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Membrane Inovation for Proton Exchange Membrane Fuel Cells (PEMFCs) Based on Duck Feather Keratin Extract as Environmentally Friendly and Efficient Alternative Energy

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ABSTRACT

The interest in renewable and sustainable energy is increasing because it offers a clean and environmentally friendly approach. One system that produces renewable energy is a fuel cell, which produces electricity through electrochemical reactions. In fuel cells, the membrane has an important role in delivering the protons produced from the anode to the cathode. The membrane used can be replaced with an alternative membrane. Recent research into alternative 'green' membranes has become an exciting area of study. In this research, a proton-conducting keratin-based membrane was made from duck feather waste. Duck feather keratin extraction is carried out by dissolving the feathers in an alkaline solution and precipitating the supernatant obtained at the triple point. The phases contained in duck feathers and dry keratin that have been prepared, have been researched and are known to be related to the amide functional group which has an a-helical and β-sheet structure. Dry keratin exhibits agglomerated flat particles with a size of several microns. This indicates the potential of keratin derived from duck feathers for applications in fuel cell membranes.

INTRODUCTION

To support Indonesia's commitment to realize net zero emissions by 2060, efforts are needed to provide electrical energy that is environmentally friendly, renewable and efficient (Berghuis,2020). One of the technologies to produce green electricity in the future is fuel cell. Fuel cell is an electrochemical device that reacts hydrogen and oxygen with by-products of heat and water without any combustion process. Fuel cell technology is similar to batteries. The difference lies in the fuel cell system which is designed to have energy continuously (Safitri, 2016).

The type of fuel cell that is currently a global interest is PEMFCs (Proton Exchange Membrane Fuel cells) because it has a high current density, is easily distributed, and operates at low temperatures (Soon, 2023). In the PEMFCs system, the electrolyte used is a nafion-based membrane. However, the use of this membrane has several disadvantages, including not being environmentally friendly because it contains fluorine elements, and has high methanol diffusion from the anode so that it can reduce fuel cell efficiency (Safitri, 2016). Therefore, alternatives to nafion membranes are needed that are safer for the environment. One of them is keratin-based membrane material (Ma, 2016). Keratin contains cysteine which can be converted into sulfonic acid groups through post-oxidative treatment to obtain proton conductivity properties so as to produce membranes that can be applied to fuel cells (Ma, 2016).

Research related to keratin extraction from chicken feathers has been conducted by researchers from Nanyang Technological University (NTU) and produced Open Circuit

Voltage between 0.95 - 1 V with a maximum power of 25 mW which is able to turn on LED lights (Ma, 2016). However, until now, there has been no research on the utilization of keratin from duck feathers as a fuel cell membrane material. Duck feathers have keratin content equivalent to chicken feathers (~97%) and contain preening oil that can improve the hydrophilic or hydrophobic properties of the membrane (Dewi, 2021) The potential of duck feathers as an environmentally friendly fuel cell membrane material is enormous. In addition, the high interest of Indonesian people in raising and consuming processed duck produces significant duck feather waste. Based on BPS data, the duck population in Indonesia reached 58,243,335 heads in 2020 with the percentage of feathers in each cutting as much as 6% of the live weight (Sharma, 2018). The addition of duck feather keratin-based membranes to Fuel Cells is expected to produce alternative energy that is environmentally friendly and efficient. In this Program Kreativitas Mahasiswa – Riset Eksakta (PKM-RE), research is aimed at knowing the potential of duck feather keratin extract as a fuel cell membrane material in an effort to produce environmentally friendly and efficient alternative energy. The addition of duck feather keratin-based membranes to Fuel Cells is expected to produce optimal conductivity as in the use of synthetic membranes. In addition, several tests were carried out to determine the structure and performance of the membrane formed, namely X-Ray Diffraction (XRD), Scanning Electron Microscophy-Energy Dispersion X-Ray (SEM-EDX), Fourier Transform Infrared (FTIR), and Electrochemical Impedance Spectroscopy (EIS).

RESEARCH METHOD

The duck feathers used in this research came from a local market in Surabaya, Indonesia. The duck feathers were first washed to remove dirt and then dried in the sun. The calamus and rachis were manually separated to obtain the soft part of the feather known as the vane (Njoku, et al., 2019). The collected feathers are then minced to obtain fine feather fiber. A schematic of the process in preparing duck feathers before keratin extraction is shown in Figure 1(a).

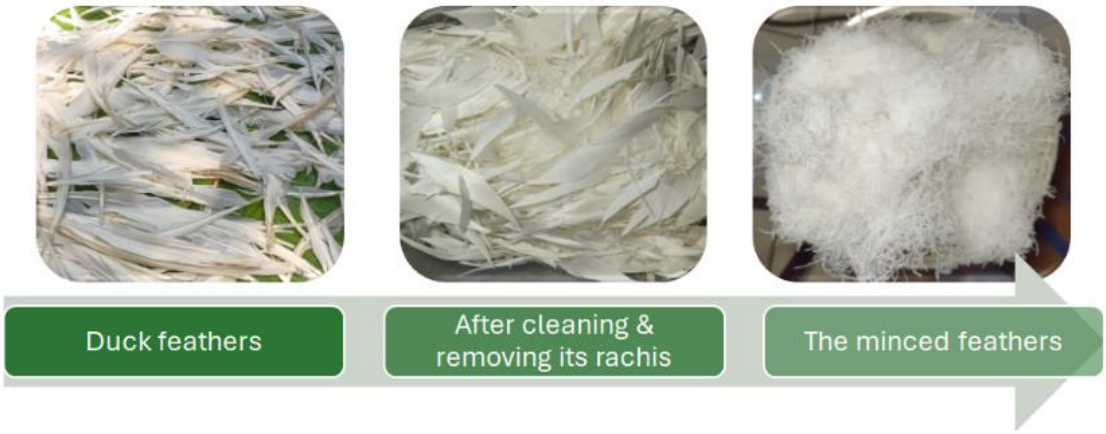


Figure 1. Process scheme (a) dries the duck feathers in the sun (b) separates the feather from its calamus and rachis, and (c) minces the duck feathers.

Keratin Extraction from Duck Feathers

The working steps in utilising duck feather keratin extraction, namely 14.4 g of duck feather waste was collected and soaked in water for fifteen minutes, to minimise dirt. Then washed with soap water and dried. The dried duck feathers were blended, then stored in a closed container and tested for FTIR, SEM-EDX, and XRD. A total of 14.4 grams of fine duck feathers were added to a solvent (8M urea, 10% l-cysteine by weight of duck feathers, 50%NaOH) with a pH of 13, and heated using a hot plate at 75°C with a stirrer speed of 550 rpm for twelve hours. Then the solvent was filtered and centrifuged at 10000 rpm for twenty minutes. The supernatant was added with hydrochloric acid and sodium sulphate until it reached pH 4, and formed a precipitate. The precipitate formed was filtered and washed with distilled water for three repetitions. The fresh keratin solids obtained were collected and weighed. Afterwards, FTIR, XRD, and SEM-EDX tests were carried out on the powder solids.

Duck Feather Membrane Fabrication

In making the membrane, fresh keratin was added to solvent (0.1M sodium carbonate and sodium bicarbonate) with the ratio of keratin and solvent (15:85)%. The mixture was then added SDS with variations of 40% and 60% of the weight of dried keratin. Subsequently, the mixture stood for 24 hours at room temperature. The mixture was heated by microwave at 90°C for one hour, after which glycerol was added as much as 10% of the weight of dried keratin and the mixture was heated again at 60°C. The membrane was incubated in solution (10% ethanol, 10% acetic acid) and 10% glycerol from the solution before further use.

Characterisation

Membrane characterisation was carried out by XRD testing to observe the phases formed, FTIR to analyse chemical bonds and functional groups, DSC/TGA to determine heat characteristics, and SEM/EDX to observe the morphology and elemental content of the membrane. In addition, EIS test was conducted to determine the conductivity of the sample. The voltage effectiveness test begins by connecting the fuel cell electrode equipped with a duck feather keratin membrane to an electric cable. Afterwards, the cable was connected to a digital multimeter to measure the amount of voltage. The multimeter used, the UX-389 type, has AC and DC current measurements with resistance of 200 ohms to 200 kilo ohms. Furthermore, data processing is carried out using the formula for current strength, voltage, and electrical power so that the data generated after processing can show the effectiveness of the fuel cell.

RESULTS AND DISCUSSION

Development of learning media (overlay visualization)

The structure and thermal stability of duck feathers were examined prior to keratin extraction. Previous studies have indicated that duck feathers contain fibrous keratin, abundant in the barbs, calamus, and rachis, making them a suitable source for the production of high-value keratin-based products (Tesfaye, 2018). The structure of duck

feathers was first examined before extracting keratin and applying it as a precursor for preparing fuel cell membranes. Figure 2(a) displays the FTIR spectrum of duck feathers, showing the presence of amide-A, amide-I, amide-II, and amide-III functional groups at wavenumbers 3264.4, 1620.9, 1532.8, and 1235.0 cm-1, respectively. Additional peaks were also observed, associated with symmetric CH3 stretching at 2959.5 cm-1, CH2 scissoring at 1448.4 cm-1, and C-S stretching at 595.2 cm-1. This spectrum is consistent with those previously reported for French mullard duck feathers (Tesfaye, 2018) and chicken feathers (Dewi, 2021 and Alvarez, 2023) The observed peaks and their assignments are listed in Table 1.

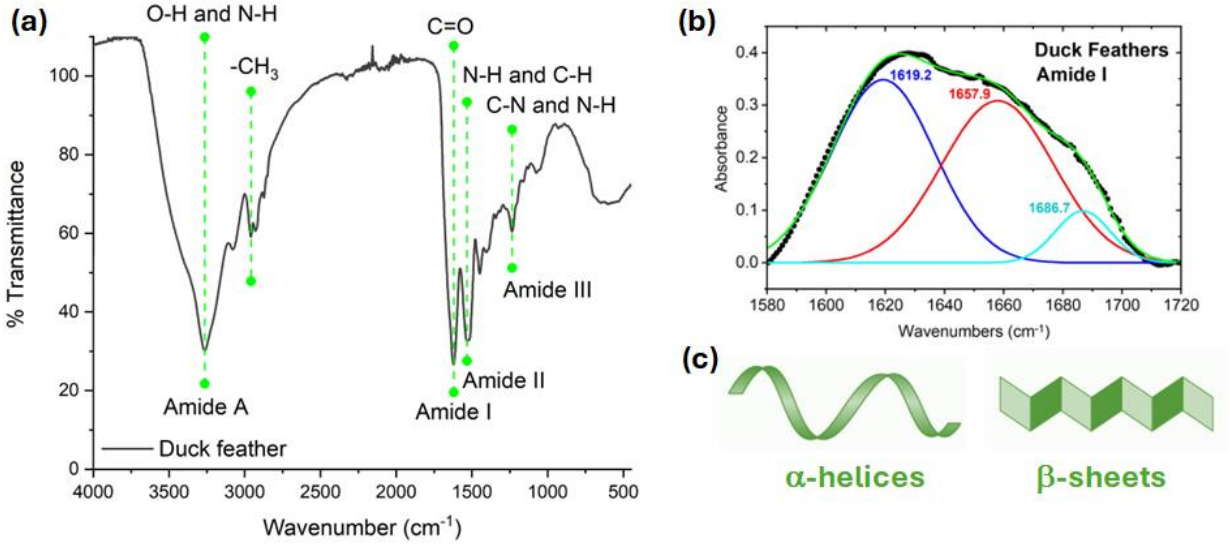


Figure 2. (a) FTIR spectrum of duck feathers. (b) Deconvolution of the spectrum within Amide I region. (c) Illustrations of α-helices and β-sheets.

Table 1. Summary of the observed FTIR peaks and their assignments, consistent with the previous reports in (Dewi, 2021), (Tesfaye, 2018), (Alvarez, 2023)

Wave Number (cm^{-1})	**Assignment**
3264.44	O-H and N-H stretching (Amide A)
2959.50	Symmetrical CH_3 stretching
1620.97	C=O stretching (Amide I), containing of *a*-helices (46.9 %), *β*-sheets (45.9 %), and turns (7.2 %)
1532.87	N-H bending and C-H stretching (Amide II)
1448.43	CH_2 scissoring
1235.04	C-N stretching and N-H in plane bending (Amide III)
595.20	C-S stretching

The Amide-I peak of duck feathers was deconvoluted to further examine the secondary structure of the protein. As shown in Figure 2(b), the deconvolution resulted in three peaks at 1619.2, 1657.9, and 1686.7 cm-1, corresponding to α-helices, β-sheets, and turns, respectively. An illustration of the α-helices and β-sheets structures is displayed in Figure

2(c). The relative percentage (%fraction) of each structure was estimated from the area of each peak. It was found that the %fraction as shown in Table 2.

Table 2. Summary of the FTIR deconvolution spectrum in Amide I region

Wave Number (cm^{-1})	Assignment	%Fraction
1619.2	β -sheet + random coil	46.9
1657.9	α -helix	45.9
1686.7	Turns	7.2

Figure 3(a) is the XRD pattern of duck feathers shows a broad peak from ~9° to ~20°. The first part is related to α-helices, and the latter has an asymmetric shape resulting from overlapping peaks between α-helices at 17.8° and β-sheets at 19.5°. The XRD pattern of duck feathers closely resembles the XRD pattern of chicken feathers (Ma, et al., 2016), confirming the presence of the protein secondary structure from the FTIR analysis. Additionally, the broad peak feature indicates a low degree of crystallinity in duck feathers. The crystalline index (Cr×I) is determined by the ratio of the crystalline peak area to the total area. The estimated Cr×I is approximately 0.47, meaning that ~47% of duck feathers consist of crystalline α-helices and β-sheets, and the remaining ~53% is amorphous phase. The Cr×I of duck feathers is comparable to that observed in chicken feathers (Dewi, 2021).

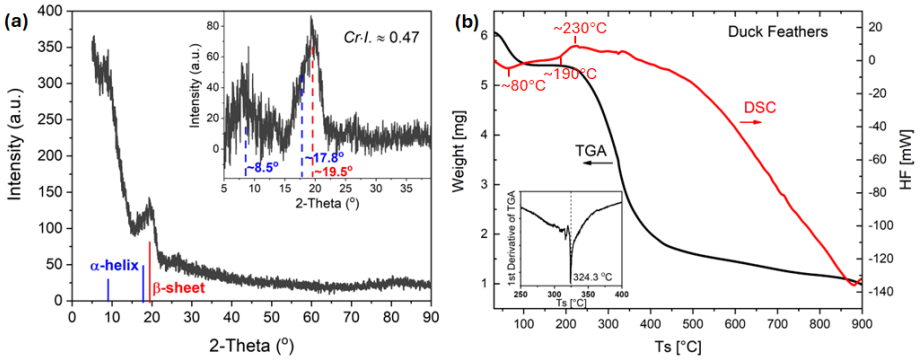


Figure 3. (a) XRD pattern of duck feathers. Inset is a close view of the baseline-subtracted pattern within 5°-39°. (b) DSC/TGA curves of duck feathers under N2 atmospheric gas.

The thermal stability of duck feathers was examined using DSC/TGA curves, as presented in **Figure 3(b)**. A broad, low-intensity endothermic peak is visible on the DSC graph. A broad dip below 100 °C indicates the amount of water bound within the keratin structure of duck feathers, and another dip is observed at ~200 °C, implying the melting of the crystalline keratin structure. A small exothermic peak at ~230 °C is associated with the irregular decomposition of α-helices. These results are consistent with those reported for chicken feathers (Tesfaye, et al., 2018). TGA analysis confirms two weight loss steps, the first being ~11% at temperatures below 100 °C, and the second being a substantial weight loss (~63%) at 200-400 °C, similar to what occurs in chicken feathers.

Structural Analysis of Dried Keratin

Figure 4(a) is the XRD pattern of dried keratin shows peaks from ~9° to ~20°. The α-helices are present at peaks around ~9° and ~17.3°, while the β-sheets are present at a peak around ~19.4°. Peaks marked with (*) indicate sulfur, hypothesized to be derived from the Na_2SO_4 precursor. The peaks observed in dried keratin are narrower compared to those in duck feathers, indicating that dried keratin has higher crystallinity than duck feathers. In Figure 4(b), the FTIR spectrum of dried keratin shows the presence of amide A, amide I, amide II, and amide III groups, similar to those in duck feathers, with the addition of CH_2, C-O, C-S, and C-S-C groups. Figures 5(a) and 5(b) show magnified images of dried keratin. Figure 5(c) represents the data in Figure 5(d), which shows that carbon (C) is the most abundant element in the keratin structure, with a weight percentage of 49.5% and an atomic percentage of 65.96%. The presence of sulfur (S) in the EDX data is hypothesized to originate from the Na_2SO_4 precursor.

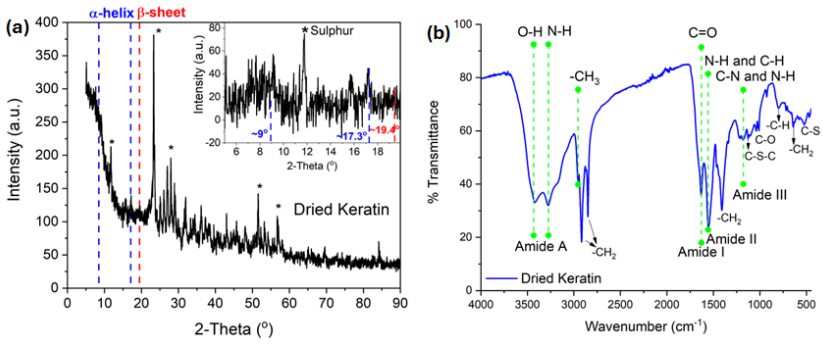
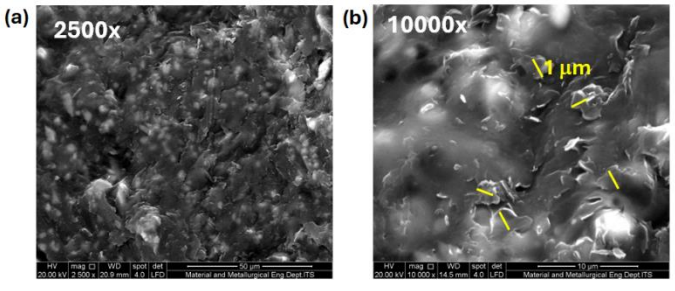


Figure 4. (a) XRD pattern and (b) FTIR spectrum of dried keratin extracted from duck feathers. Inset in (a) is the enlarged view of the XRD pattern within 5°-20°.



Element	wt%	at%
C	49.50	65.96
O	14.57	14.58
Na	7.49	5.21
Al	0.37	0.22
Si	0.97	0.55
S	26.04	13.00
Cl	1.07	0.48

Figure 5. SEM micrographs of dried keratin with the magnifications of (a) 2500× and (b) 10000×. (a) EDX graph of the observed SEM micrograph (inset) and (d) its elemental component.

Electrical Properties

Measurements using a multimeter on both processed and unprocessed duck feather samples showed that neither sample's resistance, current, nor voltage could be measured. This indicates that both processed and unprocessed duck feathers are insulative and do not conduct electrical current. The impedance data for duck feathers and dried keratin form semicircular graphs. The radius of the semicircle is related to the resistivity of the sample. A smaller radius indicates higher conductivity of the sample. Dried keratin has higher conductivity than duck feathers. This is because conductivity is closely related to the density of the sample. Dried keratin is in the form of agglomerated granules, whereas duck feathers are in the form of fibers, which are not as dense as dried keratin. The real impedance (Z') of the membrane is higher compared to that of dried keratin. This indicates that the membrane offers more resistance to the flow of alternating current than dried keratin. The electrical conductivity of the membrane is lower than that of dried keratin. This suggests that the membrane is less efficient at conducting electricity compared to dried keratin, likely due to its material properties and structure. There is a need to assess and possibly improve the fabrication process of the membrane. The current method may be leading to suboptimal electrical properties, affecting its performance. Membranes containing 60% SDS (sodium dodecyl sulfate) exhibited random impedance data, which is not displayed. This randomness is hypothesized to result from the membrane becoming highly insulative, indicating that a high concentration of SDS may adversely affect the membrane's conductivity.

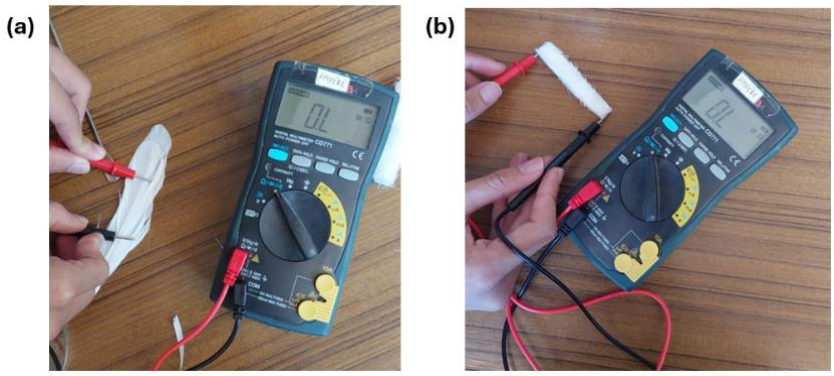


Figure 6. Resistance of duck feathers (a) before compression and (b) after compression.

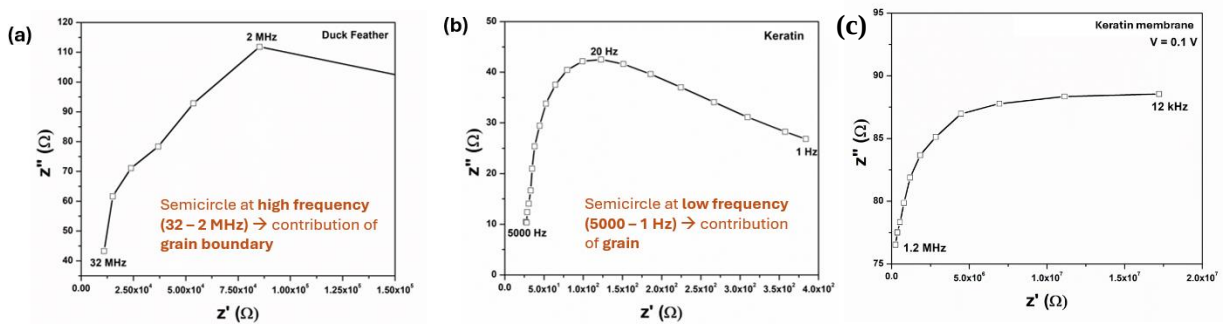


Figure 7. Impedance of (a) duck feathers (b) dried keratin and (c) keratin membrane with 40% SDS variant.

CONCLUSION

Duck feathers, whether treated or untreated, are inherently insulative. However, when processed into a keratin extract solution, the solution exhibits better conductivity compared to untreated duck feathers. Extraction of dry keratin from duck feathers produces keratin that is more conductive than unextracted duck feathers. The resulting membranes have lower electrical conductivity compared to dry keratin.

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