Research Paper

# Physical and Environmental Degradation Properties of Arrowroot (*Maranta arundinacea*) Fibre reinforced Arrowroot Starch Composite Films

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**Abstract:** This research is driven by strict government environmental regulations that requires the use of environmentally friendly products. In this study, the physical, environmental as well as sand barrier properties of arrowroot fiber (AF) reinforced arrowroot starch (AS) composites film were investigated by using different percentage of arrowroot fiber (2, 4, 6, 8 and 10%). This result showed that the control AS film with 0% of arrowroot fiber were translucent, rigid, brittle and slightly sticky and not easily peel off from the petri dish. While for other of arrowroot fiber reinforced starch composites film with different fiber percentage showed the film has fiber attached to film, more flexible and peelable. While for the film density, the arrowroot fiber reinforced starch composites film with different percentage of arrowroot fiber showed lower density value compared to control AS. Upon increasing the fiber percentage from 2 to 10%, film density decreases from 1.472 g/cm³ to 1.056 g/cm³. The moisture content of control AS film showed the lowest moisture content percentage compared to arrowroot fiber reinforced starch composites film with different percentage of arrowroot fiber. For the water absorption analysis, the result showed that the increment of fiber percentage resulted to increment in water absorption properties of the biocomposites film. The solubility arrowroot fiber reinforced starch composites film was increased from 13.86 to 34.74%, as the fiber percentage increase from 2 to 10%. For soil burial test, result showed that the decomposition of AS/AF (10%)) film was higher than that of AS/AF film with fiber percentage (control, 2, 4, 6, and 8%) samples at all given points of time. The physical, environmental as well as barrier properties of arrowroot fiber reinforced starch composites were affected by the increment of the fiber percentage. In brief, the findings of this research provide insights into the development of biodegradable food packaging.

**Keywords:** Arrowroot fiber; arrowroot starch composites, environmental degradation, solution casting, water absorption

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## 1. Introduction

Due to awareness related to environmental issues and preservation of natural resources has been led to development of natural material especially natural fiber as one of alternative as replacement of synthetic material as well as renewable raw materials [1]. The composites manufacturing now focused on invented the plant based natural fiber reinforcement as the alternatives to replace the synthetic fiber for example jute, sisal, kenaf, banana, hemp, and flax. For the automotive component, aerospace parts, sports goods as well as building construction, the natural fiber reinforced composites has been used for those applications. This is resulted from few advantages of natural materials that has high strength, low in weight, can be deform, and has high resistance to corrosion and fatigue [2].

The use of natural fiber in biocomposites gain more attention compared to synthetic composites due to environmental issues nowadays. For the hybrid composites reinforced, the cellulose fiber or natural fiber often combined with synthetic fiber such as glass fiber has demonstrated a good mechanical performance [3]–[5]. The natural fiber reinforced composites are invented to replace the uses of glass fiber reinforced composites as natural fiber are cheaper than glass fiber. The using of natural fiber in reinforced composites also does not or less cause to health problem compared to glass fiber which can cause skin irritations and can causing lung cancer [6].

Thus, this research focused on the fabrication process of arrowroot fiber reinforced arrowroot starch composite, and their physical, structural and barrier properties. The starch and fiber in arrowroot plant has few characteristics which necessary in fabricate the composites films where it has excellent digestibility, gelling ability and has highest amylose content which is 40.86% compared to other plant resources which are corn with 28-33% of amylose content, potato with 18-20% of amylose content, wheat starch with 30-32% of amylose content and cassava starch with 16-19% of amylose content [7]. Based on previous research, the fabrication of bio composite film is depending on amylose content which form more strong and stiff film due to hydrogen bonding in the linear chain of the film composites. Thus, the percentage of amylose content in arrowroot fiber play an important role develops stronger film composites compared to other plant resources. According to literature, arrowroot rhizomes comprise 38.1% of bagasse fiber [8]. Branco et al. [8] found that the arrowroot bagasse fibers are coarser as well as longer in comparison to cassava bagasse fibers. The chemical composition analyses showed that arrowroot bagasse fiber (ABF) has higher cellulose content (45.97%) than arrowroot husk fiber (AHF) of (37.35%), cassava bagasse (10.04%), and corn hull (15.30%) [9]. However, ABF has lower lignin content (2.78%) and density (1.11 g/cm$^3$) compared to AHF, corn hull, and cassava fibers. Thermal analysis revealed that ABF has a significantly higher decomposition temperature (343.8°C) compared to AHF (294.03°C). The morphological study by SEM revealed that the longitudinal section of ABF was characterized by the presence of Trichomes, (a perforated deep structure) that could improve the adhesion ability between fiber and matrices in biocomposite manufacturing. The crystallinity index of ABF was 38.2%, which was higher than the 35.1% of AHF.

Furthermore, there are only few research on application of arrowroot fiber reinforced arrowroot starch composites film especially in food packaging industries. Therefore, for this research we are focusing to fabricate the arrowroot fiber reinforced arrowroot starch composites by using different weight percentages of arrowroot fiber which are 2,4,6,8, and 10%. The effect of fiber percentages of physical properties, structural properties, and barrier properties of arrowroot fiber reinforced arrowroot starch composites were investigated.

## 2. Materials and Methods

### *2.1. Materials*

The raw material used in this research are arrowroot starch and fiber which extracted from arrowroot tubers purchased from Norient Jaya Sdn Bhd, Kuala Lumpur Malaysia. The sorbitol plasticizer with 99.5% purity was supplied by Evergreen Engineering & Recourse Sdn Bhd, Selangor, Malaysia and distilled water was used as solvent in this process.

### *2.2. Film preparation and characterizations*

The solution casting method was used to prepare starch-based films using a film-making solution that included 7.5 g of arrowroot starch in 150 ml distilled water by heating the solution at 80 ± 3 ℃ for 15 mins under slow constant stirring in a thermostatic bath to allow the starch to gelatinize. After that, the sorbitol was added into gelatinized solutions at 2,4, 6, 8 and 10% (w/w, fiber basis) for 5 mins at 80 ± 3℃. Afterward, the solution was poured in 15 cm diameter glass petri dishes. These petri dishes were placed in an air-flow oven at 45 ℃ for 18 hours to dry the films. The composites films were peel off from the petri dish and kept at the room temperature in a plastic box for a week before undergo few characterization. Figure 1 shows the fabrication process of arrowroot fiber reinforced starch composites using solution casting method. Table 1 shows processing weight for arrowroot fiber reinforced arrowroot starch composites.

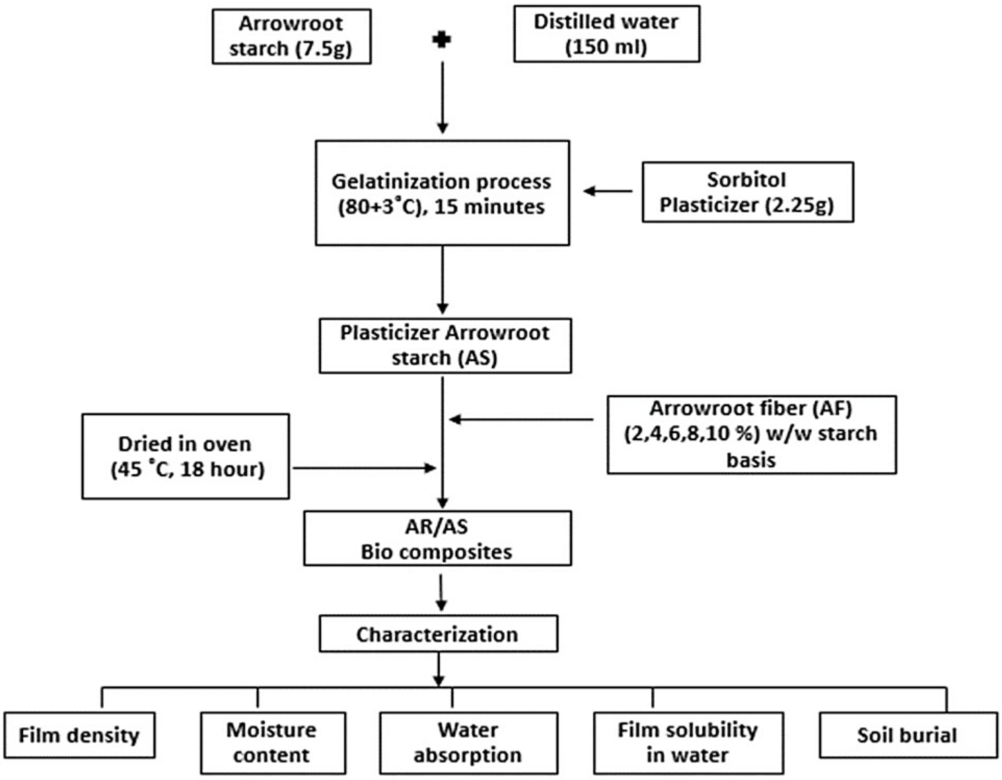


**Figure 1**. Flow chart of fabrication process for arrowroot fiber arrowroot reinforced starch composites.

**Table 1.** Processing weight for arrowroot fiber reinforced arrowroot starch composites.

| Sample | Arrowroot starch (gram) | Arrowroot fiber (gram) | Distilled water (ml) | Sorbitol (gram) |
|---|---|---|---|---|
| Control AS | 7.5 | 0 | 150 | 2.25 |
| AS/AF (2%) | 7.5 | 0.15 | 150 | 2.25 |
| AS/AF (4%) | 7.5 | 0.30 | 150 | 2.25 |
| AS/AF (6%) | 7.5 | 0.45 | 150 | 2.25 |
| AS/AF (8%) | 7.5 | 0.60 | 150 | 2.25 |
| AS/AF (10%) | 7.5 | 0.75 | 150 | 2.25 |

### *2.3. Physical characteristics*

#### *2.3.1. Film density*

The density of the film samples was determined utilizing Densimeter and ASTM D792-00. After that, the calculation of the preliminary dry matter of every sample was carried out. The film samples were weighed before (m) before being immersed into the liquid of volume (V) solvent. The density (ρ) of the sample was determined by using equation 1.

$$\rho = \frac{m}{v} \tag{1}$$

#### *2.3.2. Moisture content*

A digital weight scale was used to evaluate the moisture content of three replicates of each film sample. All the samples were weighed before ($W_i$, gram) and then dried at 105 ˚C for 24 hour and reweighed ($W_f$, gram). To calculate the moisture content of each film sample using equation 2.

$$\text{Moisture content} = \left(\frac{Wi - Wf}{Wi}\right) \times 100 \tag{2}$$

### *2.3.3.Water absorption analysis*

The water absorption analysis was conducted using ASTM D570-98(1998)59. A temperature of 50 °C was used to dry the film samples for 24 h and the dried samples were placed in a desiccator for cooling ensuring consistent weight. After that, the films were weighed (Mi, gram) and then submerged at room temperature in distilled water. A clean piece of cloth was used to wipe the immersed film samples and reweighed ($M_f$, gram). The difference between initial and final recorded masses was calculated using equation 3.

$$\text{Water absorption (\%)} = \left(\frac{Mf - Mi}{Mf}\right) \times 100 \quad (3)$$

### *2.3.4.Film solubility in water*

A strip of (30 ×10 mm) dimension was cut out from each film sample in triplicate and dried in an oven at 105 °C for 24 h. To determine the initial dry weight (Wi, gram) of each sample, the strips were weighted. Afterwards, each sample was immersed in a beaker containing 50 ml distilled water under constant magnetic stirring at 500 rpm and kept at room temperature (23±2 ℃) for a period of 6 h. The film’s insoluble part was separated from the beaker and placed in an oven for 24 h at 105 ℃. To evaluate the weight of solubilized matter ($W_f$, gram), the dried samples were weighed again. Equation (4) was used to calculate the water solubility of each sample.

$$\text{Solubility (\%)} = \left(\frac{Wi - Wf}{Wi}\right) \times 100 \quad (4)$$

### *2.3.5. Soil burial degradation*

The biodegradability analysis of a film sample in soil was conducted by calculating the weight loss of control and G-plasticized arrowroot-based films buried in compost soil under restrained humidity conditions. The tests were performed in triplicate, where each film sample with 20 × 20 mm dimension was buried 100 mm underneath the surface of the soil. The samples were buried periodically at a time interval of 2, 3, 5, 7, 10, 12, 14,16, 18, and 20 days and every sample were buried out from the compost simultaneously. After that, the sample was cleaned with water and dried in a vacuum oven at 65 °C to get a consistent weight using Equation 5, the weight loss of the sample was evaluated.

$$\text{Weight loss (\%)} = \left(\frac{W0 - Wt}{W0}\right) \times 100 \quad (5)$$

Where, ($W_0$) prior burying weight and ($W_t$) is post burying weight.

## 3. Results and Discussion

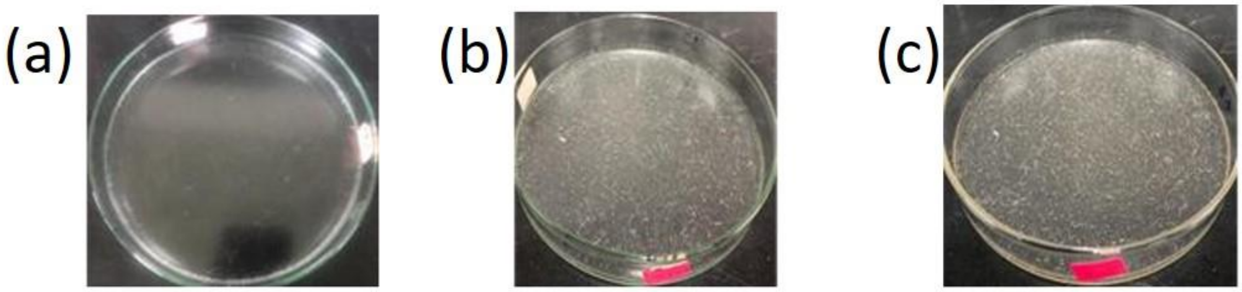
### *3.1. Appearance of arrowroot fiber reinforced starch composites*

Figure 2 presents the graphic images of the control and arrowroot fiber reinforced starch composites, and Table 2 illustrates their visual presence. Without arrowroot fiber, arrowroot starch (control) film were rigid, brittle, and translucent and slightly sticky. The control film crash into bits during peeling process due to the strong intramolecular hydrogen bonds, which gave less mobility to the macromolecular chains, and resulted in fragile and stiff surface broken films. This finding also supported by the research of Sappakul et al. [10] and Talja et al. [11], who carried investigation on starch-based biopolymers by using natural fiber from cassava and potato starch.

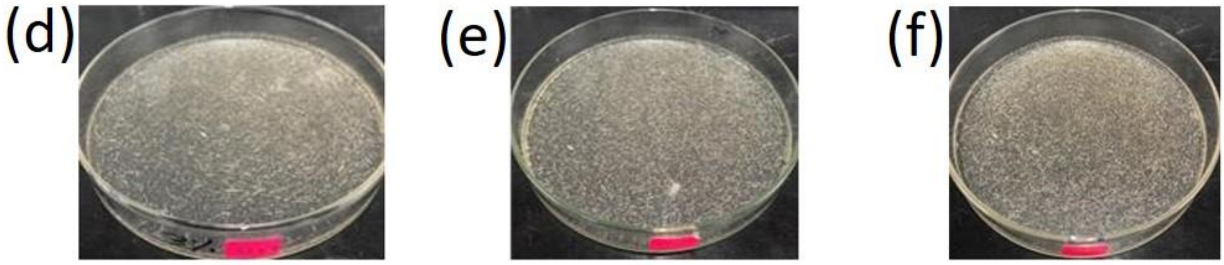
While for arrowroot fiber reinforced starch composites with different percentage of arrowroot fiber, it shows the sample form with fiber attached to the film, homogeneous, more flexible and easier to peel. It was observed that AS/AF composites with 10% of fiber has more fiber attached to the film, homogeneous, more flexible and easier to peel compare to other. Consequently, the flexibility of AS/AF film has improved as the percentage of the arrowroot fiber increase. The flexibility of AS/AF film increase due to smaller molecular size of fiber which allowed them fill in the spaces between molecules of polymer chains, which reduced the strength of hydrogen bonds between molecules, and resulted in active movement of molecules in the composites. Based on this research also, it shows that as the percentage of arrowroot fiber increase from 2 to 10 % resulted in decrement in intermolecular chains hydrogen bond of AS/AF films.

**Table 2**. The visual present of control and arrowroot fiber reinforced starch composites.

| **Sample** | **Arrowroot fiber (%)** | **Film appearance** |
|---|---|---|
| AS (Control) | 0 | Translucent, rigid, brittle and slightly sticky |
| AS/AF (2%) | 2 | Fiber attached to film, not brittle, flexible, easy to peel |
| AS/AF (4%) | 4 | Fiber attached to film, more flexible, peelable |
| AS/AF (6%) | 6 | Fiber attached to film, more flexible, peelable |
| AS/AF (8%) | 8 | Fiber attached to film, more flexible, peelable |
| AS/AF (10%) | 10 | More fiber attached to film, more flexible, very easy to peel |



AS (control) AS/AF (2%) AS/AF (4%)



AS/AF (6%) AS/AF (8%) AS/AF (10%)

**Figure 2.** Graphic images of the (a) AS control and arrowroot fiber reinforced arrowroot starch composites, (b) AS/AF 2%, (c) AS/AF 4%, (d) AS/AF 6%, (e) AS/AF 8%, and (f) AS/AF 2%.

### *3.2. Film density*

The arrowroot fiber reinforced starch composites film with different percentage of arrowroot fiber showed lower density value compared to control AS. The impact of fiber percentage on the density of the AS/AF films are shown in Figure 3. As the fiber percentage increase from 2 to 10%, the film density decreases from 1.472 g/cm$^3$ - 1.056 g/cm$^3$. The decrement in film density could be resulted due to increase of thickness of film composites and volume of fiber as we increase the fiber percentage in film composites. This finding was supported by the research made by Nur Adilah et al. [12] on polylactic acid bio composites foam reinforced with different kenaf filler loading (PLA/KF). This is expected in which the presence of kenaf fiber may disturb the crystallinity in PLA matrix and the amount of gas absorbed in the composites in which the dissolved gas is dependent on the fiber loading in composites system [13]. As the fiber percentage increase, the interaction between fiber-matrix is also affected due to insufficient polymer matrix to wet out the fiber thus leads to the fluctuate density trend of PLA/KF composites foam [12]. The result show that, the density of the foamed composites showed a fluctuate trend as the kenaf fiber percentage increase from 0 to 20%, however when the percentage of kenaf fiber used between 30 to 40 %, it resulted in an increase of the composites density. This trend is closely similar to the previous study done by Teymoorzadeh and Rodrigue [13] on morphological, mechanical, and thermal properties of injection molded polylactic acid foams/composites based on wood flour.

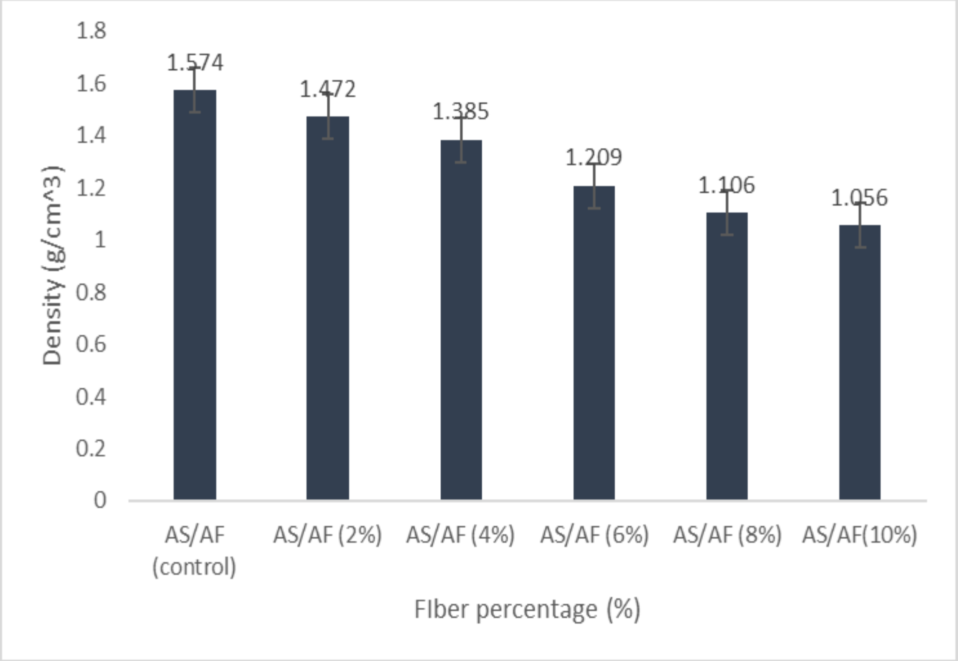


**Figure 3.** The density of control and arrowroot fiber reinforced starch composites film with differ ent percentage of arrowroot fiber.

### *3.3. Moisture content*

Based on Figure 4 the moisture content of control AS film showed the lowest moisture content percentage compare to arrowroot fiber reinforced starch composites film with different percentage of arrowroot fiber. Moisture content for arrowroot fiber reinforced starch composites film with (2,4,6,8, and 10%) of arrowroot fiber enhanced significantly with enhancing the percentage of arrowroot fiber percentage from 2% to 10% as shown in Figure 8. The hydrophilic behaviors of arrowroot fiber reinforced starch composites films were increased as the fiber percentage increase. For this research, the moisture content of arrowroot fiber reinforced starch composites film with (2,4,6,8, and 10%) were 6.104, 8.364, 9.623, 11.358 and 14.565 %.

Based on study carried by Jawaid et al [14] on cellulosic/synthetic fiber reinforced polymer hybrid composites, the hybrid arrangement will discourage the moisture absorption into the composite and pack arrangement of the fiber fill up the voids which form during fabrication of composite. This resulted from hydrophilic behaviors of cellulose fiber that enable the film composites to absorb moisture content from the surrounding environment. The hydrophilic behavior of cellulose fiber and capillary action resulted in high water intake when the composites film sample were immersed in the distilled water and resulted in increment in film dimension [15]. This show that as the fiber percentage increase, the thickness of the film composites also increases due to swelling effect. The moisture content in composites film also cause dimensional variation and will affect the mechanical properties of the composites film. During the fabrication process of thermoset-based composites, the moisture content leads to void content and affect fiber-matrix bonding and resulted in decrement in mechanical properties of the composites [2], [16]–[18].

The hydrophilic behavior and poor resistance to moisture has make the natural fiber incompatible compared to hydrophobic polymer matrix. This behavior results in poor fiber-matrix interface which in turns lead to decrement in the mechanical properties of the composites [14]. Thus, to overcome this problem, few alternatives can be done for example by chemical modification of fiber where it will help to make it less hydrophilic. Several studies conducted on chemical modification of cellulose fiber in depth and concluded that most promising approach of chemical modification seemed to be the one that gave rise to continuous covalent bonds between fiber surface and matrix [19]–[21]. Chemical treatment or surface modification of fiber improves adhesion between fiber and matrix which is the critical issue to develop advance composites [22]–[24]. The treatment of the fiber may be alkali, acetylation, bleaching and grafting of monomer. Besides surface treatment of fiber, compatibilizer of coupling agents such as silanes, maleated polypropylene (MAPP), and titanates are commonly used to improve fiber-matrix interface [21], [25]–[27].

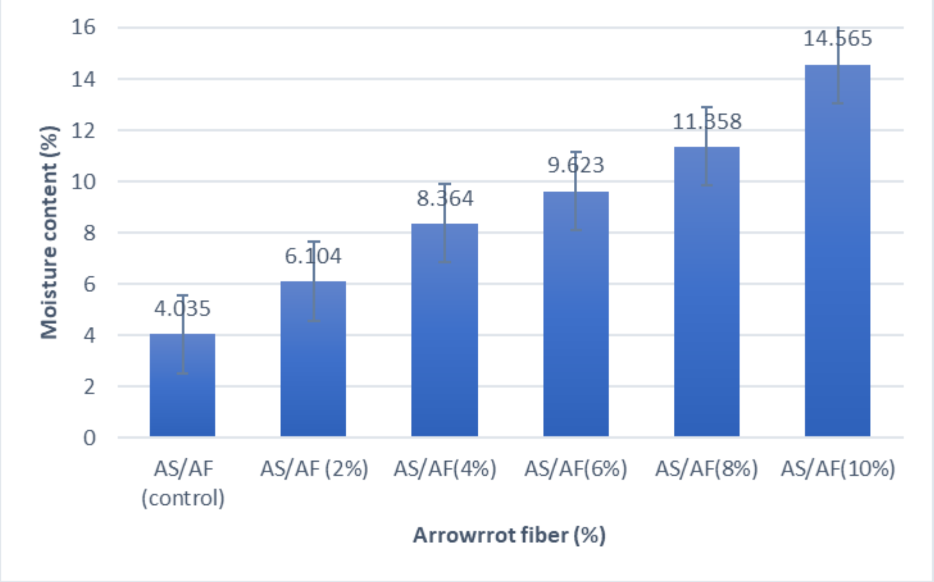


**Figure 4.** The moisture content of control and arrowroot fiber reinforced starch composites film with different percentage of arrowroot fiber.

### 3.4. *Water absorption*

The water absorption of the control and arrowroot fiber reinforced starch composites film is presented in Figure 5. Based on Figure 5, after 30 minutes, the films absorb maximum water at room temperature. For control AS film and AS/AF (2%) film absorbed about 45% and 52% respectively after 60 minutes and started to dissolve when reach 60 minutes. While for AS/AF (4%) film and AS/AF (6%) film absorbed about 56% and 60 % after 60 minutes and started to dissolve when reach 90 minutes. Lastly for AS/AF (8%) and AS/AF (10%) film absorbed 67% and 81% after 70 minutes and continue shown constant reading until 140 minutes, the film also does not dissolve and break completely compare to another sample. As can be seen, for all the samples, the water absorption was increase at first 0 to 20 minutes, then continue slows after 30 minutes and finally after extended immersion time, the film sample has approach to the saturation stage and the reading remains the same.

As the percentage of fiber increase from 2 to 10% for AS/AF fill composites, the rate of water absorption for the composites films also increased. This can be explained by the hydrophilic behaviors of arrowroot fiber itself, as they are in cellulose fiber class [28]–[30]. The hydrophilic behaviors cause the film swell when soaked or exposed to the water due to water absorption of the fiber in the composites film. As the fiber swell, the microcrack can appear in the composites film as result of largest transport of water through the fiber matric interface. Besides that, an active capillary mechanism also allows the water molecules to flow through the fiber matric interface and resulted in higher rate of diffusivity [31]. The rate of water diffusion for some film composites that has different weight percentage of fiber can be explained due to natural behavior of the natural fiber itself whether they are hydrophilic or hydrophobic.

Based on the result below, it showed that the rate of water absorption and water content of composites film are influence by the weight percentage of the fiber. The rate of water absorption increases as the weight of percentage fiber increase [32], [33]. For the samples that has highest weight percentage of fiber, which is 10% show that it has the highest rate of water since it has the higher content of fiber. For the film sample that has lower weight percentage of fiber, the microcrack may be appear due to the fiber swell and allow high amount of water transport between the fiber matric interface.

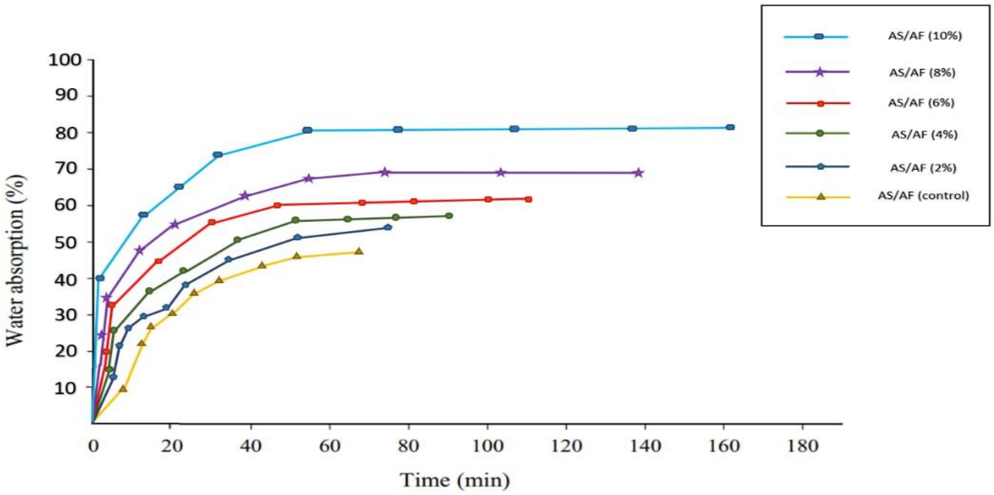


**Figure 5.** Water absorption of the control and arrowroot fiber reinforced starch composites film.

### *3.5. Film solubility in water*

For the food packaging industry, one of the important criteria needed to produce food container or wrappers are the film solubility of the composites. Thus, for this aspect, the hydrophobic behaviors of composites film are needed to improve shelf life of food product. However, the high-water solubility in composites film also needed for the edible coating and highly processed items. The film solubility in water's result for control film and arrowroot fiber reinforced arrowroot starch composites with different fiber percentage are showed in Figure 6. For the control film, the percentage of solubility in water which is 8.56% showed lowest reading compared to the arrowroot fiber reinforced arrowroot starch composites film. The result also showed that as the weight percentage of fiber increase from 2 to 10% in the arrowroot fiber reinforced arrowroot starch composites film, the percentage of film solubility in water also increase from 13.86 to 34.74%.

This result is proved by the finding from Basiak et al. [34] studied on the Effect of starch type on the physico-chemical properties of edible films. They found that the film solubility in water also increase as the fiber percentage increase. This higher rate of water solubility is influence by the amylose content in the fiber itself. While for the research by Sothornvit et al. [35] has described about the function of plasticizer in biopolymer in order to modified the intermolecular bonding in fiber matric interface. The present of plasticizer can reduce the intermolecular attraction forces of fiber matric interface and allow the higher water permeation in the film composites as the grid in film composures become less dense [30], [36]–[39]. For the features of ingesting food packaging, the higher solubility of the composites film is very useful since it in more edible and biodegradable, however for the food packaging application especially involve with liquid food product, this property is unsuitable.

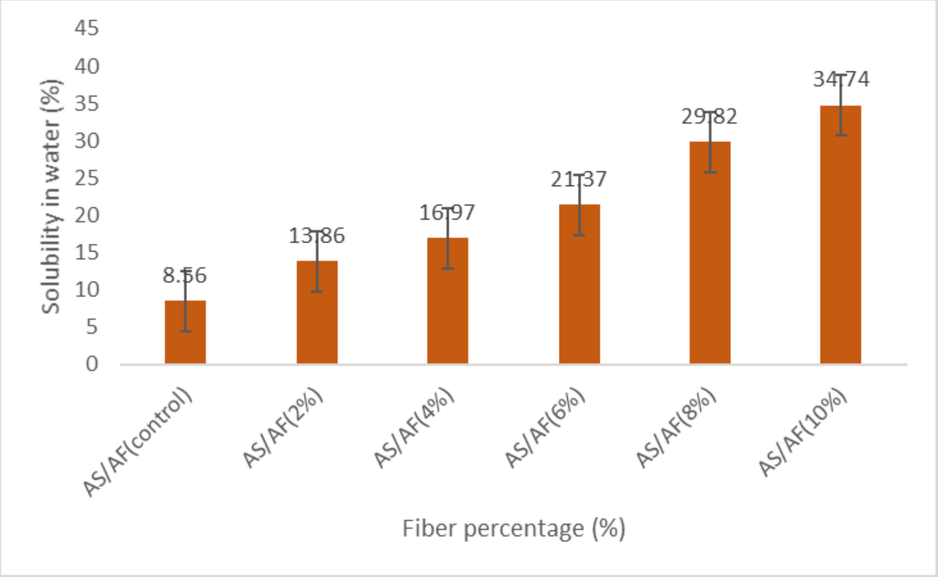


**Figure 6.** Films solubility in water.

### *3.6. Soil burial degradation*

For the soil burial degradation of composites film, bacteria, fungi, microorganism and biological factors play an important role for the decomposition process of the material. In this process, microbial organism will interact with biodegradable film during the first step of decomposition process [40]. Then, the composites film breaks down into smaller compound through the metabolic process by the action of the microbial organism with the film composites, and the average molecular weight of composites film will be reduced [41]–[43]. After that the composites film will undergo mineralization which is the complete process of the decomposition of the material [33]. For this research, a soil burial degradation test was carried for control film and arrowroot fiber reinforced starch composites film with fiber percentage (2, 4, 6, 8 and 10%) for 22 days, as shown in Figure 7.

As shown in Figure 7 for the second day, the weight loss of film samples exhibited gradual increment and began to decelerate for consequent days with degradation time for both control and arrowroot fiber reinforced starch composites film with fiber percentage (2,4,6,8, and 10%) samples. At the end of 22 days, the weight loss of AS/AF (10%) and AS/AF (8%) were found at 75% and 73%, respectively while the AS/AF (6%) and AS/AF (4%) film sample had lost 31% and 26%, respectively. While for AS/AF (2%) and control AS film sample had lost 22% and 20%. It was also calculated that the degradation rate of AS/AF (10%) and AS/AF (8%) films were 3.409 and 3.318%/day, while AS/AF (6%) and AS/AF (4%) degradation rates were 1.409 and 1.181%/day, lastly for AS/AF (2%) and control AS film sample, the degradation rates were 1.000 and 0.909%/day. It was also noticed that the decomposition of AS/AF (10%) film was higher than that of AS/AF film with fiber percentage (control, 2, 4, 6, and 8%) samples at all given points of time. Gonzalez et al. ascribed this phenomenon to the close relationship between moisture content and microbial action of the soil. In other words, the rate of degradation increased as the water content increased in films [44]. This finding was supported by the current study of water absorption results.

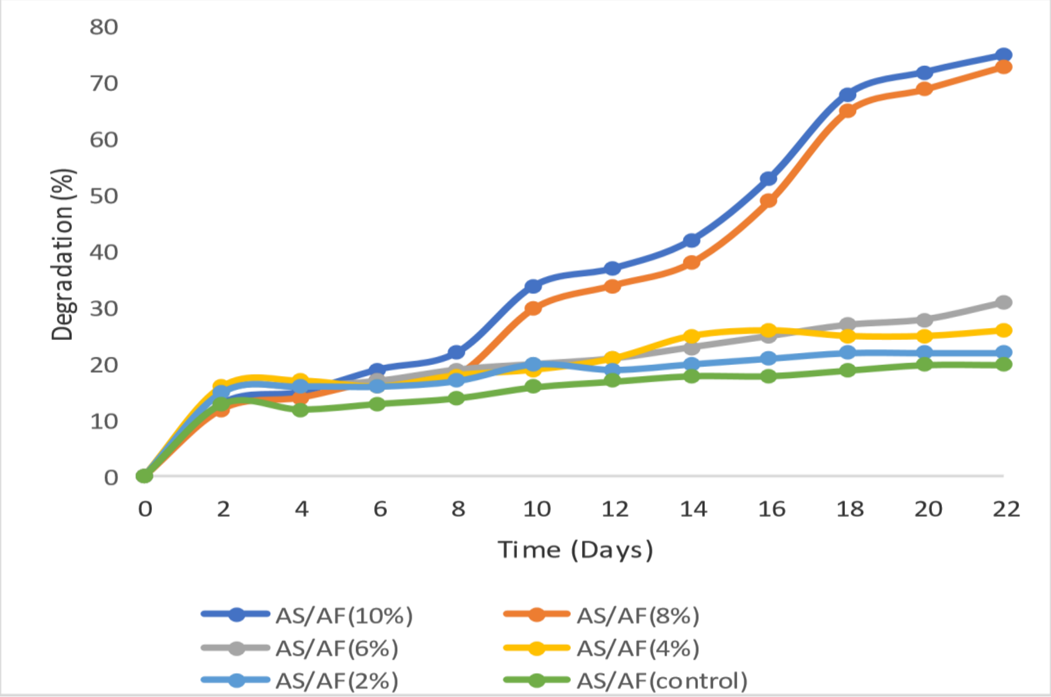


**Figure 7.** Degradation of control and arrowroot fiber reinforced starch composites film as a function of soil burial time.

## 4. Conclusions

The physical, environmental as well as sand barrier properties of arrowroot fiber reinforced starch composites film were investigated by using different percentage of arrowroot fiber. This result showed that the control AS were translucent, rigid, brittle and slightly sticky and not easily peel off from the petri dish. While for other of AS/AF biocomposites showed the film are more flexible, and peelable. Thus, the flexibility of AS/AF film was increase as the percentage of the AF increase. While for the film density, the AS/AF biocomposites showed lower density value compared to AS control. Upon increasing the fiber percentage from 2 to 10%, film density decreases from 1.472 g/cm$^3$ to 1.056 g/cm$^3$. The decrement in density could be resulted by increased thickness and volume due to increment in the fiber percentage. The moisture content of control AS film showed the lowest moisture content percentage compare to AS/AF biocomposites. Moisture content for AS/AF biocomposites enhanced significantly with enhancing the percentage of AF percentage from 2 to 10%. The hydrophilicity behaviors of AS/AF biocomposites were improved as the percentage of fiber increased. For the water absorption, the result showed that increment if fiber percentage resulted to increment in water absorption of the film. For the sample AS/AF (8%) and AS/AF (10%) film absorbed 67% and 81% after 70 minutes and continue shown constant reading until 140 minutes, the film also does not dissolve and break completely compare to another sample. Solubility in water test showed that the control AS film has lower solubility compared to AS/AF biocomposites. The solubility AS/AF biocomposites was increased from 13.86 to 34.74%, as the fiber percentage increase from 2 to 10%. For soil burial test, result showed that the decomposition of AS/AF (10%) film was higher than that of AS/AF film with fiber percentage (control, 2, 4, 6, and 8%) samples at all given points of time. The physical, environmental as well as barrier properties of AS/AF biocomposites were affected by the increment of the fiber percentage. In conclusion, the increment of fiber percentage led to improvements in the overall functioning of AS/AF biocomposites. In brief, the findings of this research provide insights into the development of biodegradable food packaging.

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