increased the viscosity.

In addition, the viscosity of the ALG/HEMA/CD

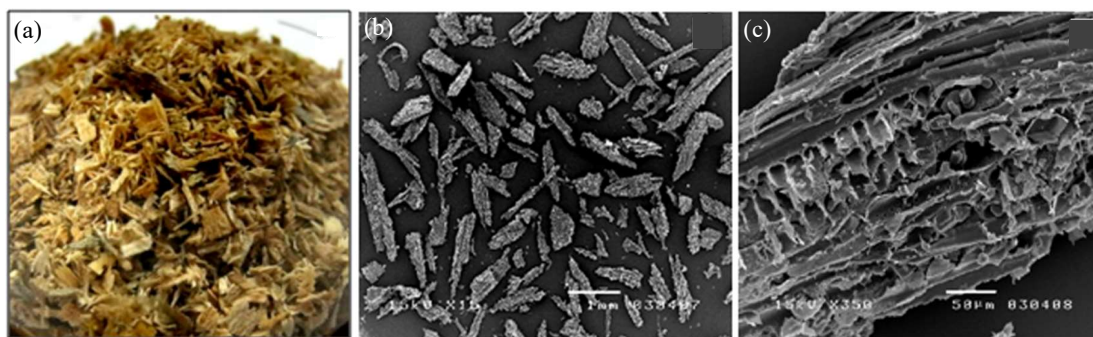
slurries (at CD:SD = 1:0) was higher than the ALG/HEMA/SD slurries (at CD:SD = 0:1). This indicated that the small particle size and high surface area of the CD caused the high content of H-bond between the CD and the HEMA. This induced the high viscosity of the ALG/HEMA/CD unlike the SD with large particle size and low surface area.



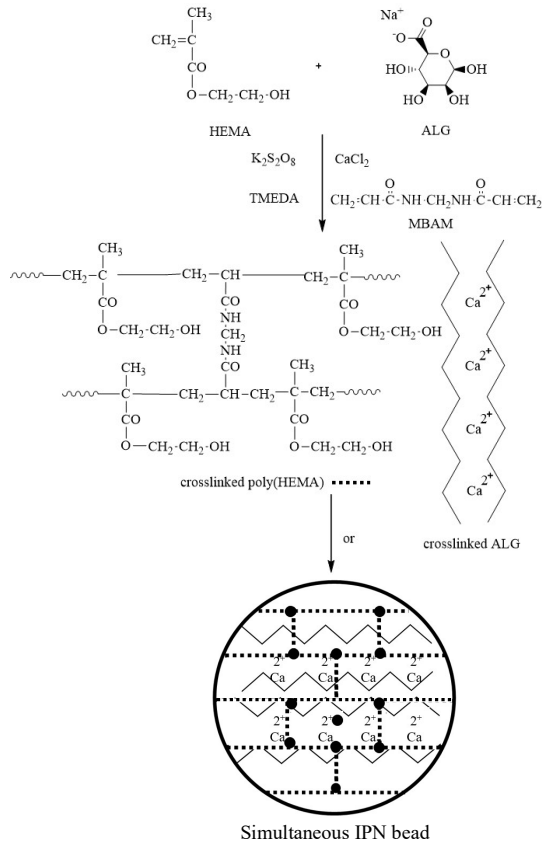
**Figure 4.** Effect of the CD, SD (2% by wt of ALG solution) and HEMA on the viscosity of alginate solution.



**Figure 5.** Photograph and SEM micrographs of the CD: (a) photograph of the CD, (b) and (c) SEM micrographs of the CD.



**Figure 6.** Photograph and SEM micrographs of the SD: (a) photograph of the SD, (b) and (c) SEM micrographs of the SD.



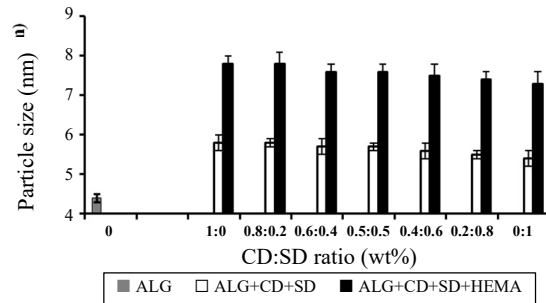
**Scheme 1.** Formation of the simultaneous IPN bead.

### 3.2.3 Effect of fillers and HEMA on beads formation

Figure 7 shows the mean particle size of the ALG beads mixed with the HEMA, CD, and SD at various ratios (2 wt% of the ALG solution). Mixing of the HEMA, CD and SD with the ALG increased the particle size of the beads. This resulted from the polymerization reaction of the poly(HEMA) in the ALG matrix. Also, the particle size of the ALG beads slightly decreased with the increasing of the SD content. This resulted from the decreasing in the ALG solution viscosity (see Figure 4). For this reason, low viscosity liquid had less mass flow from the orifice and produced small particle. The ALG solution mixed with the CD differed from the SD in that the high viscosity of the slurry (see Figure 4). It took more time to flow from the orifice and led to more mass in the beads [15]. Because of this the particle size of the ALG bead mixed with the 100%CD was larger than mixed with the SD.

For the addition of the HEMA in the slurries of the ALG mixed with the CD and SD, the crosslinking of the ALG with  $\text{CaCl}_2$  and the polymerization of the HEMA were occurred simultaneously. This caused the particle size of the beads to increase. Figure 8 shows the FTIR spectra of the ALG bead, CD, SD and ALG composites mixed with the HEMA. In the FTIR spectrum of the ALG bead (see Figure 8(a)), the

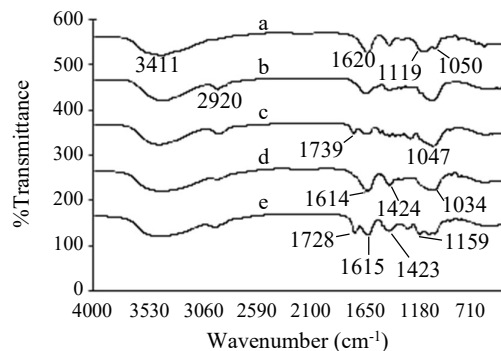
absorption band at  $3411\text{ cm}^{-1}$  was attributed to the absorbance of the  $-\text{OH}$  functional groups, that at  $1620\text{ cm}^{-1}$  was due to  $-\text{COO}$ , and that at  $1114\text{ cm}^{-1}$  was the  $\text{C-O-C}$  in  $\text{C-O-C}$  [16].



**Figure 7.** Particle size of the hydrogel beads and hydrogel composite beads at various the CD and SD ratio.

In the case of the CD (see Figure 8 (b)), the absorption band at  $3411\text{ cm}^{-1}$  was attributed to the absorbance of the  $-\text{OH}$  functional groups, that at  $2920\text{ cm}^{-1}$  was due to the  $-\text{CH}_2$ . The peak at  $1427\text{ cm}^{-1}$  was the result of the  $-\text{OH}$  functional groups, whereas that at  $1060\text{ cm}^{-1}$  was the absorption of the  $\text{C-O}$  of primary alcohol [17].

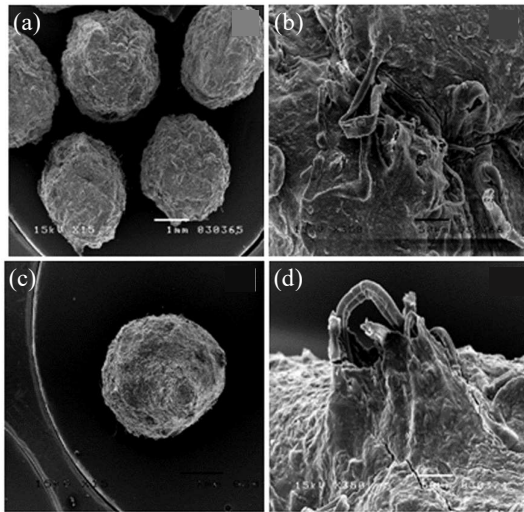
In the case of the SD (see Figure 8 (c)), the FTIR spectrum was similar to the CD, but the SD consisted of high content of lignin (17.23%) and hemicelluloses (16.25%). It appeared the  $-\text{C}=\text{O}$  groups at  $1739\text{ cm}^{-1}$ , and the vibration of the aromatic ring at  $1600\text{--}1400\text{ cm}^{-1}$ , that at  $1600\text{ cm}^{-1}$  was due to the phenolic groups [18].



**Figure 8.** FTIR spectra of beads and fillers: (a) ALG bead, (b) CD, (c) SD, (d) ALG/CD/SD bead (CD:SD = 1:1), (e) ALG/CD/SD/HEMA bead.

In general, the FTIR spectrum of the poly (HEMA) consisted of the absorption band of the  $-\text{OH}$  functional groups at  $3556\text{ cm}^{-1}$ , the blending of the  $-\text{OH}$  at  $1454\text{ cm}^{-1}$ , the stretching of the  $-\text{C}=\text{O}$  at  $1740\text{ cm}^{-1}$  [19]. Also, in the spectrum of the ALG composite beads mixed with the HEMA, it could be seen the spectra of the ALG, CD, SD and poly(HEMA).

The SEM micrographs of the ALG composite beads with and without HEMA are shown in Figure 9. Mixing of the HEMA into the ALG solution induced the large particle size of the beads with a rough surface.



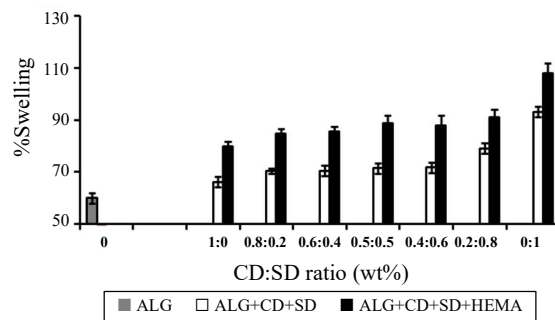
**Figure 9.** SEM micrographs of the hydrogel composite beads with and without HEMA: (a) and (b) without HEMA; (c) and (d) with HEMA (CD: SD ratio 1:1).

### 3.3 Swelling property

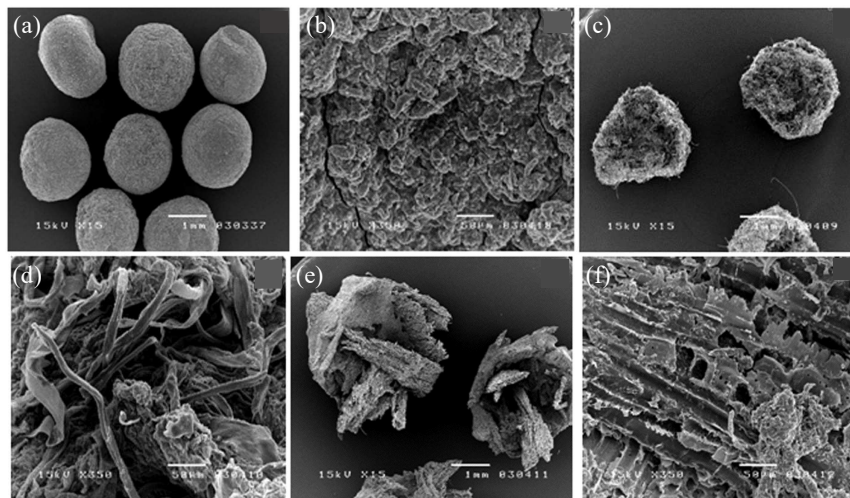
Figure 10 shows the swelling property of the beads. The alginate bead had %swelling about 60.06. The swelling of the alginate bead was due to the water diffusion between the polymer chain and caused the bead to swell. From Figure 10, it shows that the addition of the HEMA, and fillers in the ALG solution increased the %swelling. This caused by the hydrophilicity of these materials. Also, the results showed that the %swelling increased with an increase in the SD ratio.

The cross-section of the ALG bead with and without the fillers is shown in Figure 11. From Figures 11 (a-b), it can be seen that the ALG bead showed dense packing. The addition of the CD (see Figures 11 (c-d)) and SD (see Figures 11 (e-f)) in the ALG beads increased void space in the matrix. This resulted in an increase in the %swelling of the ALG composite beads. The large particle size of the SD caused the high volume of water diffused into the ALG/SD beads more than the ALG/CD beads. However, from this figure, the collapsing of the ALD/CD beads caused the particle size was smaller than the ALG/SD beads.

The effect of the HEMA on the swelling property of the ALG beads was examined. The results showed that the mixing of the HEMA into the ALG solution increased the %swelling (see Figure 11). In general, the poly(HEMA) is the presence of the hydrophilic functional groups (-OH groups). These functional groups formed H-bonding with water molecules and caused an increase in the % swelling of the beads [20,21]. Hence, the swelling of the beads resulted from the CD, SD and HEMA. From this the results, the composite beads will be applied for the water-absorbing material in agriculture.



**Figure 10.** Swelling property of the hydrogel beads and composite beads.



**Figure 11.** SEM micrographs of the hydrogel bead and composite beads: (a) ALG beads, (b) cross section of ALG bead, (c) ALG/CD beads, (d) cross section of ALG/CD bead, (e) ALG/SD beads, and (f) cross section of ALG/SD bead.

#### 4. Conclusions

The alginate beads and alginate composite beads mixed with the cotton dust and sawdust were prepared. The viscosity of the alginate solution and the formation of the beads depended on the concentration of the alginate solution, particle size and content of the fillers e.g. cotton dust and sawdust, also, the mixing of the 2-hydroxyethyl methacrylate. The solution viscosity induced the occurrence of the elongational flow. The shape of the beads changed from the spherical to long-tail beads as the increase in the alginate concentration. The addition of the cotton dust, sawdust, and 2-hydroxyethyl methacrylate had an effect on the bead formation. In addition, the cotton dust, sawdust, and 2-hydroxyethyl methacrylate promoted the swelling property of the beads.

#### 5. Acknowledgements

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