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Title	COMPOSITE BEADS OF CHITOSAN/BENTONITE AS A MATRIX FOR CONTROLLED PHOSPHATE RELEASE FERTILIZER (Piluharto, V.Suendo, I.Maulida, Asnawati)
Abstract	Good enough
Introduction	Need more sentence to explain what kind of fertilizer used, and the reasons authors use it
Methodology	Good
Results	Need more evaluation on site of bentonite due to this site will effect on size of Chitosan
Discussion	For each investigation author should explain first the significant important on using the investigation why water adsorption to the done was not clear
How well is the paper integrated with current research :	This research is old
Bibliography/References:	Need more care on structure of references
Adequacy of literature review	Need improvement
Figures:	Good enough
Tables:	Good enough
Overall evaluation on the paper:	Published in journal of International and the English should be improved

Composite beads of chitosan/bentonite as a matrix for controlled phosphate release fertilizer

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ABSTRACT. *Composite beads of chitosan-based have prepared as matrix to study phosphate-release properties. The composite beads contain of chitosan and bentonite. In here, Chitosan with various molecular weight (MW) (low, medium and high) were used as variable to study their physical properties. Meanwhile, swelling degree and phosphate release of composite beads were investigated in various of pH. As the result, composite beads with high MW of chitosan show that qualitatively have higher mechanical stability than medium and low MW chitosan. Swelling degree of composite beads increase with increase MW of chitosan. Compared to native chitosan, composite beads showing higher swelling degree. Phosphate-release measurement shows that controlled-release behavior was affected by pH of solution media with pH in order pH 3 > pH 7 > pH 10.*

keywords: composite, bead, chitosan, phosphate release, swelling degree

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1. Introduction

Controlled- release system are the release system that used organic or inorganic materials as barrier matrix to control rate and pattern of nutrients release. The system was widely used in medical, pharmacy and agriculture fields. In the agriculture field was called known as controlled-release fertilizer (CRF). Compared to conventional fertilizer, CRF more efficient and reduce soil pollution that caused consumed fertilizer excess. [1,2].

CRF consist of two main component, the matrix solid and active material. The matrix solid plays important role as diffuse barrier that controlled release of nutrients. Rate release depend on structure of solid, type of nutrients, and environmental factaors (temperature, pH, and moisture) [3] . The matrix solid usually was polymer or blend polymer or composite materials. However, polymers that used as matrix was synthetic polymers that have negative impact in environment due to not

biodegradable. Therefore, developing polymers for CRF should be emphasized on natural polymer because of more degradable and so environmentally benign [3,4].

Chitosan is one of the natural polymers that obtained from deacetylation of chitin. Chitosan have both of amino and hydroxyl groups that have cationic properties when protonated. Chitosan have cationic characteristic so it can interact to negatively charge molecule or polymers. However, since high absorbed to water, chitosan have poor mechanic stability [5,6,7]. Therefore, the needed blend or composite with the other materials to overcome that drawbacks. In this work, we prepared chitosan/bentonite composite as a matrix material to regulate phosphate release. The composite with different weight content of chitosan/bentonite was used as variable to study water uptake, morphology and phosphate release performance in the water.

1. Experimental

1.1. Materials. Chitosan with various molecular weight: low (50-190 kD), medium (190-310 kD), high (310-375 kD) molecular-weight chitosan powder was purchased from Sigma-aldrich, and acetic acid glacial, sodium hydroxide, ammonium hydrogen phosphate, bentonite, sulfuric acid, ascorbic acid, ammonium molibdate obtained from Merck, respectively.

1.2. Preparation bead of chitosan/bentonite composite. Composites beads preparation procedure according to previously described by Teofilovic [8]. 50 mL chitosan 2% (w/w) in acetic acid 2% was added bentonite 2% and gently stirred for 24 hours. Bead was obtained by drop chitosan/bentonite mixture into NaOH 2% under stirring. The beads were filtered and washed by distillate water. Finally, beads dried in oven 40 ^{o}C for overnight. The similar procedure was carried out for composite beads with medium and high molecular weight of chitosan.

1.3. Water uptake. An amount dry beads was weigh and then immersed into water distillate for 24 hours. All water on the surface of beads were remove by wipe using tissue paper. Water uptake can be calculated using the following formula [9]:

$$WU = \frac{W_w - W_d}{W_d} \times 100\%$$

Where WU, W_w, W_d represent water uptake, wet sample mass (gram) and dry sample mass (gram) respectively.

1.4. Morphology analysis. Cross section structure of chitosan and chitosan/bentonite composite beads were studied using Scanning electron microscopy (SEM) with magnification 500x at 15 kV.

1.5. Release phosphate in chitosan/bentonite composite beads. Release phosphate procedure according to previously described by Thomaszewska et.al. [10], 0.25 g of composite bead was added into a 50 mL beaker glass 100 mL, then 50 mL of distilled water was added. Beaker glass kept on the room temperature,. The measurements were carried out for 7 days with every day were measured. The concentration of phosphate (P) was determined by spectrophotometry method.

2. Result and Discussion

2.1. Composite chitosan/bentonite bead preparation.

All chitosan/bentonite composite beads have successfully prepared with various molecular weight (MW) of chitosan. The appearance of composite beads was different physically. Figure 1 show different shape of beads with different MW of chitosan. It appears that the composite bead with low molecular weight (50-190 kD) have fragile bead, with medium molecular weight have form flat and tail. Meanwhile, composite bead with high MW of chitosan have spherical shape and stable mechanically. Compared to the composite bead with low and medium MW of chitosan, the composite bead with high MW of chitosan is better. These phenomena can be explained that chitosan with high MW have more reactive groups than medium and low MW, it lead to a more intermolecules interaction in the composites. Therefore, the chitosan beads with high MW mechanically more stable.

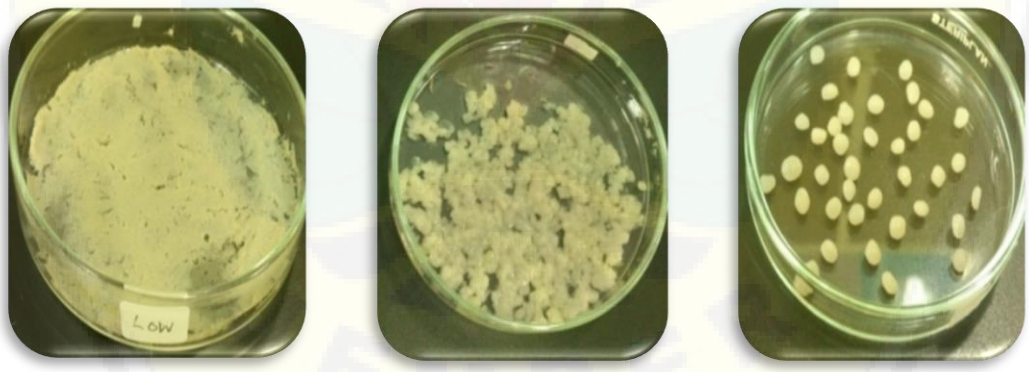

Figure 1. The chitosan/bentonite composite beads with various MW of chitosan: (a) low MW; (b) medium MW; (c) high MW

Water uptake of chitosan and composite of chitosanbentonite

In here, water absorbency that measured is chitosan bead and chitosan/bentonite composite beads. Figure 2 show that water absorbency of both of them increase with increase MW of chitosan. Compared to chitosan bead, chitosan/bentonite composite beads have a lower water absorbency. Introducing bentonite in the chitosan matrix reduces the water uptake due to the moleculare interaction between chitosan and bentonite. The amino and hydroxyl groups of chitosan interact with

polar groups of bentonite. These interaction reduce the interaction chitosan with water, thus water absorbency decrease.

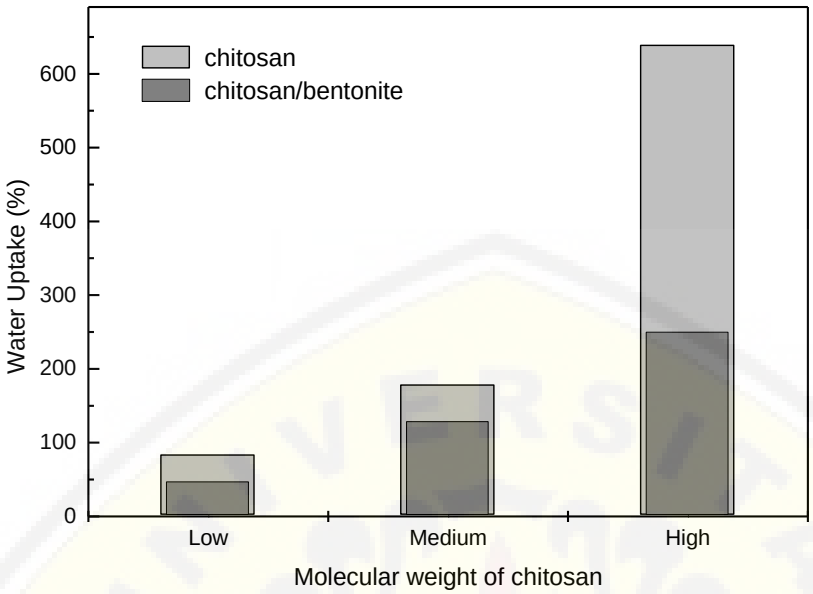

Figure 2. Water uptake of chitosan bead and chitosan/bentonite composite beads

Release phosphate in chitosan/bentonite composite beads

Phosphate (P) release in chitosan/bentonite composite beads were carried in various pH media solution (pH 3, 7 and 10). Figure 3 shows phosphate release as function of time (day). It appears that, pattern of release on each pH is similar where phosphate release increase with time. It also appears that phosphate release depend on pH, where decrease with pH in order pH 3 > pH 7 > pH 10. This fact can be explained that in the acidic media (pH 3), chitosan can be protonated and that induced swelling, thus increasing the phosphate release as the consequence.

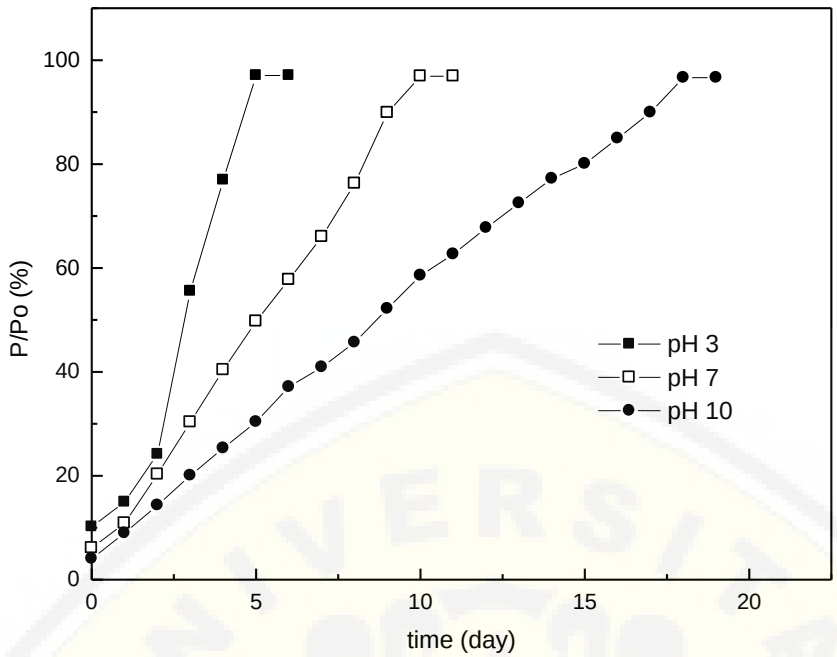

Figure 3. Release phosphate as function of time (day)

Cross section morphology of chitosan/bentonite composite beads.

Figure 4 show cross section morphology of beads. According to Figure, cross section structure of chitosan bead is more denser than chitosan/bentonite composite beads. Meanwhile, chitosan/bentonite composite beads have higher pore size and porosity than chitosan beads. On composite beads, agglomeration of bentonite particles, were observed. Aglomeration was formed due to intermolecular attraction between bentonite molecules.

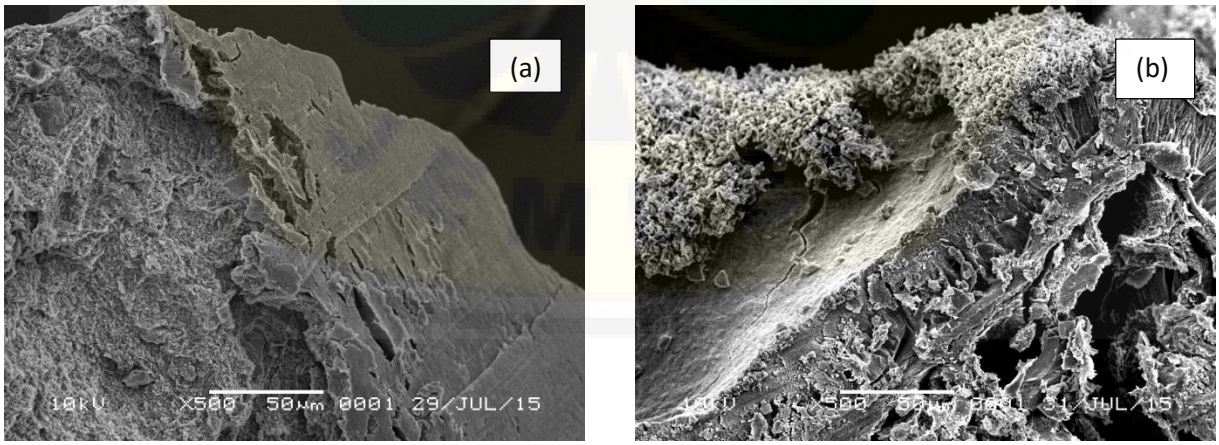

Figure 4. Cross section of beads: (a) chitosan; (b) chitosan/bentonite composite. The white bars represent 50 µm.

3. Conclusion

The chitosan/bentonite composite beads with various the molecular weight of chitosan have been prepared. Compared to low and medium molecular weight of chitosan, the composite with high molecular weight of chitosan have spherical shape beads that show a better mechanical stability. Water absorbency of composite beads increase with molecular weight of chitosan, meanwhile the rate phosphate release increases with the decrease of pH of the media solutions.

Conflict of interests

The authors declare that there is no conflict of interests regarding the publication of this paper.

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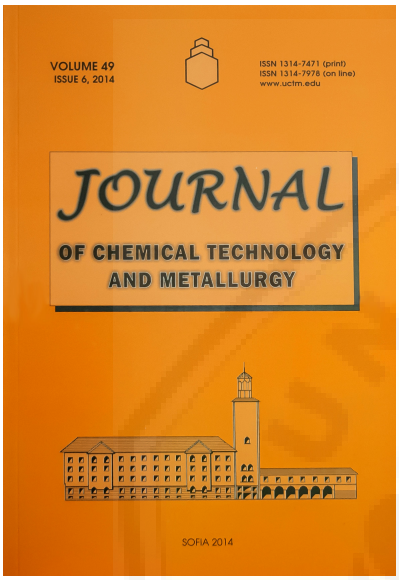

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