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Preparation of Biodegradable Starch-Polyvinyl Alcohol Blended Film with Slow-Release Phosphate Fertilizer

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Abstract

The use of slow release fertilizer has become a new trend to save fertilizer consumption and to minimize environmental pollution. This paper discusses the preparation of starch (St)-polyvinyl alcohol (PVA) blended films by direct wet mixing process as the effect of different PVA loadings on phosphate fertilizer. Degree of swelling and water retention properties were performed to measure the amount of water intakes of the fertilizer. For a given blend composition, the degree of swelling mostly increased with longer immersion time. However, at 5 g PVA content (St-PVA5), the maximum degree of swelling was still 618.43% at 60 min, and water retention of 16.74% at 60 min.The phosphorus release behavior of prepared blended films in water was investigated. A soil degradation test was performed to measure the amount of weight loss. It was found that the degradation rate increased with PVA loading. Finally, the effect of the St-PVA blended film on the plant growth was also studied. The prepared St-PVA blended film has an effect on some agronomical characteristics of the selected plant for cultivation.

Keywords: Degree of swelling, polyvinyl alcohol, slow-release fertilizer, soil degradation, starch

Introduction

Overuse of fertilizers and pesticides is one of the causes of the degradation of the environment and soil. Slow release fertilizers are the newest and most technically advanced way of supplying mineral nutrients to crops (Subbarao et al., 2013). Slow release fertilizer (SRF) and controlled release fertilizer (CRF) are often used interchangeably. SRF is known as low solubility compounds with a complex/high molecular weight chemical structure that release nutrients through either microbial or chemically decomposable compounds, whereas CRF can be defined as "products containing sources of water-soluble nutrients, the release of which in the soil is controlled by a coating applied to the fertilizer (Lawrencia et al., 2021).The use of conventional fertilizers may lead to concentration levels that are too high for effective action. A high concentration may produce undesirable side effects either in the target area, which could lead to crop damage, or in the surrounding environment (Han et al., 2009). Polymer coatings may either be semi-permeable or impermeable membranes with tiny pores. The main problems in the production of polymer-coated fertilizers are the choice of the coating material and the process used to apply it. The nutrient release through a polymer membrane is not significantly affected by soil properties, such as pH, salinity, texture, microbial activity, redox-potential, and ionics (Naik et al., 2017).

The coating materials used should be inexpensive and exhibit good coating properties. In addition, they should undergo testing for degradation in the soil which might form any toxic substance that could affect the crop. The type of coating is responsible for the mechanism of elements release from encapsulated fertilizers (Tomaszewska et al., 2002). The major nutrients for plants are nitrogen (N) which supports leaf growth and forms chlorophyll and protein; phosphorous (P) which assists in root growth, fruit, and flower growth; and lastly potassium (K) which is useful for synthesizing proteins and induces stem and root growth (Cheng and Sun, 2005). The demand for more productive agricultural systems has required new technologies that increase the efficiency of used inputs. The management of phosphate fertilizers is a key factor in crop production, which is especially the case in highly weathered tropical soil. However, in more adsorbing tropical soils, the accumulation of P by plants is usually limited due to soil sorption processes (Teixeira et al., 2016).

Polyvinyl alcohol (PVA) is a synthetic polymer, non-toxic, flexible, soluble in water, and biodegradable. PVA exhibits excellent mechanical and barrier properties and is compatible with starch. The physical properties of PVA, such as resistance, water solubility, thermal characteristics, and gas permeability, vary with the degree of crystallinity, which is highly dependent on the degree of hydrolysis and average molecular weight of the polymer. Among the natural polymers, starch is a totally biodegradable polymer, has a low-cost, is renewable, and can be a promising candidate for the development of sustainable materials (Râpă et al., 2014).

In this study, the slow-release fertilizer (SRF) hydrogels were prepared from polyvinyl alcohol and polyvinyl alcohol/starch, using formaldehyde as a cross linker. Water absorbency, water retention, and phosphorus release behavior of such SRF hydrogels in water and soil were investigated. The water and soil samples were analyzed for total amounts of phosphorus by UV-visible spectrophotometry. The objective of this research was to obtain materials with controlled release properties by covering

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Table 1. The prepared different starch/ PVA treatment SRF hydrogel films and nomenclature

Sample treatment	Nomenclature
10 g starch + 1 g polyvinyl alcohol	St-PVA1
10 g starch + 2 g polyvinyl alcohol	St-PVA2
10 g starch + 3 g polyvinyl alcohol	St-PVA3
10 g starch + 4 g polyvinyl alcohol	St-PVA4
10 g starch + 5 g polyvinyl alcohol	St-PVA5

granules of mineral fertilizer with a layer of starch, an example of a biodegradable, natural polymer, with the use of a laboratory method.

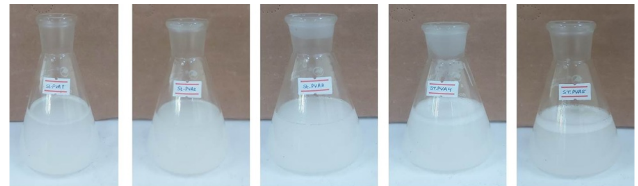
Materials and Methods

Soluble starch was pre-dried in the vacuum oven at 60 °C for 10 h. Polyvinyl alcohol (molecular weight: 20, 000, degree of hydrolysis: 98%) was purchased from the British Drug House (BDH) Chemical Ltd., England. All additional chemicals were analytical reagent grade. The standard methodologies and procedures, which incorporated both conventional and modern approaches, were employed in all investigations.

Preparation of Slow Released Fertilizer (SRF) Hydrogel Films

Samples were prepared through the direct wet mixing method using an experimental set up which consisting of a beaker, hotplate, and magnetic stirrer. The calculated amounts of starch, PVA, and a little glycerin were mixed at various ratios (PVA content is from 1- 5 wt%), and the mixture was slowly added to distilled water at room temperature under continuous stirring. When completely suspended, the temperature of the mixture was slowly raised to 80°C while maintaining stirring; a small amount of butanol was used to avoid frothing. And then, 2% of formaldehyde was added to the mixture, and stirring was maintained for 3 h to completely gelatinize the starch. The volume was maintained by adding water. After raising the temperature to 95°C, the mixture was removed from the heat and the foam was skimmed off (Figure 1). The fertilizer solution was prepared by dissolving 2.0010 g of ammonium nitrate, 3.3015 g of diammonium phosphate, and 2.5275 g of potassium nitrate to make a 100 mL aqueous solution.

The prepared starch-PVA solution and fertilizer solution were mixed in appropriate proportions under constant stirring to obtain a series of SRF hydrogel films. After homogeneous mixing, the gel was formed within 30 min. After drying the solution at room temperature, the films were allowed to dry in a hot-air oven at 60°C for 8 h and the dried starch/PVA films were removed from the mold. Films were cast and stored in polyethylene bags before use in further studies. The prepared sample was coded in Table 1.



St-PVA1 St-PVA2 St-PVA3 St-PVA4 St-PVA5

Figure 1. Prepared starch based polyvinyl alcohol hydrogel solutions

Determination of Degree of Swelling

The prepared SRF hydrogel films (1 × 1 cm2) samples with initial weight of Wd were dipped in 25 mL of water at a room temperature for various time interval. The wetted films were removed from water, blotted gently with tissue paper and reweighed (W_s). Based on these values, swelling (%) was determined. Each experiment was replicated three times. The degree of swelling (DS) was calculated according to equation (1) where (W_s) and (W_d) referred to the weights of swollen and dry SRF hydrogels, respectively.

$$DS\ (\%) = \frac{W_s - W_d}{W_d} \times 100\% \qquad \text{--------------------} \quad (1)$$

Determination of Water Retention of Soil

A dry sample of SRF hydrogel was buried in dry soil, which was placed in a cup (A). The other of the dry soil without CRF hydrogel was placed in an identical cup (B), and then each cup was weighed (W). After that, distilled water was added to both cups and reweighed (Wo). The cups were kept under identical conditions at room temperature and were weighed every day (Wt) over a period of 14 days. The water retention (% WR) of soil was then calculated by Equation (2) (Cheng and Sun, 2005).

$$WR\ (\%) = \frac{W_t - W}{W_o - W} \times 100\% \qquad \text{--------------------} \quad (2)$$

Determination of Phosphorus Release Behaviors

The release behaviors of phosphorus from the CRF hydrogels, both in deionized water and in soil, were investigated by UV-visible spectrophotometry. A 5 mL fertilizer sample solution was pipetted into a 25 mL volumetric flask, and 5 mL of molybdovanadate reagent was added. Deionized water was also added to make a 25 mL solution. After 30 min, at the room temperature, the absorbance of the sample solution was measured at a wavelength of 420 nm by a UV spectrophotometer. The amount of phosphorus in the sample solution was calculated using the calibration curve (AOA, 1990).

Figure 2. Samples for soil degradation test

Determination of Soil Degradation

The biodegradability of the samples in soils was studied by evaluating the percentage weight loss of the samples (Hamid, et al., 2013). In this study, samples with dimensions of 10 mm diameter and 5 mm thickness were weighed and placed in beakers containing agricultural soil (Figure 2). The initial weight of each beaker, (Wi) before samples were left for 14 days at room temperature was recorded. Then, the current weight of the beakers was taken every day after 24 h (Wd). The degradation rate was determined by the decrease in weight with time, which is directly represented by weight loss (WL) in Equation (3).

$$W_L\ (\%) = \frac{W_i - W_d}{W_i} \times 100\% \qquad (3)$$

Investigation of Agronomical Characteristics of Lady Finger Plant and Pea Plant at Different Time of Cultivation

Plant height, number of branches per plant, and fruit were involved in the investigations of the agronomical characteristics of lady finger and pea plants. Three randomly selected plants were taken to record average plant height, branches, and fruits in all investigations.

Results and Discussion

The acetal reaction mechanism is used for the crosslinking (Cordes, 1967). Both starch (St) and PVA contain free hydroxyl units, which make the crosslinking reaction possible. The reaction is catalyzed by the acidic environment provided by dissolving PVA and starch in water. In an acidic environment, formaldehyde's hydroxyl groups protonate. The nucleophilic hydroxyls of the St-PVA mixture attack the electrophilic center that forms as the protonated group and leaves as water. The excess of St-PVA in the reaction causes a similar mechanism to occur simultaneously on the other part of the formaldehyde. Thus, acetal bonding, which connects two polymer chains, produces crosslinking and forms a crosslinked structure. The mechanism is shown in Figure 3 (Gadhave et al., 2020).

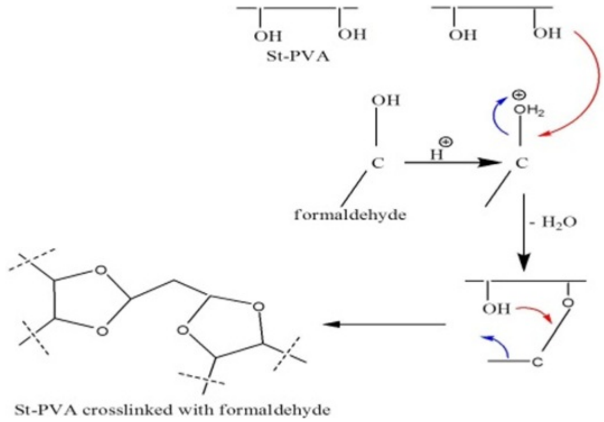


Figure 3. The mechanism showing crosslinking between St-PVA blend and formaldehyde

Degree of Swelling of SRF Hydrogel Films

First, we discuss the degree of swelling analysis of all samples at different PVA loading shown in Table 2 and Figure 4 as a function of immersion time. As seen in Table 2 the highest degree of swelling of St-PVA1, St-PVA2, St-PVA3, St-PVA4 and St-PVA5 are 539.98, 542.33, 572.12, 597.01, and 618.43% at 60 min, respectively. The water absorbency of the hydrogel films increases as the amount of hydrophilic component increases in a compound. However, the weight difference increases with PVA content. It is clearly observed that high PVA loading (5 g) in the fertilizer had significantly increased the degree of swelling. Hydrophilic groups are responsible for such results. This is because PVA is more hydrophilic than starch. Figure 2 shows that the higher PVA content in the SRF hydrogels, the higher water absorbency increased. It can be seen that all the SRF hydrogels reached in 60 min with a maximum amount of swelling within 1.5 h. However, water absorbency is an important criterion for slow release fertilizers (Wu et al., 2008) since the presence of water will cause a gradually release of phosphorus into the environment. It can be concluded that the swelling ratio of the St-PVA5 system was found to be higher than that of other systems.

Water Retention Behaviour of SRF Hydrogel Films

In this study, water retention analysis was conducted for a period of 14 days. The water retention percentage of fertilizers for both gelatinization temperatures at different PVA contents is presented in Figure 5 and Table 3. The St-PVA films were investigated for water retention in the soil. In general, the water retention slowly decreased with time. Table 3 shows that after 14 days, the water contents of the soil samples buried with St-PVA1, St-PVA2, St-PVA3, St-PVA4, and St-PVA5 hydrogel films have remained around 2.37, 6.47, 11.76, 14.59, and 16.74%, respectively. It is found that St-PVA5 (5 g of PVA) shows the best water-retention capability. These St-PVA5 films exhibit a slightly higher ratio of water retention percentages during the 14th day compared to other films. From Figure 5, it can be seen that there is no obvious difference in the pattern of water retention percentages for the different gelatinization temperatures. Therefore, the water was stored in the fertilizer and slowly released with the decrease in soil moisture (Mo-

Table 2. Degree of swelling of SRF hydrogel films at different time intervals

Time	Degree of Swelling (%)				
(min)	St-PVA1	St-PVA2	St-PVA3	St-PVA4	St-PVA5
10	176.29	182.39	197.45	234.12	251.47
20	269.29	280.74	330.98	351.65	363.80
30	274.23	325.07	348.64	382.91	407.97
40	354.47	381.92	409.91	443.45	457.94
50	362.19	410.21	492.11	517.12	530.75
60	539.98	542.33	572.12	597.01	618.43
70	510.19	528.36	544.54	575.44	604.11
80	486.13	501.82	524.20	550.76	589.25
90	478.98	497.67	510.22	543.13	570.32

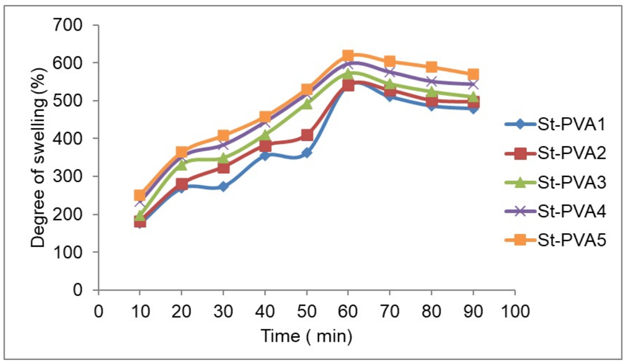


Figure 4. Degree of swelling of SRF Hydrogels at Different Time Intervals

able 3. Water retention of soils treated with SRF hydrogel films

Time	Water retention (%)				
(day)	St-PVA1	St-PVA2	St-PVA3	St-PVA4	St-PVA5
1	56.90	63.23	68.10	76.34	89.89
2	53.43	50.21	63.64	64.37	77.29
3	47.19	49.11	54.30	61.27	72.35
4	42.85	44.45	51.86	59.99	60.36
5	38.22	40.12	47.37	52.24	58.90
6	37.93	39.76	41.88	44.54	50.81
7	23.92	28.60	29.33	31.65	36.24
8	19.64	26.71	28.11	30.54	34.44
9	18.17	23.45	25.57	29.13	33.86
10	16.41	21.33	23.74	27.94	29.78
11	14.54	20.26	22.89	26.70	27.88
12	9.23	16.27	19.84	21.79	23.38
13	6.98	13.86	15.49	17.57	19.43
14	2.37	6.47	11.76	14.59	16.74

hamad et al., 2013). According to the results, the St-PVA5 SRF hydrogels affected the highest water retention, and the St-PVA1 SRF hydrogel affected the lowest water retention. It can be concluded that the water retention of the soil increased with increasing PVA contents.

Phosphorus Release Behavior of SRF Hydrogel Films

The phosphorus release behavior of the SRF hydrogel films in the distilled water at the room temperature are shown in Table 4 and Figure 6. Figure 6 shows the percent phosphorus release versus time graph for the duration of the 14th day observation period. It was found that all of the SRF hydrogels exhibited an initial increase in release. The cumulative release of phosphorus on day 14 was 55.22, 64.34, 67.58, 69.82, and 72.90% for the St-PVA1, St-PVA2, St-PVA3, St-PVA4, and St-PVA5 hydrogel films, respectively. The release behavior of the St-PVA5 film was higher than that of the other films (Table 4). It is observed that the PVA hydrogels is appeared to show the highest release amount of phosphorus due to its high hydrophilicity. Phosphorus (in the form of diammonium phosphate) in the SRF hydrogels released after the hydrogels absorbed water, leading hydrolysis process taking place. The phosphorous release behavior suggests that it is partially diffused through a swollen network and water-filled pores in the fertilizer gels (Al Rohily et al., 2020).

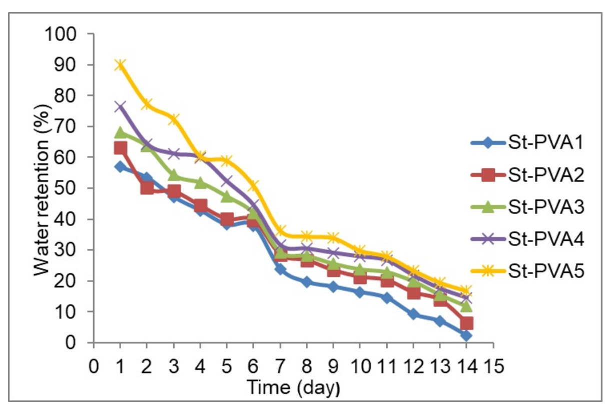


Figure 5. Water retention of soils treated with SRF hydrogel films

Table 4. Release behavior of SRF hydrogel films in water

Time	% of Phosphorus Released				
(day)	St-PVA1	St-PVA2	St-PVA3	St-PVA4	St-PVA5
1	16.13	19.32	23.76	24.43	27.34
2	21.22	23.39	28.67	27.45	32.60
3	24.45	26.74	31.80	31.62	34.19
4	29.19	32.54	35.34	39.11	41.47
5	35.24	46.43	49.50	50.64	52.48
6	47.23	52.33	55.67	57.38	62.93
7	47.89	58.24	61.23	63.55	68.73
8	57.36	60.89	64.34	66.38	72.49
9	59.75	61.34	68.47	68.39	75.66
10	63.94	64.87	65.76	72.73	77.34
11	67.22	69.25	71.82	74.29	87.58
12	68.12	69.27	70.56	71.76	82.34
13	59.74	67.23	69.12	69.75	75.89
14	55.22	64.34	67.58	69.82	72.90

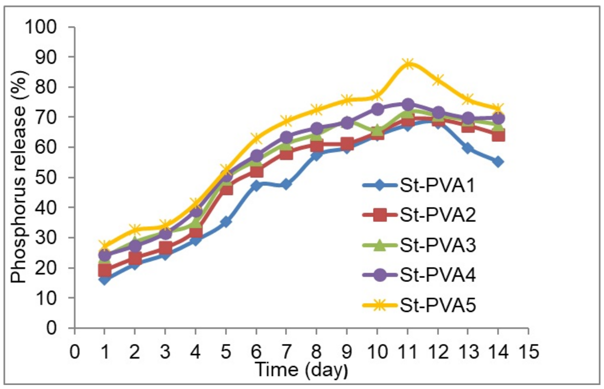


Figure 6. Phosphorus release behavior of SRF hydrogel films in water

Table 5. Weight loss (%) of SRF hydrogel at different times intervals

Time	Weight Loss (%)				
(day)	St-PVA1	St-PVA2	St-PVA3	St-PVA4	St-PVA5
1	1.21	1.54	2.47	3.82	5.15
2	4.11	4.37	4.64	6.27	7.68
3	8.26	11.18	10.72	13.28	16.83
4	11.65	15.45	16.86	21.36	26.74
5	12.78	18.77	18.49	26.45	28.90
6	16.96	21.38	23.87	29.43	34.76
7	27.73	30.84	35.47	36.78	49.54
8	29.36	36.97	43.76	43.24	51.53
9	32.72	39.44	44.71	51.84	55.69
10	36.92	44.84	47.36	57.29	58.96
11	42.16	47.37	51.67	58.95	65.57
12	43.44	49.62	52.46	62.35	71.59
13	46.12	50.74	54.67	67.49	74.71
14	51.66	62.72	66.94	74.68	76.54

St-PVA degradation in soil

The degradation rates of fertilizers based on weight loss percentage in 14 days are shown in Table 5 and Figure 7. It was found that the sample degraded rapidly at the initial stages and then slowly after 10 days. After 14 days, the samples decreased in size. It can be suggested that the weight loss experienced by the fertilizer is solely due to constant environmental factors and microbe's activity that breaks down the fertilizer (Leja and Lewandowicz, 2010). So the degradation rate was observed to occur very rapidly in the first five days and decrease with time until the whole weight of sample degraded.

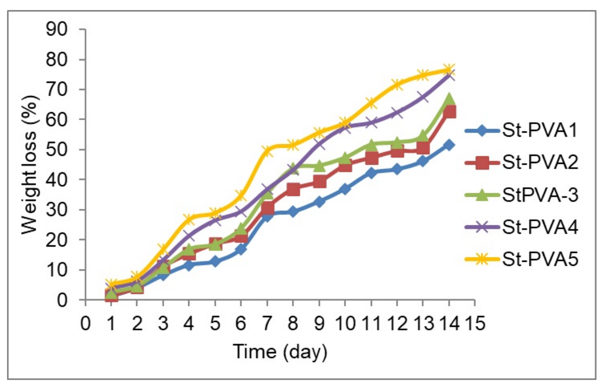


Figure 7. Weight loss (%) of SRF hydrogel at different times intervals

Effect on plant growth

The growing plant such as lady finger and pea using SRF hydrogel were recorded by photographs in Figures 8 and 9. As seen in Tables 6 and 7, in all cultivations of the agronomical characteristics of the lady finger plant and pea plant, SRF hydrogel showed longer plant height, and an increased number

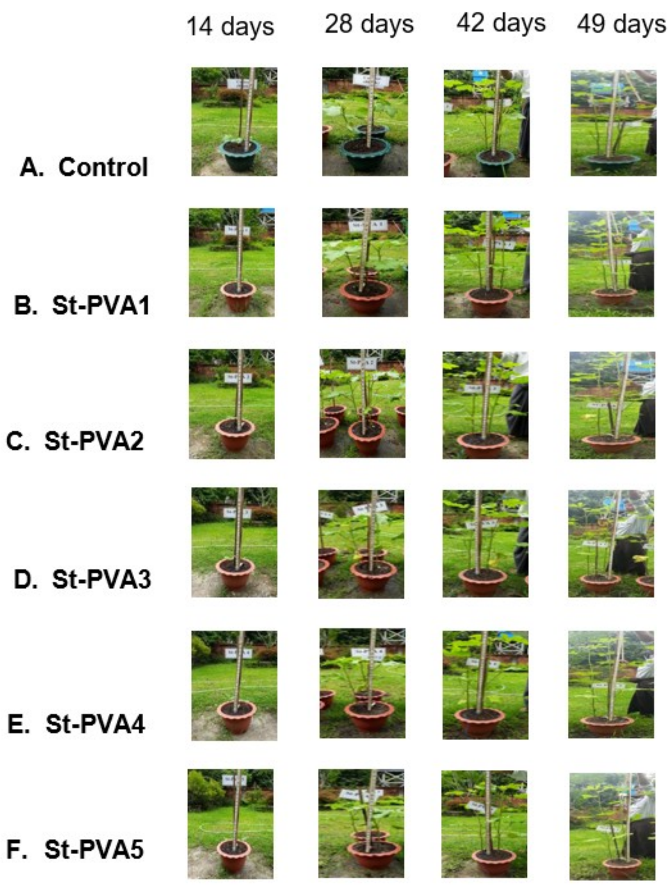


Figure 8. The growth of ladyfinger using SRF hydrogel film for A, B, C, D, E and F

of branches and fruits when compared with the control. Table 6 shows the agronomical parameter of all SRF hydrogel films increased than control. According to the data, St-PVA3 and St-PVA4 are more effective than the other films. So all SRF hydrogel films have a greater cumulative effect than the control. It can be concluded that SRF hydrogel films have a greater cumulative effect and reduce environmental pollution.

Conclusion

This study provides a scientific understanding of the potential of starch as a biodegradable binder in phosphorus fertilizer. Overall, the degree of swelling, water retention, and weight loss percentage of produced phosphorus fertilizers based on starch were found to be directly proportional to the increasing PVA contents due to the increase in hydrophilic properties. St-PVA 5 hydrogel exhibited the highest percent cumulative release of phosphorus in soil among the SRF prepared hydrogels. The degradation rates of fertilizers based on weight loss percentage after 14 days are studied. It was found that biodegradation has taken place in almost 76.54% of St-PVA 5. Lady finger and pea plants were cultivated using SRF hydrogel together with con-

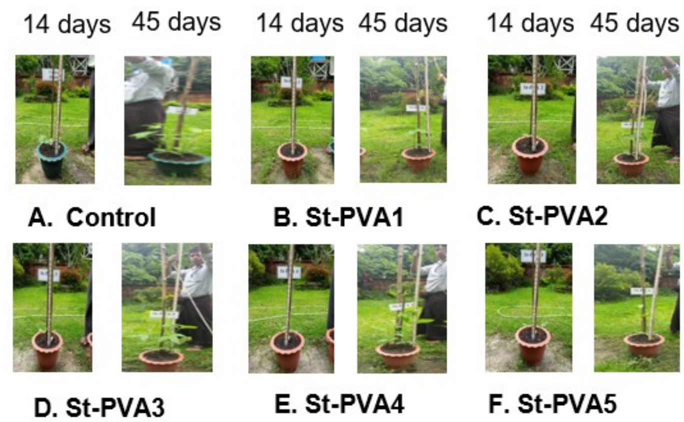


Figure 9. The growth of pea using SRF hydrogel film for A, B, C, D, E and F

Table 6. Agronomical characteristics of lady finger plant

Sample	Plant heigh (ft) Day			Number of branches per plant Day			Number of fruits per plant Day		
	28	42	49	28	42	49	28	42	49
control	1.6	2.3	3.8	4	6	6	0	1	3
St-PVA1	1.6	2.3	3.9	5	7	8	0	1	3
St-PVA2	1.7	2.5	3.8	5	7	7	0	1	3
St-PVA3	1.8	2.3	3.6	5	7	7	0	1	3
St-PVA4	1.8	2.4	3.10	4	5	6	0	1	3
St-PVA5	1.7	2.4	3.10	5	6	7	0	1	3

Table 7. Agronomical characteristics of pea plant

Sample	Plant heigh (ft) Day		Number of branches per plant Day		Number of fruits per plant Day	
	14	45	14	45	14	45
control	0.4	2.6	1	3	0	4
St-PVA1	0.6	2.10	1	3	0	5
St-PVA2	0.6	3.3	1	3	0	5
St-PVA3	0.6	3.1	1	3	0	6
St-PVA4	0.6	2.7	1	3	0	6
St-PVA5	0.6	2.7	1	3	0	5

trols. Various agronomical characteristics measured revealed that SRF hydrogel showed significant changes when compared with the control. So the SRF hydrogel film has a greater cumulative effect than the control. Finally, it can be concluded that the starch-PVA blended film has the potential to improve fertilizer's degradation rates in order to obtain slow–release properties, to become a new trend to save fertilizer consumption, and to reduce environmental pollution and safety application of agriculture but this may require further investigation.

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