Food Research 8 (Suppl. 3) : 117 - 123 (2024)
Journal homepage: https://www.myfoodresearch.com

FOOD RESEARCH

RESEARCH PAPER

Mechanical, structural and barrier properties of starch-based film reinforced with cellulose microfibres extracted from midribs of *Musa Saba'*

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Article history:
Received: 16 August 2023
Received in revised form: 26 January 2024
Accepted: 5 February 2024
Available Online: 22 June 2024

Keywords:
Filler,
Cellulose microfibre,
Starch-based film,
Packaging,
Midrib,
Musa Saba'

DOI:
https://doi.org/10.26656/fr.2017.8(S3).12

Abstract

Starch-based films have poor mechanical strength and high-water vapour permeability. This research aimed to develop biopolymeric films that contain wheat starch, glycerine, reinforcing agent, and cellulose microfibres (CMR) at 0.5 and 1 wt.%, through a casting technique. The CMR was derived from chemically treated banana midrib residues (*Musa Saba*' sp.). The effect of CMR filler on structural, mechanical and barrier properties was studied. The results show that the incorporation of CMR into the starch-based films has improved the fragility of films. The increased concentration of CMR has contributed to an improvement in film thickness (3.5–7.6%), tensile strength (8.1–55.4%), Young's modulus (4.2–32.5%), and vapour barrier permeability (12–17.5%). The good compatibility and homogenous dispersion of CMR fillers were corroborated using FESEM (EDX). In conclusion, CMR fillers have improved the properties of starch-based films and potential to be used as reinforcing elements in various polymer composites.

1. Introduction

Packaging industry is the largest and most significant application category in Malaysia's plastics market. Better wear and chemical resistance, ease of moulding, recyclability, puncture resistance, and high mechanical strength are the primary reasons for the expanding usage of plastics in the packaging industry. The packaging consumption in Malaysia is high among Southeast countries, at 16.78 kg/person. In 2020 itself, an estimation of 148 thousand metric tons of annual food plastic packaging was used (Statista, 2022; Global Information, 2023). Bioplastics are preferred due to their biodegradability and from renewable sources. Many researchers have developed packaging from starch owing to their abundance, renewability, biodegradability, and low cost (Ribba *et al.*, 2017). Starch is a polymeric carbohydrate that mainly consists of a mixture of two biopolymers; amylose (straight chain) and amylopectin (branched chain) (Hoover *et al.*, 2010). Films derived from starch are resistant to fats and oils which makes it useful for various applications in the food industry. However, its application is not in practice yet due to a number of drawbacks such as poor mechanical strength and high water vapour permeability (WVP), both of which results from the structure of starch (Ribba *et al.*, 2017).

Comprehensive and current debates regarding environmental pollution have reawakened interest in natural materials with a focus on renewable raw materials. Cellulosic fibres have received growing research interest in various engineering applications owing to their appealing properties such as biocompatibility, renewability, low cost and non-toxicity. Cellulosic fibres possess distinctive features such as low density, excellent mechanical strength, and adaptable surface properties (Camarero Espinosa *et al.*, 2013; Rajinipriya *et al.*, 2018; Bahloul *et al.*, 2021). Researchers have studied non-wood plant biomass such as residues of tomatoes, apples, carrots, cucumbers and bananas as a source of cellulose (Szymańska-Chargot *et al.*, 2017; Tibolla *et al.*, 2018). Over the recent decades, there has been an increasing trend in the research on the application of natural fibres as reinforcing fillers in polymer matrices (Salehudin *et al.*, 2014; Pelissari *et al.*, 2017; Punia Bangar *et al.*, 2022). The fibres can be applied as reinforcement in the long form, short form or derived form (i.e. cellulose microfibre, cellulose nanofibre, cellulose nanocrystals) (Elanthikkal *et al.*, 2010).

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Banana (Musaceae family), is an important fruit crop, ranked fourth after rice, wheat and maize in developing countries (Tan, 2022; Zhang *et al.*, 2022). The midrib is a by-product of the application of banana leaves for food packaging; cooking, wrapping and food serving. Banana midribs contain high cellulose content (62-64%), low lignin content (5%) and hemicellulose content (20%) (Fadarina *et al.*, 2019).

Therefore, this research aims to exploit the available sources of CMR from midrib produced from chemical treatments (steam explosion alkaline treatment, bleaching or delignification and acid hydrolysis treatment) as an application of filler or reinforcement in starch composite films. Furthermore, the films reinforced with CMR were studied in terms of their structural, mechanical and barrier properties that can be used as packaging.

2. Materials and methods

2.1 Materials

Sulfuric acid (H_2SO_4) (97%), sodium chlorite ($NaClO_2$), sodium hydroxide (NaOH) pellets, acetic acid, wheat starch and glycerine were of analytical grade, purchased from MERCK supplied by local chemical supplier and used without modification. Distilled water is used to prepare the standard solutions. Banana crop residues of midribs (*Musa Saba'* sp.) were collected after harvest in a banana plantation farm in Bintulu, Sarawak, Malaysia.

2.2 Isolation of cellulose microfibres

2.2.1 Grinding and sieving

The isolation of cellulose microfibres from banana midrib fibre requires several procedures, as summarized in Figure 1. Upon receipt, the midrib was chopped into smaller pieces and dried in a convection oven at 50°C for 24 hrs. The dried midrib pieces were then further ground into fine powder using a household blender and sieved through a 100 µm sieve. The fibre powders were then stored in an airtight storage until further application.

2.2.2 Pre-treatment and bleaching treatment

Pre-treatment and bleaching processes were required for the removal of hemicellulose, lignin and other extractives such as waxes and ashes to produce high-purity cellulose fibre (Salehudin *et al.*, 2014). The fibres were soaked in hot distilled water, known as the retting method at 70±1°C in a water bath for 3 hrs. The retting method removes impurities and large particles (Suradi *et al.*, 2009). The swollen fibres were then filtered. After that, the swollen fibres were chemically treated using 5 wt. % of NaOH in an autoclave (Hirayama HVE-50, Japan) at 121°C for 20 mins to remove the amorphous

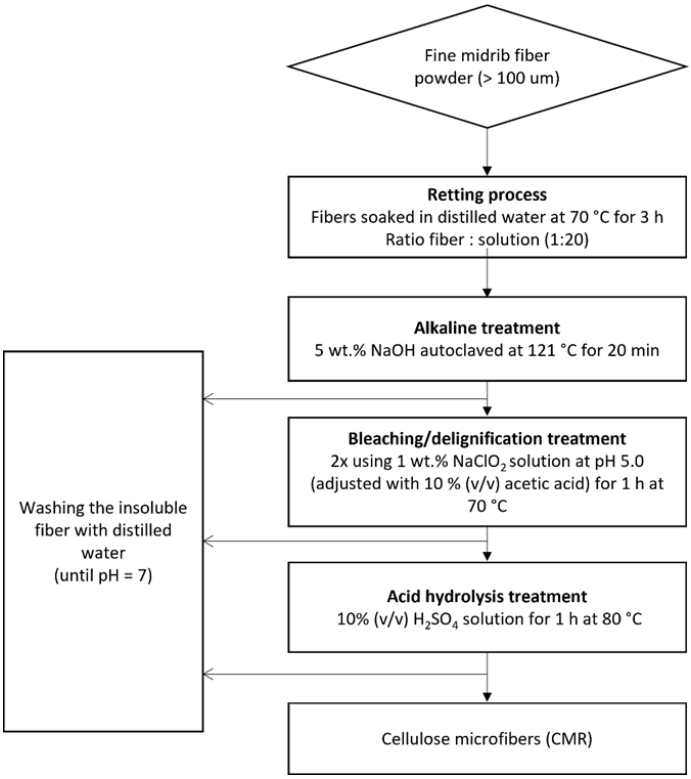

Figure 1. Process of isolation of cellulose microfibres from midrib fibres.

region of hemicelluloses and lignin from the cellulosic fibres. This approach is known as the alkali steam explosion method. Alkaline-treated fibres were separated from the NaOH solution using vacuum filtration with filter paper. The fibres were then bleached or delignified twice using 1 wt. % $NaClO_2$ solution at pH 5.0 (adjusted with 10% (v/v) acetic acid) for 1 hr at 70°C. Then, the fibres were rinsed with distilled water until the pH 7 was reached.

2.2.3 Acid hydrolysis treatment

The insoluble residue after bleaching was then subjected to acid hydrolysis treatment using 10% (v/v) H_2SO_4 for 1 hr at 80°C while being stirred vigorously using a magnetic stirrer to remove any remaining mineral residues and hydrolyze the amorphous cellulose, which only produced the required CMR. The hydrolysis treatment was stopped by the addition of cold distilled water. The suspensions were filtered using vacuum filtration with filter paper and washed repeatedly with distilled water until the pulp reached pH 7.0. The resulting microfibres were diluted in distilled water and chilled at 4°C in an enclosed Schott bottle stored in a refrigerator prior to application.

2.3 Preparation of films

The film-forming solution was prepared using the casting method. The control film without CMR, SCMR0 was prepared without the addition of cellulose microfibre. A total of 10 g of wheat starch, 5 g of glycerin, and 95 ml of distilled water were mixed

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together using a magnetic stirrer bar with medium stirring and heat was added gradually until the solution was gelatinized at 70°C. Film reinforced with CMR, SCMR0.5 and SCMR1.0 were developed by first, dissolving 10 g of starch in hot water at 50°C in a beaker, heated on a magnetic stirring hot plate with medium stirring. In a separate beaker, CMR suspension containing 0.5, and 1.0 wt.% (dry basis of total weight of solution) was homogenized at 500 rpm for 15 mins using high speed homogenizer (Wise Mix Homogenizer HG-15, Germany). These homogenized suspensions and 5 mL of glycerin as plasticizers were mixed in the starch solution under continuous stirring at 70°C until the starch was gelatinized. Then, 20 g of the solution was casted onto a disposable sterile petri dish (Ø 9 × 15 mm) and were dried in the oven at 60°C for 4 hrs. For mechanical properties studies, the mass of solution was fixed at 100 g per glass mould tray (20 cm × 20 cm × 5 cm). The solution was dried in the convection oven at 60°C for 5 hrs. The films were peeled off from their mould and stored in a sealed bag, placed in the desiccator before being characterized. The concentration of CMR (dry basis) in each prepared film was 0 wt.% (SCMR0), 0.5 wt.% (SCMR0.5) and 1.0 wt.% (SCMR1.0) respectively.

2.4 Structural properties

Field Emission Scanning Electron Microscope (JOEL JSM 7800F Schottky FESEM, Japan) was used to analyse the surface morphologies of the films. The voltage was conducted at 5 kV with an LED detector. Cross sections of the extruded films were prepared by fracturing the samples in liquid nitrogen. Platinum was sputter-coated in a thin layer onto the fracture cross sections. Energy dispersive X-ray spectrometry (EDX) was performed using X-Max 50 mm^2 Oxford instrument (United Kingdom) at 15 kV and a working distance of 10 mm.

2.5 Mechanical properties

The thickness of the film was quantitively measured using a digital micrometre (0-25 mm INSIZE 3109-25A, China (±0.001)). The thickness value was taken from an average of six random measurements of each film. Mechanical properties of the film such as tensile strength, percentage of elongation and Young's modulus were tested using texture analyzer (AMETEK Brookfield CT3 Mecomb, Malaysia), equipped with a dual grip fixture referring to standard D882-02. The size of the film specimen was cut (75 mm × 25 mm) using a scalpel, which was then mounted between the tensile grips. Both ends of the films were taped using masking tape for better grip on the specimen. The initial grip separation, trigger load and test speed were operated at 60 mm, 0.5 N and 1.0 mm/s respectively. Tensile strength (load/cross-sectional area of the sample) and elongation at break were calculated from the strength-to-elongation curve. Young's modulus was estimated as the slope of the initial linear portion of this curve. A total of 6 film samples were tested for each mechanical parameter and their average value was recorded.

2.6 Water vapour barrier properties

The water vapour permeability of the film was analyzed using the package method standard of ASTM D895-79 with few modifications. The thickness of the film was measured using a digital micrometre (0-25 mm INSIZE 3109-25A, China (±0.001)). Clear cylindrical glasses were used as the medium for this analysis and its parameter such as the diameter, surface area and its weight were recorded. The glass was filled with distilled water and the opening was covered by the films and the sides were sealed with cellophane tape. The sealed glass container was weighed and placed in the incubator at 38° C. The test was carried out in triplicate and reported as mean values. The weight of the glass containers was weighed after 2, 4, 6 and 8 days of analysis period. A graph of change in weight against time in days was plotted. The determination of the water permeability of the film was expressed in the formula given:

$$WVP = \frac{\Delta W}{\Delta t}$$

Where water vapour permeability (WVP) is the slope of the graph (g H_2O/d), ΔW is the change in weight of the container (g) and Δt is the change in time (days).

Meanwhile, to obtain the water vapour transmission rate, the formula was expressed as follows:

$$WVTR = \frac{WVP}{A}$$

Where WVTR is the water vapour transmission rate (g/m^2.d), WVP is the water vapour permeability (g H_2O/d) and A is the surface area of the container (m^2).

3. Results and discussion

3.1 Structural properties

FESEM images and EDX spectrum of the prepared film composites were presented in Figure 2. Film composites that contain midrib CMR (SCMR0.5 and SCMR1.0) were significantly different from the control film without the CMR (SCMR0). The surface image of SCMR0 film shown in Figure 2 and Figure 3 (a)(d) exhibited more transparent, uniform and smooth surface as compared to film reinforced with CMR. In contrast, films containing CMR fillers, SCMR0.5 and SCMR1.0 (Figure 3 (b)(c)(e)(f)) showed a clearly visible particulate distribution over the surface of the films. The

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distribution of the particulates (marked with yellow arrows) increased from SCMR0.5 to SCMR1.0 which implies that the particulates were originated from the presence of CMR into the film composites. CMR particulates were uniformly dispersed without aggregation owing to the application of mechanical homogeniser (Pelissari *et al.*, 2017; Tibolla *et al.*, 2020) EDX analysis was obtained to evaluate the elemental composition presented in the films (Figure 3(g) - 3(i)). The EDX spectra generated intense peaks that were associated with carbon and oxygen which means there is a presence of carbon and oxygen in the films.

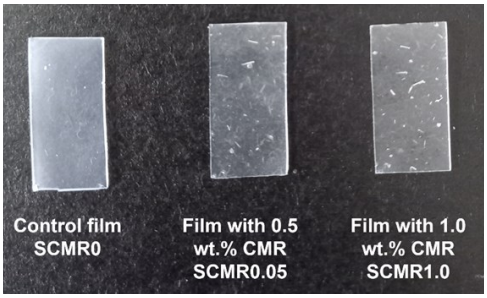

Figure 2. Images of SCMR0, SCMR0.5 and SCMR1.0.

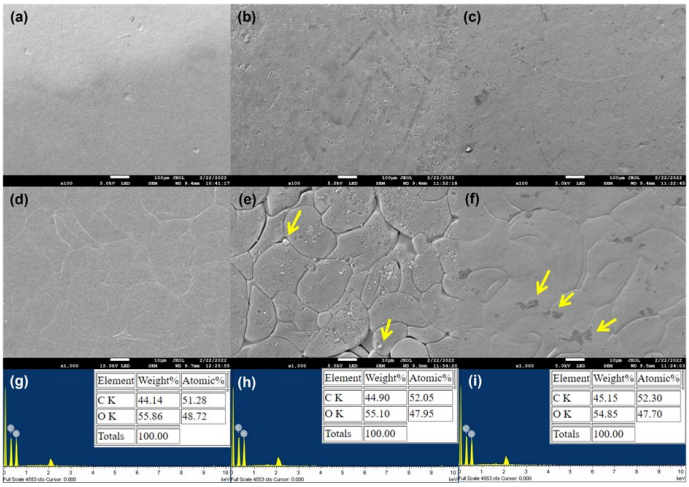

Figure 3. FESEM images (a)(b)(c) 100× magnification with scale bar 100 µm; (d)(e)(f) 1000× magnification with scale bar 10 µm; (g)(h)(i) EDX spectra of film composite: (a)(d)(g) SCMR0; (b)(e)(h) SCMR0.5; (c)(f)(i) SCMR1.0.

3.2 Mechanical properties

Mechanical properties are important for plastic products and are often quantified by tensile tests, which include tensile strength, Young's Modulus and elongation percentage. One of the drawbacks of starch-based films is their mechanical strength. An application of cellulose microfibre as a filler in film composites may improve the mechanical properties of the film. Figure 4 shows the mechanical properties including tensile strength, Young's modulus and elongation percentage of the prepared films. In this research, 1 wt.% of CMR incorporation into the film composites is the optimal amount that results in the most excellent reinforcement capability. The tensile strength and Young's Modulus of SCMR1.0 have improved significantly by 32% and 55% respectively. However, the elongation has reduced by 9.6%, showing that the control film SCMR0 has a higher elasticity.

There are few possible factors that may have influenced the improvement in mechanical strength with the addition of the isolated CMR. As supported by the FESEM results shown in Figure 3, the CMR was homogenously dispersed throughout the film. This is also possible due to stable interfacial bonding between the fibres and matrix which consequently restricts the chain motion of the starch matrix (Mahardika *et al.*, 2019). Besides, an even dispersion and distribution of CMR fillers in the film matrix due to the application of mechanical homogenizer also contributes to the improvement in the tensile properties of the film (Norrrahim *et al.*, 2021). Lastly, the optimisation of hydrogen bonds in the film composites and the interaction between polysaccharides and CMR fillers is favourable, owing to the chemical similarity and intermolecular hydrogen bonds between hydroxyl groups of macromolecules (Cao *et al.*, 2008; Chen *et al.*, 2009).

3.3 Water vapour barrier properties

Vapour permeability measures the film's ability to allow vapour to pass through it and it is one of the important characteristics of films as it greatly influences their applicability in food industries (Versaperm, 2014; Wang *et al.*, 2018). Packaging that has effective barriers may prolong product shelf life (Wang *et al.*, 2018). Table

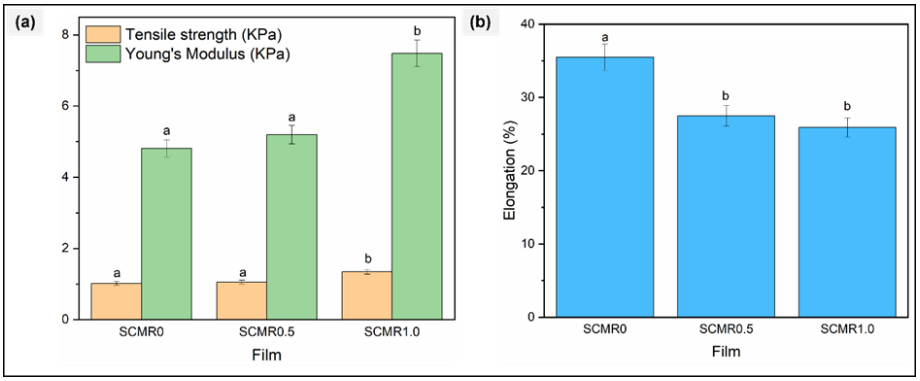

Figure 4. Mechanical properties (a) tensile strength and Young's Modulus, (b) elongation % of the films. Bars with different notations within the same graph are statistically significantly different ($p<0.05$).

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Table 1. Physical and water vapour permeability properties of films.

Properties	Units	Film sample		
		SCMR0	SCMR0.5	SCMR1.0
Thickness	µm	170±0.001[a]	176±0.001[a]	183±0.001[a]
WVP	E-09 g/m s Pa	1.776[a]	1.562[b]	1.465[b]

Values with different superscripts within the same column are statistically significantly different ($p<0.05$).

1 tabulates the results of water vapour permeability of the prepared films. SCMR0 has a higher WVP as compared to the CMR-reinforced films owing to the highly hydrophilic property of starch-based films (Ribba *et al.*, 2017). The presence of CMR has deduced the contact water with hydrophilic groups and CMR has a lesser affinity to water molecules (Müller *et al.*, 2009). The water molecules may have difficulties penetrating the cellulose crystalline region in the film matrix, associated with the enhanced material tortuosity due to the presence of the fibres and lesser hydrophilic sites available (Lavoine *et al.*, 2012; Yu *et al.*, 2017). Hence, the microfibre-network hinders the contact between starch hydrophilic groups and water molecules, therefore lessen the diffusion of water to pass through the film matrix (Lomelí Ramírez *et al.*, 2011; Yu *et al.*, 2017). This result shows that CMR fillers may become an effective vapour barrier material for packaging applications.

4. Conclusion

A sustainable source of cellulose fibres, banana midribs have the potential to be used as reinforcing components in biodegradable films. The mechanical strength (i.e., Young's modulus and tensile strength) and water vapour barrier properties of the starch-based films increased with the addition of cellulose microfibre filler. The optimum concentration of cellulose microfibre in this work was 1 wt.% which showed the highest mechanical strength and lowest water vapour permeability. The improvement in film performance was owing to the chemical similarities between starch and cellulose, hydrogen bonding interactions between the CMR fillers and matrix and homogenous and even dispersion of CMR *via* mechanical homogenizer throughout the matrix. The films produced in this work are suggested for food application as they are non-toxic and biodegradable.

Conflict of interest

The authors declare no conflict of interest.

Acknowledgements

The authors acknowledge the financial support provided by Ministry of Higher Education Malaysia (MoHE) Malaysia for the FRGS grant (FRGS/1/2020/TK0/SWIN/03/5), Swinburne Sarawak Research Supervision Grant and Project (2-5224) from Swinburne University of Technology Sarawak Campus and the Research Management Centre (RMC) of Universiti Putra Malaysia for the grant (GP-IPM 9653300).

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