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# Development of Biodegradable Hydrogel for the Slow Release of Urea

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**Abstract—***The starch-alginate-clay based hydrogels were found to be promising for the slow release of urea and prevention of the loss of fertiliser from the fields immediately after their application. The ease of hydrogels for mechanical metering, keeping the soil moist for long period of time by holding and releasing of irrigation water and slow release of nitrogen were found to be the distinct advantages of the hydrogels. A study was carried out to develop a process technology for the production of hydrogels for the slow release of nitrogen from urea, and determine its various physical and engineering properties. Soluble starch, sodium alginate, kaolin powder and urea were mixed in proportion of 12:1:0 to 12:1:3 and dispersed in 100 mL hot water. Solution was cross linked with 0.1 M $CaCl_2$ solution. The bead size, moisture content, bulk density, pH, yield, entrapment efficiency, equilibrium swelling and static nitrogen release characteristics were determined. There was no significant difference between the characteristics of the hydrogels when orifices of 0.84 and 2.50 mm diameters used. Moisture content, bulk density, pH, yield of hydrogels, and entrapment efficiency of the beads after 24 hours of drying at room temperature during the production of hydrogels varied from 11.11 to 26.58 % (d.b.), 602 to 810 kg/$m^3$, 5.90 to 8.10, 65.50 to 75.64 %, and, 49.24 to 63.45%, respectively. The hydrogels had the percentage equilibrium swelling in the range of 32.50 to 55.00 %. The diameter, moisture content, bulk density, pH of the beads, yield and entrapment efficiency of the hydrogels significantly changed with change in proportion of clay in the hydrogel. As the clay content in the hydrogels increased, the release of nitrogen was slow and it varied from 88.38 to 94.88 % in 336 hours. The release of nitrogen from hydrogels substantially decreased after 240 hours.*

## 1. INTRODUCTION

Adoption of new agricultural technology has a significant role in strengthening the farmers hold on resources and thereby enabling them for better harvest. The new agricultural strategy also improves the way in which the inputs are effectively combined and agricultural operations managed so as to get maximum returns. Specifically, the new agricultural technology encompasses the use of high yielding variety seeds, fertilizers, manures, pesticides, farm machines etc. Of the various components of agricultural technology, use of fertilizer is most important in boosting agricultural production and productivity. Introduction of high yielding and hybrid varieties brought optimism about fertilizer response superiority of modern varieties [11].

It is estimated that the surface application of N, P, K rich fertilizers results in the loss of about 40–70% of nitrogen, 80–90% of phosphorus, and 50–70% of potassium of the applied normal fertilizers to the environment[19]. This contributes greatly to non-point source pollution and eutrophication of surface water, lakes and reservoirs, besides large economic and resource losses. Recently, the use of slow and controlled release fertilizers is a new trend to save fertilizer consumption and to minimize environmental pollution. Slow release fertilizers are made to release their nutrient contents gradually and to coincide with the nutrient requirement of a plant.

Biodegradable hydrogel polymers have attracted a great deal of attention due to their capability to absorb large amount of water and nutrients, and release nutrients in controlled and slow manner to the soil. In all these studies, sodium alginate-glutaraldehyde or starch-alginate or starch-alginate-clay were used to prepare the hydrogels. However, published reports on the development of hydrogels in India for the controlled and slow release of urea are not available.

Keeping these factors in view, an investigation was carried out to develop a suitable process technology for the production of starch-alginate-clay based hydrogels for the slow release of urea and analyse the merits and demerits of the production and utilization of the hydrogels to *develop a suitable process technology for the production of starch-alginate-clay based hydrogels for the slow release of urea, to study the physical and engineering properties of the developed hydrogels and to estimate the material cost of the hydrogels.*

The outcome of the present work will be very useful to researchers, manufacturers and users of the fertilizers for further modification and improvement in the efficiency of application of urea without causing many environmental concerns. Further, project outcome has the potential for its adoption by the high-end horticulturists and large farmers for the cultivation of horticultural crops.

## 2. MATERIALS AND METHODS

### 2.1. Materials Used

The following materials were used for the preparation of starch-alginate-clay based hydrogels for the slow release of urea : (i) Water soluble starch, (ii) Sodium alginate, (iii) Clay (Kaolin powder), (iv) Calcium chloride, (v) Distilled water, (vi) Urea, (vii) Glassware (viii)Kjeldahl apparatus and (ix) Measuring instruments like vernier calliper, digital electrical balance, pH meter etc.

### 2.2. Preliminary Experiments

Preliminary experiments were conducted to identify (i) the appropriate proportion of starch, alginate and clay required for the production of hydrogel, (ii) orifice size and, (iii) effect of washing of hydrogel after their preparation. Trials were conducted based on the proportions of starch, alginate and clay used by various researchers [15].

It was observed that the increasing the starch content above 12 % m/v in 1:1 to 1:3 alginate: clay produced soft hydrogels. On the other hand, when the starch content was decreased hydrogels produced had the low swelling. Increasing the clay content above 1:3 in alginate: clay mix produced hard hydrogels. Therefore, it was decided to produce hydrogels with starch-alginate-clay in the proportion of 12:1:1 to 12:1:3. It was observed that washing of the hydrogels in distilled water after its preparation would result in the loss of fertiliser. Hence, the hydrogels was dried directly without washing. Urea was added to the extent of 15 g in the hydrogels prepared using starch-alginate-clay in the proportion of 12:1:1 to 12:1:3. The total weight of solid materials used for the preparation of hydrogel varied from 28 g (without the use of clay) to 32 g.

### 2.3. Independent and Dependent Parameters for the Experiment

The independent and dependent parameters and their levels considered for the experiment are presented in Table 1

**Table 1: Independent and dependent parameters for the experiment**

| Sl. No. | Parameters | No. of levels | Values |
|---|---|---|---|
| | *Independent Parameters* | | |
| 1. | Proportion of starch-alginate-clay | 4 | 12:1:0, 12:1:1, 12:1:2, 12:1:3 |
| 2. | Orifice size | 2 | 0.84 mm, 2.5 mm |
| 3. | Urea | 1 | 15 g |
| 4. | Calcium-chloride concentration | 1 | 0.1 M |
| | *Dependent Parameter* | | |
| 1. | Bead size | | |
| 2. | Moisture content | | |
| 3. | Bulk density | | |
| 4. | pH | | |
| 5. | Yield | | |
| 6. | Entrapment efficiency | | |
| 7. | Equilibrium swelling | | |
| 8. | Static nitrogen release characteristics | | |

### 2.4 Preparation of Hydrogels

Water soluble starch, sodium alginate, kaolin powder and urea were mixed in the required proportion and dispersed in 100 mL hot water. The mixture was stirred for 15 minutes to form a homogeneous solution. The solution was taken in a 50 mL syringe fitted with a needle of required orifice size. The solution was added drop by drop from a 30 cm height, into 100 mL of 0.1 M calcium chloride ($CaCl_2$) solution under constant stirring. The drops immediately turned into beads of about 4 mm diameter.This are due to the formation of calcium alginate as sodium alginate reacts with calcium chloride. The other product of the reaction is sodium chloride or common table salt.

$$\text{Na(Alginate)} + CaCl_2 \rightarrow NaCl + \text{Ca(Alginate)}$$

The beads were removed from the $CaCl_2$ solution after 30 minutes. They were allowed to dry at room temperature for few days till the beads get completely dry. They were stored in plastic bags for further use.

### 2.5. Determination of Physical and Engineering Properties of Hydrogels

#### 2.5.1. Bead size

The diameter of 15 completely dried round beads (after 24 hours of drying at room temperature) was measured using a vernier calliper (range 0-20 cm, least count 0.001 cm).

#### 2.5.2. Moisture content

The moisture content of the hydrogels after 24 hours of drying at room temperature was measured by oven drying method (105ºC for 24 h).

**2.5.3. Bulk density**

Bulk density of the hydrogels was measured after 15 days of production by pouring the hydrogels into a measuring cylinder up to 50 mL volume while tapping lightly on the hand palm. The beads were dried at 105ºC for 24 hours and their weight was measured. The ratio of weight and volume (50 mL) gives the bulk density of the hydrogels.

**2.5.4. pH**

The pH of hydrogels was measured using a probe type digital pH meter.

**2.5.5. Yield**

Yield of hydrogels is determined by measuring the weight of hydrogels produced and computing the amount of hydrogels that can be produced when 100 g total solids are used. This is expressed in percent.

**2.5.6. Entrapment Efficiency**

It is calculated by measuring the concentration of urea left in the $CaCl_2$ solution during preparation of hydrogel. The nitrogen content of the $CaCl_2$ solution is measured by Micro-Kjeldahl method. The procedure followed for the nitrogen content determination is based on the principle that the nitrogen in any organic material is converted to ammonium sulphate by $H_2SO_4$ during digestion. This salt on steam distillation, liberates ammonia which is collected in boric acid solution and titrated against standard acid.

The nitrogen content of the sample can be calculated by the formula given below:

Ng/kg = ((mL HCL- mL blank) ×Normality × 14.01)/ weight (g)

Since urea contains 46% nitrogen,

urea left per kg of $CaCl_2$ solution = 100/46 × Ng/Kg.

Urea loaded in 1000mL (1kg) water =150 g.

Therefore, entrapment efficiency = (150 – urea left in $CaCl_2$ solution)/ 150 ×100

**2.5.7. Equilibrium Swelling**

Swelling of the beads was carried out in an aqueous medium at room temperature. Known amount of beads were immersed in excess amount of water for 48 h at room temperature or till swelling becomes constant. The beads were then removed and wiped with tissue paper to remove excess water and weighted immediately. The percent equilibrium swelling of the beads was calculated from the mass changes.

Swelling ratio, % = $100 \times (W_s - W_d) / W_d$

Where, $W_s$ = weight of swollen beads

$W_d$ = weight of dry beads

**2.5.8. Static Nitrogen Release Characteristics**

The release characteristics of nitrogen from the hydrogels was carried out by keeping dried and loaded samples of each formulation (9g) in 50 mL of distilled water at room temperature. The amount of nitrogen released was measured using Micro-Kjeldahl method. The release of nitrogen was measured at every 48 h interval up to 336 h (14 days). The cumulative percent release of nitrogen was plotted over a period of time.

**2.5.9. Statistical Analysis**

All the physical and engineering characteristics of the hydrogels viz., moisture content, bulk density, pH, yield, entrapment efficiency and equilibrium swelling were measured in triplicate to reduce the experiment and measurement errors. The effect of orifice size on the physical and engineering characteristics of the starch-alginate-clay based hydrogels was studied using two sample t-test (assuming unequal variances) in the data analysis facility of Microsoft Excel package. Duncan's multiple range test(Statistical Package for the Social Sciences, SPSS for Windows version 10.0) was conducted to study the difference in the proportions of clay in the starch-alginate-clay based hydrogels bring in the physical and engineering characteristics of hydrogels.

**2.6. Estimation of Material Cost of Hydrogels**

The cost of materials for the production of hydrogels for the slow release of urea was calculated excluding the cost of processing, labour, transportation, equipment and other fixed and variable costs. This is because, hydrogels in the present study was produced manually in small quantities using simple equipment like syringe and beaker. However, the cost of materials required calculated in this way gives an idea of the minimum cost of hydrogels for field applications and assist in economic analysis of hydrogels.

## 3. RESULTS AND DISCUSSION

The physical and engineering properties of the hydrogels were determined and cost analysis was performed. The results of the work is done under the following heads: (i) Effect of clay content and orifice size on the physical and engineering properties of the starch-alginate-clay based hydrogels for the slow release of urea. (ii) Nitrogen release characteristics of the starch-alginate-clay based hydrogels for the slow release of urea (iii) Material cost for the production of hydrogels.

**3.1. Effect of Clay Content and Orifice Size on the Physical and Engineering Properties of the Starch-alginate-clay Based Hydrogels for the Slow Release of Urea**

The effect of clay content and orifice size on the production of starch-alginate-clay based hydrogels was evaluated on the basis of bead size, moisture content, bulk density, pH, yield, entrapment efficiency and swelling ratio. The average diameter of 15 completely dried beads, moisture content after 24 h of drying at room temperature, bulk density, and pH are presented in Table 2 and Fig. 1 and figure2.

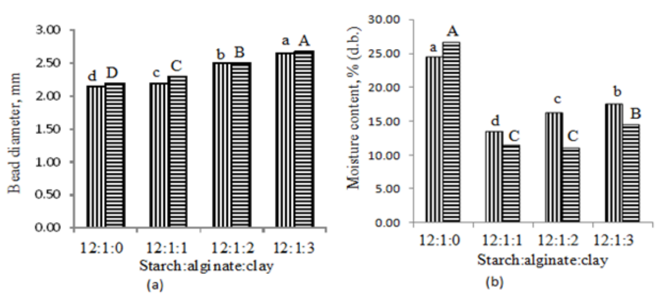
**Table 2: Average diameter, bulk density, moisture content and pH of beads**

| Sl. No. | Proportion of starch-alginate-clay | Beaddiameter, mm | Moisture content, % (d.b.) | Bulk density, kg/m$^3$ | pH |
|---|---|---|---|---|---|
| | | Orifice diameter = 0.84 mm | | | |
| 1. | 12:1:0 | 2.15 d | 24.48 a | 620 d | 7.56 a |
| 2. | 12:1:1 | 2.20 c | 13.60 d | 834 a | 7.26 a |
| 3. | 12:1:2 | 2.50 b | 16.27 c | 738 b | 6.30 b |
| 4. | 12:1:3 | 2.65 a | 17.60 b | 650 c | 6.55 b |
| | | Orifice diameter = 2.50 mm | | | |
| 1. | 12:1:0 | 2.20 D | 26.58 A | 602 D | 8.10 A |
| 2. | 12:1:1 | 2.30 C | 11.52 C | 810 A | 5.90 C |
| 3. | 12:1:2 | 2.50 B | 11.11 C | 706 B | 5.92 C |
| 4. | 12:1:3 | 2.67 A | 14.50 B | 649 C | 7.14 B |

The different alphabets after the values indicate significant difference at 5% level.

### 3.1.1. Bead size

The bead diameter varied from 2.15 to 2.65 mm for starch-alginate-clay based beads when orifice of 0.84 mm diameter was used. The bead diameter varied from 2.20 to 2.67 mm when orifice of 2.5 mm diameter was used for the preparation of hydrogel (Table 1). Further, it was found that there is no significant difference between the diameter of the beads produced when orifices of 0.84 and 2.50 mm diameters were used (t-test). The orifice size of 0.84 mm produced beads of 2.5 to 3.2 times larger diameter than its own size. The data of bead size presented in Table 1 and Fig. 1 indicates that the diameter of the beads significantly increased with increase in proportion of clay in the hydrogel for both orifice sizes. It was reported that bead diameter ranging from 1.07 to 1.34 mm for the starch-alginate-clay (12:1:0 to 12:1:4) hydrogels with addition of thiram (12 mg) when 1.2 mm diameter orifice was used for their preparation[15]. Bead diameter of 1.12 to 2.14 mm was reported for the neem leaf powder-alginate-clay hydrogels (1.5:1:0 to 1.5:1:4) with addition of thiram (30 mg) for the same orifice size[16][17]. Further, increase in size of beads has been reported with increase in clay content in the beads.




**= Orifice diameter = 0.84 mm, = Orifice diameter = 2.50 mm**

**Fig. 1: Variation in (a) bead size and (b) moisture content after 24 hours of drying with proportion of starch : alginate : clay and orifice diameter**

### 3.1.2. Moisture Content

Moisture content of the beads after 24 h of drying at room temperature during the production of hydrogels varied from 13.60 to 24.48 % (d.b.) for starch-alginate-clay beads when orifice of 0.84 mm diameter was used. The moisture content of the beads varied from 11.11 to 26.58 % (d.b.) when orifice of 2.50 mm diameter was used for the preparation of hydrogel (Table 1). Fig. 1(b) and Table 1 indicates that the hydrogels without clay dried very slowly and had significantly higher moisture content after 24 h of drying at room temperature. Further, addition of clay to the hydrogels significantly increased the moisture content of beads after 24 hours of drying at room temperature. Therefore, addition of clay to the hydrogels slowed down the process of drying of beads after their formation.

### 3.1.3. Bulk density

Bulk density of the beads varied from 620 to 834 kg/m$^3$ for starch-alginate-clay beads when orifice of 0.84 mm diameter was used. The bulk density of the beads varied from 602 to 810 kg/m$^3$ when orifice of 2.50 mm diameter was used for the preparation of hydrogel (Table 1). Further, t-test indicated that there is no significant difference between the bulk densities of the hydrogels when orifices of 2 different sizes were used for their preparation. The hydrogels without clay had the lowest bulk density. The bulk density of the beads significantly decreased with increase in proportion of clay in the beads. This is due to the presence of large number of micro pores with increase in proportion of clay in the beads.

### 3.1.4. pH

The pH of the beads varied from 6.30 to 7.56 for starch-alginate-clay beads when orifice of 0.84 mm diameter was used. There was no significant difference between the pH of the beads when 0.84 mm diameter and 2.50 mm diameter orifice was used for the preparation of hydrogel (t-test). The pH of the beads varied from 5.90 to 8.10 when orifice of 2.5 mm diameter was used (Table 1). When clay was not used in the production of hydrogels, pH was significantly higher. It significantly decreased with addition of clay.

### 3.1.5. Yield

Yield of hydrogels is varied from 66.00 to 76.45% when 0.84 mm diameter orifice was used for their preparation. The yield was between 65.50 and 75.64% when 2.50 mm diameter orifice was used (Table 2). There was no significant difference between the yields of hydrogels when 2 different sizes of orifices were used (t-test). As the proportion of clay in the hydrogels increased, yield of hydrogels significantly increased. This might be due to the decrease in leaching of reacting mixture during the synthesis of beads. It was reported the yield of 64.02 to 73.83 % for the starch-alginate-clay (12:1:0 to 12:1:4) hydrogels with addition of thiram (12 mg) [15]. The increase in yields with increase in contents has also been reported.

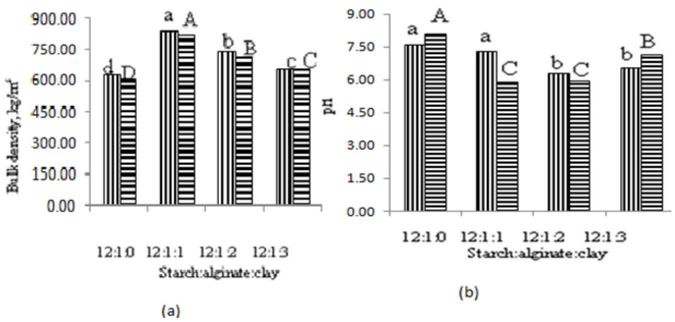



**Fig. 2: Variation in (a) bulk density and (b) pH with proportion of starch : alginate : clay and orifice diameter**

**Table 3: Average yield, entrapment efficiency and swelling ratio of beads**

| Sl. No. | Proportion of starch-alginate-clay | Beads formed, g | Yield, % | Entrapment efficiency, % | Swelling ratio, % |
|---|---|---|---|---|---|
| | | Orifice diameter = 0.84 mm | | | |
| 1 | 12:1:0 | 18.48 | 66.00 d | 51.27d | 50.00 a |
| 2 | 12:1:1 | 20.74 | 69.90 c | 55.33 c | 42.50 b |
| 3 | 12:1:2 | 22.20 | 74.00 b | 61.40 b | 37.50 c |
| 4 | 12:1:3 | 23.70 | 76.45 a | 65.48 a | 35.00 d |
| | | Orifice diameter = 2.50 mm | | | |
| 1 | 12:1:0 | 18.35 | 65.50 d | 49.24d | 55.00 a |
| 2 | 12:1:1 | 19.95 | 68.79 c | 53.33 c | 45.00 b |
| 3 | 12:1:2 | 21.44 | 71.46 b | 59.42 b | 40.00 c |
| 4 | 12:1:3 | 23.45 | 75.64 a | 63.45 a | 32.50 b |

The different alphabets after the values indicate significant difference at 5% level.

### 3.1.6. Entrapment Efficiency

The entrapment efficiency of the starch-alginate-clay based hydrogels developed in the present study varied in the range of 51.27 to 65.48 % when.0.84 mm diameter orifice was used for their preparation. The entrapment efficiency was between 49.24 and 63.45 % when 2.50 mm diameter orifice was used. The t-test indicated there was no significant difference between the entrapment efficiency when two different size of orifices were used for the preparation of hydrogel. Duncan's Multiple range test revealed that entrapment efficiency of the hydrogels increased with increase in proportion of clay during the synthesis of beads. The past researchers have reported the entrapment efficiency of more than 96 % for the fungicide thiram in the starch-alginate-clay and neem powder-alginate-clay based hydrogels [15][16][17]. Entrapment efficiency of urea in the present study is lower than that reported in the earlier studies. This might be due to the loss of urea to $CaCl_2$ solution during the process of synthesis.

### 3.1.7. Equilibrium Swelling

The starch:alginate:clay based hydrogels developed in the present study had the percentage equilibrium swelling in the range of 35 to 50 % when 0.84 mm orifice was used, and 32.50 to 55.00 % when 2.50 mm orifice was used for their preparation (Table 2). The orifice size had no significant difference on the percentage equilibrium swelling of the beads (t-test). However, the percentage equilibrium swelling of the hydrogels significantly decreased with increase in the proportion of clay in the hydrogels. Similar observations have been reported by [9] [16]. The swelling of the hydrogels increased significantly with increase in proportion of starch. As the proportion of starch in the beads increases, the number of interaction of OH-groups present in it with water increases which has increased the swelling of the beads. [15]. reported overall equilibrium swelling in the range of 31.67 to 78.33 % for the neem leaf powder-alginate beads. The increase in swelling of the hydrogels with increase in starch has also been observed by [17]. The maximum percentage of swelling of the hydrogels in the present study is lower than that reported in the earlier studies might be due to the quality of starch used in the present study.

Thus, the starch-alginate-clay based hydrogels produced using 0.84 mm and 2.50 mm diameter orifices had no significant difference in any of the physical and engineering characteristics measured. Therefore, it was decided to study the nitrogen release characteristics of the beads prepared using the 2.50 mm orifice size.

### 3.2. Static Nitrogen Release Characteristics of the Starch-Alginate-Clay based Hydrogels for the Slow Release of Urea

The total nitrogen release from the starch-alginate beads was 96.14 % in 336 hours (14 days). As the clay content in the hydrogels increased, the release of nitrogen was slow and it varied from 88.38 to 94.88 % in 336 hours. Similar observation was made by [17]. The decrease in nitrogen release with increase in clay content might be due to the greater ability of the kaolin (the mineral in clay) for intercalation with urea. The rate of release of nitrogen was faster during the initial steps.

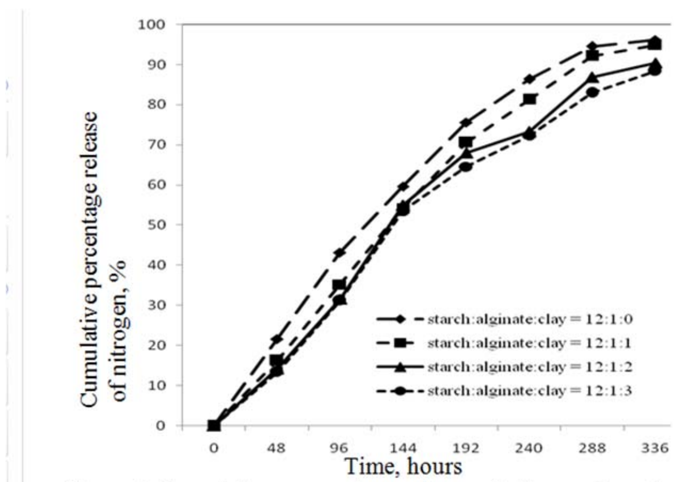



**Fig. 3: Cumulative percentage release of nitrogen from hydrogels.**

This shows that the starch-alginate-clay based hydrogel can be used for the slow release of urea to control the environment and health hazard. An amount of 50 % of nitrogen was released from the loaded starch-alginate-clay beads within 144 hours (Fig. 4.6). The release of nitrogen from hydrogels substantially decreased after 240 hours..

### 3.3 Material Cost of the Production of Hydrogels.

The purchase price of the water soluble corn starch was 367/- per 500 g, sodium alginate was 749/- per 500 g, kaolin powder was 125/- per 500 g, urea was 15/- per kg and $CaCl_2$ solution was 830/- per 500 g. The material cost estimated is shown in Table 3.3. Compared to the cost of urea, the material cost of hydrogel for the small scale manual production varied from 647.3/- to 560.59/- per kg.

**Table 4: Estimation of the material cost of starch-alginate-clay based hydrogels**

| Formulation | Starch (g/100 mL) | Alginate (g/100 mL) | Clay (g/100 mL) | Urea (g/100 mL) | 0.1 M $CaCl_2$ mL | Quantity of beads produced (g) | Cost/kg of hydrogel () |
|---|---|---|---|---|---|---|---|
| 1 | 12 | 1 | 0 | 15 | 100 | 18.35 | 647.31 |
| 2 | 12 | 1 | 1 | 15 | 100 | 19.95 | 633.87 |
| 3 | 12 | 1 | 2 | 15 | 100 | 21.44 | 601.48 |
| 4 | 12 | 1 | 3 | 15 | 100 | 23.45 | 560.59 |

The present investigation reveals that there is need to improve the production process of hydrogels for (i) the enhancement of the nitrogen content, and (ii) the reduction in loss of materials and urea during manufacture. This will bring down the application rate and cost of hydrogels, thereby reducing the application cost per unit area. Keeping in mind the environmental concerns, pollution and health hazards, the developed hydrogels (with improvement in production process) can be recommended to high-end horticulturists and large farmers for the cultivation of horticultural crops.

## 4. CONCLUSIONS

The following conclusions were drawn from the present investigation study: (i) the starch-alginate-clay based hydrogels are promising for the slow release of urea and prevention of the loss of fertiliser from the application sites immediately after their application, (ii) the ease of hydrogels for mechanical metering, keeping the soil moist for long period of time by holding and releasing of irrigation water, and slow release of nitrogen are the distinct advantages of the hydrogels, (iii) the application of hydrogels keeps the soil in near neutral condition, (iv) the high application rate, high cost of their conveyance from the production sites to the fields and high cost of production make use of hydrogels an expensive proposition as compared to urea, and (v) there is need to improve the production process of hydrogels for (a) the enhancement of the nitrogen content, and (b) the reduction in loss of materials and urea during manufacture.

## 5. ACKNOWLEDGEMENTS

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