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Bioethanol preparation and separation from pineapple peel upon combining fermentation and pervaporation through biochar-containing PDMS membranes

Preparo e separação de bioetanol a partir da casca de abacaxi combinando fermentação e pervaporação através de membranas de PDMS contendo biocarvão

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Ethanol is an alternative to replace the fossil fuels. Different sources have been studied to produce second generation ethanol, such as food waste. The goal in this work was the investigation of pineapple peel as sugar source for ethanol production by fermentation and its separation from the broth. Sugar from pineapple peel was extracted with water and the rich phase was pre-treated with filtration in cloth, and a conventional filter (14 µm). Then, the filtrate was fermented to ethanol using *Saccharomyces cerevisiae*. The fermentation broth was fed to a pervaporation system containing polydimethylsiloxane membranes, with 0 and 2 wt% biochar from pineapple waste, in order to purify bioethanol and remove it from the broth. The yield of bioethanol in fermentation broth was 88%, after 24 hours. Pervaporation tests revealed higher flux and selectivity for non-containing biochar membranes, showing values of 120 g/m^{2}h and 1.0 v/v% of ethanol in permeate. The results showed the potential of the use of pineapple waste as the sugar source for biofuels such as bioethanol.
Keywords: biochar, bioethanol, pineapple peel.

Etanol é uma alternativa para substituir os combustíveis fósseis. Diferentes materiais têm sido estudados como fonte de etanol de segunda geral, como o resíduo alimentício. O objetivo deste trabalho foi investigar a casca de abacaxi como fonte de açúcar para a produção de etanol por fermentação e sua separação do mosto. O açúcar da casca de abacaxi foi extraído com água e a fase rica foi pré-tratada com filtração em tecido, filtro convencional (14 µm). Então, o filtrado foi fermentado a etanol usando *Saccharomyces cerevisiae*. O mosto fermentativo foi alimentado em um sistema de pervaporação contendo membranas de polidimethilsiloxano, com 0 e 2 % em massa de carvão a partir do resíduo de abacaxi, a fim de purificar o bioetanol e removê-lo do mosto. O rendimento de bioetanol no mosto fermentativo foi de 88%, após 24 horas. Testes de pervaporação revelaram maior fluxo e seletividade para membranas sem carvão, mostrando valores de 120 g/m^{2}h, e 1.0 % em volume de etanol no permeado. Os resultados mostraram o potencial do uso do resíduo de abacaxi como a fonte de açúcar para biocombustíveis como o bioetanol.
Palavras-chave: biocarvão, bioetanol, casca de abacaxi.

1. INTRODUCTION

Alternative renewable energy sources are being sought to replace fossil fuels. Ethanol is already used as a substitute for gasoline. It is a biofuel with high heat of vaporization, low flame temperature, high specific energy and high octane, properties that favor its use in automobiles [1]. Although it can be produced from various raw materials, first-generation ethanol fermentation involves many socio-environmental issues [2]. Consequently, different food waste such as watermelon [3], pizza [4], banana [5], papaya [6] and pineapple [7] have been studied as carbon source for ethanol production.

Among various candidates, pineapple waste stands out due to its abundance. It is estimated that around 50% of the mass of this fruit is discarded in the industry [8, 9]. Therefore, routes for using those wastes in ethanol production can not only benefit the energy sector, but also minimize waste that can result in other forms of environmental pollution. In addition to it, there is also huge amounts of bromelain, a proteolytic enzyme, in pineapple waste [10]. For instance, the use of

pineapple waste in solid state for the production of bioethanol was investigated through three routes: direct fermentation (bioethanol content 4.7 %v/v), saccharification and fermentation (4.9 %v/v) and simultaneous saccharification and fermentation (5.4 %v/v of bioethanol), as well as the removal of bromelain by means of micro and ultrafiltration [11]. In another approach, bromelain was extracted from natural deep eutectic solvents followed by saccharification of the solid waste to ethanol with content of 12.7 g/L of the bioalcohol [12].

In addition to researching other sources of raw materials, there is also interest in increasing the energy balance of ethanol production. One strategy to achieve this objective is to replace an operation, such as distillation, with another that allows obtaining the same product demanding less energy. A process studied for this application is pervaporation [13]. Pervaporation is less capital and energy intensive than distillation and adsorption processes for small plants treating less than 5000 L/h of feed solution [14]. In pervaporation, a liquid solution is fed to the membrane, while the permeate is removed in the vapor phase. One of the main advantages of this separation process is the possibility of getting products of higher purity than the azeotrope composition [14], such as the ethanol/water mixture, which reaches the azeotrope at 95.6% by mass of alcohol [15]. Therefore, solvent dehydration is the main interest of academic and industrial research related to pervaporation [16].

The use of sorption-diffusion model for pervaporation of ethanol from ethanol/water mixtures ranging from 5 to 20 wt%, at 30 to 60ºC, showed permeance of water and ethanol of 13.3 $\pm$ 0.1 g/m^{2}hkPa and 11.8 $\pm$ 0.1 g/m^{2}hkPa, respectively. In addition to it, activation energy is lower for water (56.09 kJ/mol) compared to ethanol (60.4 kJ/mol). The results for the alcoholic fermentation broth as feed solution for bioethanol separation showed low fouling tendency [17].

The goal of this work was to investigate the use of pineapple waste as source of sugars and activated carbon, named biochar from this point on, in the preparation of bioethanol and separation from the broth using mixed matrix membrane in pervaporation. The specific goals were to address the leaching of pineapple peel by water followed by filtration of the pulp, to evaluate the solid as biochar and its use in mixed matrix membrane, to define the operational conditions to the synthesis of bioethanol from the water soluble sugars and to measure the transport properties of bioethanol separation from the broth using pervaporation.

2. EXPERIMENTAL

2.1 Materials

Pineapples were purchased at a local market in Belo Horizonte, Minas Gerais, Brazil. *Saccharomyces cerevisiae* LNF CA-11 (batch #20210829-U) yeast was used in fermentation tests. Other chemicals were used in the preparation of the broth, such as sucrose, KH_2PO_4, $MgSO_4$ and $(NH_4)_2SO_4$. Solutions were prepared with distilled water. Ethanol P.A (Exodus Científica, min. 99.8%) was used as model solvent. Polydimethylsiloxane (Sylgard 184 Dow Corning, elastomeric kit) membranes were prepared by in situ polymerization. Filter paper (J. Prolab, 14 μm) was used in pretreatment of the pineapple liquor.

2.2 Experimental strategy

The experimental route was comprised of six steps: i) pineapple peel leaching with water, ii) separation of the slurry by filtration; iii) pyrolysis of the pineapple waste to prepare biochar; iv) fermentation of the sterilized liquor to ethanol, v) preparation of the membrane upon mixing the biochar from pineapple and PDMS, vi) pervaporation of the fermentation broth by using the mixed matrix membrane. Leaching was used to transfer the remaining sugars from the pineapple peel to liquid medium with water. Filtration was used to separate the insoluble solids from the liquid phase. Pineapple residues were also investigated as biochar by means of their pyrolysis and then used as filler in the pervaporation membrane. Liquid phase was used in fermentation step as carbon source to prepare bioethanol. The bioalcohol was separated from the broth through

pervaporation, using mixed matrix membrane. The main hypothesis was the evaluation of the reuse of byproducts.

2.3 Pineapple peel leaching

The methodology for obtaining the peel liquor was adapted from Simões et al. (2022) [10]. Pineapples were cleaned with water, detergent and a small brush. The peel was removed with a knife. The cleaned peels were broken into small pieces (1 cm) with a knife before getting frozen. The pieces, still frozen, were weighed (0.261 kg) and mixed with one liter of distilled water. This solid/liquid mass ratio was lower (0.26:1) than the one described in a previous work [10], due to the difficulty of filtration step after leaching for 1:1 ratio. The mixture was left to rest for 1.5 hours to improve the solubility of the sugars. After this period, the larger particles were removed by filtration using a domestic sieve. Finally, the solution was filtered again using filter paper (14 μm) in a typical filtration system, using buchner funnel, kitasato and vacuum pump (Edwards, 0.02 mbar). The filtration area was 63 cm^2.

2.4 Fermentation

The procedure was conducted in accordance with the guidelines provided by the supplier. The glassware was sterilized in an autoclave (Stermax, 21 L) at 127°C for 30 minutes. In the fermentation of the liquor, 0.500 g of dry yeast was used, which was hydrated in a beaker containing 10 mL of warm distilled water (approximately 35°C) for 30 minutes. The mixture was inoculated into a glass bottle containing 500 mL of pineapple peel liquor. The system was sealed with tow and cotton and kept under agitation with the aid of a magnetic stirrer. Samples of 10 mL were used for analysis in specific times. Tests were compared to the sucrose broth, with the following composition: 30 g/L of sugar, 5 g/L of KH_2PO_4, 1 g/L of $MgSO_4$ and 2 g/L $(NH_4)_2SO_4$, as reported in the literature [18].

2.5 Biochar

The biochar was prepared from 0.395 kg of peel, 0.158 kg of crown and 0.063 kg of pineapple stem, different individuals, which were dried in an oven at 120°C for 18 hours. Then, pyrolysis of the solid material was carried out at 550°C for 60 minutes. The heating rate in the vertical fixed bed was 30°C/min. Once the temperature was stabilized, nitrogen (N_2) was fed to the reactor at a flow rate of 2 L/min. This sample was weighted, milled and sieved through a 400 mesh (38 μm) device. Samples were characterized by dynamic light scattering (Horiba) to determine particle size distribution. Specific surface area was determined by N_2 physisorption (Belsorp, software Belmaster).

2.6 Membrane preparation

The elastomeric kit (Sylgard 184) was used in the proportion of 9:1 by weight, as described elsewhere [19]. The mixture was homogenized and cast on a flat plastic surface. Visible bubbles were removed with a needle. The membranes were heated in an oven at 80°C for 1.5 h. For mixed matrix membrane, biochar was added to Sylgard part A and dispersed before the addition of Sylgard part B. The total amount of biochar was 2% w/w regarding to the total polymer content (part A and part B). The curing time and temperature were the same (80°C for 1.5 h).

2.7 Pervaporation

Pervaporation tests were conducted for three different feed solutions: water/ethanol solution (95:5 by weight), *S. cerevisiae* broth from sucrose and *S. cerevisiae* broth from pineapple peel, which were prepared as described before (2.4). The experimental setup is shown in Figure 1.

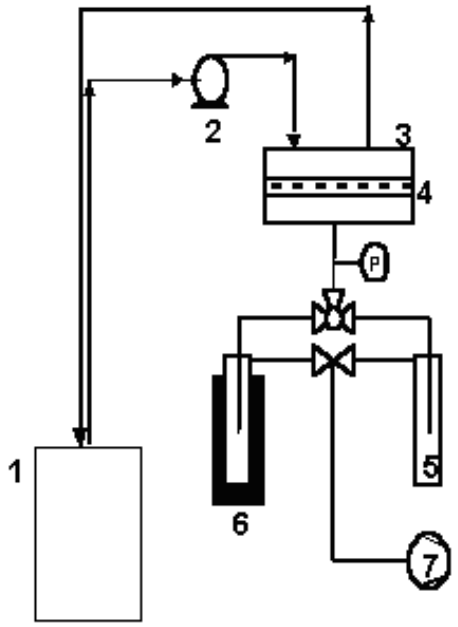

Figure 1: Experimental setup for pervaporation experiments. 1 – Jacketed feed tank, 2 – peristaltic pump, 3 – permeation cell, 4 – membrane, 5 – crystallizer, 6 – liquid N_2 bath and 7 – vacuum pump (pressure 0.02 mbar).

Feed solution was transferred to the feed tank, with temperature control of 35ºC. The suspension was pumped to the membrane module in the flow of 60 L/h. Permeate was kept under vacuum and samples were collected in a crystallizer, immersed in liquid nitrogen, -196ºC. Concentrated solution was transferred back to the feed tank. Membrane permeation area was 200 cm^2. The ratio between the area and feed solution was set in 2.5 cm^{-1}. Tests were considered after 30 minutes to attain steady state regime. Total tests time was varied from 2 to 3 hours. Permeation flux and ethanol content on permeate were reported as the average of at least 3 runs. Standard deviation was used to compare the mean values.

2.8 Characterization

The concentrations of soluble solids in the solutions were approximated by refractometry (Abbe). A calibration curve was determined for different sugar contents.

The concentration of ethyl alcohol was determined from simple distillation. The distillated fraction was submitted to refractometry to determine ethanol content.

The concentration of yeast cells was determined by using an UV-visible spectrophotometer (Bel Photonics). The sample was diluted 1:50 in distilled water and the absorbance was recorded at 600 nm. The blank was the medium with no cells.

Chemical groups in membrane surface were investigated by Fourier Transformed Infrared Spectroscopy equipped with Attenuated Total Reflectance (FTIR-ATR, Bruker Alpha, software opus 7.2). Samples were placed in the equipment and a probe with diamond in the edge was placed in membrane skin layer. The spectra were obtained from 500 to 4000 cm^{-1}, with step of 2 cm^{-1}. Results were reported as the average of 28 runs.

3. RESULTS AND DISCUSSION

3.1 Fermentation of pineapple peel liquor

The physicochemical of the broth from pineapple peel after 24 hours are presented in Table 1. The results are the average of 3 runs.

Table 1: Physicochemical properties of fermentation broth of pineapple peel 24 hours after the beginning.

Parameter	Value
pH range (-)	3.90 – 3.31
Total dissolved solids (g/L)	16.06 ± 0.01
Decrease in total dissolved solids (g/L)	1.32 ± 0.08
Final ethanol concentration (% v/v)	0.8 ± 0.2

According to Table 1, it was possible to prepare ethanol from sugars from pineapple peel liq. The yield was 88% in comparison with the theoretical yield. This was equivalent to approximately 0.450 grams of ethanol per gram of glucose in 24 hours. The decrease in pH during the experiment is expected due to the metabolism of the yeast, which leads to proton or acid of low molecular weight expelled to the medium. This production of acid can be related to the total dissolved solids.

Gil and Maupoey [11] reported a yield of 0.375 g of ethanol per gram of glucose after 72 hours of direct fermentation of a liquor prepared from pineapple residues. Nigam (1999) [20] obtained a yield of 92.5% for the continuous production of ethanol from pineapple processing factory waste, upon using different nutrients to promote yeast growth. In another approach, Tropea et al. (2014) [21] reported a yield of 96.5% for ethanol production after 30 hours of simultaneous saccharification and fermentation.

3.2 Biochar

The total weight of the biochar produced was 0.018 kg, which is 3 wt% of the initial weight. Figure 2 shows the result of particle size distribution.

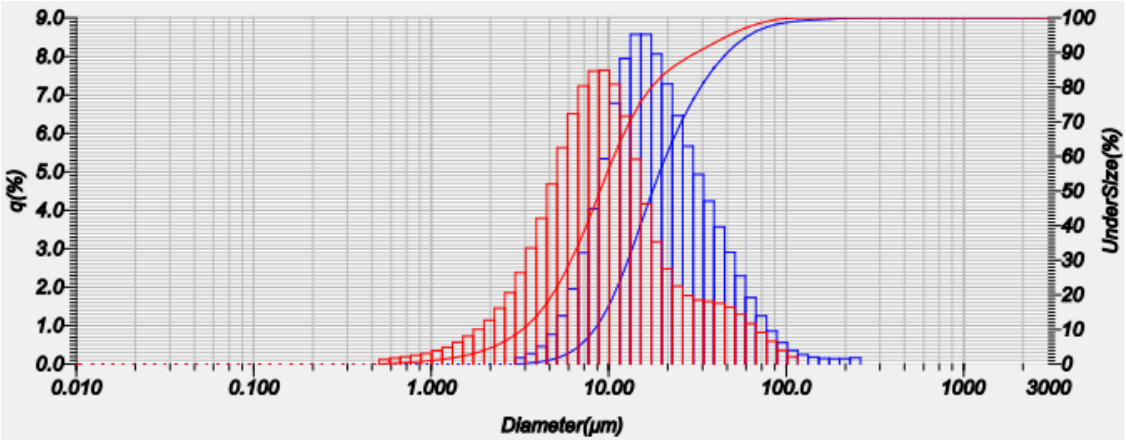

Figure 2: Particle size distribution of biochar prepared from pyrolysis of pineapple residues using diffraction light scattering method.

The average particle size was (19 ± 2) μm. Nitrogen physisorption showed specific surface area of 4.18 m^2/g, total pore volume of 0.007 cm^3/g and average pore size of 6.6 nm. This biochar

was used as the filler in mixed matrix membranes in which polydimethylsiloxane was the polymeric matrix.

3.3 Membrane characterization

The FTIR results for biochar and PDMS membranes with 0 and 2 wt% biochar is shown in Figure 3.

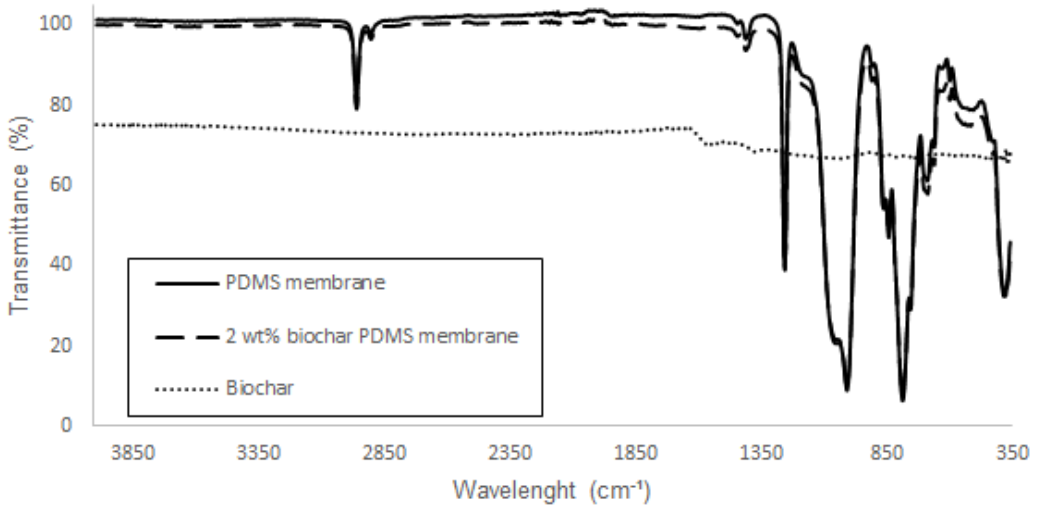

Figure 3: FTIR results of the biochar prepared from pyrolysis of pineapple waste and PDMS membrane with 0 and 2 wt% of biochar.

It was noticed that the bands in biochar were very few (1560, 1377, 870 and 743 cm^{-1}), usually related to nitrogen and oxygen aliphatic carbon chain. Both membranes show similar behavior to each other, which indicates that the corresponding bands are characteristic of the polydimethylsiloxane polymer matrix. This could also be ascribed to the low content of biochar in the mixed matrix membranes, which was not enough to show any peak related to the filler. Peaks in the region of 2903 cm^{-1} and 2961 cm^{-1} indicate C-H bonds with symmetric and asymmetric stretching [22-24]. The range of 1220 cm^{-1} and 1250 cm^{-1} are associated with the presence of the Si-CH_2CH_2 group and the Si-O bonds mark the transmittance at 1000 cm^{-1} and 1100 cm^{-1} [24]. The Si-C bonds are marked at 688 cm^{-1}, 755 cm^{-1} and 787 cm^{-1} [23, 24]. The results also show the absence of a band at 3300 cm^{-1}, a typical region of hydroxyls characteristic of the non-crosslinked polymer, which indicates that there was complete curing of the polymeric matrix during membrane synthesis [24].

3.2 Pervaporation

Flux of the membranes used in pervaporation of model water/ethanol (95/5), as well as sucrose and pineapple peel liquor broth are shown in Table 2.

Table 2: Permeate flux through the PDMS membranes with varying biochar content for different feed solutions.

Feed	Flux	
	0% biochar ($g.m^{-2}h^{-1}$)	**2 wt% biochar ($g.m^{-2}h^{-1}$)**
Water/ethanol (95/5)	(14 ± 3) x 10	25 ± 3
Sucrose	(11 ± 3) x 10	(4 ± 3) x 10
Pineapple peel liquor	(12 ± 4) x 10	9 ± 8

Average membrane thickness was 0.4 mm for both membranes, which is very high, probably leading to small fluxes. Permeate fluxes of PDMS membranes where higher than the ones noticed for the mixed matrix material, indicating that the addition of the biochar decreases the free volume fraction of the membrane. There was no difference in fluxes upon comparing the three different feed solution considering pure PDMS membrane. On the other hand, permeate fluxes for the membrane containing biochar showed a higher value for sucrose as sugar source, followed by water/ethanol (95/5) and pineapple peel liquor, which was not expected since the broth solutions should present higher membrane fouling, leading to lower fluxes. The high standard deviations of both broth feed solutions shows that this system should be investigated further to address any clue for the unusual behavior.

Gaykawad et al. (2013) [25] investigated the application of a commercial PMDS membrane in the pervaporation of lignocellulosic biomass fermentation broths at 30°C, obtaining permeate fluxes in the range of 500 to 715 $g.m^{-2}h^{-1}$. Mohammadi et al. (2005) [26] reported fluxes in the range of 520 to 900 $g.m^{-2}h^{-1}$ in the pervaporation of synthetic water and ethanol solutions in the range of 0.3 to 3% w/w at 30°C. In the work, the authors used a PDMS membrane whose dense layer and support were 0.008 mm and 0.120 mm thick, respectively. Therefore, the thickness of membranes tested in this research may have contributed to flow reduction.

The fluxes for PDMS membrane with no biochar were higher than the ones with biochar, indicating that the free fraction volume of the film was considerably reduced. It was noticed that fluxes were in the following sequence: water/ethanol, pineapple peel liquor and sucrose, although the high fluctuations do not allow to evaluate differences among them. On the other hand, the fouling was not significative due to the use of the broths (sucrose and pineapple peel liquor) compared to water/ethanol. For the 2 wt% biochar PDMS membrane, the highest flux for sucrose is also related to high fluctuation, thereby the comparison does not show relation between fouling and flux. The high experimental fluctuation can be regarded do the local variation in membrane thickness.

Gonçalves and Figueiredo (2024) [19] investigated the application of a PDMS/biochar mixed matrix membrane for pervaporation of ABE mixtures (acetone, butanol, ethanol). The authors obtained a flux of 13.21 $g.m^{-2}h^{-1}$ for the membrane with 2 wt% carbon.

However, higher fluxes can be found for mixed matrix membranes with different loads or polymer modification. Pang and coauthors [27], for example, achieved a flux of 1681 $g.m^{-2}h^{-1}$ using a fluorinated PDMS/ZIF-8 membrane for the pervaporation of a 5 wt% water and ethanol solution at 60°C.

Goethem et al. (2022) [28] studied the stability of PDMS membranes during the pervaporation of fermentation broth for ethanol production. The results found by the authors showed that the permeabilities of both unfilled and membranes and zeolite and silicate-filled membranes decreased markedly over time under the application of this type of feeding.

In addition to the permeate flux, it is important to evaluate the membrane selectivity. Table 3 shows the average permeate concentrations for the pervaporation tests.

Table 3: Average ethanol content in permeate from pervaporation for PDMS membranes with varied biochar content as a function of the feed solution.

Feed	**Ethanol concentration (%v/v)**	
	0 wt% biochar	**2 wt% biochar**
Water/ethanol	0.5 ± 0.5	0.5 ± 0.5
Sucrose	1.6 ± 0.6	0.34 ± 0.01
Pineapple peel liquor	1.02 ± 0.01	0.5 ± 0.5

The permeate concentrations showed high dispersion, which makes it difficult to analyze the results. The highest selectivities in this work were found with the unfilled membrane when applied to the pervaporation of fermentation broth, which the α values for the sucrose must and pineapple liquor were 1.4 ± 0.2 and 1.4 ± 0.1, respectively. In the literature, selectivity factors close to 4 are recorded both for water/ethanol solutions and fermentation broth [25, 28] using PDMS

membranes. Although the selectivities are low in this work, it is known that water permeance is higher than ethanol, while activation energy for water permeation in PDMS is lower than ethanol [17]. Maybe the use of a different polymer could improve the results for selectivity factors.

Although these preliminary results of biochar as filler in membrane are not conclusive, new attempts to prepare biochar with better surface properties could lead to an increase in membrane transport properties.

This performance may be related to the structure of these films. As the structure of non-commercial membranes is generally more heterogeneous when compared to the commercial ones, it is possible that the selective properties of these materials are lower. Furthermore, since water molecules are small, there are fewer barriers to the transport of these molecules through the free volume fraction of the membranes.

4. CONCLUSION

Fermentation of pineapple peel liquor to prepare bioethanol was possible with yield of 88%. Biochar (from the pyrolysis of pineapple waste) showed a small surface area and pore volume when compared to similar products studied in the literature, which limited the performance of mixed matrix membrane in pervaporation. The PDMS with no added biochar showed better results, with flux of 120 $g.m^{-2}h^{-1}$ and 1.2 v/v % of ethanol in the permeate. This work highlighted opportunities for using pineapple waste as sugar source to prepare bioethanol through relatively low complexity processes.

5. ACKNOWLEDGEMENTS

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