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### Coating of Urea Granules for Slow Release Fertilizer Using Bioblend Polystyrene / Polycaprolactone.

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#### ABSTRACT

The usage of bioblend polystyrene/polycaprolactone for coating of urea as a slow release fertilizer dosage form has been carried out. Bioblend was prepared by mixing polystyrene with polycaprolactone. The purposes of addition of polycaprolactone biopolymers on polystyrene, not only to obtain the biodegradable sealant characteristic, but also expected to create pores. Bioblend polystyrene/polycaprolactone has good compatibility with the urea granules and higher efficiency of the polymer coating on the urea, could reduce the release rate of nutrients from fertilizers. Results showed that the release rate of fertilizer products obtained were slower than the uncoated urea granules. The release of the active substance from coated urea on the soil media in formula 2 and 3 after 10 days of testing were only 18.3, and 28.0% w/w, respectively. The release profile of urea using the mud media was slower than in distilled water and soil media as well. Meanwhile, the total released of the fertilizer in aqueous media from all of formulas were average of 24 hours. It shown improper applications using the aqueous media. The percentage of urea released after 10 days of testing using mud media from formula 2 and 3 were only 7.9, and 9.5%w/w, respectively. Formulas of bioblend polystyrene/polycaprolactone coated urea obtained were suitable and fulfill as a slow release fertilizer product.

**Keywords:** urea granules, fertilizer, bioblend, polystyrene, polycaprolactone.

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## INTRODUCTION

A fertilizer is one of our most important agrochemical industries to increase production and productivity of crops [1]. The fertilizer spread on crops could not be absorbed appropriately by plants, the efficiency of urea absorbed is only about 30-50%. The rest of the unabsorbed fertilizers will be degraded or flush out away by water [2]. According to the Food Agriculture Organization (FAO) world fertilizer consumption is estimated to reach 190.4 million tons in 2015 [3]. The low efficiency using of nitrogen fertilizers has contributed in large negative impact such as; increased erosion, decline soil fertility, pollution of groundwater or a decrease in the quality of ground water in term of chemical parameters such as; pH, DO, BOD, COD, eutrophication of rivers and lakes. It also has an impact on the atmosphere and climate [4]. An alternative to increase the efficiency of urea fertilizer is usage of a slow-release fertilizer. It has several advantages compared to the conventional fertilizer as follows; improving fertilizer efficiency, reducing the fertilizer lost by rain or irrigation water, providing sustained release of the fertilizer for a longer time, saving the fertilizer consumption and minimizing environmental pollution [5,6].

Polymer Coating Urea (PCU) available in the market so far is more expensive 4-8 times compare to conventional urea fertilizers. The higher price of previous coated fertilizer because of expensive polymer used. Hence the bioblend polystyrene/polycaprolactone polymer coating is used to lessen the production cost resulting the cheaper price of final product. In addition, the polymer used is environmentally friendly [7]. Polymer coating efficiency factor is a problem because of the efficiency of the polymer coating on the fertilizer is significantly dependent on the physical and chemical interactions between polymer materials and fertilizers [8]. The limitation of coating urea with synthetic polymer and biopolymer material is less compatible. Appropriate compatibility of the polymer coating with granular urea will increase the efficiency of the coating. Polystyrene is an inexpensive polymer that can be used as an alternative coating polymer. It is a synthetic polymer that widely used, particularly as wrapping insulation materials and electronic instruments. The biodegradable polystyrene can be obtained by preparation of bioblend polystyrene. Bioblend is a mixture comprising at least one biodegradable polymer with undegradable polymers [9].

In this study, bioblend polystyrene was manufactured by mixing polystyrene with PCL. PCL is a biodegradable synthetic biopolymer most commonly used for medical use, because it has good biocompatibility properties and non-toxic in the human body so that it can be ascertained quite safe for the environment. Biresaw et. al. (2004) reported that bioblend polystyrene-polycaprolactone has more compatible characteristics than a mixture of polystyrene with other biodegradable polyesters [10]. In other hand, Salman (1988), also reported that single polystyrene coated urea had high release of nitrogen, around 95% in water with a temperature of 22°C for one week. The similar characteristic also found using thick coating layer (15%), its caused by plenty of pores (10-20µm) in the core layer of coated urea granules [11]. The addition of polycaprolactone polymers in the formulation not only to obtain the biodegradable sealant, but also to allow the formation of pores. Bioblend polystyrene has good compatibility with the urea granules, high efficiency, and reduce the release rate of nutrients from fertilizers.

The purpose of the research was to study the effectiveness of the coating of urea fertilizer using both types of coating the polystyrene bioblend. The coating process was carried out with a spray coating method. Spray coating method is the most effective method for coating granular urea [12].

## EXPERIMENTAL

### Equipments and Materials

#### Materials

Polystyrene, polycaprolactone (Aldrich Chemical), p-dimethylaminobenzaldehyde (DMAB), cassava starch (Amprotab™, Bratachem), urea granule (PT. Pupuk Sriwijaya, Indonesia) diameter of 2 mm, chloroform (Merck), alcohol (Merck), nutrient agar medium (Merck), Ehrlich reagent, aqua DM.

**Equipments**

Hotplate magnetic stirrer (Thermo Scientific), a homogenizer (IKA® RW Digital), UV-Vis spectrophotometer (UV-1700 Pharma Spec), analytical balance (Shimadzu AUX 220), Fourier transform infrared spectroscopy (PerkinElmer), pH meter (Thermo Scientific), scanning electron microscopy (SEM) JEOL-JSM-6510LV, Las Navas Instruments Termogravimetric-2000 Analyzer, oven, spray gun and pump, pan coating, glass tools and other apparatus.

**Methods**

**Preparation of Granular Urea**

Granular urea was washed using chloroform and then dried in oven at temperature of 50-60°C. Urea was then screened with a sieve 1-2 mm in size to obtain a uniform size.

**Preparation of Coating Liquid**

Polystyrene was dissolved in 50 mL of chloroform. One gram of polystyrene mixed with half gram of polycaprolactone then dissolved in 50 mL chloroform. The solution was stirred using a magnetic stirrer at speed of 380 rpm for 10 minutes. The various formulas prepared can be seen in Table 1.

Table 1: Bioblend Coating Polystyrene/PCL Formula

Formulation Code	Products	Composition		
		PS (g)	PCL (g)	Starch (g)
F1	Bioblend PS/PCL 2:1	1.0	0.5	0
F2	Bioblend PS/PCL 3:1	1.5	0.5	0
F3	Bioblend PS/PCL 4:1	2.0	0.5	0
F4	Bioblend PS/PCL 5:1	2.5	0.5	0
F5	Bioblend PS/PCL 6:1	3.0	0.5	0

**The Coating of Urea**

The coated urea was prepared by spray coating method. 25g of uncoated urea granule was sprayed with a coating solution in a coating pan. The coating pan was rotated at adjusted rotation speed and the temperature maintained at 60-70°C. Finally, coated urea granule was dried in oven at temperature of 70-80°C for 1 hour to ensure the appropriate solvent evaporation and the granule completely dry.

**Characterization of Coated Urea Granules**

**Topography of Granules**

The characterization of surface morphology of granules was performed to characterize and compatibility behaviour between the polymer coating with urea. The evaluation was conducted using SEM.

**Chemical Interaction**

The possibilities of chemical interaction between the components of coating materials and uncoated urea granules was evaluated using Fourier Transform Infrared Spectroscopy (FTIR).

**Thermogravimetry Test**

Thermogravimetry test was conducted to evaluate changing of sample mass as a function of temperature determined by oxidative thermal stability using a Termogravimetric Analyzer at temperature range of 40-950°C, heating rate 3°C per minute and flow rate of dry nitrogen at 2 L per minute [13].

## Release Rate and Determination of Urea

### Release in aqueous media

One gram coated urea obtained was dissolved in 500mL of distilled water. Sample solutions were withdrawn and filtered at 1, 3, 6, 12 and 24 hours. The absorbans of solutions were measured using a spectrophotometer [13-15].

### Release of Urea in Soil

100g of dried soil was placed in a glass funnel assembled with filter paper. Three grams of sample Formula 2, 3, and uncoated urea were planted in the funnel glass which placed in a glass bottle 27cm in height. The soil in the glass funnel was moisturized with 20mL distilled water at flow rate of 5mL/min. Sample solutions were collected and the absorbance was measured spectrophotometrically. Sampling was performed every day until day 10. Dried soil and mud media were evaluated for formula 2, 3 and uncoated urea.

### Release of Urea on Mud

100g of mud collected from the rice fields were placed in a beaker of 1000mL. Two grams of sample formulas 2, 3 and uncoating urea were planted in the mud and added with 500mL of distilled water. Samples solutions were collected and the absorbance of filtered samples were measured. Sampling was performed every day until day 10.

### Determination of The Concentration of Urea

The concentrations of urea were determined using spectrophotometric method. DMAB was used as color forming. DMAB could react with urea to form yellow-green lemon Kellyr based on Ehrlich reaction as shown in Figure 1. The color was stable for 10 minutes. The substance obtained will absorb visible light at 420nm [14,16-18]. Reagent solution was prepared by dissolving 2g of DMAB in 50mL ethanol 95% and 50mL concentrated hydrochloric acid. The calibration curve was created using standard solutions at concentrations of 5, 10, 20, 40, 60, 100, 400, 600, 800, 1000 and 1200 ppm.

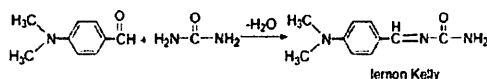


Figure 1: Reaction of DMAB with Urea [19,20].

### Coating Efficiency

Coating efficiency of the coated bioblend could be determined by dissolving the product with the mass known, M total (g) in 100mL of distilled water at room temperature ( $\pm 24^\circ\text{C}$ ). The sample solutions were filtered using filter paper. The residue was washed with distilled water, then dried at  $120^\circ\text{C}$  for approximately 4-6hours, and weighed (M polymer in g). Coating efficiency was calculated using following equation:

$$\% \text{ Coating} = \frac{M \text{ residue polymer (g)}}{M \text{ total (g)}} \times 100$$

% Coating is the percentage of the polymer coating on the urea granule. M residual polymer is the mass of the polymer residue (g), and M total is the total mass (g) [11,12].

## RESULTS AND DISCUSSION

### Morphology and Characteristics of the Coated Urea

Figure 2 shows SEM micrograph of granular coated urea polystyrene bioblend. Figure 2a and 2b show urea granule morphology with magnification of 40 and 80 times, respectively. The topography of coating

surface was generally smooth, compact and uniform, but in some parts of the coating surface is uneven and unsmooth due to fast drying effect.

Picture 2c depicted SEM micrograph profile of cross section of bioblend polystyrene coated granular urea. The picture shows the structure of two layers of bioblend polystyrene coated urea. The outer part is bioblend coating polystyrene/polycaprolactone, while in the center is urea. Polymer coating on the outside serves as a physical barrier to mass transfer, thereby reducing the rate of diffusion of water into the center and diffusion of urea out of the core and provide slow-release effect [11,21,22].

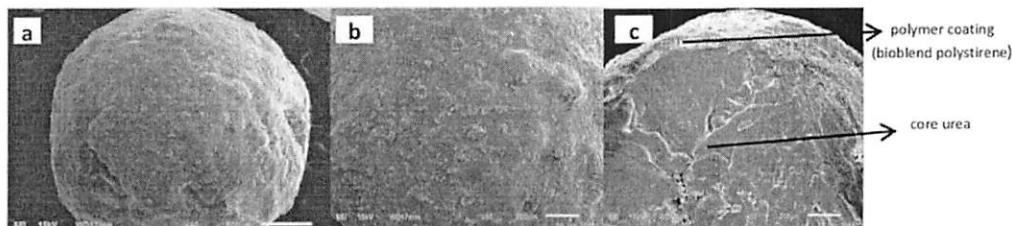


Figure 2: SEM micrograph of coated urea bioblend polystyrene granule, a). urea granule morphology with a magnification of 40 times, b). urea granule morphology magnification of 80 times, c). SEM micrograph of a cross-section bioblend granular urea coated polystyrene, enlarged 80 times.

#### FTIR Analysis of Coating Shells

The FTIR spectra of samples bioblend polystyrene/polycaprolactone coated urea shows similarities spectra of samples without coating urea (Figure 3). Peak at  $3429$  and  $3336$   $\text{cm}^{-1}$  of plain urea and the sample formulas is asymmetric and symmetric range vibrations  $\text{NH}_2$ . Peak at  $3256$   $\text{cm}^{-1}$  of the 3 samples urea, an OH vibrations of absorbed water molecules. Peak at  $1672$   $\text{cm}^{-1}$  is a carbonyl (CO) and at  $1589$   $\text{cm}^{-1}$  is n NH bending vibration and stretching vibration of CH (usually an area of bending vibrations NH)  $\text{O} = \text{C}-\text{NH}_2$ . At  $400-1500$   $\text{cm}^{-1}$  or fingerprint region, all the usual peak of urea samples and the samples look very similar [12,22,23,24].

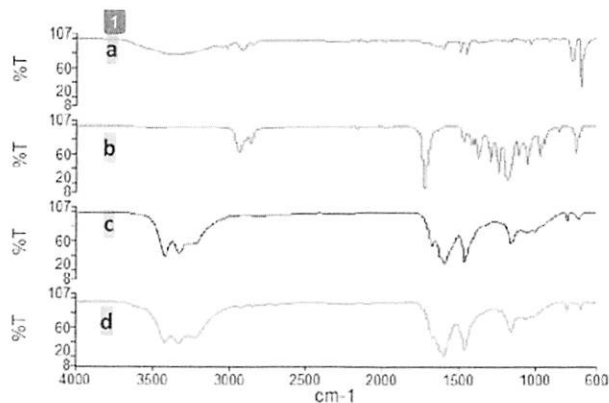


Figure 3: FTIR spectra of a). polystyrene, b). polycaprolactone, c). uncoated urea, d). bioblend polystyrene/polycaprolactone coated urea.

#### Thermogravimetric Analysis (TGA)

The thermal decomposition testing of urea showed that decomposition occurs in two stages, as shown in Figure 4. The first stage start at temperature of  $120^\circ\text{C}$  to  $250^\circ\text{C}$ , the main reaction ends at  $225^\circ\text{C}$  with the weight of 61.2%. At this first stage associated with early evaporation of urea, biuret formation,  $\text{NH}(\text{CO})_2(\text{NH}_2)_2$ . In the second stage, 38.8% of urea started to decompose at the temperature of  $390^\circ\text{C}$ . The first stage of the thermal decomposition of urea is the most important phase of this study, related to the evaporation of urea. No significant weight loss was occurred in a temperature range between  $25$  and  $150^\circ\text{C}$

associated with dehydration absorbed water, interlayer water, and water cation exchanger. The degradation characteristics of the first phase of urea coated with polymer showed similarities with uncoated urea granule. It could be concluded that the coating of granular urea fertilizer not affected the consistency of the original core. Coating with a polymer material could not change the characteristics of decomposition of urea significantly because of only small amount of urea on the surface of the polymer granules to allow modify the rate of urea decomposition characteristics [25-27].

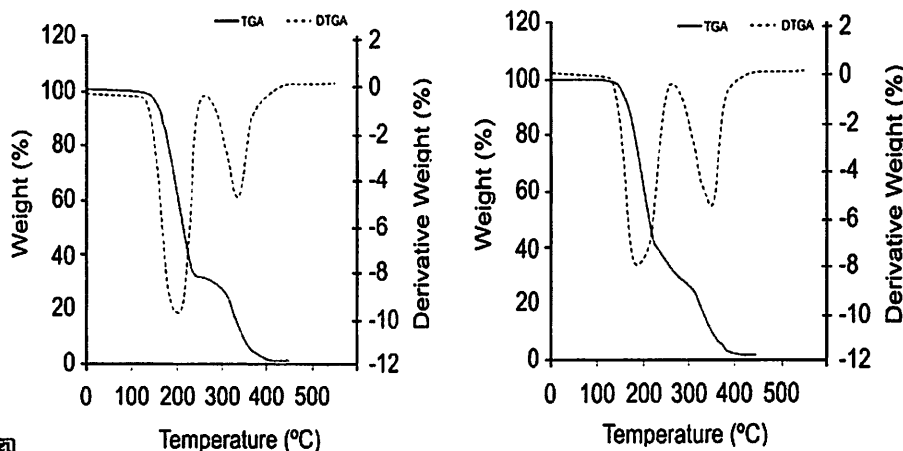


Figure 4: TGA thermogram a). urea b). polystyrene/polycaprolactone bioblend coated urea c). polystyrene/starch bioblend coated urea.

**Coating Percentage**

The higher the mass of the polymer coating in the coating solution, the higher the percentage of coating (Figure 5a). Concentrations of the solution would greatly affect the operation of apparatus and mechanisms of growth of the film coating on the surface of urea. In the Figure 5a, it was clear that the greater the mass of the coating, the higher the percentage of coating. The greater the mass of the coating, the saturation level during drying could reach a maximum so that resulting in increased formation of urea crystallization on the surface. Several theories suggested that the greater the mass of the polymer coating, the more coating bonded to the surface of urea [28]. However, in cases bioblend polystyrene polymer blending, in which polystyrene is easily loss solvent, so that it will generate dustiness. The higher the concentration of polystyrene in the mixture formula, the greater the possibility of the formation of dust, thereby decreasing the percentage of coating efficiency (Figure 5b).

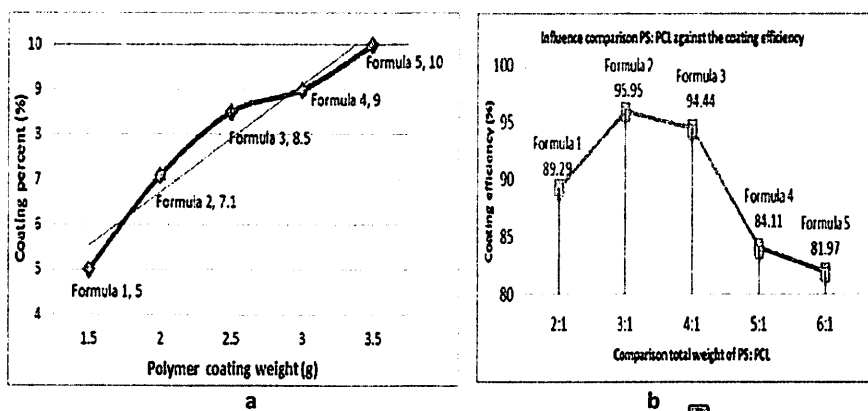


Figure 5: a). The influence of the amount of polymer coating to the percent of the coating. b). The influence of the ratio of polystyrene with polycaprolactone to the efficiency of the coating.

Dustiness occurred because the polymer coating material not strong enough to bind to the surface of the urea granules. It was could also be formed due to in-appropriate apparatus set up, especially on the temperature adjustment, where the higher the temperature would speed up the drying of the coating process. The faster drying rates would influenced the bond coating surface of urea on the polymer coating, and increase the formation of dust. So that the coating would decrease the percentage of efficiency [29]. Efficiency of the coating formula 1 and 2 were 89.3 and 96.0%, respectively. Increasing of polystyrene amounts in formula 2 also caused the stronger of film coating layer. So the efficiency of the coating would be higher. However, the addition of a larger mass of polystyrene was found to decrease the efficiency of the coating. It could be seen from the decline of the percentage of coating in formula 3. In contrary, increasing of amounts of polystyrene in formula 4 and 5, decreasing the percentage of the coating.

#### Analysis of the Formation of Coating Films

In the spray coating process the coating film formation consists of 3 steps: (1) coating the polymer droplets reach the substrate, (2) coating the polymer droplets spread on the substrate, and (3) drying polymer coating and film forming coating. This step occurs repeatedly when spray coating process until the entire film formed. The solvent of the polymer sprayed onto the substrate urea within a certain time interval will disappear to evaporate together with the flow of hot air sprayed in the certain time interval. Drying capacity was significantly affected the spray coating process. If the drying capacity is in accordance with the amount of solvent that used, it will produce a good film layer. If the drying capacity is not enough, there will be wet that may form aggregate. However, too quick drying would caused droplets to lack solvent, causing the formation of bubbles in the film related not only with high viscosity droplets but also in-appropriate spreads droplets and makes it uneven. The uneven spreading of polymer substrate on urea will lead to the formation of plenty of small pores in the coating film layer. Film layer coating formed not strong enough and not compact due to the rough surface structure and not rigid [30]. The strength of the film layer formed also depends on the physical properties of polymers, mainly for blending the two different properties polymers.

The film thickness, and the composition of the mixing of the two polymers also influenced the strength of the resulting thin film layer. This can be seen from the resulting film layer coating the polystyrene mix composition/polycaprolactone in ratio 1: 1. Results of visual observation, and the thin film coating layer produced in this formula is very fragile and easily destroyed only slight friction with the hand. The resulting film is very fragile due to the influence of tensile strength properties of the low polycaprolactone affects tensile strength polystyrene that is higher. This unbalanced mix composition results in the brittle film. The addition of a number of polystyrene (formula 1) can improve the tensile strength properties of the thin layer film bioblend polystyrene/polycaprolactone. However, with the addition of a larger amount of polystyrene will lead to the formation of plenty small pores in the coating film layer (Formula 4 and 5).

#### Bioblend Coated Urea Release Profile in the Distilled water, Soil and Mud Media

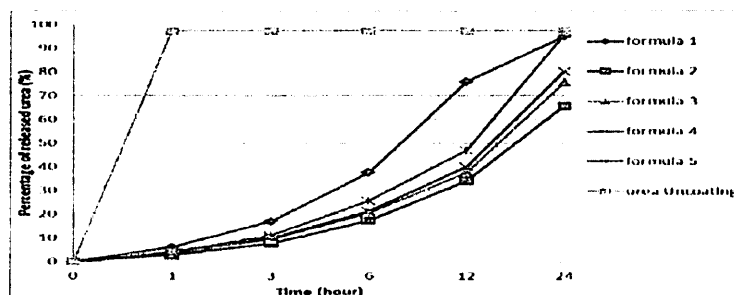


Figure 6: The Release of Urea versus Time Curve for each Formula in Distilled Water Media.

Figure 6 shows the behavior of coated urea release bioblend in distilled water at room temperature. The release of urea from bioblend formula 2 and 3 during 24 hours were around 80%. Thus, all formulas are not suitable for applications using aqueous media. Figure 7 shows a slower release profile of the formula 2, and 3 in soil and mud media. The release of urea using soil media formulas 2 and 3 after 10 days were 18.3, and 28%, respectively. The release profile of urea from bioblend were slower than distilled water and soil

media. The percentage of urea released from formula 2 and 3 after 10 days using mud media were only 7.9 and 9.5%, respectively.

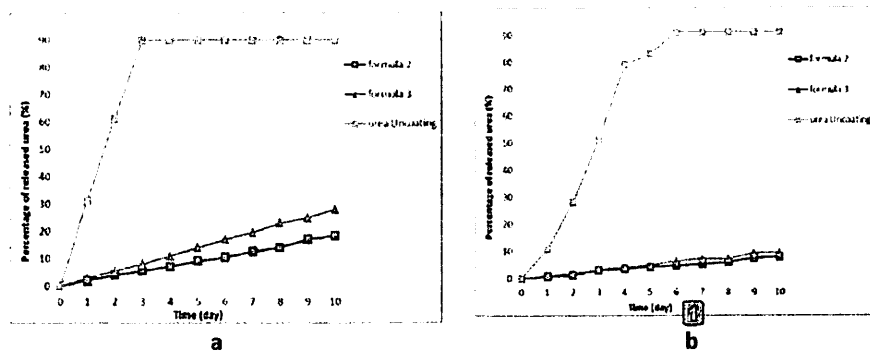


Figure 7: The Release of Urea versus Time Curve for Formula 2,3,6,7 and Un-coating Urea a). Soil Media, b). Mud Media.

#### Released Characteristics of the Coated Urea

The process of nutrient release from polymer-coated fertilizers occurs in several stages. Firstly, the water molecules penetrate through the layers of polymer, then urea in the core was dissolved lead to the formation of inner pressure, so that coating layer swells and enlarges the pore diffusion. Lastly, nutrients are released through the diffusion mechanism by concentration gradient in the layer, and/or by pressure gradients [31,32]. Based on mechanisms described above, the characteristics of the fertilizer released from bioblend polystyrene/polycaprolactone coated urea in distilled water could be divided into 3 stages; i). Lag time. Water molecules penetrate through the coating layer bioblend polystyrene, but urea not released yet. The grace period of the stage is determined by the percentage of coating by bioblend polystyrene and uniformity of coating layer. ii). Constant rate release. Urea in the core starts to dissolve and leave through the coating layer. The concentration of urea in the centre is very high at this stage and release process will finish until all the urea in the core dissolves completely. iii). Declining stage. Urea in the core dissolves completely, and urea concentration starts to decrease [21].

#### CONCLUSIONS

All formulas studied showed the release of urea in aqueous media with an average time of 24 hours. The release of fertilizer from dosage forms obtained in aqueous media were less efficient. It could be concluded that the dosage form not suitable for applications using this media. Results showed that the obtained product had a slow release characteristics fertilizer as the slower release rate than the uncoated polystyrene-PCL urea granules. The release of the active substance from coated urea on the soil media in Formula 2 and 3 were only 18.3, and 28% w/w after 10 days of testing. The release of fertilizer in the mud media was slower than in distilled water and soil media. The percentage of urea released from Formula 2 and 3 after 10 days of testing on the media mud were only 7.9 and 9.5%w/w, respectively. This finding showed that the products were fulfilled as a slow release fertilizer dosage form.

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