Journal of Food Biosciences and Technology,
Islamic Azad University, Science and Research Branch, Vol. 14, No. 1, 1-8, 2024

Preparation and Characterization of Micro Cellulose Moisture Absorbent Pad for Food Packaging Applications

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Received: 10 January 2024 Accepted: 26 March 2024

ABSTRACT: In this research, the natural film based on starch and cellulose on a micro-scale was produced and its properties were investigated in terms of sheeting ability to absorb moisture in food packaging. For this purpose, ground cellulose fibers with 60-micrometer mesh were dissolved in 1% acetic acid solvent, starch, and glycerol. The resulting solution was converted into a film by casting the evaporation method and drying at ambient temperature. The properties of biodegradable biofilms such as swelling degree (SD), water vapor permeability (WVP), tensile strength (TS), and elongation at break (EB) were evaluated at various thicknesses and times. The results indicated that the maximum amount of SD of biopolymer (5.91) was obtained in thickness group 1 (> 0.1mm) and during 30 min of storage. The relatively high strength of the micro cellulose (MC) film was significant (6.78 ± 0.76 N) and its permeability increased by increasing the thickness of the biofilm. The results showed that due to the hydration ability and strength of MC film can be used as a moisture-absorbent bio pad for food packaging purposes.

Keywords: Bio Pad, Cellulose, Moisture Absorbent, Swelling Degree, Food Packaging.

Introduction

It is very necessary to choose packaging materials with suitable barrier properties to provide quality indicators and increase the shelf life of fresh products. Natural polymers are a suitable source of biodegradable materials for packaging applications, which are usually prepared from agricultural by-products (Yousefnia Pasha *et al.*, 2023). On the other hand, active packaging directs the adverse changes inside the package to increase the shelf life and improve the safety of the product to keep optimal conditions inside the package. This kind of packaging contains components inside the package such as moisture absorbers, oxygen removers, gas generators (carbon dioxide and ethylene), antimicrobial agents, etc. (Ebadi *et al.*, 2022). The absorbents method in different forms, including absorbent pads for meat, chicken, fruit, cheese, or seafood, oven-able pads, and the bottom layer in fish containers or tray dividers, which are a major part of food packaging that can absorb all kinds of liquid, blood, odor, ethylene, etc. usually with antibacterial/antimicrobial or

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antifungal properties and improve the appearance of the product and increase its shelf life. For packaged poultry meat, the three layers' absorbent of meat exudates which were prepared of punctured polyethylene, cellulose, and polyethylene were studied by Oral *et al.* (2009).

One natural material with a high water absorption capacity is cellulose. Cellulose $(C_6H_{10}O_5)_n$ is the most abundant substance in nature that forms the cell wall of plants and is present almost pure in cotton fibers. Cellulose is used in micro and nano-forms in food packaging (Tajeddin, 2017).

Up to now, the scientific studies and reports published about the use of biodegradable materials for food packaging are mostly in the form of film and coating to prevent the penetration of moisture, gas, etc., whereas the scientific documents for the use of biopolymers in the form of pads for permeability to moisture have been reported rarely.

Fresh foods such as meat, chicken, fish, and fruit should be well-handed and packaged the they get to the consumer cleanly and without any cross-contamination. The shelf life of chicken meat increased from 3 to 5 days by using oregano essential oil in the absorbent pad inside the package and stored at 4 ^{0} C (Oral *et al.*, 2009). In a study to improve the quality of chicken meat packaging, an absorbent pad in different concentrations of gelatin (4 to 20%) was prepared in three methods (use in a foam container, textile, and standard pad) and as a suitable alternative method for packaging chicken meat was reported (İçöz1 & Eker, 2015).

However, the industry is the pioneer in this matter, and currently, a wide range of absorbent products are offered globally because absorbency is a key requirement in many different applications of food packaging. Recently, the Sirane Company has produced pads to increase the shelf life of meat, soft fruits, and seafood. These pads have the same technology basis, but according to the specific characteristics of each product in the form of how they decay and what causes their damage and corruption, the technology varies. In the case of soft fruits, in addition to being absorbent, the pad is also absorbent and cushioned to protect the fruit more (Sirane Group, 2023). For fresh food packaging, the Pomona company produces a wide range of natural absorbent pads, which have introduced an organic solution, and liquids are trapped and do not enter fresh food. The absorbent material of this company consists of cellulose nonwoven fabric, which is combined with compostable film. The film is produced by the blowing process and then placed on the surface of the absorbent (Bioplastics Magazine, 2020).

Although pad production has been commercialized for food preservation, few published scientific papers on biopolymers exist. Therefore, in this article, a natural film based on starch and cellulose is produced on a micro-scale, and its properties and ability to be sheeting to absorb moisture in food packaging are investigated.

Materials and Methods

The natural polymer of cellulose fibers milled with a mesh of 60 µm was provided by Nano Novin Polymer Company (Mazandaran, Sari). Corn starch was obtained from Sigma-Aldrich, USA, glycerol and acetic acid were purchased from Merck chemical company Germany, magnesium nitrate hexahydrate (Fluka, Germany), and anhydrous calcium chloride was obtained from Duksan, Korea.

After preliminary preparation, two forms of cellulose (raw and milled), corn starch, and glycerol in a suitable solvent (distilled water/acetic acid 1%) were used to formulate the film by optimizing the conditions. The materials were stirred at ambient temperature, then heated for about 30 min at 80-85^{0}C until wholly dissolved and gelatinized in a heater stirrer (Heidolph Persia, Germany) (Chaichi *et al.*, 2017). The stirring speed was set at about 1000 rpm. To prevent the evaporation of the materials during heating, it was covered with aluminum foil. The ultimate pH of the solution was measured by a pH meter (Metrohm-691, Swiss-made). The film was prepared by casting method. The solution was poured into the plate to form a film in this method. The plates were dried in a flat place at ambient temperature.

Biofilm samples were prepared in five thicknesses (from 0.01 to 0.30 mm). Experiments were performed in triplicate order. The structure and characteristics of cellulose biofilm such as Swelling Degree (SD), mechanical properties (Tensile Strength (TS), elongation at break (EB), and water vapor permeability (WVP) at different thicknesses in several time intervals, were evaluated.

- Biofilm characterization

Thickness: The film thickness was measured using a micrometer (Mitutoyo, Japan) with an accuracy of 0.001 mm, at least 3 points of each film, and the average thickness was calculated.

Swelling degree (SD): The 2 × 2 cm^2 cut of biofilm samples were weighed (W_i), immersed in distilled water, and again weighed after different times (W_f). The SD of biofilms was determined by the following equation (Lavorgna *et al.*, 2010):

$$SD = (W_f - W_i)/ W_i$$

- Mechanical properties

The mechanical properties of biofilms were determined by a texture analyzer (Housfield, Model H5KS, UK) with a 500 N Load cell, according to ASTM standard method D882-02 (ASTM 2007a). Tensile strength (TS) and elongation at break (EB%) were estimated using the following equations:

$$TS\ (Mpa) = \text{Maximum force} / \text{Film width} \times \text{Film Thickness}$$

$$EB\ \% = \text{Final length} - \text{Initial length} / \text{Initial length} \times 100$$

- Water vapor permeability (WVP)

The WVP was measured by ASTM method E96-00 (ASTM 2007b) as explained with some modification by Ebadi *et al.* (2021). Due to the difference in relative humidity between the two sides of the film, vapor pressure is created, and the moisture passes through the film and is absorbed by anhydrous calcium chloride powder (Figure 1). The water vapor transition rate (WVTR) and WVP were estimated by the underneath equations:

$$WVTR = \text{Curve slope}/\text{film area}$$

$$WVP = WVTR \times \text{Thickness}/ \text{Pressure difference}$$

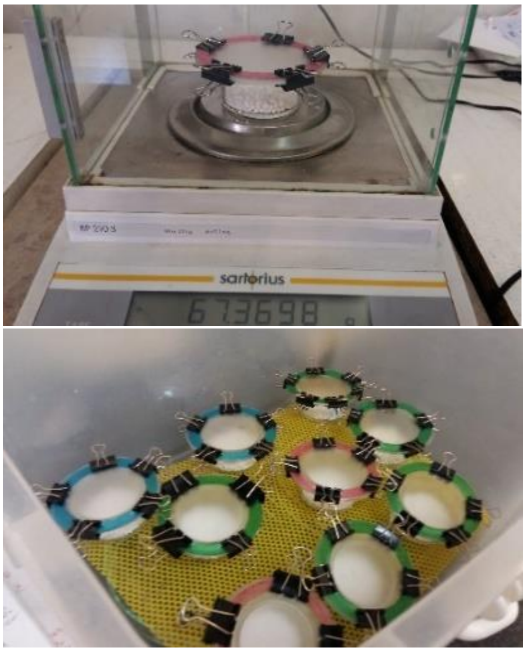


Fig. 1. Weighing the cup containing the biofilm for measurement of Water Vapor Permeability (WVP)

- Statistical analysis

Analysis of variance was performed using a general linear model (GLM) of the SAS software (version 9.4, SAS Institute, Cary, NC). Differences between means were tested using Duncan's method at the 5% level. Data were also analyzed by repeated measurement method and differences between means were tested using Duncan's new multiple range test within the SAS package.

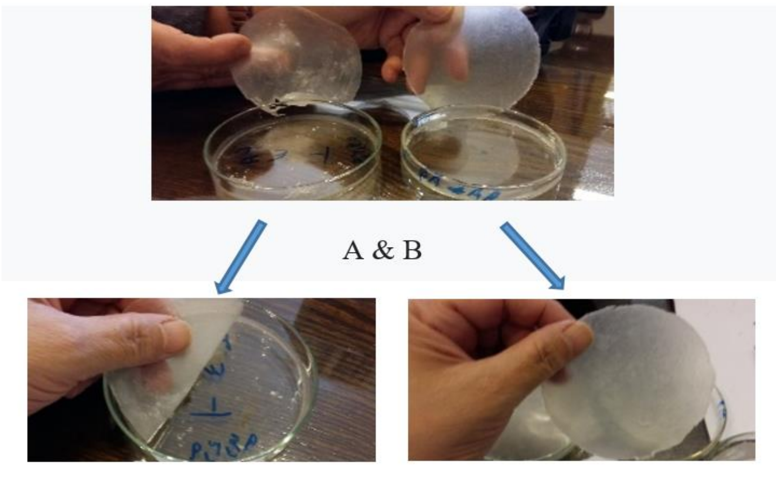
Results and Discussion

In this research, two types of raw and milled forms of micro cellulose (MC) were investigated. Initially, after preliminary preparation, the suitable experimental group was selected for film production as a moisture-absorbent pad. Due to the lack of uniformity of the film, the raw cellulose fibers were eliminated, so the micro cellulose milled sample was used for the pad preparation (Figure 2).

- Swelling Degree (SD)

The analysis showed that the SD of micro cellulose was significantly affected by the biofilm thickness in all investigated times ($p \leq 0.05$) and the results of variance analysis using the repeated measurement method show that the effect of time was significant ($p \leq 0.01$).

By thickness increasing, micro cellulose films showed that the reduced water uptake and the highest SD were obtained at thicknesses lower than 0.1 mm ($p \leq 0.05$) (Table 1 and Figure 3). It can be partly attributed to the lower surface area and interaction with water molecules in higher thicknesses (Cordeiro *et al.,* 2011). Regardless of thickness, the hydration ability of biofilms first increased and then decreased by passing the time. The highest average SD for MC biofilms was recorded after 30 min 2.03±0.56 (Table 1). The results indicated that the minimum and maximum SD were observed in the high and low-thickness samples respectively. The highest average amount of SD was obtained by 5.91 in low-thickness (> 0.1 mm) and in the storage time of 30 min. If the maximum absorption in 2 min is measured as "absorption speed" and the maximum absorption in the following times as "ultimate absorption", the values of the SD rate in the speediest time was 3.20 and the ultimate absorption was 5.91 (> 0.1 mm). This trait is remarkable for bio pad production which is probable to be utilized in food active packaging.



A **B**

Fig. 2. Comparison of milled cellulose film in different solvents (A: water and B: acid)

Table 1. Means and standard errors of swelling degree (SD) of micro cellulose in different thicknesses and time intervals

Thickness / Intervals	Mean±SE						MIN	MAX
	1	2	3	4	5	Total		
2 min	3.20±0.71^{a}	1.72±0.51^{ab}	0.96±0.24^{b}	0.69±0.01^{b}	0.75±0.06^{b}	1.47±0.34	0.68	3.92
30 min	4.92±0.98^{a}	2.35±1.10^{b}	1.16±0.12^{b}	0.93±0.12^{b}	0.79±0.04^{b}	2.03±0.56	0.75	5.91
60 min	4.27±0.80^{a}	2.29±1.29^{ab}	1.05±0.02^{b}	0.81±0.06^{b}	0.78±0.01^{b}	1.84±0.50	0.75	5.08
24 h	4.44±0.74^{a}	2.24±1.04^{b}	1.21±0.03^{b}	1.02±0.13^{b}	0.97±0.11^{b}	1.98±0.48	0.86	5.19
48 h	4.42±0.62^{a}	2.29±1.00^{b}	1.08±0.07^{b}	1.07±0.15^{b}	0.80±0.07^{b}	1.93±0.48	0.73	5.04
48 h <	3.84±0.30^{a}	1.73±0.46^{b}	1.02±0.03^{b}	1.08±0.16^{b}	0.81±0.02^{b}	1.70±0.38	0.79	4.15

The different letters indicate significant differences in a row ($p< 0.05$)
Average thickness; 1: 0.06, 2: 0.09, 3: 0.14, 4: 0.18, 5: 0.24 mm

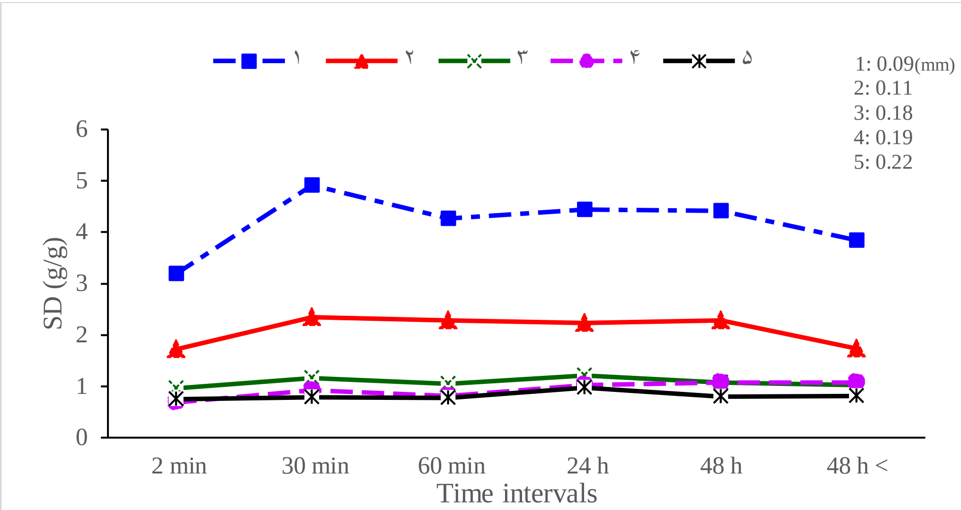


Fig. 3. Swelling degree (SD) of micro cellulose biofilms at various thicknesses and times

- Mechanical properties

Mechanical properties of cellulose biopolymer at different thicknesses including maximum load for tearing (ML), Elongation at break **(**EB), and Tensile strength (TS) are shown in Table 2 and Figure 4.

The results indicated that the average percentage of elongation at break (EB) value of MC was 15.44. The percentage of EB has a non-linear relationship with the thickness of biofilms (Figure 5) and the highest values of EB and TS were found at the thicknesses of 0.150-0.199 mm. It seems that MC biofilms were less flexible than other biopolymers such as nano cellulose and nano chitosan (Ebadi *et al.*, 2021). It can be due to the agglomeration of micro cellulose particles in the film matrix leading to strong interactions between molecules (Cao *et al.*, 2008 and Ma *et al.*, 2009). Our study is supported by the work of Ljungberg *et al.* (2005), which have described dispersion quality plays a key role in the mechanical properties of the nanocomposite and they indicated higher EB % and lesser fragility compared to including aggregated samples.

By increasing the thickness of the biofilm of MC, the ML for stretching the film increases, therefore in this study, it was 3.09 to 11.55 N for the first and fourth thicknesses respectively (Table 2). The strength for tearing the micro cellulose biofilm was significantly high (6.78 ± 0.76 N) in comparison with nano cellulose biofilms (1.84 ± 0.19 N) (Ebadi *et al.*, 2021).

- Water Vapor Permeability (WVP)

Due to the main function of water in food decomposition, WVP is a significant characteristic in the evaluation of biofilms. The results showed that the WVP of micro cellulose was 0.99×10^{-10} g/msPa and by increasing the thicknesses of the biofilm, its permeability improved slightly in nano and micro cellulose samples (Figure 5), however, there was no statistically significant difference in high-thickness. In our previous study (Ebadi *et al.*, 2021) it was concluded that the WVP of nano samples had more permeability than the micro ones. Cellulose fibers may increase the tortuosity in porous materials which can provide lower permeability, therefore micro cellulose seems to be much more effective on the film tortuosity than nano-fibrils (Ma *et al.*, 2009 and Kristo & Biliaderis, 2007).

Table 2. Means and standard errors of mechanical properties of micro cellulose biofilms

Thickness	Mean±SE		
	Elongation at break (%)	Tensile strength (MPa)	Max Load (N)
1	12.82±0.52 [b]	3.65±0.18 [b]	3.09±0.30 [d]
2	15.95±0.11 [a]	4.71±0.02 [a]	6.92±0.02 [c]
3	17.57±0.69 [a]	5.15±0.19 [a]	9.24±0.43 [b]
4	16.90±1.54 [a]	4.96±0.48 [a]	11.55±1.12 [a]
Total	**15.44±0.61**	**4.49±0.19**	**6.78±0.76**

Numbers of the same column with different letters indicate significant differences ($P< 0.05$)
Average thickness; 1: 0.099 >, 2: 0.100-0.149, 3: 0.150-0.199, 4: 0.200-0.249, 5: 0.250 < mm

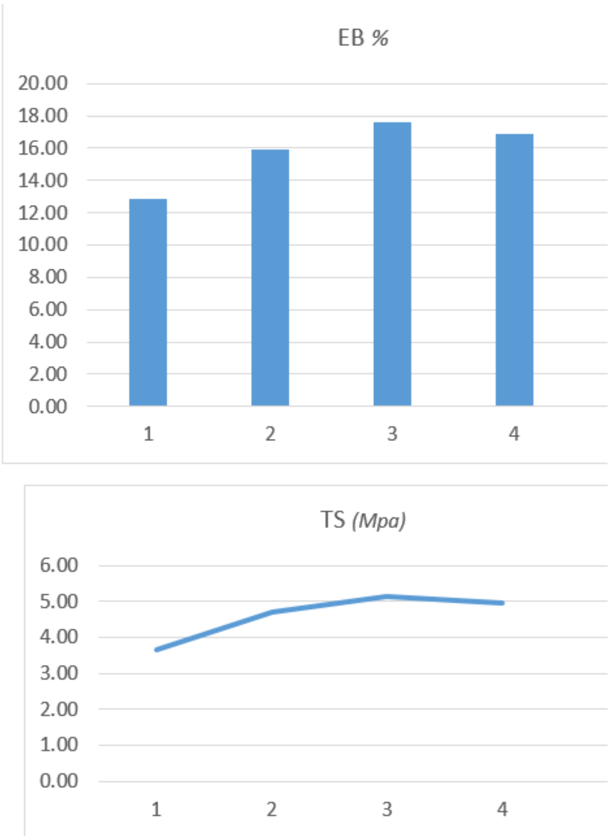


Fig. 4. Mechanical properties of micro cellulose (MC) biofilm in different thicknesses
Average thickness; 1: 0.099 >, 2: 0.100-0.149, 3: 0.150-0.199, 4: 0.200-0.249, 5: 0.250 < mm

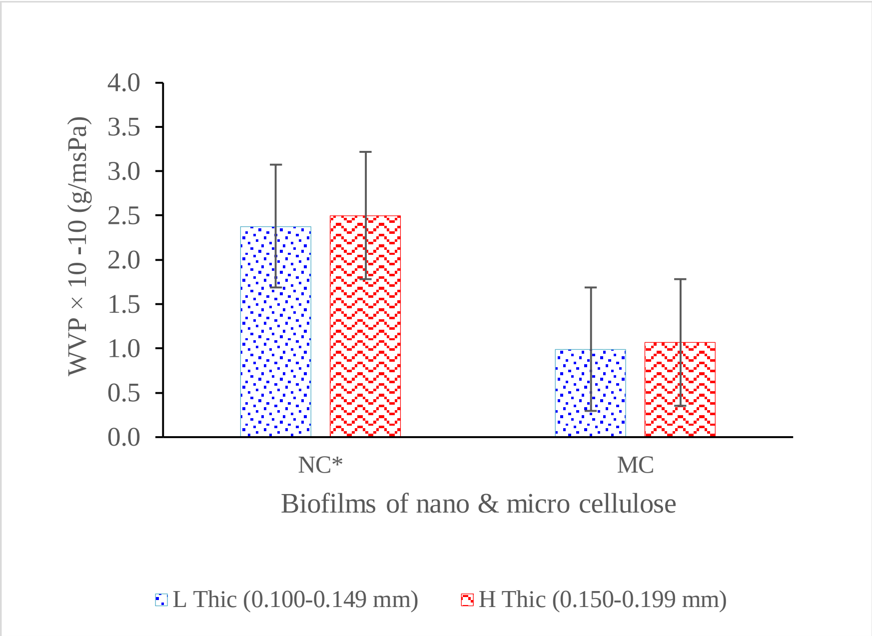


Fig. 5. Water Vapor Permeability (WVP) of nano cellulose (NC) and micro cellulose (MC) biofilms at different thicknesses
NC*: Figure adapted from Ebadi et al., 2021

Conclusion

The natural film based on starch and cellulose on a micro-scale was produced with sheeting ability to absorb moisture in food packaging. The properties of biopolymers were affected by the thickness of the MC film. The highest levels of SD were attained at low thickness in a short time. The high percentage of EB improves the flexibility of the MC biofilms at high thickness. Due to the hydration ability and strength of MC biofilm, it is suggested to use it as a moisture-absorbent bio pad in active food packaging. Moreover, the fabrication of MC films is from natural ingredients; consequently, is environmentally friendly.

Abbreviations

Micro cellulose: MC; Nano cellulose: NC; Swelling degree: SD; Water vapor permeability: WVP; Tensile strength: TS; Elongation at break: EB; Maximum load: ML; American Society for Testing and Materials: ASTM; Statistical Analysis System: SAS

Acknowledgments

We truthfully appreciate the Animal Science Research Institute (ASRI) of Iran, and the Agricultural Engineering Research Institute (AERI) of Iran, for supporting this study.

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