

ISOLATED PINEAPPLE PEEL MICROCELLULOSE AS A MATRIX FOR POLYPYRROLE-BASED CAPACITOR

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Received March 16, 2024

This study investigates the viability of utilizing microcellulose extracted from pineapple peel waste as a sustainable and cost-effective matrix material for polypyrrole (PPy)-based capacitors. A novel PPy/microcellulose composite was synthesized with varying pyrrole volumes (1-2 mL) and characterized using Fourier Transform Infrared (FTIR) and X-ray diffraction (XRD) analyses. FTIR analysis confirmed successful cellulose isolation, while XRD indicated a similarity between the extracted pineapple peel and commercial cellulose. Capacitor cells were fabricated using the synthesized composites, and their capacitance performance was evaluated. Notably, cells containing 2 mL of PPy exhibited the highest capacitance values. Additionally, the source of cellulose had minimal influence on the resulting capacitance. This study suggests that microcellulose derived from pineapple peel waste holds promise as a sustainable alternative to traditional matrix materials for high-performance capacitors.

Keywords: capacitor, microcellulose, pineapple peel, polymer composite

INTRODUCTION

One of the challenges faced when implementing Renewable Energy (RE) is preparing materials for storing the generated energy. Energy storage devices are typically manufactured using inorganic materials, such as Ni, Ni-Cd, and Li. However, these devices face challenges related to natural decomposition. Therefore, exploring material substitution in producing these energy storage devices is necessary. One potential energy storage alternative is using organic materials derived from conductive polymers.¹ Conductive polymers, such as polyaniline, polypyrrole (PPy) and polythiophene are cost-effective,² have a low environmental impact,³ exhibit high pseudocapacitance,⁴ and are easy to fabricate.⁵ However, their observation by Zhuo *et al.*⁶ and others has highlighted the significant challenge of limited cycle stability in conductive polymers for capacitor applications. This instability arises from volumetric transformations that occur during the charge-discharge process. To overcome this

limitation, researchers have actively pursued the development of novel composite materials. These composites exploit the synergistic combination of electrostatic charge storage and Faradaic redox reactions, aiming to achieve enhanced stability and overall electrochemical performance. A study by Afzal *et al.*⁷ demonstrated that carbon nanotubes (CNT) embedded in nano-sized PPy can maintain 72% capacitance after 3000 cycles (1 Ag⁻¹). Furthermore, the reduced graphene oxide or graphite oxide composites with PPy prepared could retain 85% capacitance after 1000 cycles.⁸

Conversely, agricultural waste can be used as a carbon source. Pineapple peel, which is rich in cellulose, is one such waste that can be utilized for size reduction into microcellulose. Approximately 36% of the pineapple fruit is comprised of the peel, which is often categorized as environmental waste.⁹ Therefore, utilizing microcellulose from pineapple peel can be a

sustainable solution to reduce waste, and also have potential economic benefits.¹⁰

This research aims to characterize and assess the capacitance of the composite made from pineapple peel microcellulose and PPy. The microcellulose will be characterized using Fourier Transform Infrared (FTIR) and X-ray diffraction (XRD) instruments. FTIR will confirm the isolation of cellulose from pineapple peel by comparing its functional groups with those of commercial cellulose. The crystallinity of the microcellulose isolated from pineapple peel will be determined using an XRD instrument. After assembling the composite into parallel plates, capacitance measurements will be conducted. The PPy/microcellulose composite with four variations of the pyrrole volume will be used to fabricate these parallel plates. This study also investigates the influence of pyrrole volume on the measured capacitance of the PPy/microcellulose composite. The capacitance of parallel plates using commercial PPy/microcellulose filler will also be measured and compared to establish a baseline for comparison.

EXPERIMENTAL

Materials

Hydrochloric acid, ammonium persulfate, hydrogen peroxide, nitric acid, sodium hydroxide, sodium sulfite, and commercial microcrystalline cellulose were acquired from Merck, Germany. Pyrrole ($\geq 98\%$) was purchased from Sigma Aldrich, while ethanol and sodium hypochlorite (NaOCl) were technical-grade.

The pineapple peel was obtained from the traditional market in Pontianak, West Kalimantan, Indonesia.

Instruments

In this research, the FTIR spectra were obtained using a Shimadzu Prestige-21 Spectrophotometer and were collected over the wavenumber range of 4000-400 cm^{-1} . Powder XRD analysis was performed using a Bruker D2 Phaser diffractometer. Other standard laboratory equipment employed included an 80-mesh sieve, magnetic stirring apparatus, Petri dishes, Bucher funnel, condenser, oscilloscope, oven and grinder.

Cellulose isolation

Cellulose isolation started by preparing and pre-treating the pineapple peel, which was thoroughly cleaned, air-dried, and ground into an 80-mesh powder. Ten grams of this powder were then mixed with 250 mL of 3.5% HNO_3 containing 5 mg NaNO_2 . The mixture was heated at 90 °C for 2 hours with continuous stirring. Subsequently, the mixture was filtered, and the residue was thoroughly washed with distilled water until reaching a neutral pH.¹¹ The residue was treated with 250 mL of a solution containing 2% NaOH and 2% Na_2SO_3 at 50 °C for one hour.

The mixture was then filtered, and the residue was washed with distilled water until a neutral pH was achieved. The residue was bleached using 250 mL of 1.75% NaOCl solution for 30 minutes at 70 °C. The mixture was then filtered and washed with distilled water. The bleached residue was purified with 250 mL of 7.2% H_2O_2 at 55 °C for 2 hours. The mixture was filtered, and the obtained residue was hydrolyzed with 1.5 M HCl solution at a 1:20 (w/v) ratio for 90 minutes at 75 °C under constant magnetic stirring.

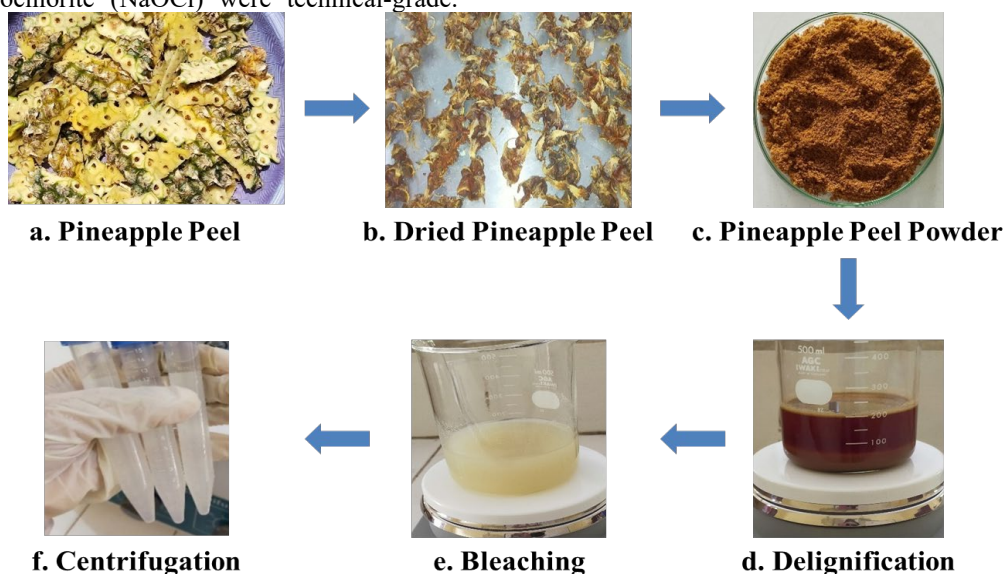


Figure 1: Process of cellulose isolation from pineapple peel

The resultant mixture was filtered, and the residue was washed with distilled water until a neutral pH was reached. The post-filtering residue was air-dried and then subjected to sonication for 1 hour, followed by centrifugation at 4,000 rpm for 30 minutes. Finally, the obtained material was oven-dried at 60 °C for 6 hours, resulting in microcellulose. The process of cellulose isolation derived from pineapple peel is presented in Figure 1.

Preparation of pyrrole composite

The pyrrole composite was prepared following established methods,^{13,14} with certain adjustments. Pyrrole monomer (1 mL) was added to distilled water (50 mL) and stirred using a magnetic stirrer for 30 minutes at room temperature. Microcellulose (0.5 g), derived from pineapple peel, was subsequently added to the mixture, followed by another hour of continuous stirring. The container was confidently sealed with plastic film during this stage to prevent the evaporation of pyrrole effectively. Polymerization was assertively initiated by adding 10 mL of a 5.8% ammonium persulfate (APS) solution, and the mixture was left undisturbed for 4 hours. Subsequently, the mixture was filtered and the resulting composite was thoroughly washed with distilled water and methanol. Finally, the composite was dried in an oven at 50 °C for 24 hours. The synthesis procedure was replicated using two additional variations, employing 1.5 mL and 2 mL of pyrrole, to examine the effect of pyrrole concentration on the composite. Moreover, for comparison purposes, we also assertively repeated the entire polymerization process using commercially available microcellulose, following the same three pyrrole volume variations (1 mL, 1.5 mL, and 2 mL).

Fabrication of PPy/microcellulose and graphite electrode plates

An aluminium plate measuring 6 cm x 3 cm was immersed in a 1 M NaOH solution, followed by air drying. A blend was prepared by combining 0.5 g of PPy/microcellulose composite with 0.5 g of polyvinyl alcohol, which was moistened with heated distilled water at 40 °C until a viscous consistency was achieved. This mixture was then uniformly applied onto the aluminium plate and left to dry at ambient conditions. Similarly, this method was employed for making another electrode plate using the same dimension as the previous aluminium plate, but using graphite instead of cellulose.

Constructing capacitor cells

Capacitor cells were constructed by assembling two electrode plates, separated by a filter paper saturated with 1 M KOH electrolyte solution, and secured with parafilm.¹⁵ These cells contained either a PPy/microcellulose composite electrode (tested for capacitance) derived from pineapple peel

microcellulose or a commercially available microcrystalline cellulose composite, both paired with a graphite counter electrode. Three PPy/microcellulose composite variations were synthesized using different pyrrole volumes (1 mL, 1.5 mL, and 2 mL). After assembly, the constructed cells were evaluated for voltage and capacitance using a multimeter and LCR meter, respectively.

Capacitance evaluations

Capacitance measurements of PPy/microcellulose composite plates were conducted using an oscilloscope. In those measurements, the electric current used was alternating current with voltage variations of 2, 3, 4, 5, and 6 V at a frequency of 50 Hz. The results measured were the electric current (amperes) that flowed through the plate, and the data gathered was analyzed to determine the capacitive reactance. The PPy/microcellulose composite plate's capacitance measurements were conducted using an oscilloscope with an alternating current (AC) at 50 Hz. Voltage variations of 2, 3, 4, 5, and 6 V were applied during the measurement. The measured data, representing the electric current (amperes) flowing through the plate, was then analyzed to determine the capacitive reactance, from which the capacitance was derived. The electric current data is presented in Table 3. The capacitance values for the composite plates, calculated using Equation (1), are shown in Table 4.

$$C = \frac{1}{2\pi f} \cdot \frac{I}{V} \quad (1)$$

where $f = 50$ Hz, which is the frequency of alternating current (AC), $C =$ capacitance in farads, $V =$ voltage in volts, $I =$ current in amperes.

RESULTS AND DISCUSSION

The initial pineapple peel powder exhibited a brown color, suggestive of high concentrations of hemicellulose and lignin compounds. Following the isolation process, the powder transitioned to a white color. This distinct color change signifies the effectiveness of the delignification, bleaching, and hydrolysis steps in reducing the content of lignin and hemicelluloses within the pineapple peel, ultimately revealing the underlying white cellulose. Other research corroborates this observation, highlighting that the brightness and whiteness of cellulose serve as an indicator of its quality, with whiter and brighter isolated cellulose signifying higher quality.¹⁶ This study successfully isolated cellulose, and from 10 g of pineapple peel powder, a yield of 8.4% was achieved. Notably, the isolated cellulose displayed a bright white appearance, further confirmed by the visual comparison of the

pineapple peel powder's color before and after isolation, presented in Figure 2.

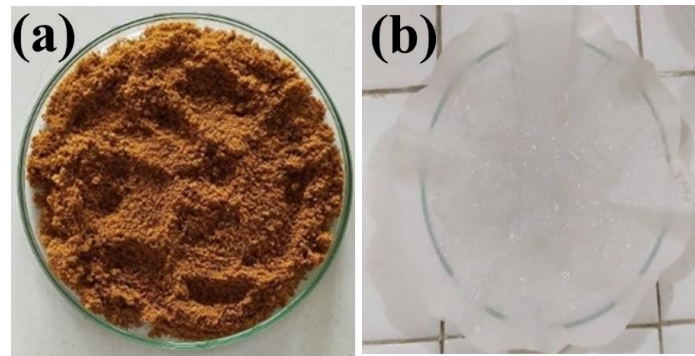


Figure 2: (a) Pineapple peel powder 80 mesh, (b) pineapple peel cellulose after hydrolysis

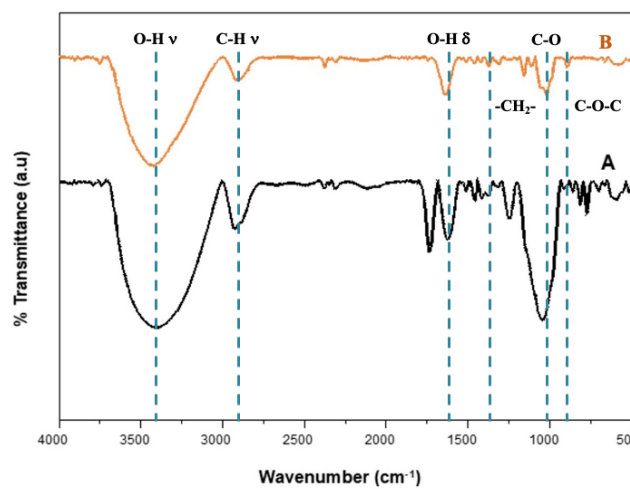


Figure 3: FTIR spectra of (A) microcellulose derived from pineapple peel; (B) commercial microcellulose

Table 1

Comparison of wave numbers and associated functional groups obtained for commercial microcellulose and microcellulose extracted from pineapple peel

Functional group	Wave number (cm ⁻¹)	
	Commercial microcellulose	Cellulose from pineapple peel
O-H stretching (ν)	3350	3442
C-H stretching (ν)	2900	2916
O-H bending (δ)	1641	1637
-CH ₂ -	1431	1425
C-O	1058	1056
C-O-C	896	896

FTIR analysis of microcellulose

FTIR spectroscopy was employed to elucidate the presence and characteristics of functional groups within microcellulose extracted from pineapple peel. A comparative analysis was conducted using commercially available pure microcellulose as a control. Figure 3 depicts the

acquired FTIR spectra, and Table 1 summarizes the corresponding assigned functional groups.

Analysis of the spectral data in Figure 3 and the details in Table 1 reveal slight variations in the absorption intensities of functional groups between microcellulose derived from pineapple peel and the commercial variant. The O-H stretching band, typically observed in the 3000-

3700 cm^{-1} region, signifies the presence of hydroxyl groups and potential hydrogen bonding, as documented in the literature.^{17,18} The analysis of the microcellulose samples revealed relatively minor differences in the O-H stretching bands, indicating that both samples fall within the typical range for this functional group in microcellulose. It is imperative to consider the slight variations in absorption intensities between the pineapple peel-derived microcellulose and the commercial microcellulose, as they could be attributed to differences in the isolation and processing methods used for each sample.¹⁹

Moreover, the presence of strong absorption bands corresponding to O-H stretching in the spectra suggests the presence of strong hydrogen bonding within the microcellulose samples, aligning with previous studies on microcellulose structure and properties.²⁰ In this study, the O-H stretching band for pineapple peel microcellulose appears at 3442 cm^{-1} , while the commercial microcellulose exhibits it at 3350 cm^{-1} . While the difference in O-H stretching frequencies between the two samples is minimal, both fall within the typical range reported for this functional group.

The consistent detection of C-H stretching bands in both spectra, characteristic of microcellulose, further supports the successful isolation of microcellulose from the pineapple peel. As detailed in Table 1, the FTIR spectra in Figure 3 confirm a common characteristic of microcellulose: the presence of C-H stretching bands. These bands typically manifest as absorption peaks around 2901-2917 cm^{-1} .²¹ The findings demonstrate that the C-H stretching band for commercial microcellulose appears at 2900 cm^{-1} , while the pineapple peel microcellulose

band is observed at 2916 cm^{-1} , consistent with previous research.²²

A peak around 1060 cm^{-1} signifies the water absorption capability of microcellulose. The pineapple peel microcellulose exhibits a peak at approximately 1056 cm^{-1} , while for the commercial one, it is observed at 1058 cm^{-1} . Additionally, observations of ether groups (C-O) between 1285-1035 cm^{-1} are crucial for microcellulose structure with notable peaks observed in both pineapple peel-derived and commercial microcellulose samples. A peak observed around 1600 cm^{-1} indicates the presence of lignin within the microcellulose, as reported earlier.²³ Both commercial and pineapple peel microcellulose spectra reveal peaks in this region, likely attributable to incomplete delignification during the isolation process. The successful isolation of microcellulose from pineapple peel is evident from detecting various vibrations and functional groups in the FTIR analysis, comparable to those observed in both commercial microcellulose and reference cellulose.

XRD analysis of microcellulose

XRD analysis was performed to characterize the crystallinity of the isolated microcellulose. Figure 4 presents the diffractograms of the isolated pineapple peel cellulose (B), commercial cellulose (C), and the untreated 80-mesh pineapple peel powder (A) for comparison. As reported in the literature,²⁴ the XRD pattern shows peaks corresponding to the intensity of X-ray scattering at specific angles (2θ) and the distance between the scattering plane and the crystalline fragment.

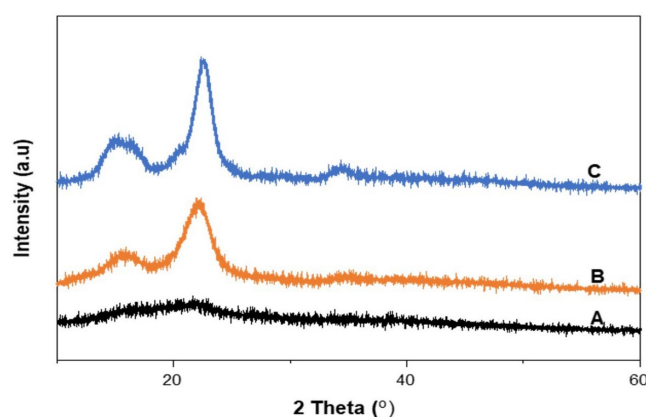


Figure 4: X-ray diffractograms of (A) untreated 80-mesh pineapple peel powder, (B) isolated microcellulose from pineapple peel, and (C) commercial microcellulose

The non-crystalline cellulose exhibits a broader and lower intensity peak, while a sharp and prominent peak in the diffraction pattern characterizes the crystalline portion of cellulose.²⁵

Figure 4 presents the X-ray diffractograms of the untreated pineapple peel powder (A), isolated microcellulose from pineapple peel (B), and commercial microcellulose (C). The untreated pineapple peel powder (A) exhibits the most pronounced peak at a 2θ angle of 15.1° , characterized by a broad and less intense signal. In contrast, the isolated microcellulose (B) displays a sharper and more intense peak at $2\theta = 22.2^\circ$ with a d-spacing of 4.0 \AA . The commercial microcellulose (C) shows the highest intensity peak at $2\theta = 22.7^\circ$. The diffraction pattern of the isolated microcellulose (B) closely resembles that of the commercial microcellulose (C). This observation aligns with previous reports,^{26,27} where the most intense peak was observed around $2\theta = 22\text{--}22.8^\circ$, accompanied by twin peaks at 14.9° and 16.5° . Additionally, an intensity spike was noted around $2\theta = 34\text{--}35^\circ$ in these studies, which is consistent with the increased intensity observed at $2\theta = 34.3^\circ$ in the isolated pineapple peel microcellulose (B).

Analysis of the composite

The composite is formed by combining microcellulose and polypyrrole. Microcellulose serves as the reinforcing matrix, offering

structural support and stability. Polypyrrole, synthesized through the oxidative polymerization of pyrrole monomers, is dispersed within the microcellulose matrix. As documented previously,²⁸ the oxidative polymerization process necessitates an oxidizing agent to accelerate the reaction. In this work, ammonium persulfate was utilized as the oxidizing agent. The functional groups present within the PPy/microcellulose composite were investigated using FTIR spectroscopy. The resulting spectra, presented in Figure 5, were analyzed to identify characteristic peaks associated with the constituent materials. Typically, the PPy ring exhibits characteristic stretching vibrations in the FTIR spectrum at 1319.3 cm^{-1} (C-N) and 1558.5 cm^{-1} (C=C).²⁹ Additionally, another study reported peaks at 1044 cm^{-1} (C-C) and 789 cm^{-1} (C-H) for PPy.³⁰ These characteristic peaks were observed in both composites with commercial or pineapple peel PPy/microcellulose, with minimal deviations. The commercial composite displayed peaks at 1056 cm^{-1} (C-C) and 792 cm^{-1} (C-H), while the pineapple peel composite exhibited peaks at 1045 cm^{-1} (C-C) and 790 cm^{-1} (C-H). Furthermore, the PPy/microcellulose analysis revealed peaks associated with cellulose at 1560 cm^{-1} (C=C) and 1454 cm^{-1} (C-N) for the commercial composite and 1552 cm^{-1} (C=C) and 1473 cm^{-1} (C-N) for the pineapple peel composite.

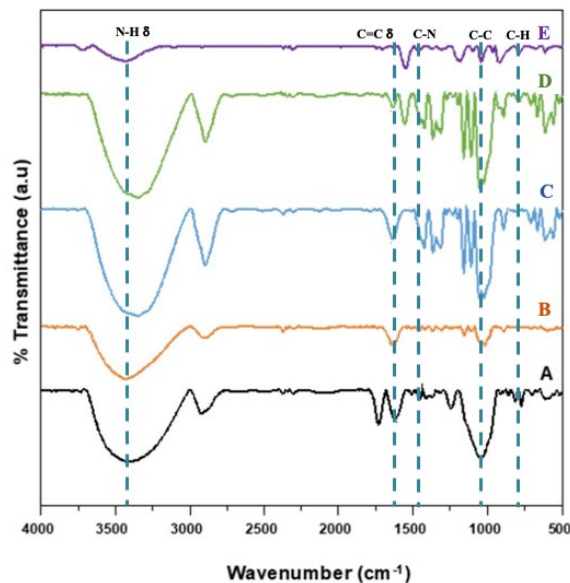


Figure 5: FTIR spectra of (A) composite of PPy/commercial microcellulose; (B) composite of PPy/microcellulose isolated from pineapple peel; (C) commercial microcellulose; (D) microcellulose from pineapple peel; (E) raw 80-mesh pineapple peel powder

Tabel 2

FTIR analysis results for PPy/cellulose composites: wavenumber and associated functional groups of PPy/microcellulose composites compared to commercial microcellulose

Functional groups	Wavenumber of PPy/commercial microcellulose (cm ⁻¹)	Wavenumber of PPy/extracted microcellulose (cm ⁻¹)	Polypyrrole wavenumber according to references (cm ⁻¹)
N-H bending (δ)	3350	3444	3405; ²⁹ 3479 ³⁰
C=C bending (δ)	1560	1552	1581 ^{29,30}
C-N	1454	1473	1477 ^{29,30}
C-C	1056	1045	1044 ³⁰
C-H	792	790	789; 600 ³⁰

Table 3

Electric current measurements of PPy/microcellulose composite capacitors at various pyrrole volumes

Applied voltage (V)	Electric current of the cell (mA) using different pyrrole volumes					
	PPy/microcellulose from pineapple peel			PPy/commercial microcellulose		
	Pyrrole 1 mL	Pyrrole 1.5 mL	Pyrrole 2 mL	Pyrrole 1 mL	Pyrrole 1.5 mL	Pyrrole 2 mL
2	1.8	1.7	1.4	1.8	2.7	4.1
3	2.2	2.4	2	2.6	4.0	6.1
4	2.8	3.2	2.8	3.5	5.0	7.8
5	3.5	4.3	4.1	4.2	6.5	10.2
6	4.1	5	4.7	4.8	8.1	12.

Table 4

Capacitance of capacitor cells fabricated with PPy/microcellulose from pineapple peel and PPy/commercial microcellulose

Pyrrole (mL)	Cell consisting of pineapple peel microcellulose (μ F)	Cell consisting of commercial microcellulose (μ F)
1	2.3	2.7
1.5	2.6	4.2
2	3.4	6.5

The small deviations observed in the wavenumbers of the cellulose peaks likely originate from the inherent variations in microcellulose extracted from different sources.

The close resemblance between the characteristic peaks of microcellulose in the composites compared to individual spectra suggests a primarily physical interaction between these components. In other words, this analysis indicates that PPy and microcellulose are not chemically bonded, but rather coexist within the composite structure.

Capacitance evaluation

The capacitance of the fabricated capacitor cells was evaluated using both a multimeter and an LCR meter at various voltage levels: 2, 3, 4, 5, and 6 V. As shown in Table 3, the measured current (mA) exhibits a direct proportionality to the applied voltage (V). This indicates that

increasing the voltage leads to a corresponding increase in the current drawn by each capacitor cell variant. The data presented in Table 3 were subsequently employed to calculate the capacitance of each cell, as detailed in Table 4.

For comparison, a capacitor cell fabricated solely with 2 mL of pyrrole (without cellulose) exhibited a capacitance of 4.7 μ F, exceeding the cell containing extracted microcellulose, but lower than the one with commercial microcellulose. This observation suggests a positive correlation between the amount of pyrrole and the resulting capacitance of the capacitor cell.

As shown in Table 4, incorporating pyrrole into both commercial and pineapple peel-derived microcellulose composites significantly enhances the capacitance compared to their pristine counterparts. This improvement can be attributed to the conductive nature of polypyrrole, which

effectively boosts the overall conductivity of the composites while maintaining stability at room temperature. Additionally, the inherent responsiveness of polypyrrole makes it well-suited for applications in electrical fields, as corroborated by other researchers.^{29,30} Notably, the use of cellulose extracted from pineapple peel appears to have minimal influence on the resulting capacitance of the composite material.

CONCLUSION

This study successfully demonstrated the isolation of microcellulose from pineapple peel waste. FTIR and XRD characterization results confirmed this achievement, as the spectra of the extracted microcellulose closely resembled those of commercial microcellulose and previously reported data. Furthermore, the study effectively synthesizes PPy/microcellulose composites, which are seamlessly integrated as electrode materials in parallel-plate capacitor cells. Subsequent capacitance measurements revealed that both PPy/commercial microcellulose and PPy/pineapple peel microcellulose composites exhibited high capacitance values, particularly for variants containing 2 mL of pyrrole, which are 6.5 and 3.4 μF , respectively. These findings suggest the promising potential of utilizing microcellulose extracted from pineapple peel waste as a matrix in PPy-based composite materials for capacitor applications.

ACKNOWLEDGMENT: The authors gratefully acknowledge the Faculty of Mathematics and Natural Sciences at Universitas Tanjungpura for their generous support in covering the research expenses through DIPA FMIPA UNTAN in the year 2023 (grant number: 2689/UN22.8/PT.01.05/2023).

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