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Physicochemical Characterisation and Biodegradability of Starch Films Reinforced with Cassava Peel Microcrystalline Cellulose

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The purpose of this study was to examine the impact of MCC from cassava peel (CP) on the characteristics of starch films, as well as to analyse the physical properties and composition of the filler in the developed biodegradable starch films. The characterisation techniques, such as Fourier transform infrared (FT-IR) spectroscopy, solubility and biodegradability tests, were used to study the film properties. The FTIR analysis indicates significant interactions and modifications within the starch matrix due to the incorporation of MCC. The solubility test revealed a decrease in water solubility as the quantity of MCC increased, with the minimum water solubility recorded at 13.37%. The biodegradability tests showed a distinct correlation between the filler content and the percentage of weight loss. As the CP-MCC content increased, the weight loss of the produced starch film rose from 10.05% to 17.53%. Our findings indicate that the MCC derived from CP has the potential to manufacture high-performance biodegradable films, hence contributing to environmental sustainability.

Keywords: Biodegradable plastics; microcrystalline cellulose; Cassava peel

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Starch is a popular substance for biodegradable plastic film because it is renewable, abundant, and cost-effective [1]. However, starch-based films have drawbacks, including poor mechanical, thermal, and gas barrier qualities, as well as limited water resistance, particularly in the presence of water and humidity. In response to these drawbacks, adding fillers like cellulose to biopolymer films has been a promising strategy. According to Othman *et al*. [2], cellulose fillers can be obtained from renewable sources. For instance, agricultural waste can be them to become sustainable reinforcements.

Debnath et al. [3] highlighted that cellulose, a relatively common plant-derived biopolymer, can be synthesised into useful derivatives such as microcrystalline and nanocrystalline cellulose. These crystalline cellulose compounds are effectively utilised in starch-based films to improve thermal resistance, mechanical strength, and resistance to gases and water. MCC is primarily utilised in food processing, pharmaceuticals, and industrial applications. It is used as a binding and dissolving agent in the pharmaceutical industry, as well as a filler or reinforcement in the manufacturing of biodegradable materials and composites.

MCC is renewable, biodegradable, and non-toxic, with excellent mechanical properties. Merci et al. [4] and Yao Désiré [5] reported that using MCC as a filler material in starch films can improve its features as a packaging material. When used optimally, MCC can significantly enhance the water-repellent properties, mechanical strength, and protective performance of biodegradable materials. Thus, extend the shelf lives of fruits and vegetables. MCC is derived from various plant biomass, including tea waste [6], sugarcane bagasse [7], rose stems [8] and sweet sorghum [9].

The global annual production of cassava is approximately 256 billion tons, primarily harvested for human consumption in the form of flour, chips, and other food products [10]. The industrial processing of cassava primarily focuses on extracting starch from the roots, resulting in significant amounts of solid waste, including bagasse and peels. Approximately 11% of the total yield consists of peels as a by-product. Cassava peel contains 20 – 42% starch which made up 83% amylopectin, a branched polysaccharide and 17% amylose, a linear polysaccharide and have a significance of fibres, such as 35 – 56% hemicellulose (of which 9–13% is xylan), 6–38% cellulose, and 7–10% lignin [11].

To the best of our knowledge, there are a few studies conducted on the use of cassava peel microcrystalline cellulose (CP-MCC) as a reinforcing material in starch-based films. Thus, here, the effects of MCC from cassava peel as filler on the properties of starch-based films were investigated. Also, a comparison was made between the obtained CP-MCC with commercial microcrystalline cellulose (C-MCC)

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to identify any notable differences. These prepared samples were analysed using Fourier Transform Infrared Spectroscopy (FTIR), water solubility test and soil biodegradability test. This study is believed to provide future researchers with baseline information on the preparation and characterisation of starch-based biodegradable plastics reinforced with microcrystalline cellulose from cassava peel for food, chemical, and pharmaceutical industries.

EXPERIMENTAL

Chemicals and Materials

For this study, the microcrystalline cellulose (MCC) extracted from cassava peel (CP) was obtained from previous research conducted in our laboratory at the Faculty of Applied Sciences, Shah Alam. Starch, glycerol (99.8%), and sodium hydroxide pellets (98%) were purchased from Chemiz, Malaysia. Finally, the Commercial MCC (C-MCC) was sourced from Sigma-Aldrich, St. Louis, Missouri, USA.

Characterization Methods

Initially, 7 g of starch was mixed in 100 mL of distilled water and whisked until the starch dissolved. Then it was heated to a consistent 70 °C while it was stirred continuously for 5 minutes to obtain a homogeneous solution. Then, CP-MCC was added to prevent it from clumping. The usual quantity of CP-MCC, to add ten grams per dry-weight starch, was altered to determine the optimal starch-to-CP-MCC ratio. The formulation is shown below in Table 1.

Finally, 1.4 ml of glycerol was added to the mixture, which was stirred for 8 minutes at a constant temperature of 70 °C. The finishing mixture was then poured into a petri dish to cool and dried in a fume hood overnight. After complete drying, the films were easily peeled off the petri dish. These samples were then immediately conditioned in a desiccator before testing. This method was repeated for C-MCC. The schematic steps of the preparation of starch films reinforced with both MCC, C-MCC and CP-MCC are shown in Figure 1.

Table 1. The formulation for starch film reinforced with CP-MCC and C-MCC.

Formulation coding	Composition		
	Glycerol (ml)	Starch (g)	MCC (g)
Pure Starch Film	1.4	7	0.0
0.2 MCC	1.4	7	0.2
0.6 MCC	1.4	7	0.6
1.0 MCC	1.4	7	1.0

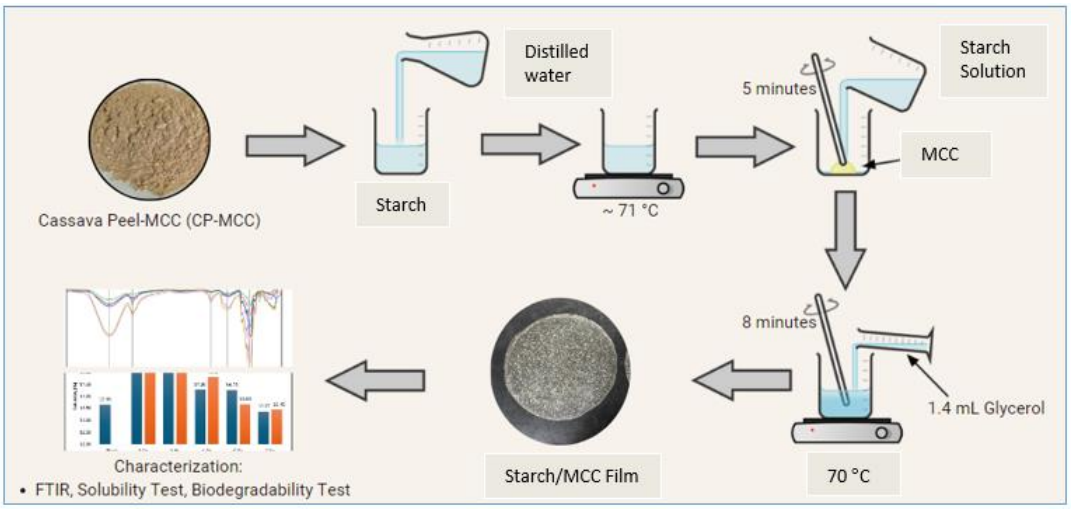


Figure 1. Schematic steps of the preparation of starch film reinforced with MCCs.

Characterisation of Sample

Fourier-transform infrared (FTIR) spectra were recorded using an attenuated total reflectance (ATR) accessory on a Shimadzu spectrometer (Shimadzu, USA). The functional groups present in the synthesised CP-MCC and C-MCC were identified by using a Perkin-Elmer instrument. The FTIR spectrum was performed in the range of 4000 cm^{-1} to 500 cm^{-1} using 8 scans.

Solubility Test

The solubility of the starch films prepared from CP-MCC and C-MCC was evaluated using the method described by Debnath et al. [12]. In determining solubility, these film pieces were precisely cut to dimensions 2 cm × 2 cm, dried in an oven at 105 °C for 2 hours, and weighed. These pieces were then immersed in 80 mL of deionised water at a controlled temperature of 25°C with constant agitation for 30 minutes at 180 rpm. Then, the remaining film pieces were dried under controlled conditions. Next, these films were filtered, dried, and weighed to determine solubility. It was calculated in triplicate using the formulae:

$$Solubility\ (\%) = \frac{Initial\ Weight - Final\ Weight}{Initial\ Weight} x\ 100$$

Biodegradability Test

The biodegradability method by Rahman et al. [13] was applied with some modifications. Film samples (CP-MCC and C-MCC) were cut into 2 cm × 2 cm pieces and weighed. These film pieces were then placed in agricultural soil contained in a pot, then covered with a plastic net with a mesh size of 1mm. They were then exposed to atmospheric conditions. During sample analysis, the weather conditions were inconsistent, with intermittent rainfall and sunny intervals, and the ambient temperature ranged between 28°C and 32°C. After 9 days, the film samples were removed carefully from the soil. The weight loss of each sample was determined by washing the samples with cold water at a temperature of 4 ºC to ensure the removal of any adhered soil particles. Following washing, the film sample was dried in an oven at 70 °C until a constant weight was achieved. The weights of the samples before and after washing were recorded. Additionally, the soil was watered for weight loss calculations. It was calculated in triplicate using the equation:

$$Weight\ loss\ (\%) = \frac{Initial\ Weight - Final\ Weight}{Initial\ Weight} x\ 100$$

RESULTS AND DISCUSSION

Functional Group Analysis using FTIR

The FTIR spectra of both CP-MCC and C-MCC with various amounts of filler were analysed to identify the presence of functional groups. Figure 2 shows the spectra of CP-MCC and C-MCC.

Figure 2 shows the FTIR spectra of starch-based film with the addition of various amounts of MCC, demonstrating potential structural changes. Based on Figure 2, the broad peak within 3200 – 3600 cm^{-1} showed the presence of stretching vibration of hydrogen bonds in the cellulose molecule [14, 12, 13]. Furthermore, the addition of MCC from 0.2 to 1.0 g to the starch-based film showed the decreased intensity of the O-H peak.

A peak was observed around 2922 cm^{-1}, which can be attributed to the presence of the amount of amylose and amylopectin present in the starch. The peaks around 1646 cm^{-1} could be associated with the water peak absorption due to strong interaction between the carbohydrates and water within starch films [15]. Other peaks, 17388 cm^{-1} is related to bending of CH and CO bonds in polysaccharides aromatic rings and the peak at 1016 cm^{-1} is due to the C-O-C stretching vibration obtained in 1,4-glycosidic links linkages of d-glucose units that appeared in all spectra at various intensities, depending on the percent content of the MCC in each sample [14, 16, 17, 18]. Overall, the synthesised CP-MCC (Figure 2 (a)) exhibits a structure similar to that of C-MCC (Figure 2 (b)), which can be attributed to the compositional similarity.

Solubility Test

Water solubility measures the ability of a film to dissolve in water. It is a crucial property for any film that may come into contact with water, as it indicates how well the film can withstand exposure to moisture. A film with high water solubility will dissolve or break down in water. Meanwhile, a film with low water solubility will be more resistant to water and remain intact for longer periods. Starch and glycerol are hydrophilic compounds present in film; therefore, they contribute to the solubility of film in water [19]. Table 2 shows the comparison between two different biocomposite films, the C-MCC and CP-MCC, and varying amounts of MCC as the parameter. The water solubility of a film depends on factors such as its chemical composition, molecular weight, and the presence of any hydrophilic or hydrophobic groups.

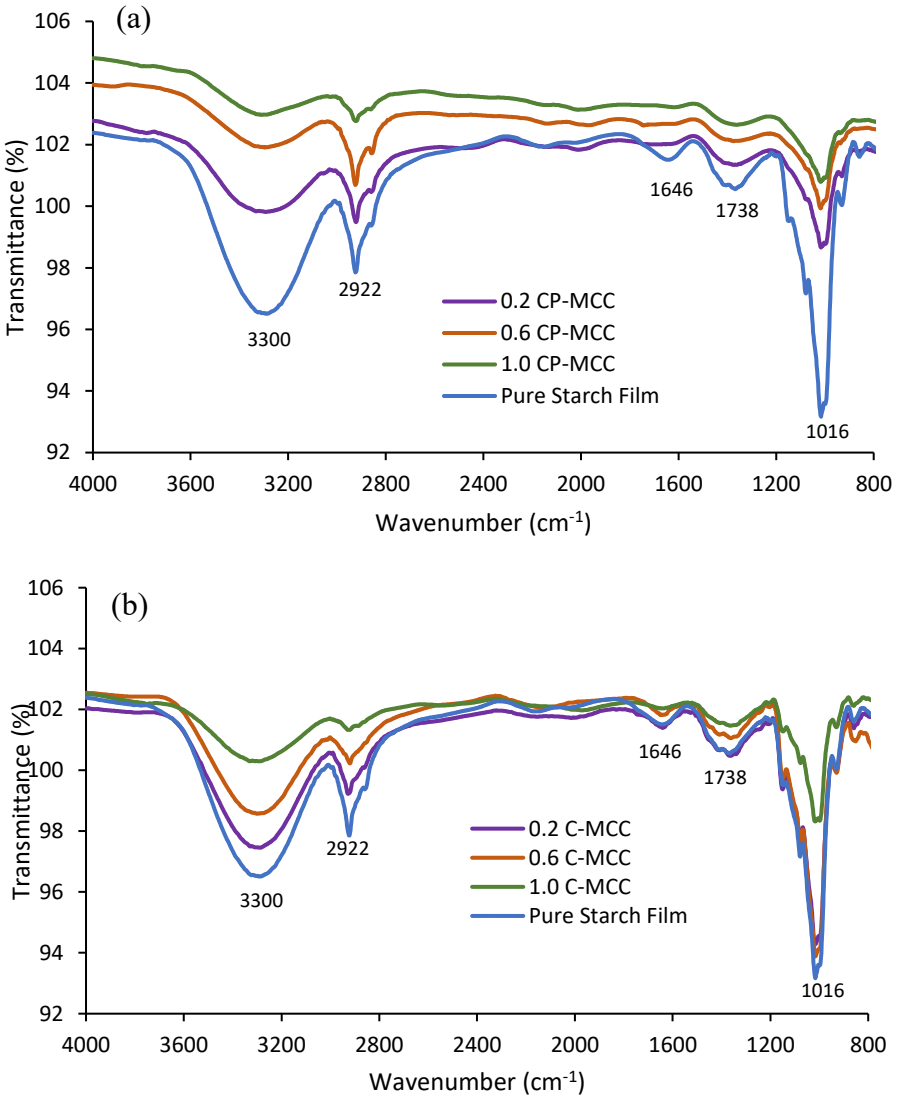


Figure 2. FTIR spectra of various amounts of filler in starch-based film (a) CP-MCC (b) C-MCC.

Table 2. Water solubility of MCCs.

Film	Water Solubility (%)
Pure Starch Film	18.65±1.90
0.2 CP-MCC	15.66±0.66
0.6 CP-MCC	14.80±1.54
1.0 CP-MCC	13.45±1.20
0.2 C-MCC	15.52±1.08
0.6 C-MCC	14.28±1.14
1.0 C-MCC	13.37±1.02

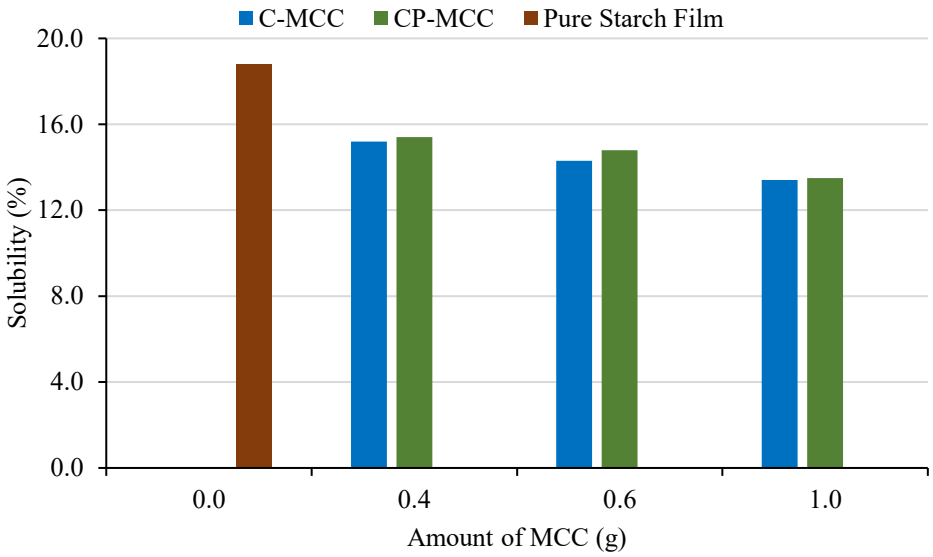


Figure 3. Water solubility (%) of starch-based films with the addition of MCC.

Pure starch films demonstrate the highest solubility at 18.65%, due to their pronounced hydrophilic properties that facilitate water absorption. The highest solubility was recorded at 15.66% for CP-MCC and 15.52% for C-MCC. Figure 3 reveals an overall trend for both CP-MCC and C-MCC films. The figure shows a decrease in water solubility as the MCC content increases, with the lowest values recorded at 13.45% and 13.37%, respectively. This decreasing value is due to adding more MCC can encourage the films to be more resistant to water. Thus, this water resistance can help in protecting against moisture, especially for packaging applications. According to Debnath et al. [12], water solubility decreased when the amount of MCC synthesised from elephant grass was increased in the packaging material. The least water solubility was 1.0g MCCs due to a lack of affinity towards the water molecules. Other than that, it may be attributed to the formation of hydrogen bonding between MCCs with the -OH group [13].

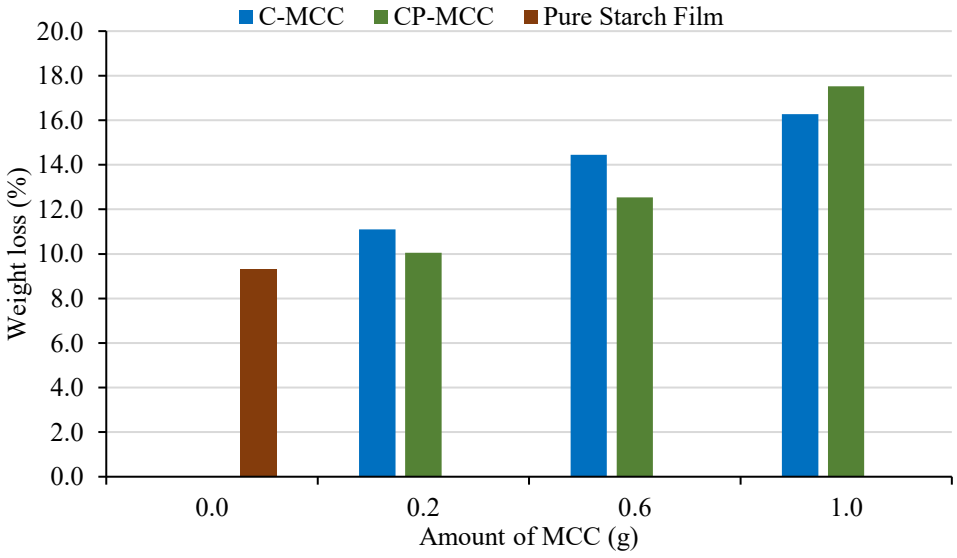


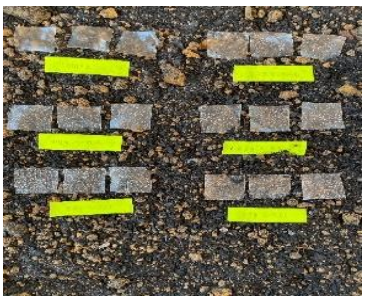
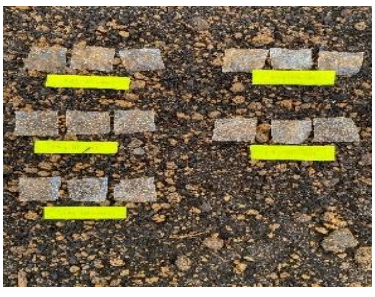
Figure 4. Weight loss (%) of starch-based films with the addition of MCC.

Biodegradability Test

Figure 4 illustrates the soil biodegradability of the prepared bioplastic samples over 9 days. The purpose of this test was to evaluate the time required for the films to degrade in the presence of soil. During the soil burial process, observation was made on the biodegradation of both CP-MCC and C-MCC films. The results indicate that the percentage of weight loss for CP-MCC and C-MCC increases with higher MCC content. The prepared film exhibited a significant increase in weight loss percentage, rising from 10.05% to 17.53% for CP-MCC and from 11.10% to 16.28% for C-MCC. Similar findings were reported by Rahman et al. [13], who observed that the lowest percentage of weight loss occurred in films with a 2.5% MCC composition, attributed to the reduced presence of O-H bonds. In comparison, the highest was observed in films with a 15% MCC composition. The addition of MCCs may contribute to effective microbial degradation of MCC, the films' increased porosity and surface area, the ease with which water can pass through them, and the combination degradation mechanism that operates together. The optimum dispersion of MCC from the starch matrix could also increase microbial access and enzymatic activity. This move will increase the overall biodegradation rate.

Table 3 displays the physical appearance of CP-MCC and C-MCC film after the soil burial test for 0 and 9 days. Starch-based films reinforced with MCC did not fully decompose within 9 days; however, their dimensions significantly reduced, and the films exhibited decrease elasticity. The use of MCC in the preparation of starch-based film has a great impact on their surface structure, either in terms of colour, shape and crack. Suklav et al. [20] reported that starch-based biocomposite films containing 10–40 wt% MCC derived from rice embryos decomposed entirely within 3 days, whereas films with 50–60 wt% MCC took 8 days to degrade fully. Nevertheless, Rendón-Villalobos et al. [21] reported that bioplastics prepared from mango cotyledon starch with 0.1% and 0.5% wt% MCC exhibited significant structural changes after 20 days of degradation. Thus, the biodegradation rate of starch-based films is influenced by the proportion of MCC added. Moreover, the biodegradation process can be affected by soil characteristics such as pH, moisture content, and microbial diversity.

Table 3. Soil biodegradability of C-MCC and CP-MCC for 0 and 9 days.

Number of Days	CP-MCC	C-MCC
0 Day		
9 Day		

In summary, microcrystalline cellulose was found to be an effective filler for enhancing the performance of biodegradable films. The optimum content of MCC depends on the type of polymer matrix used [22, 23]. Jeencham et al. [24] reported that incorporating 3% (0.25 g) microcrystalline cellulose (MCC) into starch-based films resulted in a promising bio-composite suitable for medical packaging applications, demonstrating enhanced transparency, improved mechanical strength, and increased surface hydrophobicity. In this study, due to the ongoing increasing and decreasing trends observed in solubility and biodegradability tests, determining an optimum MCC content for application in starch-based films would be a futile effort. While higher MCC content can improve strength and stiffness, excessive loading frequently leads to particle agglomeration. While increasing the MCC content may enhance film performance, excessive amounts often lead to particle agglomeration, uneven dispersion, decreased flexibility, reduced transparency, and greater sensitivity to moisture [24,25]. Therefore, careful optimisation of MCC content is critical to balance reinforcement effects with overall film performance.

CONCLUSION

In this study, the physicochemical properties of biodegradable starch-based film were enhanced by optimising the amount of MCC filler. The suitability and interaction between the starch film and microcrystalline cellulose (MCC) were evaluated through FTIR analysis, solubility tests, and biodegradability assessments. The FTIR results reveal that both starch-based films, with and without cellulose, exhibit nearly identical functional groups. However, as the MCC concentration increases, the intensity of the IR spectra decreases, notably the O-H peak at region 3300 cm^{-1}, 2922 cm^{-1} and 1016 cm^{-1}. The incorporation of MCC into the starch film improves the water resistant and biodegradability. The improved characteristics of the films can be ascribed to the establishment of a rigid hydrogen-bonded network of MCC within the matrix. The water resistant of starch films improved with the addition of MCC, while their biodegradability of starch films increased with the increment of MCC content. Therefore, the MCC content should be optimised based on the target application and the film's functional requirements.

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