Evaluation of the Biodegradation for Oil Absorbed Cassava Starch Film Filled with Kenaf Core Fiber

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Abstract. Cassava starch (CS) films filled with different loading of kenaf core fiber (KCF) were prepared and tested. Two tests were carried out; oil absorption test and biodegradation test. The oil absorption test were carried out with different loadings of KCF (0%, 0.2, 0.4, 0.6%, 0.8% and 1.0%). The results showed an increase of oil intake as the kenaf loading increased. The biodegradation test was conducted via soil burial method. Two sets of film samples were tested; film samples with and without immersion of oil. Each set of the film samples respectively compares between CS films with and without KCF loadings. The results of the biodegradation tests showed that CS films with KCF had lower amount of weight loss compared to CS without KCF. Furthermore, the result of biodegradation tests for film samples immersed in oil were lower compared to film samples with oil immersion.

1. Introduction
Nowadays, polymer based materials are used extensively in all kinds of fields due to their advantages of high formability, light weight and high strength. As a result, the consumption of polymer materials has globally increased since the 1950’s [1]. However, most of these polymer materials are produced from petroleum based plastics. Petroleum based plastics have a large timeframe of working life. Their high durability results in the massive accumulation of plastic waste over the course of time, leading to various serious environment issues which requires collection and disposal, generally by burial in landfill, composting, or incineration [2].

As a result, the interest in the use of biopolymers has been increasing. According to Peterson et al. [3], this was due to the increase in concern regarding the price of crude oil and as well as global warming which had influenced the awareness of consumers significantly. Nowadays, biopolymers are obtainable without much difficulty, for instances; biopolymers can be obtained from waste produced by agricultural feedstock, microbial activities, and marine fauna.

Although biopolymers are incapable of completely replacing the conventional synthetic polymers, biopolymers are capable of other ways which synthetic polymers could not. Starch is an example of a promising material for biodegradable plastics due to its versatility as a biopolymer, which holds an enormous potential with low price for use in non-related food industries [4]. There are a variety of sources to obtain starch, for example; maize, potatoes, rice, corn and cassava [5] [6]. Based on Souza et al. [7], they described that films which are manufactured from starch are odorless, possessed neither taste nor colors, and are capable of biodegrading naturally. In another of their study [8], they stated the
plasticization of cassava starch using glycerol. The results provided information on accurate formulation to be used to produce composite films with improved mechanical properties.

At present time, works regarding natural fiber polymer composites are increasing in number. Natural fibers can be incorporated as reinforcement filler as they are cheaper in cost and can be obtained with much ease compared to conventional mineral fillers. Natural fibers in general are polar and hydrophilic in nature, resulting in a poor mechanical properties when added into conventional plastics as they are more hydrophobic in nature [9]. In an attempt to overcome the polarity issue, Jutarat et al. [10] in one of their works studied the incorporation of starch and thermoplastic polymer blends. They reported natural fiber such as; cotton fibers, works well as reinforcing agents in their starch and polyethylene blends. Natural fibers and starch possesses good chemical compatibility since both originated from natural resources [11]. Hence, the mixing of natural fibers and thermoplastic starch will have reinforcing effects.

One of the most studied natural fibers today is the kenaf. Kenaf fibers have been used in many parts of the world since long time ago. In the past, the main usage of kenaf was to manufacture ropes, twines, coarse clothing, and paper. Even today there are some which continue such practices. Nevertheless, the kenaf are still in demand today however as reinforcing additives for polymer production [12]. The kenaf can be divided into two sections; the bast and the core. For this study, the kenaf core will be used as kenaf fibers since the core has better oil absorption properties compared to the bast section. In a report by Ridwan [13], he stated that kenaf core is a very high absorbent material and there are already plenty of commercialized products for toxic waste cleanup, remediation of chemically contaminated soils and oil spills on water. The main purpose of this study are to evaluate the effects different loading of KCF on oil absorption and natural degradation of cassava starch films via soil burial.

2. Experimental

2.1. Materials
Native cassava starch (brand: ‘Kapal ABC’ brand, imported from Thailand) was used without further purification. Analytical grade glycerol was purchased from Fisher Chemicals, UK. Kenaf core fibers were purchased from LKTN (Lembaga Kenaf Tembakau Negara). Cooking oil (brand: Knife, manufactured in Malaysia) were used to test for oil absorption of the CS films.

2.2. Film Preparation
Cassava starch films were prepared by mixing 6 g of CS, with 1.5 % (W/W) glycerol and different loading of KCF (0.2%, 0.4%, 0.6%, 0.8%, and 1% of W/W) using Isotemp stirring hot plate (Fisher Scientific) at 80 °C for 13-15 minutes or until a filmogenic solution was formed. The filmogenic solution was transferred on an polyacrylic mold (240 mm × 160 mm) via solution casting and left to dry in a ventilated oven at 40 °C for 24 hours. Two types of film samples were prepared, CS films without KCF (control) and CS films with KCF.

2.3. Measurements of Film Thickness
The thickness of each film was measured at three different positions to the nearest 0.0001 mm using dial thickness gauge (Precise brand, model: 330-101).

2.4. Oil Absorption Test
The oil absorption test were carried out by adapting the test methods from ASTM D570-98 [14]. The film samples were cut to bar shape (30 × 10 mm). The test samples were dried in a ventilated oven at 40 °C for 24 hours to remove any residual moisture and immersed into oil for 9 hours. The samples were weigh, and the weight of samples after immersed in oil were determined through the following equation:
where $O_o$ represents the percentage of weight change, $O_i$ represents the initial weight of samples before immersed in oil and $O_f$ represents weight samples after immersed in oil. The CS films with and without KCF were tested.

2.5. Biodegradation Test
The biodegradation test was carried out via soil burial method by adaptation from SR EN ISO 846/2000. The sample specimens were cut into square shape ($20 \times 20$ mm) were buried in a compost soil. The films samples were taken out of the soil, and cleaned carefully using a slightly damp tissue in order to remove adherent soil and dried on filter paper. The samples were stored in a desiccator for 24 h until they reached a constant weight. The weight of the samples was taken at regular interval of time (5 days) to record the weight loss. The weight loss degradation was determined through the following equation:

$$m_{loss} = \frac{m_i - m_f}{m_i} \times 100\%$$

where $m_{loss}$ represents the weight loss, $m_i$ represents the initial weight and $m_f$ represents the final weight of the samples after each testing periods. The effects of the presence oil in biodegradation of CS films without KCF and CS films with the highest loading of KCF films were used.

3. Results and discussion

3.1. Oil Absorption Test
Fig. 1 shows the oil absorption of the CS/KCF with differing load of fibers for 9 hours. The results show that the oil uptake increased steadily up until the 6th hour and began to slowly form a plateau. All the reinforced films absorbed more than the un-filled CS films which was only 0.9% at the 9th hour of immersion in the oil. Films with 1% KCF loading absorb the most oil which was 3.98%. Moreover, the film with 0.2% loading absorbed the least oil with only 1.79%. It is clear that the KCF increased the oil absorption uptake of the CS film. From the results, it is shown that CS film does not absorb much oil. Most of the oil are probably absorbed by the KCF. Melissa et al. [15], stated in their report that polysaccharides possesses good film-forming properties, which provides efficient barriers against oils and lipids. Bazargan et al. [16], stated in his report that natural fibers possesses many free hydroxyl groups at the molecular level. Therefore, KCF are able to attract oil molecules.
3.2. **Biodegradation Assessment**

The weight loss of film samples during 35 days of soil burial are shown in Fig 2. In all the films, the rapid increase in weight loss occurred for the first 15 days, and gradually increased following after. Based on the graph, CS films without KCF loadings lost more weight compared to CS films with KCF. The film samples were all mainly made up of either starch or glycerol. Both CS and glycerol are hydrophilic in nature. Hence, the film samples were very susceptible to the water in the soil. According to Maran et al. [17], high concentration of starch and glycerol increases weight loss of starch films during soil degradation. They reported the starch and glycerol favors the absorption of water, increasing the water activity of the films and promotes the growth of microorganisms. Furthermore, the microorganisms which are present in the soil utilize the cassava starch as a source of nutrients which results in further degradation of the film samples. Lopez-Llorca et al. [18], reported the degradation of starch films and high microbial activity in the films which was buried in a forest soil. They stated that glycerol would eventually be assimilated into the soil or metabolized by microbes, increasing the degradation of the films. Based on the results, the incorporation of KCF had significantly reduced the weight loss of the CS films during the soil burial. In a study by Antonio et al. [19], they stated that strong interaction between fiber-matrix may resist the water from the soil from diffusing into the CS matrix. This will inhibit not only the susceptibility of water towards the matrix but also microorganisms growth. The result showed a slight decrease in weight loss for both CS films with and without KCF after immersed in oil. This shows the oil absorbed are capable of influencing the degradation of the CS films. Oils are essentially non-polar, this makes the water in the soil which are opposite in polarity to repel each other. The repelling nature of the oils absorbed by KCF and water

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**Fig. 1**: Oil absorbed by CS/KCF films with different loading of KCF at different time(s).
in soil makes it difficult for any interactions between CS and water. Thus, weight loss due to water of CS with KCF immersed in oil was less during the soil burial. However, CS films without KCF immersed in oil also experienced the same decrease in weight loss. This is probably due to some oil residue remaining on the surface of the CS films.

![Graph showing weight loss over time for different CS film samples during soil burial.]

**Fig 2. Weight loss (%) of CS film samples during soil burial**

4. **Conclusion**

The effects of loading KCF on the oil absorption of cassava starch film was studied. The result of the oil absorption test showed an increase of oil absorption as the KCF loadings increased. As starch films are naturally resistant to oil, the incorporation of KCF are an improvement towards starch based plastic application for oil absorption purposes. However, incorporation of KCF in CS films showed a reduced in weight loss compared to CS films with no KCF loadings. Furthermore, the immersion of film samples in oil resulted in a slight decrease of weight loss in the soil burial test. Since the decrease in the weight loss of the films were only minor this does not impact the biodegradation properties in any major way.

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