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Research paper

Enhancement of Bio-plastic using Eggshells and Chitosan on Potato Starch Based

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Abstract

The extensive production of polymer plastics and their use in different commercial applications had burdened the municipal in cost and operation of the waste management system. This unwanted waste had also posed a significant threat to the environmental surroundings which destroyed biota. Hence, alternatives called bio-plastic evolved as the development of renewable resource by utilizing agricultural, eggshells and exo-skeleton seafood (chitosan) wastes instead of petroleum sources. The aim of this research is to use the eggshells and chitosan as fillers in potato starch to overcome the inherent drawbacks of bio-plastic. The experimental study was done on tensile strength, water absorption and biodegradability for potato starch-based bio-plastic with eggshells or chitosan. The results showed that by adding the eggshells into the potato starch-based bio-plastic had increased the tensile strength by 4.94% compared with chitosan only 1.28%. The reduction of water absorption by 10.95% was determined using eggshells as fillers. Meanwhile, the used of chitosan resulted in 27.59% reduction in water absorption. In eggshells, the weight loss in biodegradability test was 21.06% compared to chitosan of 7.9% within 20 days. It can be concluded that eggshells as fillers performed much better that chitosan in potato starch-based bio-plastic. It also can be deduced that adding fillers in starch-based bio-plastics can improve the bio-plastic performance.

Keywords: *biodegradability; bio-plastic; chitosan; eggshells; potato starch; shrimp shells; tensile strength; water absorption.*

1. Introduction

In early age, the first plastics were made by using plant through the internal chemical synthesis. One of the first synthetic plastics was made from cellulose which is a substance come from plants and trees. Plastics are the most widely used polymer in daily life, since plastic offers a lot of benefits, in a variety of shapes, for example, film, panels, sheets, or can be flexible for any applications which requires packaging [1]. Every year, the productions of petroleum-based plastic surpass 300 million tons in 2015 [2].
The polymers basically are produced from non-biodegradable petrochemical-based products over the decades ago. Moreover, those polymers take a very long time to decompose, which can create a serious problem caused by the long life of plastic waste, for example, the volume of industrial and commercial dumps in the landfills increased [3].
Bioplastics are biodegradable plastics which are made from biomass. Based on the definition, biodegradable plastic is capable to break down or decomposed through actions by bacteria or others living organisms. There are two types of bioplastics, which are plastics that made from renewable raw materials such as poly-3-hydroxybutyrate (PHB) and polyhydroxyvalerate (PHV) and plastics that contain additives to enhance biodegradation.
Bioplastic or biodegradable plastic could be obtained from starch. In the basic knowledge, by using the pure starch, it is able to absorb humidity. Thus, it is a suitable material for production in pharmaceutical sector in the making of drug capsules. To produce bioplastic from starch glycerol should be added in the heating process. The elasticity characteristic of bioplastic can be fabricated by adding glycerol as plasticizer. Hence, the flexibility of the plastic is provided.
Polymer-plastics are commonly used in daily life as it is low cost, versatile and durable. According to the Statistics of Plastic Usage, shoppers worldwide are currently using an approximate rate of 500 billion plastic bags per year. Most were predominantly from petroleum and natural gas [4]. The plastic waste problem is increasing until today. Regarding on the environmental issue, plastic waste is responsible in polluting the ocean which can affect not only marine and aquatic organisms but also human beings.
The major significance drawback of plastic is that this material is not biodegradable [2]. The degradation time depends on different types of plastic, for example, the plastic bottle takes 450 – 1000 years to decompose completely. The petroleum-based plastic such as polyethylene terephthalate (PET) basically does not decompose the same way as the organic material. Furthermore, the bottles made from PET will never biodegrade [5]. Hence, nature itself chooses another alternative in holding together large molecules of this material since the carbon-carbon bonds in polypropylene require too much energy. In this case, it is hard for organisms to synthesize carbon-carbon bonds compare to peptide bonds which can be found in the proteins or any other organic materials which link carbon to nitrogen. Since the plastic is unable to be degraded by nature, the people tend to incinerate the plastic. In fact, poisonous gases such as dioxins, furans, and mercury will be produced and released to the air. These toxic substances are very harmful to the ecosystem and surrounding environment.
Nowadays, in spite of accumulation of plastic waste due to the growth of population, food waste also becomes a significant problem to the environment. In fact, 70% of the municipal solid waste (MSW) in Malaysia comes from food waste. Generally, 90% of food waste is biodegradable. Thus, the rate to reuse and recycle the food waste is still low. In addition, there is no specific method to dispose the food waste in Malaysia [6].

The new innovative technique in the production of bioplastic can promote a sustainable solution to reduce plastic waste together with food waste in long term. Bioplastic shows a great sign to overcome the pollution since it is biodegradable. Prolong to that, bioplastic is potentially suitable to replace the plastic materials from petroleum based. Therefore, bioplastic is the best alternative to minimize the cost of solid waste management in Malaysia.

2. Materials and Methods

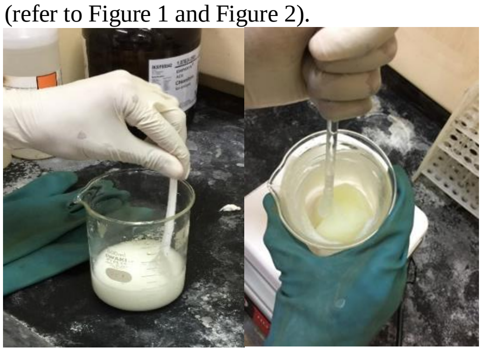
Sodium hypochlorite, glycerol, and acetic acid were purchased from Merck KGaA, Darmstadt, Germany. The chitosan (shrimp shells) filler was purchased from Sigma Aldrich brand under Merck KGaA, Darmstadt, Germany. The eggshells have been collected from the food stalls at Dataran Cendekia (UiTM Shah Alam) meanwhile potato starch was purchased from a grocery shop in Shah Alam, Selangor, Malaysia. Deionized water (Merck Millipore) has been used in this experiment in the Environmental Laboratory, Faculty of Civil Engineering, Universiti Teknologi MARA, Shah Alam, Selangor, Malaysia. The procedure of bioplastic production has been done in the laboratory based on the method from [7]. Meanwhile, eggshells filler have been prepared according to [8].

In this research, bioplastic from potato starch without fillers, bioplastic from potato starch with the additives of filler from eggshells, bioplastic from potato starch with additives of fillers from chitosan, and bioplastic from potato starch with both eggshells and chitosan as additives have been produced.

2.1. Preparation of Bio-Plastic

An amount of sixty (60) ml of distilled water was measured using 100 ml measuring cylinder and poured into the 100 ml beaker. Secondly, ten (10) g of potato starch was measured using weight balance and placed in the beaker with distilled water. The mixture was stirred and mixed together using glass rod. After that, five (5) ml of glycerol and 5 ml of acetic acid were measured using 10 ml measuring cylinder and poured into the beaker with the mixture.

The mixture was stirred again. At the same time, the hot plate was turned on and set at 70˚C. When the mixture was well mixed, the beaker was placed on the preheated hot plate. The mixture was stirred continuously. Then, a milky white liquid appeared in the beaker. When it becomes sticky and almost transparent, the hot plate was turned off and the mixture was spread on aluminium foil. Lastly, the mixture was left for four (4) days to cool to room temperature (refer to Figure 1 and Figure 2).



A B

Fig. 1: A) The mixture is stirred, B) The mixture becomes sticky

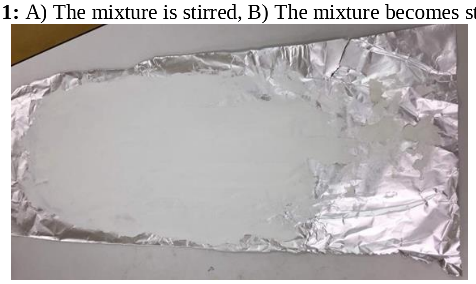


Fig. 2: The mixture is laid on aluminium foil and cooled

The procedures for producing potato starch based bioplastic have been repeated by adding 2.5 g of eggshells powder or/and chitosan powder into the mixture. In order to prepare the eggshells powder, first, the eggshells were crushed into smaller pieces and placed in a beaker. Next, sodium hypochlorite was poured into the beaker with eggshells and stirred using glass rod until the mixture becomes hot and releases bubbles.

The beaker was closed using aluminium foil and left for 24 hours. After 24 hours, the sodium hypochlorite was removed and the eggshells were washed using water. At the same time, the oven was turned on and set to 50˚C. The eggshells were placed in a ceramic bowl. After the oven has reached the required temperature, the ceramic bowl with eggshells was placed in the preheated oven and left for 24 hours.

After 24 hours, the oven was turned off. The ceramic bowl with eggshells was taken out and left to cool to room temperature (see Figure 3a). Then, the eggshells were ground using grinder until it becomes powder (see Figure 3b). After that, the eggshell powder was sieved using sieve size 63μm to get a constant size. Lastly, the eggshells powder was stored in a container.

A B

Fig. 3: A) Eggshells after crushed, B) Eggshells after grinded

2.2. Tensile Test

After the production of bioplastic, the tensile test was conducted in the laboratory as shown in Figure 4. This test is conducted to determine the mechanical properties of bioplastic on Tensile Strength (TS) and Young's Modulus (YM). In this test, ASTM-D638-77 will be used as the standard method to determine the mechanical properties of bioplastic [9]. However, the required dimensions of bioplastic are 100mm x 30mm [3].

In the beginning, five (5) samples with dimensions of 100mm x 30mm from four types of bioplastic were prepared. Next, the sample was hung using thread to a ring with attached hook at the bottom part of the sample to place the loads (see Figure 4). Then, the load was applied until the sample failed. The total loads were recorded and the total length of the failure sample was measured and recorded. Young's Modulus will be calculated. The following equation is to calculate Young's Modulus (YM);

$$\text{Young's Modulus } (N/mm^2) = (F/A) / [(L_1 - L_o)/L_o] \quad (1)$$

Where F is the total applied load (N), A is the area of bioplastic (mm^2), L_1 is the total length of bioplastic after breaking (mm) and L_o is the initial length of bioplastic (mm). The steps will be repeated for the next sample of bioplastic. Finally, all data were calculated and averaged. The graph was plotted to compare the results of the different type of bioplastics.

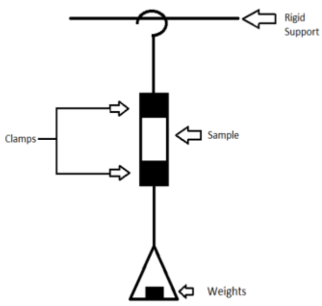


Fig. 4: Illustration of Tensile Test on the bioplastic

2.3. Water Adsorption Test

The water absorption test was conducted in Environment Laboratory, Faculty of Civil Engineering, Universiti Teknologi MARA, Shah Alam. This test is to determine the water absorption behaviour on bioplastic. In this test, ASTM D570-63 was used as the standard method in order to determine the water absorption of bioplastic [10]. Thus, to test the bioplastic, 100mm x 100mm dimensions are required [3]. Hence, to conduct the overall test a maximum period of 50 days is required.

In an early step, a sample of bioplastic with dimensions of 100mm x 100mm from four types of bioplastic was prepared. An oven was turned on and set at 80˚C. Once the oven reached to the required temperature, the samples were placed in the oven and left for 24 hours to be dried. Next, after 24 hours of drying, the oven was turned off and the samples were taken out. The samples were cooled to a room temperature by using desiccator for one (1) minute. Next, the samples were weighted using weight balance and the data was recorded as an initial data. Then, all the samples were immersed in distilled water at room temperature for 50 days (see Figure 5). Within ten (10) days interval, the samples were removed and were wiped using tissue paper in removing the remaining water from the surface of the samples. The sample was weighted and data was recorded. The following equation was used to calculate water absorption;

$$\text{Water Absorption (\%)} = [(W_t - W_o) / W_o] \times 100 \quad (2)$$

Where W_t (g) is the weight of bioplastic at the time being and W_o (g) is the initial weight of bioplastic before being immersed in distilled water. The steps were repeated for the next sample of bioplastic. Finally, all data were calculated and averaged. The graphs were plotted to compare the results of the different type of bioplastics.

Fig. 5: The bioplastic is being immersed in the distilled water

2.4. Biodegradability Test

The biodegradability behaviour test is to determine the biodegradability of bioplastics using compost soil. In this test, ASTM D6003-96 was used as the standard method to determine the weight loss of bioplastics [10]. Hence, the limitation of this test was limited to 20 days to observe the behaviour of biodegradability.

In the beginning, sample of every four (4) types of bioplastic were prepared. All the samples were weighted using weight balance. The weight data was recorded as an initial data. Next, the samples were buried for 64 days in natural soil and compost soil (refer to Figure 6). With seven (7) days interval, the sample was removed from the soils. The sample was washed with distilled water and dried in an oven of 60˚C for 24 hours. After 24 hours, the sample was weighted and the data was recorded. The following equation was used to calculate weight loss;

$$\text{Weight Loss (\%)} = [(W_o - W_t) / W_o] \times 100 \quad (3)$$

Where W_o (g) is the initial weight of bioplastic before buried in soil while W_t (g) is the weight of bioplastic at the time. The steps were repeated for the next sample of bioplastic. Finally, all data was calculated and averaged. The graphs were plotted to compare the results of the different type of bioplastics in a different type of soils.

Fig. 6: Bioplastics buried under compost soil

3. Result and Discussion

The result has been evaluated based on the experiment carried out for the preparation of bioplastic and the test run for this material.

3.1. Bio-plastic Trial Result

Generally, first (1st) trial to third (3rd) trial, the production of bioplastic had failed. However, on the fourth (4th) trial, the bioplastic had been successfully produced with the dimensions of 100mm x 100mm. The failure of the early bioplastic was affected by several factors. Basically, the first (1st) trial to the fourth (4th) trial was based on the method from [7].

On the first (1st) trial, the production of bioplastic had failed due to the incorrect ratio used. After the process of making the bioplastic and four (4) days period for hardening process, the sample was still in a wet condition. Therefore, the wet condition indicated that the failure result of bioplastic production in first (1st) trial.

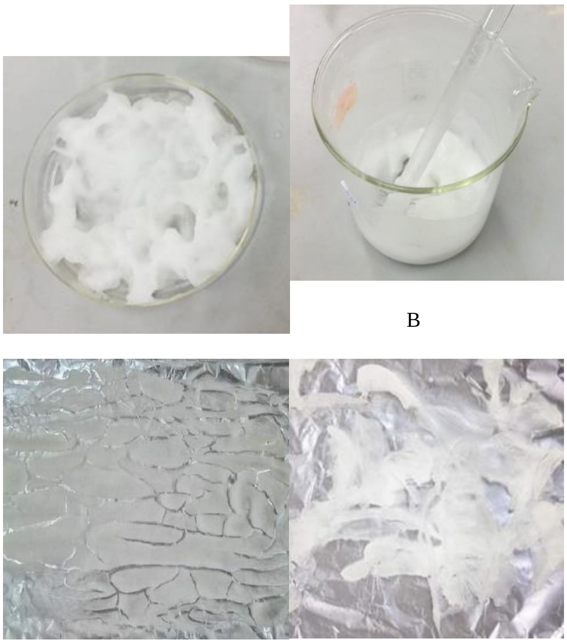
On the second (2nd) trial, the glycerol was measured in 1 ml instead of 5 ml. The used of glycerol is to act as a plasticizer to hold the bond of molecules in the bioplastic. As for the incorrect measurement of glycerol in the first place, it leads to the brittleness of bioplastic. Hence, the sample was cracked right before its rigid state.

On the third (3rd) trial, the production had failed due to the process of heating the mixture. The moisture content of the mixture was lost due to the excessive time of heating process. The mixture became over-sticky and hard or unable to be stirred. Furthermore, the mixture was merely hardened in the beaker before it is laid on to the aluminium foil.

On the fourth (4th) trial, the procedure requirements fully fulfilled the precise measurement; hence the production of bioplastic was successfully produced with dimension 100 mm x 100 mm (see Table 1).

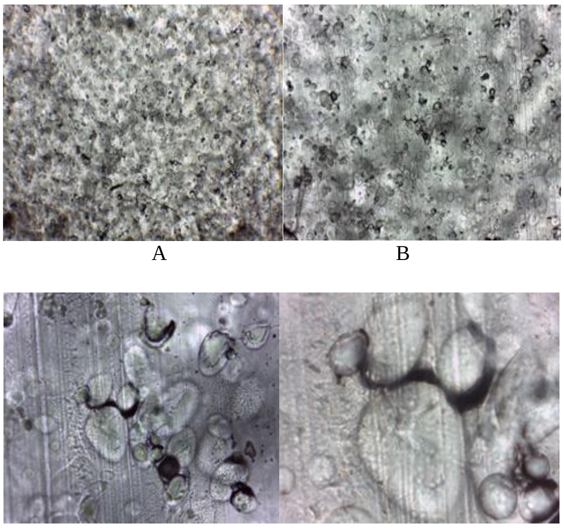
Table 1: Formulation Proportion Materials of Bioplastic

Materials	Proportions			
	Potato Starch Based Bioplastic Without Filler	Potato Starch Based Bioplastic with Egg Shells as Filler	Potato Starch Based Bioplastic with Chitosan as Filler	Potato Starch Based Bioplastic with Egg Shells and Chitosan as Filler
Distilled water	60 ml	60 ml	60 ml	60 ml
Potato Starch	10 g	10 g	10 g	10 g
Glycerol	5 ml	5 ml	5 ml	5 ml
Acetic acid	5 ml	5 ml	5 ml	5 ml
Egg shells	0 g	2.5 g	0 g	2.5 g
Chitosan	0 g	0 g	2.5 g	2.5 g
Dimension				
Length	140 mm	160 mm	170 mm	170 mm
Breadth	140 mm	140mm	150 mm	160 mm



A B C D

Fig. 7: A) 1st trial – Wet condition, B) 3rd trial – Early hardening, C) 2nd trial – Cracking before its rigid state, D) 2nd trial – Cracking after its rigid state



A B C D

Fig. 8: A) 50x magnifier, B) 100x magnifier, C) 400x magnifier, D)1000x magnifier of bioplastic in microscope

Figure 8 shows the potato starch-based bioplastic with different size dimension under the microscope. The image shows the inner details of bioplastic sample. The bioplastic was light and flexible to be bent. There was some bubbles trap in the bioplastic due to it is not properly compressed during the laying process. However, the bubbles did not affect the flexibility of the sample.

3.2. Mechanical Properties

Based on Figure 9, the graph indicates that potato starch-based with the addition of eggshells filler has the highest Young Modulus value with 0.0333 N/mm^2. The higher the Young Modulus value, the stronger and stiffer the bioplastic would be. It indicated that the filler of eggshells able to hold the particle bond, hence the bioplastic become more rigid.

In Figure 10, the graph also indicates that potato starch with the addition of eggshells filler able to cater greater load compared to others. This is because every eggshell has a membrane layer and it is divided by two types of the layer which lie beneath the shell, outer and inner shell membranes [11]. Both of the membranes are made up of protein fibres; exactly lie parallel to the surface of the egg [12]. The membrane of the eggshells holds the bond between the molecules exists in the bioplastic. Therefore, the theory of the eggshells membrane able to enhance the bioplastic is accepted in this research.

Meanwhile, based on Figure 9 and Figure 10, potato starch-based bioplastic with the addition of eggshells and chitosan filler indicate the lowest value in Young Modulus and tensile strength. The proportion of the production of this bioplastic is the main cause of reducing the strength of bioplastic. The amount of plasticizer such as glycerol is lacking and lead to the brittleness of the bioplastic. Basically, plasticizer like glycerol or water has a low molecular weight and volatility which can facilitate the process of hardening by decreasing the intermolecular force's chain and hence increasing their mobility [13]. Therefore, to increase the strength of bioplastic, the consideration amount of glycerol should be reconsidered again in order to accommodate the amount of filler such as potato starch, eggshells, and chitosan.

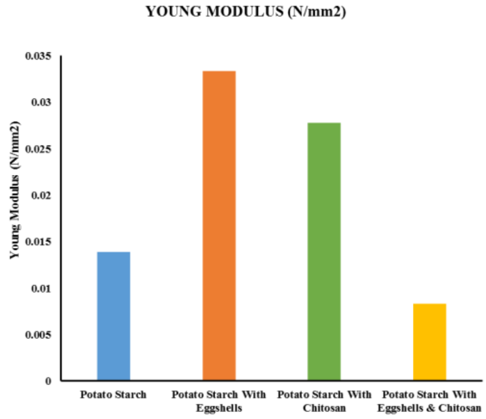


Fig. 9: Young Modulus bar chart

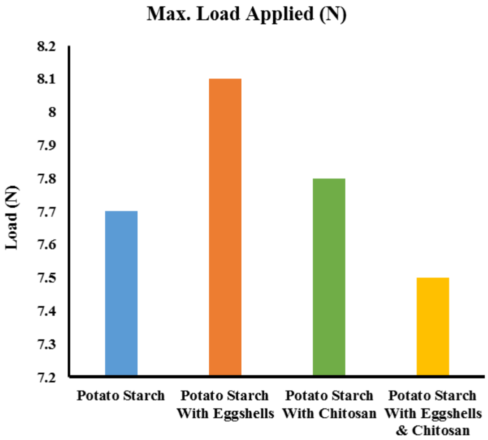


Fig. 10: Maximum load applied bar chart

3.3. Water Adsorption Test Result

Figure 11 shows that the optimum reading of potato starch-based bioplastic of 16.71 g with the maximum rate of water absorption 55.88% on day 50. Specifically, behavioral of potato starch-based bioplastic until day 20 presented a rapid increased of water absorption level. Further demonstrated a gradually increase of water uptake and finally reached to an equilibrium point. This graph also has a similar trend to the previous study from [3].

Nevertheless, as for potato starch-based bioplastic with eggshells as filler, the optimum reading is 14.29 g with the maximum rate of water absorption 44.93% on day 50. However, the bioplastic started to show the optimum reading on day 30 which is 14.20 g with the rate of 44.02%. This indicated that by adding eggshells filler into potato starch-based bioplastic was able to reduce the water absorption content in the bioplastic. This is because the filler capable to cover up the pores contained in the bioplastic. Hence, it reduced the rate of water absorption.

In the same way, potato starch-based bioplastic with chitosan as filler, the graph of Figure 12 indicates the optimum reading of 12.97% with the maximum rate of water absorption (28.89%). Up to day 10, it shows the rapid increase of water absorption level and on day 10 up to day 50 in water uptake demonstrated a gradually increase until it finally reached to an equilibrium point. Chitosan is known as a non-starch organic material, thus has the same structure to cellulose [14], therefore, this research finding agreed with the previous research result from [3]. Hence, by comparing the two types of bioplastic above, the addition of chitosan was far better due to the lowest rate of water absorption and minimum water content absorbed. The chitosan filler has a film-forming based which able to absorb lesser water [15].

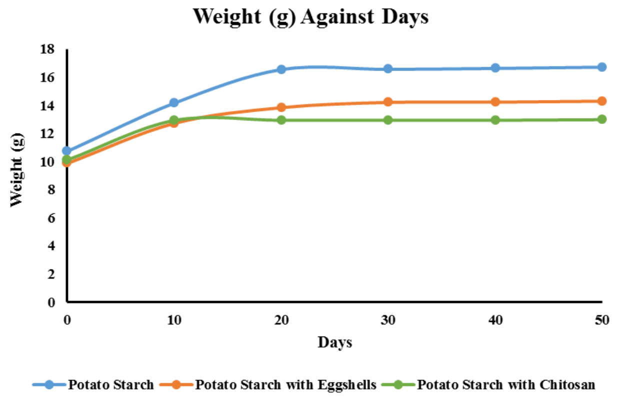


Fig. 11: Weight (g) against days for water adsorption test

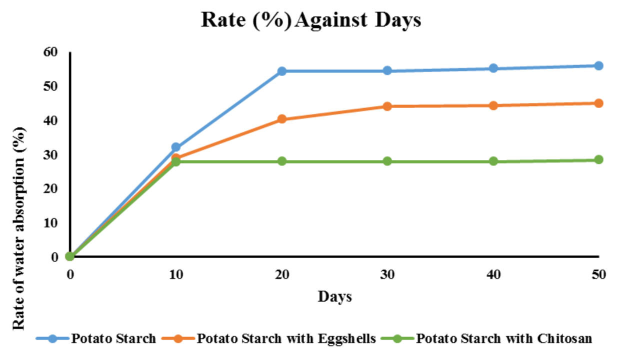


Fig. 12: Rate of water absorption (%) against days for water adsorption test

Based on Figure 13, the sample was taken out after 10 days undergone the process of water absorption test. Fungus started to grow on the sample due to the damp condition on the bioplastics surface. This happened due to air voids produced during the heating process of bioplastic in which the deionized water in the content of the mixture was heated up and evaporated. This cause bubbles in the end of bioplastic production. During the immersion of bioplastic in deionized water, the air voids become a space for the water to penetrate it. Since the fillers were added into the mixture, the fillers replacing the void of the bioplastic. Therefore, the rate of water absorption was reduced since the water was unable to penetrate and filled the void of the bioplastic.

Fig. 13: A sample of bioplastic after ten (10) days undergo water absorption test

3.4. Biodegradation Behaviour Test

The biodegradability test of bioplastic was determined after 20 days. This test carried the result of bioplastic mass reduction in natural soil and compost soil. Based on Figure 14, it indicates the biodegradability behaviour in reducing the mass of bioplastics. The initial mass of potato starch-based bioplastic was 7.77 g on day 0 and reduced to 1.05 g on day 20 with the optimum rate of weight loss of 86.49%. Therefore, in Figure 15 the value of 86.49% is the optimum rate of weight loss which indicated that the potato starch-based bioplastic had a good ability to biodegrade under the compost soil. Hence, the result obtained is similar with the previous research which mentioned that bioplastic is able to be decomposed completely within 24 days [16].
Figure 15 shows the addition of eggshells filler into potato starch-based bioplastic has slowed down the rate of biodegradability of bioplastic with the optimum rate of weight loss of 65.43%. Based on Figure 14, the initial mass of the sample is 5.41 g and reduced to 1.87 g on day 20. Eggshell is largely made up of calcium carbonate which has the major content up to 95% following with 3.5% minor amount of organic matrix in the eggshell [17]. Therefore, the microorganisms degrade the bioplastic but the calcium carbonate remains. Since it has an average rate of biodegradability, this would deduced that by adding the eggshells filler, it takes more time for microorganisms to break the bond chains between nutrients in bioplastic.

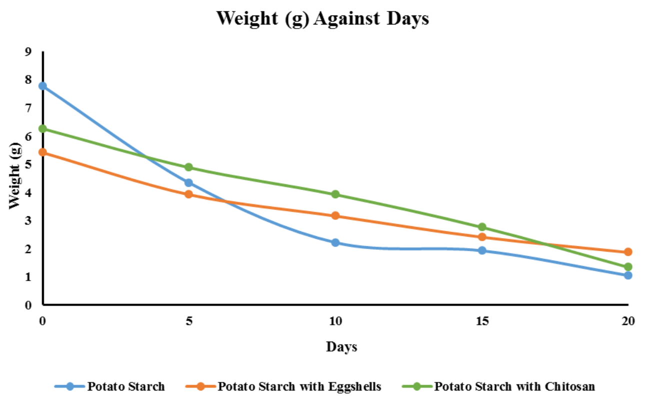


Fig. 14: Weight (g) against days for biodegradability behaviour test

On top of that, by referring to Figure 15, the addition of chitosan into potato starch-based bioplastic also has a good ability to biodegrade under the compost soil with the optimum rate of 78.59%. Based on Figure 14, the initial mass of the sample is 6.26 g and able to reduce to 1.34 g on day 20. It is capable to assume that the sample can completely decompose within 25 days. However, in order to accelerate the biodegradation time of potato starch-based bioplastic with chitosan filler, the enzymes known as chitinase and chitosanase are needed [18].
During the biodegradability behaviour test, some observation was made. In the beginning, on day 5, the compost soil began to grow fungus on the surface area of the soil. This indicates that more fungus grew in the compost soil, the higher the mass reduction of bioplastic. Hence, the higher rate of weight loss of bioplastics to be decomposed. There are at least 90 species of microorganisms that responsible for biodegradation of bioplastics such as aerobes, anaerobes, photosynthetic bacteria, archae-bacteria and lower eukaryotic [2].

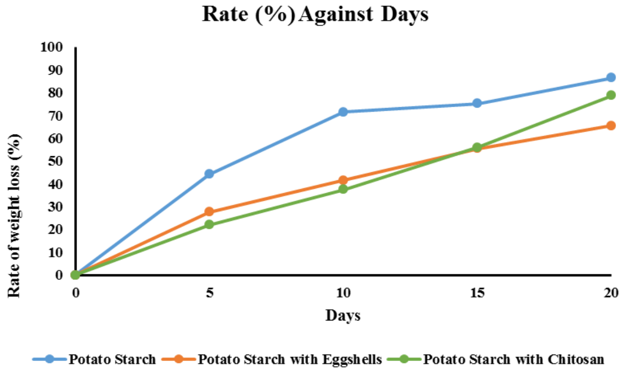
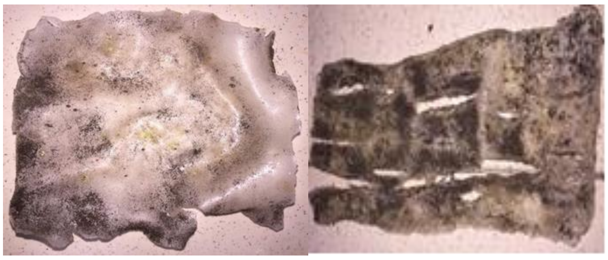


Fig. 15: Rate of weight loss (%) against days for biodegradability behaviour test



A B

Fig. 16: A) Potato starch-based bioplastic taken out from compost soil after 5 days, B) Bioplastic with the addition of eggshells filler taken out from compost soil after 5 days

Based on Figure 16 a, the image of potato starch-based bioplastic after five (5) days planted under the compost soil. Therefore, the sample is thinner compared to its original thickness before undergone the process of biodegradability behavioural test. In contrast, by referring to Figure 16 b, shows the image of potato starch-based bioplastic with the addition of eggshells filler after five (5) days buried under the compost soil. In contrast with Figure 16 a, instead of becoming thinner the sample from Figure 16 b has shredded from the effect of being decomposed by microorganisms in compost soil.

4. Conclusion

The potato-starch based bioplastic with egg shells and chitosan as filler have been produced. Then the mechanical properties, water absorption and biodegradability of the bioplastic also have been determined. From the result obtained, it can be presumed that by adding the eggshells filler into the potato starch-based bioplastic is able to increase the tensile strength by 4.94%, reduce the water absorption by 10.95% and increase the rate of weight loss in biodegradability by 21.06% within 20 days. Meanwhile, adding the chitosan filler is able to increase the tensile strength by 1.28%, reduce water absorption up to 27.59% and increase the weight loss in biodegradability by 7.9%. As for adding both fillers of eggshells and chitosan, it slightly decreased the tensile strength and Young Modulus by 2.6% and 40% respectively due to no trial and error in conducting the experiment. As for water absorption, the less water absorbed indicated a good sample of bioplastic and for biodegradation, the higher rate of weight loss, the faster time is taken to decompose completely. As for the conclusion, after producing and conducting several testing on the bioplastic, it can be presumed that the use of eggshells and chitosan as filler does improve the potato starch-based bioplastic.

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