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B.ENG. (HONS) CHEMICAL ENGINEERING

MAY 2013

INVESTIGATION OF DEGRADATION OF STARCH COMPLEX FOR CRF APPLICATION

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by

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Dissertation is submitted as partial fulfillment of

the requirement for the

Bachelor of Engineering (Hons)

(Chemical Engineering)

MAY 2013

Universiti Teknologi PETRONAS

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CERTIFICATION OF APPROVAL

Investigation of Degradation of Starch Complex for CRF Application

By

Chong Win Sin

A project dissertation submitted to the

Chemical Engineering Department

Universiti Teknologi PETRONAS

in partial fulfillment of the requirement for the

BACHELOR OF ENGINEERING (Hons)

(CHEMICAL ENGINEERING)

Approved by,

____

(DR. NURLIDIA BT. MANSOR)

UNIVERSITI TEKNOLOGI PETRONAS

TRONOH, PERAK

May 2013

CERTIFICATION OF ORIGINALITY

This is to certify that I am responsible for the work submitted in this project, that the originality work is of my own except as specified in the references and acknowledgements and that the original work contained herein have not been undertaken or done by any unspecified sources or persons.

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(CHONG WIN SIN)

ABSTRACT

Controlled-release fertilizer (CRF) is applying a biodegradable material as a coating material to control release the nutrients from the urea. One of the cheap and easily available biodegradable materials is starch, and these properties enable it to be used as coating material for CRF application. However, starch itself has a few disadvantages and thus needed to be modified in order to overcome such drawbacks. Researches are yet to be done to determine a starch full potential as CRF. In this study, tapioca starch is chemically modified with urea in the presence of borate.

This study uses two important conditions for biodegradation to take place, which are moisture content and temperature as the study's parameter for a period of 10 days. It is found that the soil's different moisture content does not take any major part in the degradation of the starch complex as degradation takes place slowly and evenly throughout the experiment whereas a high temperature environment allows the starch complex to degrade faster. It then degrades steadily and slowly after Day 2.

However, an analysis shows that the nitrogen release by the starch complex film in semi-solid state under the two parameters is below 2%. The results might have been affected by the semi-solid samples, short time frame of sample's collection and lack of microorganisms' presence.

ACKNOWLEDGEMENT

First and foremost, I would like to express my deepest gratitude to Dr. Nurlidia bt. Mansor, whom is my Final Year Project (FYP) supervisor, for her valuable guidance and advice along the duration of this project. During this period, her willingness to both guide and motivate me allows the project to run smoothly and reaches its objectives. Besides that, my deepest appreciation goes to the FYP coordinator, Dr Nurhayati Mellon, for providing sufficient information that is required to complete this project. My sincere thanks to the Chemical Engineering Department of Universiti Teknologi PETRONAS (UTP) for providing me a great opportunity to undertake this project that uses the knowledge and skills learned throughout the 4 years of studies in the university. I also would like to thank the lab technologists and graduate assistant from the Chemical Engineering Department for assisting me throughout this project and special thanks to Mr. Zahid Majeed who provides valuable information and necessary helps that I need for the project. Last but no least, an honourable mention goes to everyone who directly and indirectly helps and supported me during the time working on this project.

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ABBREVIATIONS AND NOMENCLATURES

ASA	Alkenyl Succinic Anhydride
CHNS	Carbon-Hydrogen-Nitrogen-Sulfur
CRF	Controlled-Release Fertilizer
FTIR	Fourier Transform Infrared
PVA	Polyvinyl Alcohol
SUB	Starch-Urea-Borate
WFPS	Water Filled Pore Space

CHAPTER 1
INTRODUCTION

1.1 Background Study

1.1.1 CRF

A controlled-release fertilizer (CRF) is defined as an insoluble, granulated fertilizer that releases its nutrient gradually into the soil. Since a CRF is not water-soluble, the nutrients will not be able to disperse quickly into the soil as compared to the conventional fertilizers. Its granules can prevent the dissolution while slowly allowing the nutrients to flow outward. Such fertilizer can normally be prepared by coating the fertilizer granules with a thin film or by mixing the fertilizer with a medium to form a single phase or matrix and thus reducing its dissolution rate (Han, Chen, & Hu, 2009). Most of the CRF are using a biodegradable material as the coating material due to its environmental friendly properties.

1.1.2 Biodegradable Material

As biodegradation is defined as the chemical dissolution of the materials by bacteria or any other biological means, thus biodegradable materials could be of any organic materials such as plant and animal matter, and other substances originating from living organisms, or of artificial materials that are similar to the organic materials and can be put to use by the microorganisms. One of the cheapest available biodegradable materials in the market is starch.

1.1.3 Starch

Starch is a carbohydrate consisting of a large number of glucose units joined by the glycosidic bonds. It is in the carbohydrate organic compounds class with the carbon, hydrogen and oxygen ration of 6:10:5 ($C_6H_{10}O_5$). Starch is a glucose polymer with the linkages between glucose units that are formed during condensation. It is consist of two types of molecules, which are the linear amylose and the branched amylopectin, and this makes it can be considered as a crystalline material. Starch is commonly found in the human diet, as it is contained in large amounts in the staple food (Eliasson, 2004).

As a natural polymer, starch is available in large amounts from several renewable plant sources, and it can be produced in abundance beyond market availability (Das et al., 2009). It is the cheapest available biopolymer and it is a completely biodegradable polysaccharide. However, starch itself can not be satisfactorily used as a coating material due to its hydrophilicity and brittleness which will lead to its poor mechanical properties (Kweon et al., 2000). Thus, as the consequence, it needs a chemical modification to overcome such drawbacks.

1.1.4 Starch Complex

The material used in this project is a starch complex mixture of starch-urea-borate (SUB) (Sarwono, Man, & Bustam, 2013). The tapioca starch is chemically modified with urea in the presence of borate. Urea as the fertilizer produces nutrients necessary for the plant growth such as nitrogen. Urea contains high nitrogen content (46%) and has comparatively low cost of production and is most widely used as fertilizer (Chen et al., 2008). The utilization efficiency and plant uptake of the urea is generally low due to surface runoff, leaching and vaporization, where nitrogen released from the conventional fertilizer escapes to the environment. This causes a huge economic and resource losses as it cannot be absorbed by the crops, and also serious environmental pollution issues.

1.2 Problem Statement

Urea will be completely coated with the tapioca starch, where it will act as a protective layer for the urea. This layer of starch will be biodegraded first before the urea is exposed to the soil environment and starts to release its nutrient into the soil. This process will set a time delay or control the release of nutrient from the fertilizer and hence provide sufficient time for the crops to absorb the nitrogen. It will help in reducing the environmental pollution as well.

However, there is lack of information on the biodegradability of this starch complex, especially it's exposure to the surrounding moisture and temperature. Since the starch will act as a medium to delay the fertilizer's release, thus it is important to determine how the condition of moisture and temperature can affect its rate of biodegradation, before the nutrients from the fertilizer will be released into the soil.

1.3 Objective

Basically, there are three objectives that need to be achieved in this study. They are:

- To determine the influence of moisture content of the soil on the starch complex
- To analyse the influence of the temperature on the starch complex
- To investigate the potential of starch complex as a coating material on urea as fertilizer

In order to achieve the first and second objective, the starch complex will be buried into the soil with different moisture content and temperature in this study. The data and results obtained from this study will be used to determine its biodegradation rate when expose to such parameter, and thus further investigate its potential as a coating material on the urea and as a controlled-release fertilizer, and thus completing the third objective for the study.

1.4 Scope of Study

The scope of this study is basically consisting of preparing the tapioca starch and the starch-urea-borate mixture as our starch complex for the experiment. The experiment will be conducted on both starches in order to determine how different moisture content and temperature of the soil would affect its biodegradation. In the first part, the degradability of the tapioca starch will be studied and the result will be used for comparison with the degradability of the starch complex, and hence determining its suitability as coating material.

In addition, for the second part, the degradability of the starch complex under the parameters of study will be investigated accordingly. The results can be used to understand how each different parameter affects its degradation rate and also what is the best condition will be for the starch complex in order to degrade over time.

Lastly, the ammonia release activities of the urea from the starch complex will be investigated. Since the source of the information available regarding the degradability of the starch complex is limited, this study is considered as an innovative approach in order to provide a better understanding and parameter control for the starch complex.

CHAPTER 2
LITERATURE REVIEW

2.1 Urea as Fertilizer

Fertilizer is the vital input material for the sustainable development of crop production. They are added into the soil to release nutrients necessary for the plant growth (Akelah, 1996).

However, due to the various environmental and economic drawbacks associated with the use of conventional fertilizer, it turns into a focus of worldwide concern (Ni, Liu, & Lu, 2009). Thus, both controlled- and slow-release fertilizers are used as they show potential and advantages over the conventional types, such as decreasing the fertilizer loss rate, supplying nutrient sustainably, lowering application frequency, and minimizing potential negative effects associated with over dosage. Thus, coated fertilizer can be prepared by coating granules of conventional fertilizers with various materials to reduce their dissolution rate (Al-Zahrani, 2000) and thus, making the coated fertilizers as the major category of controlled- and slow-release fertilizers (Ibrahim & Jibril, 2005). And as compared to the controlled-release fertilizers, the conventional fertilizers should be avoided as it causes high nutrient concentrations in the runoff water and also increases the total nutrient runoff in the vegetated roofs system (Emilsson et al., 2007).

Among all nitrogen fertilizers, urea is one of the most widely used due to its high nitrogen content and comparatively low cost of production. However, the degree of utilization of such fertilizers for its nitrogen content is in the range below 50%

(Ibrahim & Jibril, 2005). However, as a neutral organic molecule, urea cannot be absorbed easily by the charged soil particles before hydrolyzing. Thus, this results in a great quality of urea running off and causing serious environmental hazards.

As urea is very water-soluble, one of the methods used to obtain the controlled-release of the fertilizer is by controlling the solubility of the fertilizer itself (Ramirez-Cano et al., 2001) such as using the urea to react with various aldehydes to reduce the solubility of the material. Though, another method to regulate the plant nutrient release is by applying coating.

It is no surprise that many would prefer controlled-release fertilizers as an alternative method to improve the nutrients use efficiency while minimizing the environmental hazards.

2.2 Controlled-release Fertilizer

Controlled-release fertilizers (CRF) are made to release their nutrient contents gradually and if possible, to coincide with the nutrient requirement of the plant. Its physical barrier is used to reduce their dissolution rate which is normally being prepared by encapsulation of water soluble granular plant nutrients such as coating or matrix formation, with low permeability of hydrophobic membranes (Hauck, 1985). One of the pre-requisites of producing well acting CRFs is to use high quality granular fertilizers that are regularly shaped and have surfaces that are as smooth as possible (Shaviv & Mikkelsen, 1993). The coating of active soluble component with a membrane as a diffusion barrier would create a good CRF. This physically prepared CRF by coating fertilizer granules with various materials are the major categories for the controlled- and slow-release fertilizers (Tomaszewska, Jarosiewicz, & Karakulski, 2002).

2.2.1 Characteristics

The rate of release of the CRFs, the pattern and reproducibility are reasonably governed by a proper choice of few control parameters such as fertilizer type, thickener type, thickener concentration in the dry mixture and the geometry of the device and its opening (Shavit et al., 1997). The ability to control both the rate and the time pattern of the release through different combination of the parameters is demonstrated and it differs for different parameter involved. Besides the mentioned parameters, the coating materials can be classified as a function of their properties such as liquid barrier and water vapour for choosing the best material for coating purpose (Devassine et al., 2002).

However, it is noted that the variation in the characteristics of the polymers in terms of physical and chemical properties can be utilized in producing controlled release compound fertilizer that will fit the requirements of the growing plants involved (Hanafi, Eltaib, & Ahmad, 2000).

2.2.2 Coating Materials

There are many materials that have been reported to be used as coatings, such as polysulfonate (Jarosiewicz & Tomaszewska, 2003), polyvinyl chloride (Hanafi, Eltaib, & Ahmad, 2000), and polystyrene (Liang & Liu, 2006). However, it was found that after the release of the fertilizers, these remaining coating materials in the soil are very difficult to be degraded and hence, it could accumulate over time to become a new type of pollution.

As compared to these biopolymers, lignin is amorphous and a relatively hydrophobic material and there are plenty of patents using combination of urea and lignin for controlled release coatings. There are controlled release formulations of urea with the combination of lignin and ethylcellulose (Fernandez-Perez, et al., 2008). The use of bioplast despersions lignin with an addition of alkenyl succinic anhydride (ASA) as the hydrophobic compounds and crosslinker decreases the release of urea in water, though the complete release of urea occurred within an hour (Mulder, et al., 2011). Thus, further research is necessary to improve the coating process and quality for lignin.

2.3 Starch as Coating Material for CRF

Starch, an easily biodegradable polymer, cannot be used as a packaging material due to its hydrophilic character. There are researches done to modify the starch to improve such characteristic. The modified starch products are found to be an effective new material for encapsulating water-soluble urea as fertilizer for controlled release from few hours to 1 day (Chen et al., 2008).

Starch can also be blended with other biodegradable material to be used as a coating material for fertilizers. A starch/polyvinyl alcohol (PVA) blend film could biodegrade in the soil environment where the water absorbency and permeability of the films increased along with the PVA content while decrease in starch content affect its compatibility (Han, Chen, & Hu, 2009).

It can also be mixed with the biodegradable polymers as a coating formulation. The polysulfone-starch mixture for coating can be applied effectively for CRF formulation as the increase of starch concentration in the film forming solution causes the increase in the coatings porosity and enhances the hydrophilicity (Tomaszewska & Jarosiewicz, 2004). Thus, it is found that the presence of starch in the fertilizer coating results in an increase of the water diffusion rate to the granule inwards and minerals solution outwards.

2.4 Soil Moisture

Over a certain period of time, emission of gases such as nitrous oxide and nitric oxide which results from soil microbial activity where it is produced via nitrogen-based fertilizer. Soil moisture could be one of the factors that control the gas emission rate from the soil. As the water filled pore space (WFPS) percentage increases, there was a positive relationship between nitrous oxide emission and soil water content but a negative relationship for nitric oxide for the non-coated and coated urea treatments (Hou et al., 2000). The significant effect on the two gases emission from the soil moisture indicates it can control the emission after the nitrogen fertilization from the urea. Nitrogen production occurred at the highest soil moisture level at more than 90% WFPS is observed (Ruser et al., 2006).

2.5 Soil Temperature

One of the factors that will influence the degradation of the starch is the temperature of the soil. It is found that heat treatment generally increased the rate of starch degradation at higher temperature (Sveinbjornsson, Murphy, & Uden, 2007). The reactivity of different starch is found to be different in a thermal analysis of rising temperature method of evaluation (Aggarwal & Dollimore, 1996), and this can be used to differentiate the origin of the starch. However, it also requires higher temperature in order to increase the reactivity.

CHAPTER 3
METHODOLOGY

3.1 Flow of the Experiment

The general flow of the whole experiment is described in the Figure 3.1 as follow:

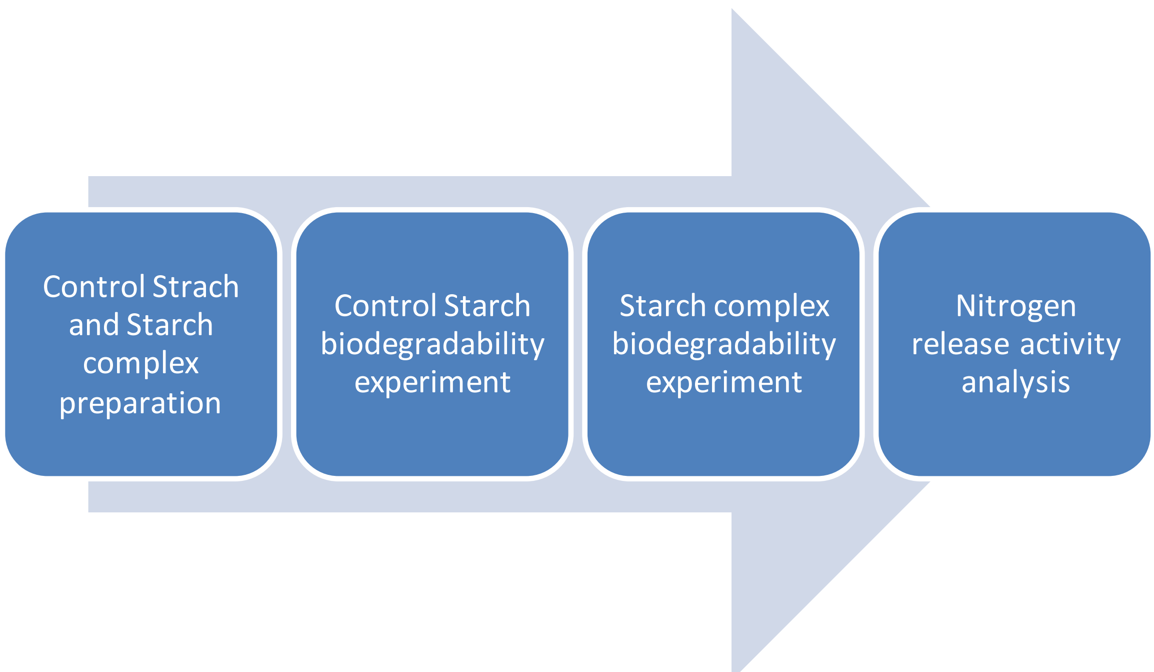

Figure 3.1 Flow of the Experiment

According to the Figure 3.1, the experiment is commenced with the preparation of the control starch and starch complex in the form of film for the experiment purpose. In the first part of the experiment, control starch which consists of tapioca starch is being used. A set of control starch films will be placed under two different parameters which are the soil's moisture content and temperature to determine its biodegradability under such condition. In the second part of the experiment, the control starch will be replaced by the starch complex, which is a mixture of tapioca starch and urea, and this set of starch complex will undergo the same conditions in the first part of the experiment to study its degradability. Then the nitrogen release activity by the starch complex in the second part of the experiment will also be analyzed.

3.1.1 Materials Used

The materials to be used for starch complex and experiment purpose are:

- Tapioca Starch
  - It is a food grade starch.
  - It is already commercially available in the market.
- Urea
  - It is the fertilizer used in the starch complex.
  - It is responsible for the nitrogen release.
- Borate
  - It acts as a cross-linker and catalyst in the starch complex.

3.1.2 Preparation of Starch Complex

The following are the general step-by-step methods in order to prepare the starch complex (Sarwono, Man, & Bustam, 2013).

1. An aqueous solution of tapioca starch will be prepared with the concentration of 5% of the total weight of the solution.

Figure 3.2 An aqueous solution of starch

2. The starch solution will then be stirred and gelatinized at 80^oC for 30 minutes.

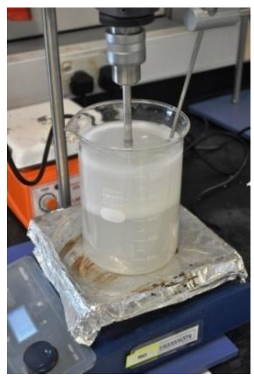

Figure 3.3 Stirring process on hot plate

3. The colour of the solution will change from white into opaque indicating of it being fully gelatinized.
4. Both the urea and borate will then be added into the gelatinized starch and this mixture will be stirred for another 3 hours at temperature of 80^oC.

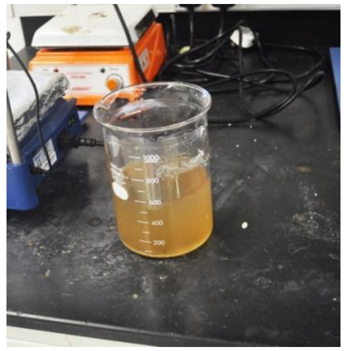

Figure 3.4 Gelatinized SUB solution

5. The solution samples will be poured onto a flat container (Refer to Appendix 3.1) with the weight of about 200g each (Refer to Appendix 3.2) and it will be dried overnight in an oven at the temperature between 40^{o}C and 50^{o}C.
6. The dried samples will then be further dried at 120^{o}C in the oven for 2 hours.
7. The starch complex consisting mixture of starch, urea and borate is prepared.

Figure 3.5 SUB dried films

The prepared starch complex will be cut into circle-shape pieces for the both parts of the experiment. The remaining and unused starch complex should be stored and kept dry from the surrounding in a container to avoid it being biodegraded.

3.1.3 Experimental Procedure

The experiment will begin by preparing the control starch and the starch complex in the form of films in the shape of a circle. Firstly, the soil that will be used in this study will be oven-dried at 120^{o}C and weight until constant weight is obtained, in order to fully eliminate any possible water and moisture contained within the soil. The soil will then be placed into a small container acting as a pot in the study.

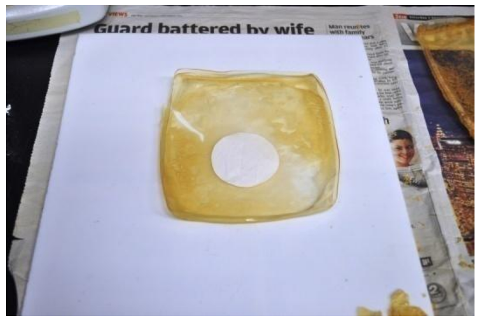

Figure 3.6 SUB film and the circle reference

The film shall be placed between two pieces of dark cloth in order to better differentiate the degraded sample from its surrounding. And since this study contains two different parameters, thus two different sets of experimental work are needed to be prepared and carried out.

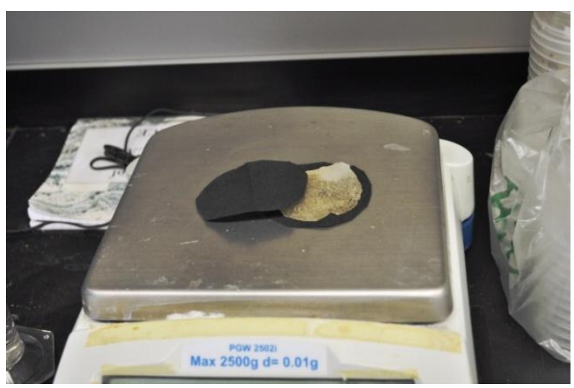

Figure 3.7 Film is placed between two dark clothes and weighted

(a) Experiment on the different moisture content of the soil

- Different water percentage will be added onto the soil, based on the net weight of the soil after being oven-dried and at constant weight, producing 2 sets of sample for each starch.
- The control starch and the starch complex is a form of film will then be buried into the soil and will be placed inside a box at room temperature condition.
- After a period of 24 hours, the samples will be taken to measure the rate of biodegradation in terms of weight loss for both the control starch and starch complex.
- Samples will be taken each day for the duration of 10 days to obtain the weight loss data.

Figure 3.8 Pots are kept inside a box at room temperature condition

(b) Experiment on the different temperature of the soil

- The same set of soil from the previous experiment will be reuse in the second part of the experiment but with the use of new control starch and starch complex.
- These samples will then be placed into the oven and its temperature shall be set and maintain at $40^{o}C$ throughout the experiment.

- After a period of 24 hours, the samples will be taken to measure the rate of biodegradation in terms of weight loss for both the control starch and starch complex.
- Samples will be taken each day for the duration of 10 days to obtain the weight loss data.

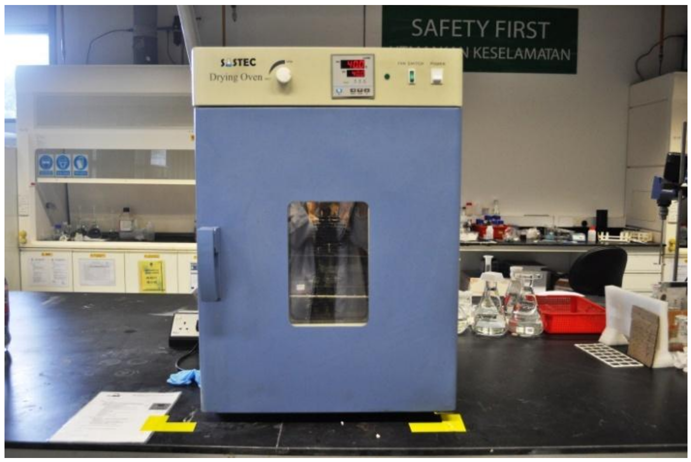

Figure 3.9 Pots are kept inside the oven at 40^{o}C

(c) Nitrogen contain analysis

- The samples containing starch complex from both experiments of different moisture content and different soil's temperature will be taken in order to determine the nitrogen release activity.
- Samples will be taken each day for the duration of 7 days to determine the nitrogen release activity by the starch complex.

Two sets of data will be collected from each set of both the parameters for drying and they will be measured and an average value will be obtained as the final result. All the data shall be taken every 24-hour for 10 days and the rate of biodegradation for both starches will be compared. In addition, the sample containing the starch complex will be used to analyze the nitrogen released by fertilizer.

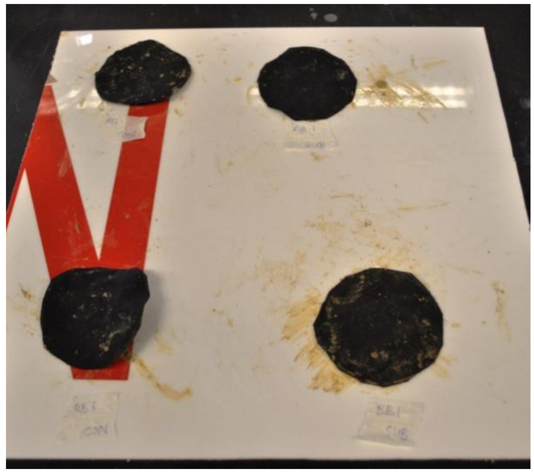

Figure 3.10 Samples after an overnight drying

3.1.4 Experiment Matrix

There are 3 experiment matrices that will be used in this study. As for the first part of the experiment, the samples of two different moisture content of the soil will be placed in a box in a room for the constant temperature, which is the room temperature.

Table 3.1 Experiment Matrix 1

Rate of Biodegradation at T = 25^{o}C	Time Frame of Data Measurement	Moisture Content			
		40%		60%	
		Control Starch	Starch Complex	Control Starch	Starch Complex
	0-hour				
	24-hour				
	48-hour				

Based on Table 3.1, the moisture content of the soil would be of 40% and 60% of the net weight of the oven-dried soil at the temperature of 25^{o}C. The sample will be taken every 24-hour for measurement for 10 days and the data will then be used for comparison.

Table 3.2 Experiment Matrix 2

Rate of Biodegradation at T = 40^{o}C	Time Frame of Data Measurement	Moisture Content			
		40%		60%	
		Control Starch	Starch Complex	Control Starch	Starch Complex
	0-hour				
	24-hour				
	48-hour				

According to Table 3.2, the temperature of the soil will be changed from 25^{o}C to 40^{o}C while maintaining the moisture content of the soil. Similar to previous matrix, the sample will also be taken every 24-hour for measurement for 10 days and the data will then be used for comparison.

The rate of biodegradation for both starches in both the experiment can be determined and calculated via the weight loss of the starches as this is one of the main parameters used (Muthukumar, Aravinthan, & Mukesh, 2010).

Table 3.3 Experiment Matrix 3

The Nitrogen Release Activity Analysis	Time Frame of Data Analysis	Starch Complex			
		Moisture Content = 40%		Moisture Content = 60%	
		T = 25^{o}C	T = 40^{o}C	T = 25^{o}C	T = 40^{o}C
	0-hour				
	24-hour				
	48-hour				

Table 3.3 shows that the experiment matrix used for the analysis of nitrogen release activity of the starch complex for both the parameters. Since the nitrogen release activity

is being controlled and delayed due to the starch coating, it is important to determine the time frame for its first day of nitrogen release.

3.1.5 Analytical Equipment

In order to determine the rate of biodegradation of the tapioca starch and the starch complex and the nitrogen release activity by the latter, it is important to use the suitable equipment that will provide desirable information for the study. Thus, the analytical equipments involved in this study are:

- Weighting machine
  - Weighting machine will be used to measure the weight of the soil before and after oven-dry for characterization of the soil for moisture content.
  - It will be used to measure the initial mass of the tapioca starch and the starch complex before the experiment commences.
  - It will also be used to determine the rate of biodegradation of both starches.
  - This can be calculated via the weight loss of the starches according to the following formula:

$$Rate\ of\ Biodegradation = \frac{|Weight\ measured\ after\ X\ day - Initial\ Weight\ measured\ |}{Initial\ Weight\ measured}$$

- CHNS Analyzer
  - CHNS analyzer can be used to determine the carbon, hydrogen, nitrogen and sulfur content of a sample.
  - It will be used to determine the nitrogen content of the starch complex for this study.

CHAPTER 4
RESULTS AND DISCUSSION

4.1 Preparation of Control Starch and Starch Complex

In this study, each batch of starch complex is prepared by using 1000mL of ionized water added into 50g of tapioca starch. Once the starch solution is completely gelatinized, 4.5g of borate and 20g of urea will be added into it and will be continue stirred at 80^{o}C.

This batch of mixture would produce approximately 800g of solution at the end of the stirring process, which it will produce 4 sets of 200g solution in the flat container. The lost in weight of the solution mixture is due to the vaporization to surrounding. However, this solution could suffer more weight loss due to the inaccuracy of the thermostat used with the hot plate resulting in higher temperature being maintained in the stirring process and thus, allowing more solution being vaporized.

After the 4 sets of 200g solution are completely dried, it would produce a film weighing between 14g and 20g for each set. This film will further be cut into 4 or 5 pieces of round shape circle per set that could fit into the pot. Each pieces of film weighing between 1g and 2g.

The same procedures are repeated for the preparation of control starch without the addition of urea into the gelatinized starch solution. However, it only could produce the control starch film piece weighting between 0.5g and 1.3g. This is due to the noninvolvement material urea and also the vaporization that takes place during the stirring process.

4.2 Experiments at Room Temperature with Different Moisture Content

Table 4.1 Sample's Weight Lost Data at 25^{o}C and 40% Moisture Content

Day	Control Starch	Starch Complex
	Average Weight Lost (%)	
1	2.9571	10.3014
2	29.0198	38.7163
3	35.0219	54.4479
4	71.9356	71.8625
5	75.8376	78.2289
6	78.6149	80.2853
7	77.1757	80.8336
8	79.2293	84.1537
9	86.6712	87.0892
10	88.6284	88.9753

Table 4.2 Sample's Weight Lost Data at 25^{o}C and 60% Moisture Content

Day	Control Starch	Starch Complex
	Average Weight Lost (%)	
1	6.5178	13.1432
2	35.1844	49.2673
3	62.2577	63.3772
4	65.0124	69.1577
5	68.3762	74.9149
6	73.3524	77.2688
7	77.7254	82.7576
8	80.2562	84.6369
9	87.4268	85.6367
10	90.4822	89.2860

An average weight lost of the sample is calculated as shown in the table 4.1 and table 4.2 above (Refer to Appendix 4.1 and Appendix 4.2 for the detailed data). The pots containing the sample were placed inside a box in a room with the temperature set to 25^{o}C. The dark clothes storing the film in it is removed from the respective pot daily and will then put for an overnight drying at 40^{o}C to eliminate the water absorbed by both the clothes and film. The dry sample will then be weighed and the reading obtained is recorded.

4.3 Experiments at higher Temperature with Different Moisture Content

Table 4.3 Sample's Weight Lost Data at 40^{o}C and 40% Moisture Content

Day	Control Starch	Starch Complex
	Average Weight Lost (%)	
1	5.2226	19.5771
2	13.3412	56.0521
3	74.6381	91.6065
4	79.7425	93.3579
5	81.3720	94.1905
6	85.0085	95.5313
7	85.4011	94.6495
8	85.7134	93.5013
9	85.2377	95.1907
10	85.7660	95.6960

Table 4.4 Sample's Weight Lost Data at 40^{o}C and 60% Moisture Content

Day	Control Starch	Starch Complex
	Average Weight Lost (%)	
1	15.5212	19.5265
2	52.1519	86.7333
3	65.5293	87.2662
4	66.6812	87.0205
5	67.9731	88.1427
6	71.2646	89.5295
7	73.5708	88.5968
8	73.8489	89.8222
9	74.7190	92.2950
10	75.6690	91.9827

In the second part of the experiment, all the pots filled with wet soil of the fixed moisture content are reused and placed inside an oven. The temperature is then set to 40^{o}C throughout the duration of the experiment. The average weight lost of the samples this part of the experiment is shown in the Table 4.3 and Table 4.4 above (Refer

Appendix 4.3 and 4.4 for detailed data). This data will be used to plot a graph to show its degradation rate.

4.4 Control Starch and Starch Complex Degradation Rate Analysis

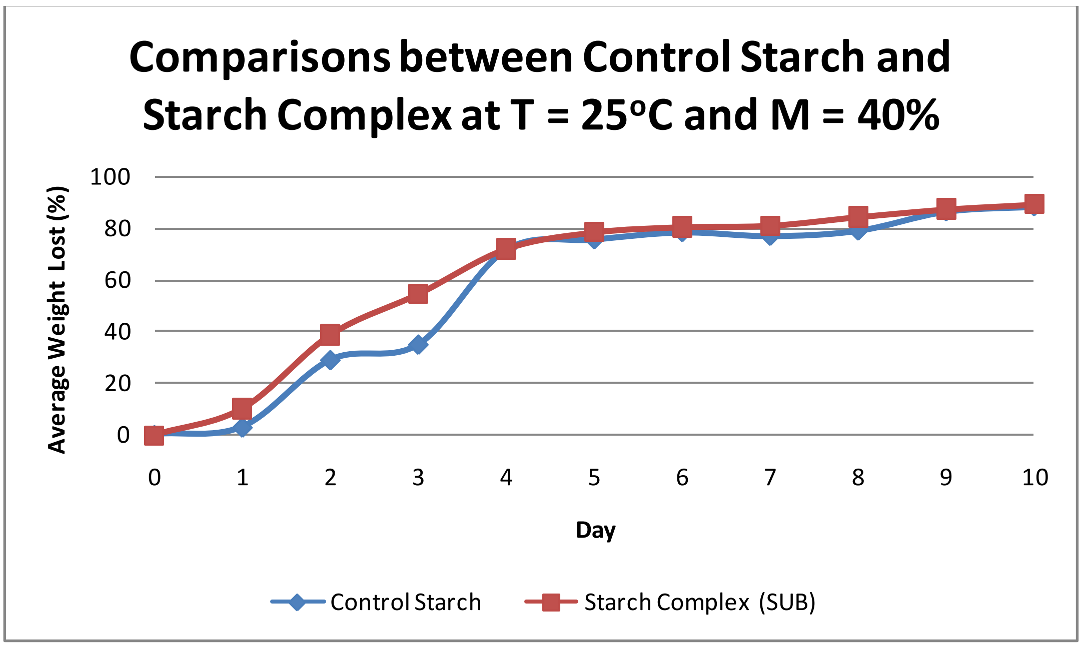

Figure 4.1 Comparisons between Control Starch and Starch Complex at T = 25°C and M = 40%

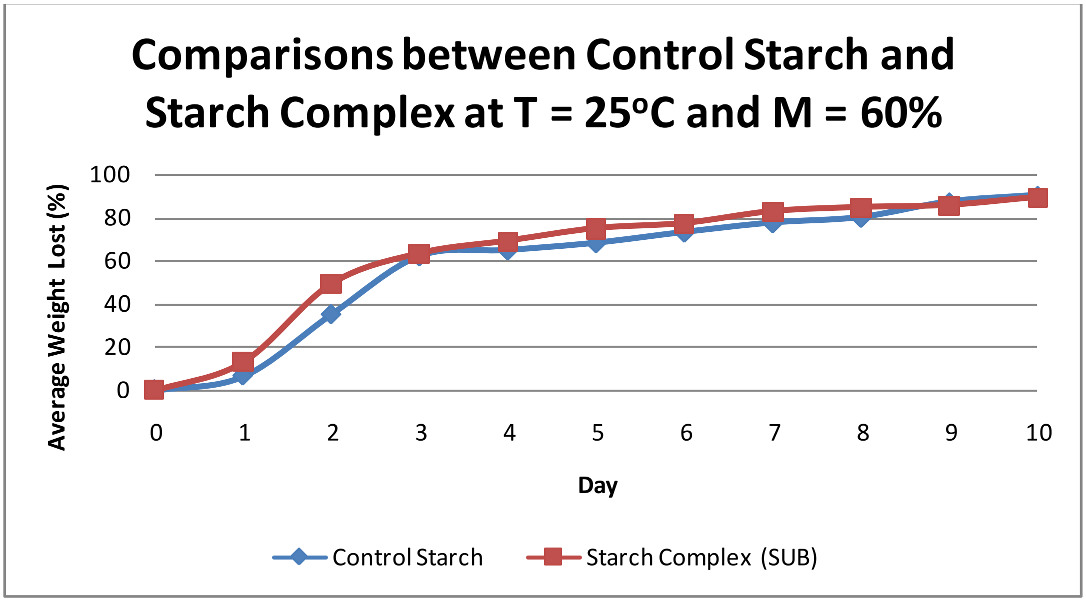

Figure 4.2 Comparison between Control Starch and Starch Complex at T = 25°C and M = 60%

Figure 4.1 above shows the comparisons between control starch and starch complex at temperature of 25°C with 40% soil's moisture content. The degradation rate of both the control starch and starch complex shows a similar trend of increase in percent of average weight lost over day.

When the soil's moisture content is increase to 60% while maintaining the temperature at 25°C, similar trend is observed in Figure 4.2, which is the increase in the average weight lost percentage

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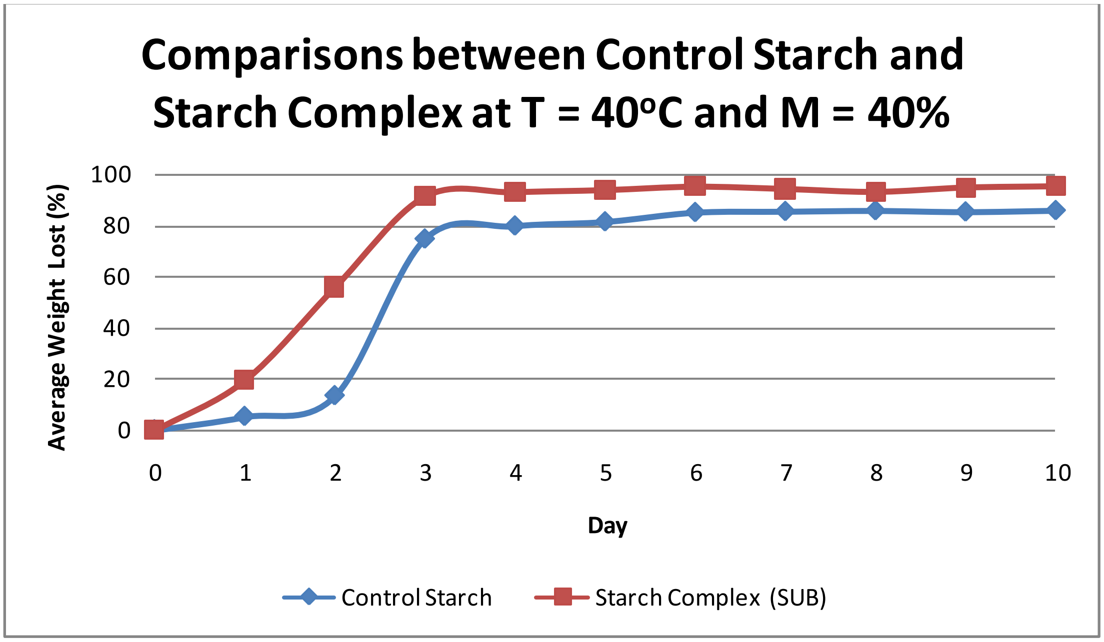

Figure 4.3 Comparison between Control Starch and Starch Complex at T = 40^{o}C and M = 40%

As the samples are placed inside an oven for the temperature at 40^{o}C with 40% soil's moisture content, the degradation pattern shows a significant difference between both the starches despite having the same trending line, as seen in Figure 4.3.

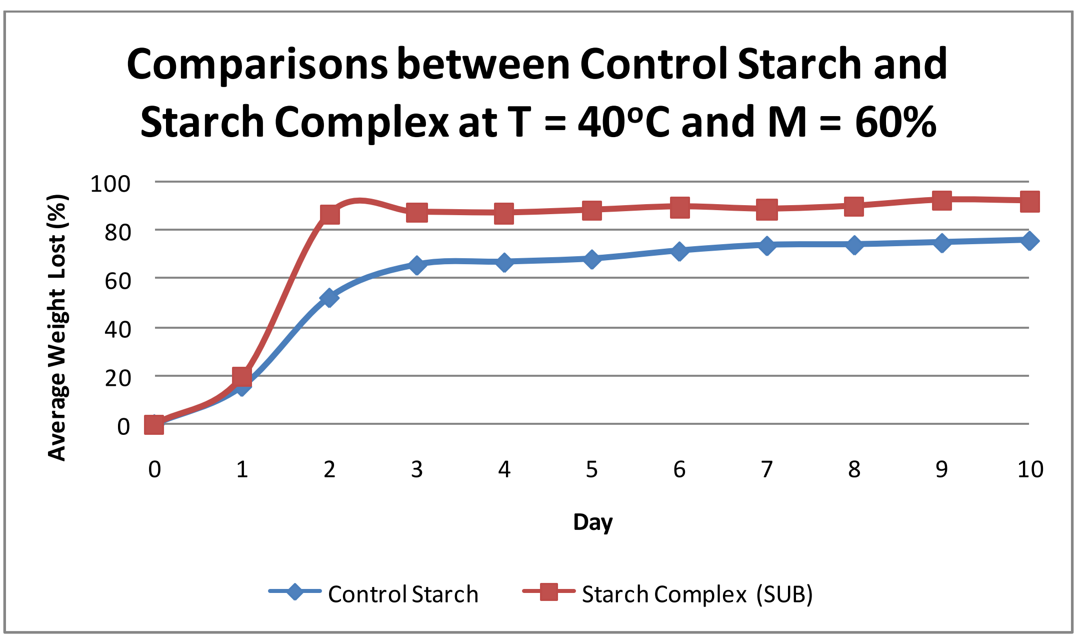

Figure 4.4 Comparison between Control Starch and Starch Complex at T = 40^{o}C and M = 60%

By changing the moisture content to 60% while maintaining the temperature at 40°C, both starches show the similar degradation rate trend, while having an obvious difference between them, as observed in the Figure 4.4 above.

The similarity of the degradation rate line between the control starch and the starch complex, as shown in Figure 4.1 and Figure 4.2, shows that the rate of degradation is not affected by the addition of urea into the starch mixture. Although the degradation rate is showing a significant difference in the Figure 4.3 and Figure 4.4, but both the degradation rate line is having the same trend. The difference in the rate of degradation only occurred due to the large difference of the films' weight used in this part of the experiment.

4.5 Starch Complex Experimental Analysis

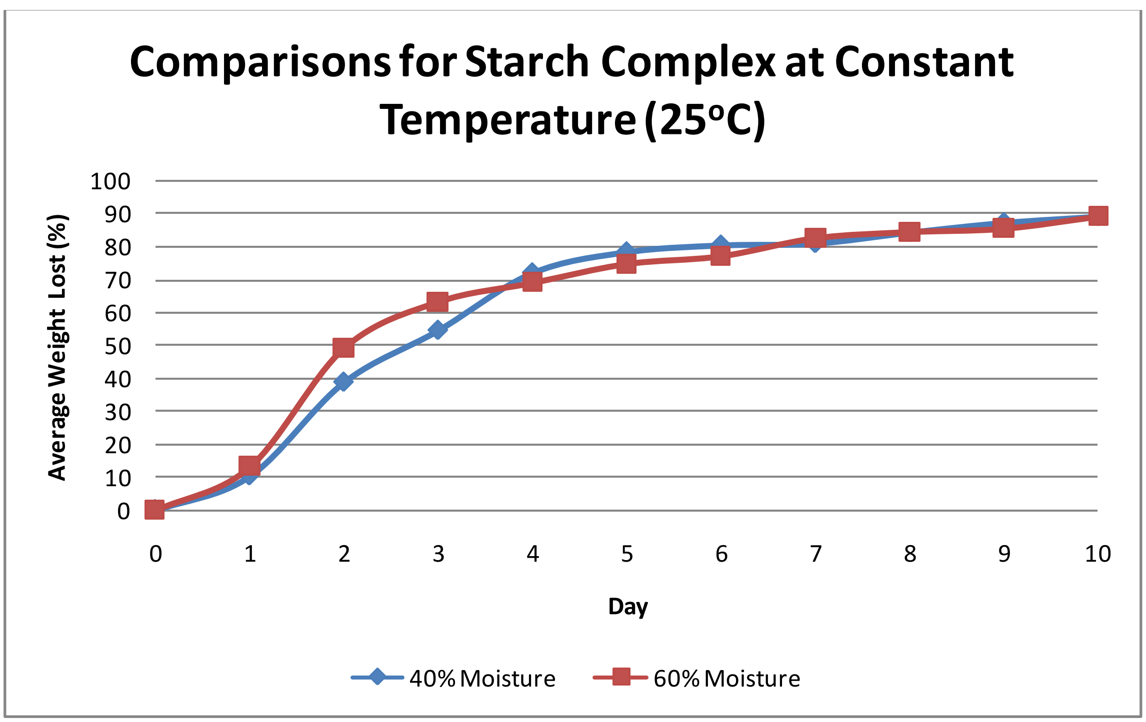

Figure 4.5 Comparisons for Starch Complex at Constant Temperature (25°C)

Figure 4.5 shows the degradation rate of starch complex at the constant temperature of 25°C with 40% and 60% of soil's moisture content respectively. On the first 4 days, it is observed that the samples with higher moisture content tend to degrade faster compared with lower moisture content's samples.

Despite having higher rate of degradation for the samples with 60% of moisture content, it seems that the difference of the amount of water present in the soil will only affect the starch complex's degradation rate as early as Day 4. From Day 5 onwards, the degradation rate increases slowly but steadily, with both sets of samples having a similar degradation rate.

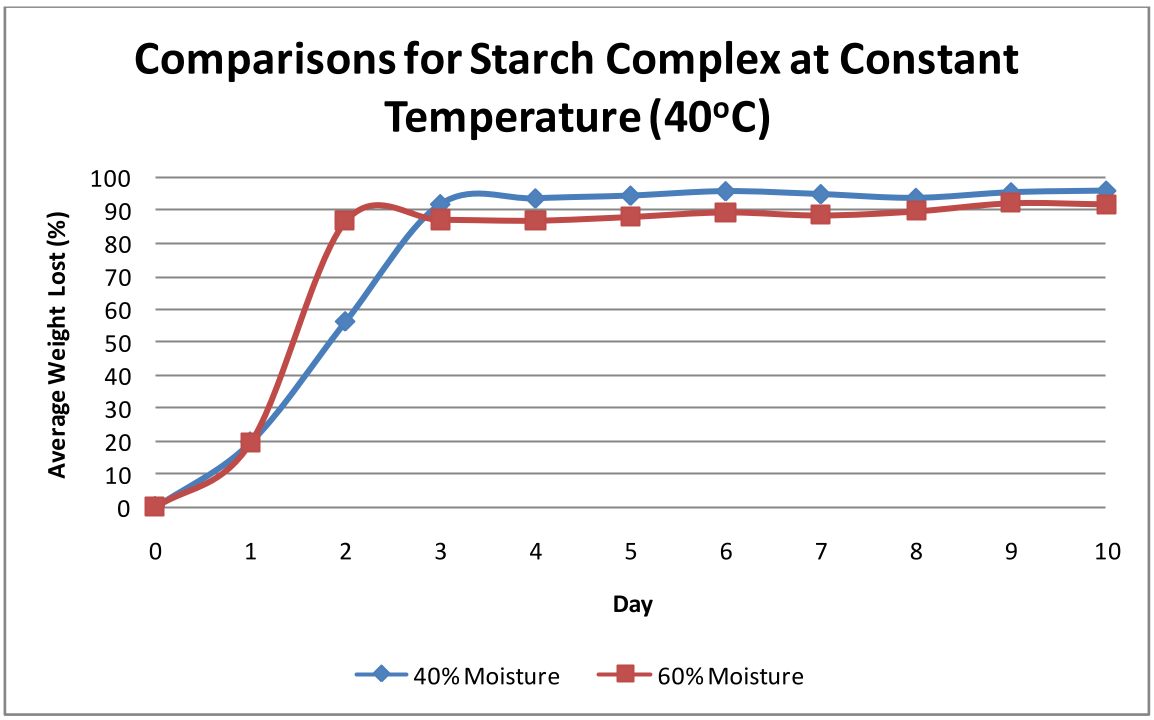

Figure 4.6 Comparisons for Starch Complex at Constant Temperature (40°C)

Figure 4.6 shows the rate of degradation for starch complex at a constant temperature of 40°C with two different moisture contents, which are 40% and 60%. Similar to Figure 4.5, the rate of degradation is faster with 60% soil's moisture content on the earlier part of the experiment. By Day 2, samples with 60% soil's moisture content give a higher degradation rate. And as for the remaining days on the experiment, the degradation rate for both samples remains almost constant and only increases slightly over time.

Despite having the similar pattern, the degradation rate showed by the 60% moisture content samples is higher during the earlier part of both experiments as it provides a suitable environment for the microorganisms to decompose the fertilizer to produce nitrogen. The degradation rate for the starch complex is not much affected by the moisture content of the soil as both experiments have the similar degradation rate at Day 10. However, the emission of nitrogen of the starch complex may be affected by the soil's different moisture content where higher WFPS will have higher nitrogen production and release (Ruser et al., 2006).

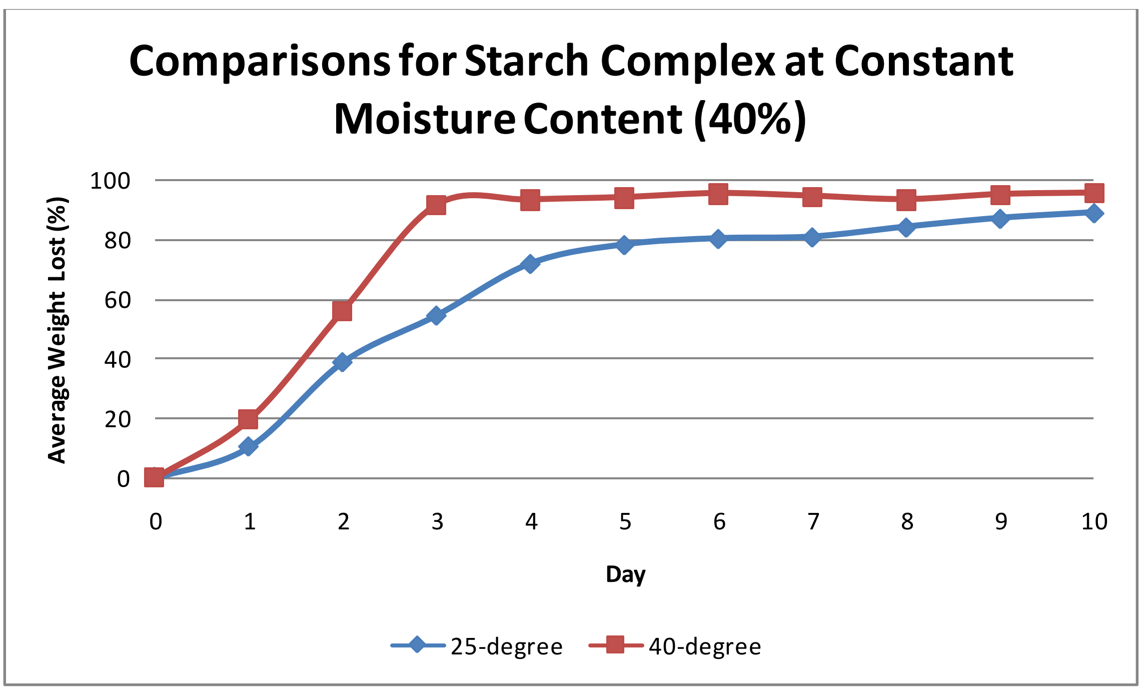

Figure 4.7 Comparisons for Starch Complex at Constant Moisture Content (40%)

Figure 4.7 shows the degradation rate of starch complex at the constant soil's moisture content of 40% at temperature 25°C and 40°C respectively. It is observed that the degradation rate for the samples at 40°C is higher than the samples at 25°C throughout the experiment period.

It is also observed that by Day 3, the degradation rate for the samples at 40°C reaches its maximum degradation rate and continue degraded slowly for the remaining days of the experiment. The samples at 25°C however require 10 days to reach the same degradation rate of 40°C samples at Day 3.

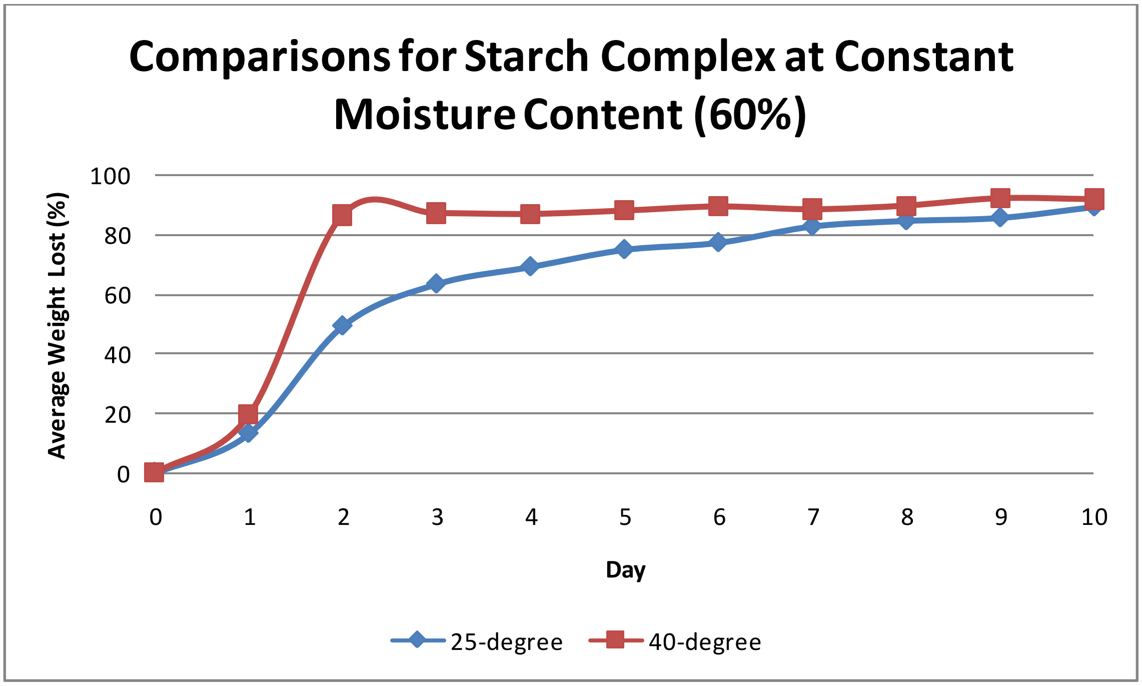

Figure 4.8 Comparisons for Starch Complex at Constant Moisture Content (60%)

The degradation rate of starch complex at the soil's constant moisture content of 60% at the temperature of 25°C and 40°C respectively is shown in the Figure 4.8 above. Similar to the previous experiment, the samples at 40°C shows a faster rate of degradation throughout the study. However, it reaches its degradation peak by Day 2 and continues to slowly degrade for the remaining time of the experiment. On the other hand, the samples at 25°C require 8 days in order to reach the same degradation peak of 40°C samples.

Both data shows that the degradation rate of the starch complex is higher at the higher temperature (Sveinbjornsson, Murphy, & Uden, 2007) and could reach their degradation rate peak as early as Day 2. The reactivity of this tapioca starch is also found to be faster at higher temperature (Aggarwal & Dollimore, 1996) between the two temperatures and 40^{o}C could be its optimum temperature for reactivity.

4.6 Nitrogen Release Analysis

7 samples from each parameter were then taken for analysis to detect the presence of nitrogen in the starch complex. Table 4.5 shows the result of the CHNS's analysis.

Table 4.5 Starch Complex's Nitrogen Release Analysis

Time Frame of Data Analysis (Day)	Nitrogen Release (%)			
	Moisture Content = 40%		Moisture Content = 60%	
	T = 25^{o}C	T = 40^{o}C	T = 25^{o}C	T = 40^{o}C
0	0.000	0.000	0.000	0.000
1	1.854	1.464	0.814	0.624
2	0.520	1.710	0.945	0.915
3	1.113	1.153	0.808	0.599
4	1.051	0.994	0.861	0.968
5	1.101	1.213	0.821	0.815
6	0.964	0.660	1.373	0.571
7	1.282	1.420	0.958	0.639

All the samples show the presence of nitrogen in the degraded starch complex not more than 2% under any the conditions. Although the data shows that the fertilizer did not immediately release the nitrogen in a large amount within the 7 days, it shows that the decomposition of the urea was delay or rather slowed down by the presence of the tapioca starch.

Since the films were exposed to water present in the soil, it swelled and turned into a semi-solid state. This semi-solid sample does not contain an even concentration or component in it as the reactivity and degradation do not take place evenly. Thus, even by performing the analysis via CHNS to determine the presence of nitrogen in it, the results obtained will not show an increasing trend of nitrogen content in it as it can be seen in Table 4.5 above.

The starch complex might require more than 7 days to able to complete release the nutrients from the urea. As CHNS only manage to detect less than 2% of nitrogen content from the starch complex films' sample, it should be able to produce a higher amount of nitrogen since urea contains high nitrogen content (46%) in urea alone (Chen et al., 2008). Despite being chemically modified with both starch and borate, the nitrogen content wouldn't reduce to as low as only 2%. Thus, it might require a longer time period before full decomposition of urea takes place.

Besides that, as the soil was first being oven-dried at 120^{o}C, there are high possibilities that most of microorganisms inside the soil had been killed and destroyed at this temperature. The microorganisms would produce the enzyme which will decompose the urea into the nutrients needed for plant growth, including nitrogen. Since these microorganisms are responsible for decomposing the urea, thus by eliminating its presence in the soil, it is hardly for the urea to produce any nitrogen by its own as little microorganisms could survive such temperature.

CHAPTER 5
CONCLUSION AND RECOMMENDATION

This study is designed according to the three main objectives which have been set at the commencement of the study. The first objective of this study is to study the influence of the soil moisture on the starch complex, which is made of starch-urea-borate. Besides that, the effect of the temperature of the soil on the starch complex will be determined as well. While the starch complex's rate of degradation does not show any major differences while being placed in different moisture content environment, the rate of degradation of the starch complex in this study is found to show a significant difference when the samples are placed in two different temperatures, whereas the samples at higher temperature the degradation rate occurs rapidly and reached a steady rate as early as Day 2 while the samples in lower temperature will degrade slowly throughout the study's period. It took between 8 and 10 days for these samples to reach the degradation rate of the higher temperature's samples.

The third objective to be achieved in this study is to investigate the potential of the starch complex as coating material on urea as fertilizer. However, the analyzing of nitrogen release by the starch complex via CHSN shows less than 2% of nitrogen had been detected in the samples. These numbers cannot justify if the soil's different moisture content could play a role in influencing the nitrogen production from the fertilizer. Three possible factors which cause the lack of nitrogen been detected had been identified.

The semi-solid starch complex films did not degrade evenly and might not have the even nitrogen concentration in it. Secondly, the starch complex might require more than 7 days in order to fully decompose and for the urea to start producing its nutrients, including nitrogen. Lastly, it might due to the lack of microorganisms' presence in soil for urea's decomposition due to the soil's heating at high temperature in the beginning of the experiment. This high temperature heating basically killed and destroyed most of the soil's microorganisms. An alternative method could be designed and used to determine the nitrogen release activity and justify the starch complex applicability as CRF.

In order to further improve the design of this study, there are some suggested future works which can be done for expansion and continuation. Firstly, the soil used for the purpose of this study can be fully characterized so that this information can be used to further determine the factors that will affect the biodegradation of the starch complex, such as the pH value and the component of the soil. Secondly, since biodegradation of starches is done by the enzyme, it can be used as one of the parameter for the future study.

Furthermore, the starch solution can be produced with different tapioca starch concentration and different starch complex thickness can be used. Nevertheless, the time frame used for the study can be designed longer in order to detect the presence of nitrogen from the starch complex's films.

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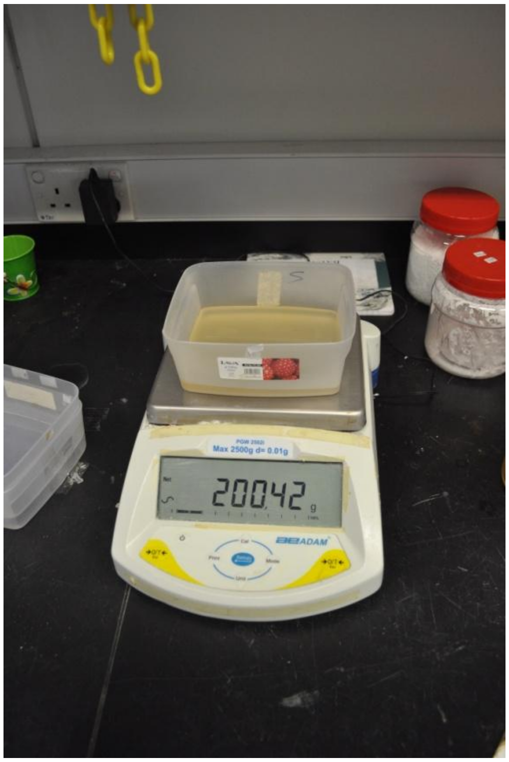
APPENDICES

Appendix 3.1

Appendix 3.2



Appendix 4.1

Day	Set	Control Starch						Starch Complex(SUB)					
		Sample Original Weight (g)	Weight of Cloth (g)	Total Weight (g)	Final Weight (g)	Weight Loss (%)	Average Losses (%)	Sample Original Weight (g)	Weight of Cloth (g)	Total Weight (g)	Final Weight (g)	Weight Loss (%)	Average Losses (%)
1	A	1.2526	0.6848	1.9374	1.8928	3.5606	2.9571	1.5217	0.6928	2.2145	2.0621	10.0151	10.3014
	B	1.0367	0.7042	1.7409	1.7165	2.3536		1.4583	0.6884	2.1467	1.9923	10.5877	
2	A	1.1084	0.7138	1.8222	1.5268	26.6510	29.0198	1.4632	0.6737	2.1369	1.6085	36.1126	38.7163
	B	0.9873	0.6853	1.6726	1.3627	31.3886		1.5075	0.7091	2.2166	1.5937	41.3201	
3	A	0.9731	0.7236	1.6967	1.3751	33.0490	35.0219	1.3817	0.6893	2.0710	1.3478	52.3413	54.4479
	B	1.1826	0.6843	1.8669	1.4294	36.9948		1.4639	0.6938	2.1577	1.3298	56.5544	
4	A	0.9847	0.6754	1.6601	1.0153	65.4819	71.9356	1.6482	0.6883	2.3365	1.1686	70.8591	71.8625
	B	0.9685	0.7032	1.6717	0.9125	78.3893		1.3448	0.7184	2.0632	1.0833	72.8659	
5	A	0.9754	0.6848	1.6602	0.9214	75.7433	75.8376	1.6826	0.6893	2.3719	1.0439	78.9255	78.2289
	B	1.0516	0.6943	1.7459	0.9474	75.9319		1.5983	0.7147	2.3130	1.0738	77.5324	
6	A	1.1742	0.6891	1.8633	0.9451	78.1979	78.6149	1.5359	0.6732	2.2091	0.9915	79.2760	80.2853
	B	0.9648	0.7261	1.6909	0.9284	79.0319		1.6375	0.6969	2.3344	1.0032	81.2947	
7	A	0.9638	0.6718	1.6356	0.8734	79.0828	77.1757	1.7234	0.6729	2.3963	1.0073	80.5965	80.8336
	B	1.0982	0.6823	1.7805	0.9539	75.2686		1.4982	0.7016	2.1998	0.9852	81.0706	
8	A	0.9546	0.7034	1.6580	0.8881	80.6516	79.2293	1.4315	0.6861	2.1176	0.9243	83.3601	84.1537
	B	1.0242	0.6627	1.6869	0.8900	77.8071		1.5532	0.6672	2.2204	0.9010	84.9472	
9	A	0.9357	0.7018	1.6375	0.8051	88.9601	86.6712	1.4057	0.6983	2.1040	0.8719	87.6503	87.0892
	B	1.1583	0.6529	1.8112	0.8338	84.3823		1.3517	0.6806	2.0323	0.8627	86.5281	
10	A	0.9529	0.6935	1.6464	0.7802	90.9015	88.6284	1.6326	0.6704	2.3030	0.8316	90.1262	88.9753
	B	0.7109	0.7252	1.4361	0.8222	86.3553		1.3527	0.6839	2.0366	0.8486	87.8244	

Appendix 4.2

Day	Set	Control Starch						Starch Complex (SUB)					
		Sample Original Weight (g)	Weight of Cloth (g)	Total Weight (g)	Final Weight (g)	Weight Loss (%)	Average Losses (%)	Sample Original Weight (g)	Weight of Cloth (g)	Total Weight (g)	Final Weight (g)	Weight Loss (%)	Average Losses (%)
1	A	0.8023	0.6934	1.4957	1.4385	7.1295	6.5178	1.3248	0.6717	1.9965	1.8038	14.5456	13.1432
	B	0.7958	0.6783	1.4741	1.4271	5.9060		1.2793	0.6983	1.9776	1.8274	11.7408	
2	A	0.5238	0.7197	1.2435	1.0739	32.3788	35.1844	1.0739	0.7073	1.7812	1.2742	47.2111	49.2673
	B	0.6378	0.6828	1.3206	1.0783	37.9900		1.2655	0.6927	1.9582	1.3087	51.3236	
3	A	0.7256	0.6853	1.4109	0.9862	58.5309	62.2577	1.1493	0.6831	1.8324	1.1481	59.5406	63.3772
	B	0.7673	0.7104	1.4777	0.9714	65.9846		1.0846	0.7027	1.7873	1.0583	67.2137	
4	A	0.8103	0.6931	1.5034	0.9674	66.1483	65.0124	1.3697	0.6936	2.0633	1.1947	63.4153	69.1577
	B	0.7646	0.6827	1.4473	0.9589	63.8765		1.2749	0.7143	1.9892	1.0343	74.9000	
5	A	0.7835	0.6893	1.4728	0.9475	67.0453	68.3762	1.0278	0.7184	1.7462	0.9992	72.6795	74.9149
	B	0.7477	0.7016	1.4493	0.9281	69.7071		1.3707	0.7016	2.0723	1.0148	77.1504	
6	A	0.7612	0.7137	1.4749	0.9027	75.1708	73.3524	1.2641	0.6907	1.9548	0.9719	77.7549	77.2688
	B	0.7184	0.6947	1.4131	0.8992	71.5340		1.1892	0.7052	1.8944	0.9813	76.7827	
7	A	0.6978	0.7247	1.4225	0.8913	76.1250	77.7254	1.2963	0.6816	1.9779	0.9273	81.0461	82.7576
	B	0.7386	0.6924	1.4310	0.8451	79.3258		1.1738	0.7115	1.8853	0.8938	84.4692	
8	A	0.7193	0.7612	1.4805	0.9094	79.3966	80.2562	1.0421	0.7151	1.7572	0.8770	84.4641	84.6369
	B	0.7403	0.6718	1.4121	0.8116	81.1158		0.7992	0.6943	1.4935	0.8157	84.8098	
9	A	0.7251	0.6673	1.3924	0.7789	84.6090	87.4268	0.9367	0.6971	1.6338	0.7923	89.8367	85.6367
	B	0.7811	0.6948	1.4759	0.7710	90.2445		0.8673	0.7105	1.5778	0.8715	81.4366	
10	A	0.7127	0.6947	1.4074	0.7215	96.2397	90.4822	1.0104	0.7037	1.7141	0.8009	90.3800	89.2860
	B	0.6887	0.6758	1.3645	0.7810	84.7248		1.1543	0.6483	1.8026	0.7846	88.1920	

Appendix 4.3

Day	Set	Control Starch						Starch Complex (SUB)					
		Sample Original Weight (g)	Weight of Cloth (g)	Total Weight (g)	Final Weight (g)	Weight Loss (%)	Average Losses (%)	Sample Original Weight (g)	Weight of Cloth (g)	Total Weight (g)	Final Weight (g)	Weight Loss (%)	Average Losses (%)
1	A	0.8324	0.6314	1.4638	1.4083	6.6675	5.2226	1.3827	0.6884	2.0711	1.8010	19.5342	19.5771
	B	0.7200	0.6899	1.4099	1.3827	3.7778		1.4261	0.6948	2.1209	1.8411	19.6199	
2	A	0.7681	0.7064	1.4745	1.3807	12.2120	13.3412	1.3452	0.6873	2.0325	1.2974	54.6461	56.0521
	B	0.7118	0.6846	1.3964	1.2934	14.4704		1.3053	0.6727	1.9780	1.2280	57.4581	
3	A	0.9982	0.7169	1.7151	1.0028	71.3584	74.6381	1.7808	0.6986	2.4794	0.8628	90.7794	91.6065
	B	1.1978	0.6892	1.8870	0.9537	77.9178		1.8820	0.7131	2.5951	0.8555	92.4336	
4	A	0.8793	0.6931	1.5724	0.8515	81.9857	79.7425	1.5609	0.6897	2.2506	0.7847	93.9138	93.3579
	B	0.8422	0.7013	1.5435	0.8908	77.4994		1.3198	0.6961	2.0159	0.7911	92.8019	
5	A	0.9706	0.7095	1.6801	0.8674	83.7317	81.3720	1.7463	0.6972	2.4435	0.7792	95.3044	94.1905
	B	1.0873	0.6873	1.7746	0.9155	79.0122		1.7015	0.6723	2.3738	0.7901	93.0767	
6	A	1.2717	0.7175	1.9892	0.8916	86.3097	85.0085	1.4552	0.7098	2.1650	0.7558	96.8389	95.5313
	B	1.1232	0.6822	1.8054	0.8652	83.7073		1.5598	0.6982	2.2580	0.7883	94.2236	
7	A	1.0940	0.6825	1.7765	0.8304	86.4808	85.4011	1.7562	0.7088	2.4650	0.8043	94.5621	94.6495
	B	0.9548	0.7052	1.6600	0.8549	84.3213		1.8126	0.7284	2.5410	0.8238	94.7368	
8	A	1.1083	0.6779	1.7862	0.8325	86.0507	85.7134	1.4679	0.7184	2.1863	0.7861	95.3880	93.5013
	B	1.1235	0.6934	1.8169	0.8577	85.3761		1.3130	0.7258	2.0388	0.8359	91.6146	
9	A	1.1336	0.6963	1.8299	0.8558	85.9298	85.2377	1.3607	0.7274	2.0881	0.7874	95.5905	95.1907
	B	1.1013	0.6830	1.7843	0.8532	84.5455		1.5780	0.7187	2.2967	0.8009	94.7909	
10	A	0.8679	0.6955	1.5634	0.7965	88.3627	85.7660	1.5258	0.6946	2.2204	0.7526	96.1987	95.6960
	B	0.7623	0.6753	1.4376	0.8036	83.1694		1.7725	0.7014	2.4739	0.7866	95.1932	

Appendix 4.4

Day	Set	Control Starch						Starch Complex (SUB)					
		Sample Original Weight (g)	Weight of Cloth (g)	Total Weight (g)	Final Weight (g)	Weight Lost (%)	Average Losses (%)	Sample Original Weight (g)	Weight of Cloth (g)	Total Weight (g)	Final Weight (g)	Weight Lost (%)	Average Losses (%)
1	A	0.6958	0.6887	1.3845	1.2715	16.2403	15.5212	1.3816	0.6991	2.0807	1.7937	20.7730	19.5265
	B	0.5810	0.6790	1.2600	1.1740	14.8021		1.4163	0.6811	2.0974	1.8385	18.2800	
2	A	1.3381	0.7166	2.0547	1.3393	53.4639	52.1519	1.4318	0.7042	2.1360	0.9039	86.0525	86.7333
	B	1.1369	0.6970	1.8339	1.2559	50.8400		0.9161	0.6761	1.5922	0.7914	87.4140	
3	A	0.9952	0.6693	1.6645	0.9980	66.9715	65.5293	1.4573	0.6936	2.1509	0.8822	87.0583	87.2662
	B	1.0506	0.6913	1.7419	1.0686	64.0872		1.7835	0.6762	2.4597	0.8996	87.4741	
4	A	0.9893	0.7058	1.6951	1.0002	70.2416	66.6812	1.6838	0.7084	2.3922	0.8746	90.1295	87.0205
	B	0.8235	0.6815	1.5050	0.9852	63.1208		1.7528	0.7488	2.5016	1.0308	83.9115	
5	A	1.1824	0.6837	1.8661	1.0636	67.8704	67.9731	1.6430	0.7143	2.3573	0.9092	88.1376	88.1427
	B	1.0456	0.7050	1.7506	1.0388	68.0757		1.7305	0.7014	2.4319	0.9065	88.1479	
6	A	1.0976	0.7062	1.8038	1.0205	71.3648	71.2646	1.4712	0.6752	2.1464	0.8395	88.8322	89.5295
	B	1.2710	0.6818	1.9528	1.0483	71.1644		1.2125	0.6869	1.8994	0.8054	90.2268	
7	A	0.7854	0.6833	1.4687	0.8926	73.3512	73.5708	1.3831	0.7104	2.0935	0.8529	89.6971	88.5968
	B	0.6593	0.7041	1.3634	0.8769	73.7904		1.4636	0.6858	2.1494	0.8688	87.4966	
8	A	0.7063	0.7067	1.4130	0.8921	73.7505	73.8489	1.5728	0.6885	2.2613	0.8393	90.4120	89.8222
	B	0.7243	0.7130	1.4373	0.9017	73.9473		1.4460	0.7013	2.1473	0.8570	89.2324	
9	A	0.7387	0.7218	1.4605	0.8981	76.1337	74.7190	1.5731	0.7107	2.2838	0.8556	90.7889	92.2950
	B	0.7814	0.7034	1.4848	0.9120	73.3043		1.6148	0.7006	2.3154	0.8007	93.8011	
10	A	0.8532	0.7149	1.5681	0.9472	72.7731	75.6690	1.7497	0.6994	2.4491	0.8373	92.1186	91.9827
	B	1.0884	0.7310	1.8194	0.9643	78.5649		1.8005	0.6785	2.4790	0.8253	91.8467	