

Release Behavior and Degradation Research of Controlled-Released Urea Encapsulated in Starch-Alginate Hydrogel

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Abstract

This paper presents the synthesis of urea encapsulated in starch- and alginate- based controlled released fertilizer, using calcium chloride (CaCl₂) as crosslinking agent. A series of micro-spheres-like beads of different composition were prepared by varying the amounts of starch, sodium alginate, urea and CaCl₂, in accordance to robust parameter design (RPD) at four factor designs - L₉ arrays with a total of nine formulations. It was found that the urea release rate was highly correlated with the swelling behavior of the samples, at a R² of 0.737. Sodium alginate content played the most significant role in creating the size and shapes of the hydrogel; low alginate content create irregular shapes of the beads formed; high alginate content create viscos solution that could not be extruded via syringe. The urea release rate could be controlled by adjusting the starch and CaCl₂ concentration. Increased in starch and alginate content, create a greater intermolecular interaction between sodium alginate and starch that lead to a significant reduction in the biodegradation process.

Keywords: *Controlled-released, N fertilizer, Starch-alginate beads, Urea release rate.*

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I. INTRODUCTION

Fertilizers, herbicides and pesticides are the main players in the agrochemical industry. The growth of plants and their quality are mainly a function of the quantity of fertilizer and water. However, depending on the effectiveness on the soil management, the use of the conventional fertilizer often leads to uncontrolled over-dosage that causes crop damage and other environmental pollution; in returned, results in financial losses for the agriculture businesses. One effective solution to reduce these damaging effects is the use of controlled-released fertilizer (CRF). The CRF were designed to release their content of nutrients gradually and if possible to coincide with the nutrient requirements of a plant. In general, these fertilizers were prepared by coating the active soluble component with a membrane layer that serves as a diffusion barrier, in which reduces their dissolution rate.

Many researches are focusing on finding a suitable

encapsulation membrane material that can be safely used in agro-industries.

Starch is a natural biodegradable, inexpensive polymer that is available in large amounts from several renewable plant sources. Starch-based product has gained great attention in exploring the non-food usage such as encapsulating matrix in the controlled release for agrochemicals due to its biodegradability in soil and ease of availability. Starch-encapsulated agrochemicals for controlled release can be created by the dispersal of the agrochemicals into an aqueous paste of gelatinized starch and the performance of a procedure of retrogradation or crosslinking. A large amount of research has been done on methods for encapsulating various agrochemicals within natural or modified starch matrix.

Over the year, hydrogel-based controlled-release devices have caught the attention of the researchers due to their interesting properties that ease of

production. They consist of an agrichemical in a polymer network in the form of a microcapsule or granule. Hydrogels are 3-dimensional polymeric networks that swell quickly by imbibing a large amount of water or de-swell in response to changes in their external environment [1]. Hydrogels exhibit a thermodynamic compatibility with water, which allows them to swell in aqueous media and make them potential candidates for agrichemicals release devices. Studies has shown that superabsorbent hydrogels help to decrease irrigation water consumption, enhance fertilizer retention in the soil, decline the death rate of plants, and increase plant growth rate.

There are several ways to synthesize CRF, including encapsulating active ingredient in to the superabsorbent materials. This is a simple yet economical method to produce CRF. Due to the research findings which demonstrated the good properties of starch, sodium alginate and calcium chloride (CaCl_2), it is worth the investigation. Sodium alginate forms insoluble metal-alginate complex with heavy metal ions and starch-alginate- Ca^{2+} beads were prepared by using CaCl_2 as cross linker. The increase in starch contents, was reported to cause irregular trend in the bead physical size, however, the cross linker concentration was observed to have a much significant effect of the size compare to starch content. Increasing the cross linker concentration was reported to reduce the size of the starch-alginate beads. The application of these superabsorbent materials was only tested on several active ingredients mainly as pesticide and fungicide. However, there is currently limited research on the application of these materials for fertilizer.

Nevertheless, the objective of this project was to study the effect of each material variation on urea release rate for a total of 9 formulations. The project scope includes the release behavior study which covers the mechanism of swelling, dissolution as well as the diffusion of urea into distilled water and its biodegradability. Physical characterization was

also carried out to justify the release behavior of each formulation.

II. MATERIAL AND METHODS

A. Material

Native cassava starch manufactured by Thailand-Cap Kapal ABC was obtained from local grocery store. All the other reagents are of analytical grade; Urea (99% purity, granular), Sodium alginate and the crosslinking agent, CaCl_2 anhydrous granular C.P. were obtained from R&M Chemicals, UK. All materials were used as received. Submit your manuscript electronically for review.

B. Synthesis of Starch-Alginate-Urea beads

A defined amount of starch, alginate and urea was mixed in 25 mL distilled water as discussed in the previous paper presented by the authors with slight modification in processes to due to different encapsulated active ingredient. The solution was heated up and stirred constantly at 400 RPM using a hot plate magnetic stirrer (Thermos Scientific MSH-300) for 15 – 30 min to achieve homogenous state. Cross-linking solution was prepared by dissolving CaCl_2 in 100 mL of distilled water. This was followed by the dripping of starch-alginate-urea mixture using a 2-mm diameter syringe into CaCl_2 solution for crosslinking action to occur. The formed starch-alginate-urea (St-Alg-U) beads were rinsed with distilled water, filtered using 200-mesh screen. The beads were dried in the natural convection oven (Mettler UN75) at 70°C overnight. Beads of different starch, alginate contents were prepared in different concentration of crosslinker. The experiment was arranged in robust parameter design (RPD) at four factor designs - L9 arrays with a total of nine formulations (that is SA1 to SA9) as shown in Table I, and were used to study the effect of each factor on the urea release dynamics and the biodegradability.

Table I Formulation arranged in robust parameter design (RPD) at four factor designs - L9 arrays and its yield and average beads size

#	St (%w/v)	Alg (%w/v)	Urea (%w/v)	CaCl ₂ (M)	Yield (%)	Avg Beads Diameter (mm)
SA1	2	2	30	0.2	56.997	0.893
SA2	2	5	50	0.6	67.42	1.71
SA3	2	8	70	1	67.715	2.097
SA4	5	2	70	0.6	57.839	1.027
SA5	5	5	30	1	68.898	1.903
SA6	5	8	50	0.2	76.323	1.927
SA7	8	2	50	1	59.324	1.507
SA8	8	5	70	0.2	77.193	1.78
SA9	8	8	30	0.6	78.617	1.807

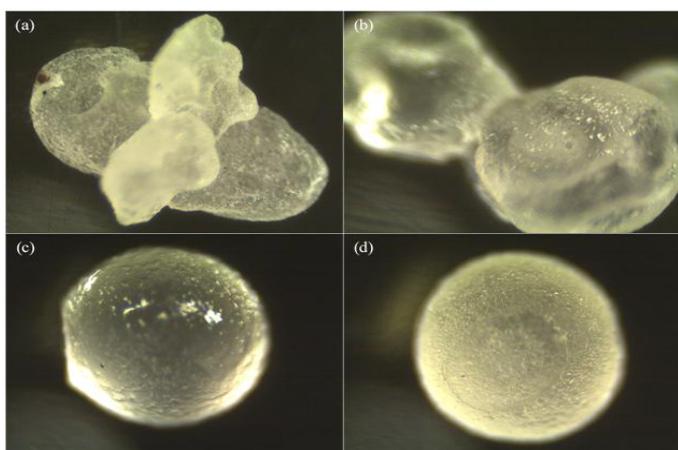


Fig.1 Microscopic samples for (a) low alginate SA1 (b) SA4; (c) medium level alginate content SA5; (d) high level al

C. Encapsulation efficiency and beads size measurement

The encapsulation efficiency of the St-Alg-U measured using a Mitutoyo Digital Caliper and the average sizes of the beads are presented in the last column in Table 1.

D. Swelling ratio and solubility analysis

The swelling study consists of setting the initial weight (W_o) of the beads at 5 g which was immersed into 100 mL of distilled water over a fixed interval of time (1 hour, 2 hours, 4 hours, 6 hours, 1 day, 2 days, 7 days, 14 days, 21 days and 30 days) under static conditions and room temperature of 25°C. The swollen weights of the hydrogels were measured at W_w and the swelling percentage was calculated beads is calculated as:

$$\text{yield}(\%) = \frac{(\text{amountofproductformed})}{(\text{amountofreactanttaken})} \times 100\%$$

Six dried beads of each formulation were using Eq. (2) as shown below:

$$\text{Swelling \%} = \frac{W_w - W_o}{W_o} \times 100\% \quad \square \quad \square \quad \square$$

For the dissolution analysis in water, the swollen beads were dried using oven under the same temperature of 80°C for 24 hours where the new dried weight was recorded as W_D . Thus, the solubility of samples in water can be determined using Eq. (3):

$$\text{Dissolution \%} = \frac{W_o - W_D}{W_o} \times 100\%$$

E. Urea release rate in water

The amount of urea released into distilled water was determined through the calibration curve of the known concentration of pure urea. This was carried out using Thermo Scientific™ GENESYS 10S Ultraviolet/visible (UV/VIS) light absorption Spectrophotometer at 210 nm wavelength for urea detection. At the fixed interval of time mentioned in section 2.4.1, 3.5 mL of solution was sampled from the swelling ratio study to conduct the release rate analysis and a 3.5 mL of distilled water was added to the original solution in order to maintain a liquid constant level.

F. Biodegradability study

Biodegradation of the beads was studied by obtaining the loss in weight during the soil burial method as proposed by Thakore et al. One kg of gardening soil was mixed with 100 mL activated sewage sludge, which obtained from the wastewater treatment plant in the Subang Jaya municipal district, followed by a 24-hours sun-dried process. The soil samples went through the sieving process to remove big pieces of solid such as pebbles, metals, non-soil materials, etc. to produce uniformed compost. Numbers of plastic gardening pot of about 200 mL capacity were filled with compost. Weighed amount (1 g) of beads of each formulation were wrapped in a synthetic net was buried in the compost at depth of about 5 cm. The compost was

kept moist by sprinkling water at regular time intervals to maintain humidity and stored at room temperature. The excess water was drained through the bottom of the pots. The degradation of the samples was studied at regular time intervals by removing the samples carefully from soil and was clean gently with distilled water. The samples were dried at 60°C in the oven for 24 hours. Weight loss of the beads was recorded as measure of degradation. All experimental work was carried out in replicates of 3.

III. RESULTS AND DISCUSSION

A. Physical appearances and encapsulation yield

In this study, the hydrogel beads were prepared by varying starch and alginate contents, crosslinked in different concentration of CaCl₂ to study the effect on various formulation characteristics of the hydrogel-controlled-released fertilizer.

The percentage yield of samples were not significantly affected by the increases in starch content or CaCl₂ concentration, but was found increases with the increment in alginate amount at a P-value of 0.04, which agreed with the result produced by B. Singh et al. [23]. It was observed that the solution's viscosity increases alongside with the increment of the alginate amount. Although the pair T-test shows that there are no significant differences between the samples diameter sizes after cross-linked; physically, under microscopy images, irregularities in shape as shown in Fig. 1 proved that there sodium alginate content varies the shape of the hydrogel. At a low alginate presents in the solution 2 w/v%, the beads formed at irregular shapes and shows a tendency of binding to each other as shown in Fig. 1(a) and (b); whereas, with the increment of alginate in the solution from 2 to 5 w/v% and above, the beads formed shows mainly of spherical shapes (Fig. 1(c) and Fig. 1(d)). Nonetheless, further increment of sodium alginate in the solution creates a thick viscos liquid that could not be extruded via the syringe. This result agreed with the previous study done by the authors on the encapsulation of

phosphorus and other researchers, that the content of starch and sodium alginate must be increased at the same time to allow a more spherical shape whereas the increment of Sodium alginate itself create the highly viscous mixture that alter the shape of the beads. This occurred due to the availability of free cations from the starch to form hydrogen bonds with the sodium alginate.

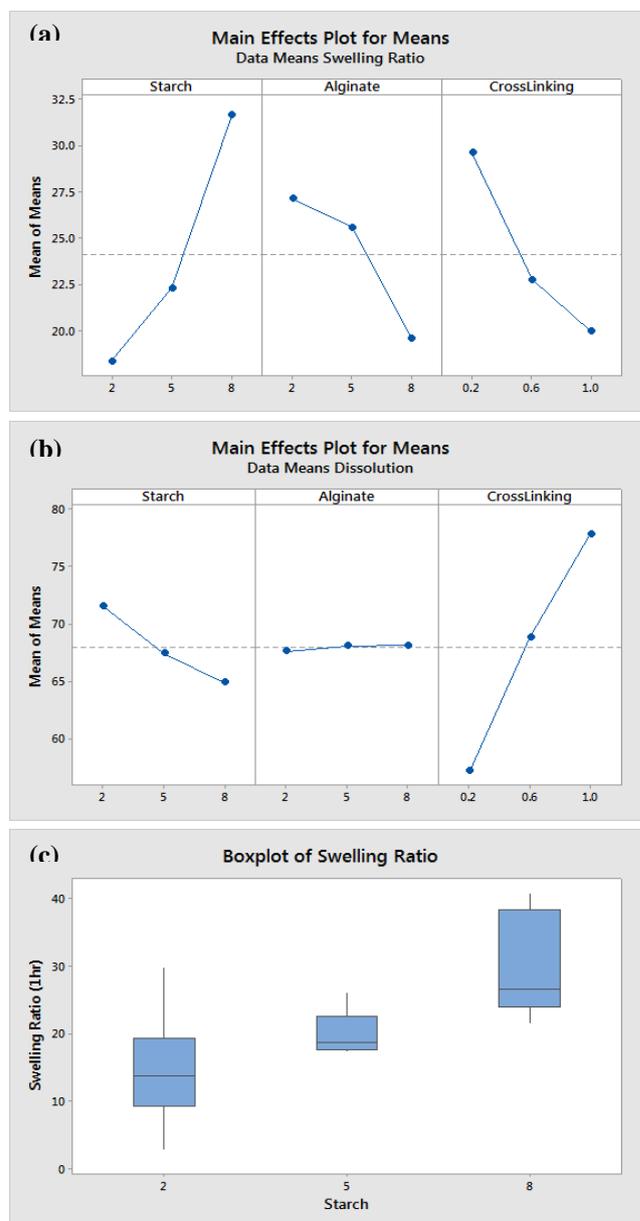
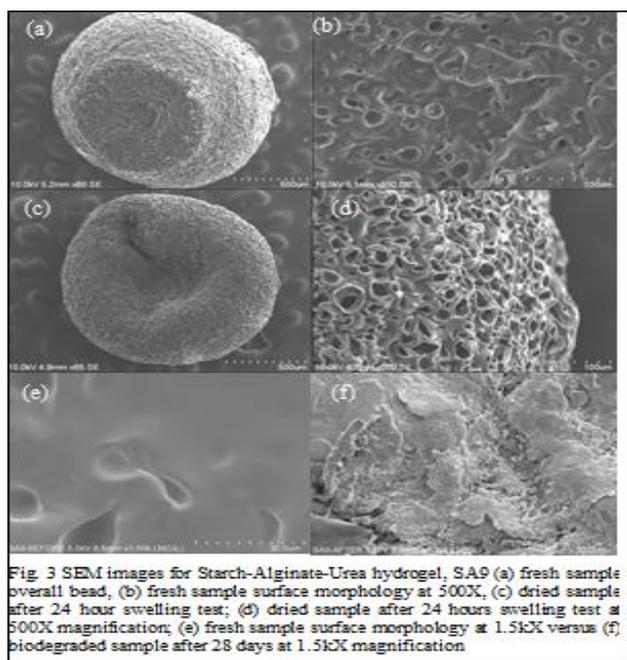
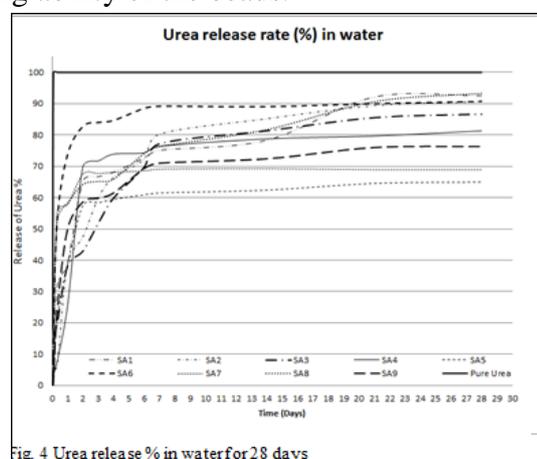


Fig. 2 Main effect plot for means of means on (a) beads diameter (b) swelling ratio (c) samples' solubility in water and (d) Boxplot for swelling ratio of the samples



Besides affecting the physical shape of the hydrogel beads, alginate content in sample also played a significant role in the swelling effect of the samples. In general, the increment of starch amount in the starch-alginate beads increases the swelling ratio, as shown in the main effect plot and the boxplot for different starch content at the P-value less than 0.004. This may cause by the hydrophilic nature of the starch present in the beads. As the amount of starch increases, the number of interaction of -OH groups present in the starch with water increases; in which, results in the increment of swelling ability of the beads.



The increment of the concentration in crosslinking solution and alginate content results in a drop of swelling of the samples (Fig. 2(b)), possibly due to its effect in creating a denser molecular chains in hydrogel matrix; in other word, created a longer path

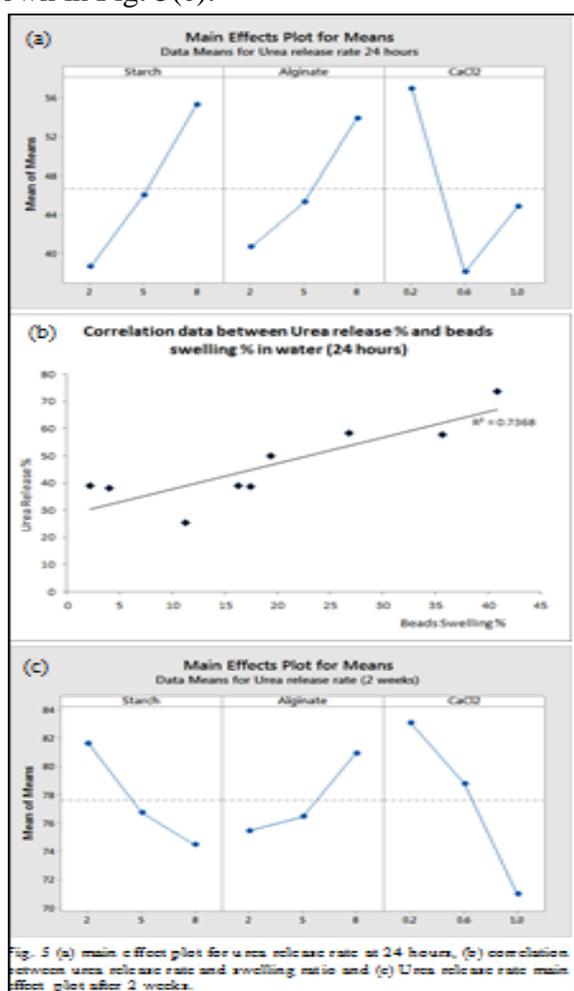
of diffusion of molecules into and out of the polymeric matrix. The surface morphologies of the samples were observed using FESEM as shown in Fig. 3. It can be seen from Fig. 3(a), (b) and (e) that the fresh hydrogel surface has a highly porous network with poor mechanical integrity. After a 24-hours swelling and dissolution testing, the porosity of the beads surface increases dramatically, as presented in Fig. 3(d), compare to the fresh sample's surface (Fig. 3(b)). This may be due to the dissolution of the materials in water, and at the same time, losing urea concentration as it diffused through the metal-polymer matrix into the water.

Opposing the swelling trends of controlled-release formulation, an increment of starch content in the matrix decreases the solubility of the hydrogel, as shown in Fig. 2(b). This phenomena was reported by A. Roy et al. whereby there are more intermolecular interactions between the starch and alginate molecules. With this, the polymer matrix becomes more compact, thus hindering the water penetration. There is a high tendency for the hydroxyl group from starch and the carbonyl group from sodium alginate to form strong hydrogen bonds. On contrary, the dissolution improves with increasing CaCl_2 content due to its high solubility property. The Ca^{2+} ion readily forms bond with the O-H group from water molecules, thus increasing the interaction for dissolution to occur.

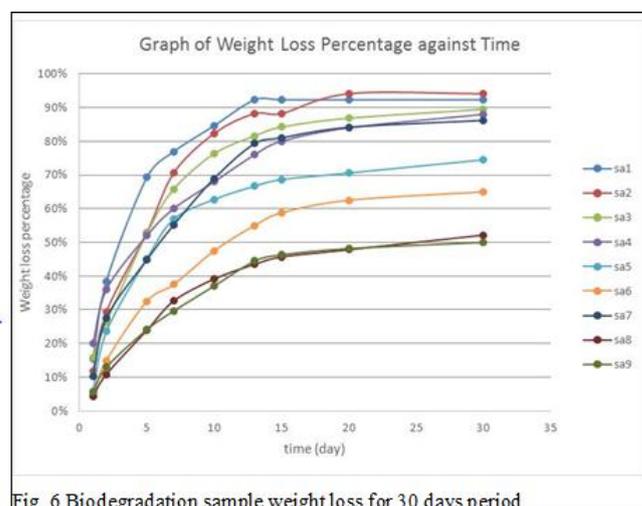
B. Urea Release behavior in Water

It is known that pure urea dissolves in water completely within seconds. The common trend of the urea release rate for the St-Alg-U samples is shown in Fig. 4; an exponential increment in releasebehaviour of theureainto waterwas observed for the first 24 hours, followed by a slower release rate in the following weeks until it reaches a constant value. The release of the active ingredient for the first 24 hours was found closely correlate to the swelling characteristics of the samples as presented in Fig. 5(b). In theory, the release of the water-soluble fertilizers, entrapped in the starch-polymer matrix, occur only when water penetrates the network to swell the polymer and dissolve the

chemicals, followed by diffusion along the aqueous pathways to the surface of the device. Fig. 5(a) shows that the release rate of urea increases with the increment of starch concentration, where starch was reported as the main factor of affecting the swelling degree of the hydrogel. Similar result was reported by A. Roy et al. on the controlled-release of pesticide using St-Algbiocomposite whereby the same phenomena was caused by the increase in the hydrophilic nature of starch. At the same time, the non-ionic content of the hydrogel is reduced, thus the crosslinking of CaCl₂ became underdeveloped. Therefore, the penetration rate of water into the hydrogel accelerated, causing a higher diffusion rate of urea out of the matrix. On a contrary, the effect of starch concentration reverses after a long time swelling in water. Samples with larger amount of starch may have dissolved in water that potentially causes a highly saturated solution, in which, affects the dynamic of release to become slower over time as shown in Fig. 5(c).



In general, the urea release rate decreases significantly with the increment of the crosslinking agent, as this has been observed by other researchers previously. The observation shows that the release of urea was well controlled in a sustained manner with increasing CaCl₂ content. Increase in crosslinking concentration has increased the crosslinking density, that directly increase the compactness of the network; in other words, reduced the penetration of water molecules into the hydrogel [23]; hence, reduce the urea release rate from the matrix. The path of diffusion in the matrix is increased as more COO⁻ from sodium alginate is able to crosslink with Ca²⁺ available in CaCl₂. B. Singh et al. also described the same effect on testing with controlled-release thiram fungicide formulation. It is suggested that the main controlling parameter is the amount of crosslinking agent used in the synthesis.



C. Biodegradability

Waste water treatment sludge contains various types of microorganism which activates the process of biodegradation. It is observed that generally, all St-Alg-U beads show a gradient weight loss in 30 days. A close-up surface morphology of the fresh sample and biodegraded sample after 28 days is as shown in SEM in Fig. 3 (e) and (f). In general, the biodegradation mostly occurs in the unstructured areas due to the greater mobility of the chains and hence, higher accessibility to the microorganism. Samples with low starch and alginates content was

found to degrade faster than other samples, where its losses nearly 90% of the original weight within first two weeks. As discussed in the earlier, low alginate contents produces irregular shapes of the samples, seems to produce a faster and higher degree of degradation compares to the rest of the samples. It is observed that higher starch and higher alginate content reduces the biodegradability of the samples, possible due to the present of anionic polymer that increases the number of polar chains, causing a greater intermolecular interaction between sodium alginate and starch macromolecular [22]. In results, starch and alginate bond tightly together at the same time retard the penetration of microorganisms into the surface. Both the components form strong hydrogen bond between carbonyl group of the sodium alginate and hydroxyl group from the starch. Besides, the increment in alginate content on lowering biodegradability may be attributed to the increase in crosslinking between the alginate and Ca^{2+} ion in the beads, in which results into a more compact structure and hence reducing the rate biodegradation [23].

IV. CONCLUSIONS

In this research, the encapsulation of urea fertilizer using starch and sodium alginate has been successfully synthesized and tested for its release behavior and biodegradability. The results obtained from this research proposed that there are differences in the physicochemical and morphological properties of all the 9 formulations for starch-alginate-urea hydrogel beads. A balanced formulation between starch and sodium alginate was important for the overall shape of the bead. The swelling and the dissolution studies further explained the effect of each material variation on the release rate. It can be observed that the swelling characteristics of hydrogels for the first 24 hours are closely associated to the urea release rate, which is the key is determining the chemical structure of the hydrogels. Each of the chemical component exhibit a different role in the hydrogel formation, where starch content affects its swellability,

biodegradability and urea release rate, alginate played an important role in sizing and shapes of the matrix, where varying CaCl_2 could significantly control the release of the urea through the matrix.

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