S.T. Yau High School Science Award

Research Report

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Title of Research Report

Monosulfide and Norbornene-Derived Crosslinking of Polydicyclopentadiene Membranes: Toward Selective Fatty Acid Methyl Ester Separations

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Allen Ma

Abstract

Fatty Acid Methyl Esters (FAMEs) are essential components in biofuel production. Different FAMEs have different properties, and separating them allows fine-tuning of cetane number, oxidative stability, and cold flow. Traditional methods such as distillation and recrystallization are energy-intensive or limited in scope, and membrane-based separation of FAMEs remains largely unexplored. In this work, we synthesized polydicyclopentadiene (PDCPD) membranes crosslinked with either S_2Cl_2 in a mixture of dichloromethane (DCM) and N,N-dimethylformamide (DMF), or S_2Cl_2 and a dinorbornene crosslinker in DCM. We systematically investigated the effects of S_2Cl_2 equivalence (0.5 to 10.0 mol per mol of DCPD) and DCM/DMF solvent composition on membrane selectivity. The permeation of *p*-nitrobenzaldehyde (PNB) and triphenylmethane (TPM) was studied as a model separation, followed by the separation of methyl stearate and methyl linoleate. We found that a PDCPD membrane crosslinked with S_2Cl_2 in 5% DMF (19 mL DCM, 1 mL DMF) achieved a maximum observed selectivity of 5.23 for PNB and TPM, and that a PDCPD membrane crosslinked with both S_2Cl_2 and a dinorbornene crosslinker in DCM achieved a maximum observed selectivity of 21.7 for PNB and TPM. However, the same membrane showed only a selectivity of 1.4 for methyl stearate and methyl linoleate, indicating that the similar cross-sectional areas of methyl stearate and methyl linoleate made them difficult for the synthesized membranes to separate effectively. These results demonstrate the potential of crosslinked PDCPD membranes for organic solvent nanofiltration and provide insight into how crosslinking conditions can be optimized to achieve more effective FAME separation.

Keywords: Organic Solvent Nanofiltration, Fatty Acid Methyl Esters, Polydicyclopentadiene, Membrane separation

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I would like to thank Professor Bowden for guiding me in brainstorming potential crosslinking conditions for S_2Cl_2.
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I would like to thank Wynn Tompkins and Jett Tjaden for synthesizing the dinorbornene ester crosslinker used in my experiments.

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3. observe the common standard of academic integrity adopted by most journals and degree theses.
4. have declared all the assistance and contribution we have received from any personnel, agency, institution, etc. for the research work.
5. undertake to avoid getting in touch with assessment panel members in a way that may lead to direct or indirect conflict of interest.
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Name of team member: Allen Ma

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Introduction

Biodiesel is a renewable energy source made from organic raw materials such as vegetable oil or animal fats (Nisar et al., 2021). It reduces greenhouse gas emissions, has a high combustion efficiency, and benefits farmers' income (Nisar et al., 2021). Fatty acid methyl esters (FAMEs) are major components in biodiesel (Gopinath et al., 2015). The structural properties of individual FAMEs, including carbon chain length, saturation, degree of saturation, and double-bond position, significantly contribute to the characteristics of the biodiesel mixture (Gopinath et al., 2015). For example, saturated FAMEs such as methyl stearate have high cetane numbers and oxidative stability but poor low-temperature properties, while unsaturated FAMEs like methyl linoleate have better viscosity and cold-flow properties but are prone to oxidation (Gopinath et al., 2015). It is therefore important to be able to control and separate these components to adjust the properties of the biodiesel.

Traditionally, methods such as distillation and recrystallization are used to carry out these separations (Long et al., 2011). However, each of these methods has limitations. Distillation requires significant energy input to increase and stabilize a high temperature, making it expensive and environmentally costly (Long et al., 2011). Recrystallization works well for certain molecules, but other molecules are unable to be recrystallized. (Long et al., 2011) Organic Solvent Nanofiltration (OSN) is a promising alternative to these methods. OSN is an efficient, low-energy method to separate solute molecules with molecular weights of 200-1000 $g \cdot mol^{-1}$ using size differences (Su et al., 2025). FAMEs are also soluble in organic solvents but insoluble in water. However, to purify a solvent containing multiple solutes, the molecular weights of the solutes typically need to differ substantially in order for most membranes to separate them effectively (Xiao et al., 2024). As a result, the separation of molecules with the same carbon chain length using OSN remains largely unexplored, as they have almost identical molecular weights.

Polydicyclopentadiene (PDCPD) serves as a particularly interesting polymer material. Unlike other polymer membranes, PDCPD separates molecules based on their cross-sectional area instead of their molecular weights (Long et al., 2011), giving it the potential to achieve difficult FAME separations. PDCPD is a polymer of the dicyclopentadiene unit formed via ring-opening metathesis polymerization (ROMP) (Long et al., 2011), and its polymerization leaves free volume in between, allowing small molecules to selectively permeate through. PDCPD is a sturdy yet flexible material that can be swelled by organic solvents, such as dichloromethane (DCM), without suffering permanent damage or deformation. This allows S_2Cl_2 or dinorbornene ester crosslinkers to be added to the swollen membrane, forming crosslinks between polymer chains and decreasing PDCPD's accessible free volume. As a result, molecules with smaller surface areas tend to pass through the membrane while molecules with larger surface areas stay, increasing selectivity relative to an uncrosslinked PDCPD membrane, which has lower selectivity between molecules of different sizes.

S_2Cl_2 is an electrophilic crosslinker that is able to connect PDCPD chains with disulfide bonds (Kang et al. 2022). In many organic solvents, S_2Cl_2 forms disulfide bonds between chains. However, it is proposed that in N,N-dimethylformamide (DMF), S_2Cl_2 forms monosulfur bonds.

Despite the potential that PDCPD offers for size-based organic separations, it remains unclear how crosslinking conditions and crosslinker equivalence influence its selectivity between different molecules. In particular, the effect of DMF/DCM solvent composition during S_2Cl_2 crosslinking has not been systematically evaluated.

We hypothesized that crosslinking PDCPD membranes with S_2Cl_2 in DMF would reduce the membrane's free volume and enhance selectivity. Furthermore, we hypothesized that increasing the DMF composition in the solvent (DMF/DCM ratio) and the presence of a dinorbornene crosslinker would increase membrane selectivity.

In this work, we report the synthesis of PDCPD membranes crosslinked with S_2Cl_2 and a dinorbornene crosslinker. We systematically investigated the effects of crosslinker concentration and solvent composition on membrane properties. P-nitrobenzaldehyde (PNB) and triphenylmethane (TPM) were used as model solutes to evaluate the effectiveness of various crosslinked membranes. With its three coplanar benzene rings, TPM has a significantly larger cross-sectional area of 0.51 nm^2 than PNB's 0.060 nm^2 (Long et al., 2011). The smaller PNB is therefore able to permeate the PDCPD membrane more easily and at a higher flux. After identifying the membrane with the highest selectivity for PNB and TPM, we conducted a separation of methyl stearate and methyl linoleate, two C18 FAMEs with identical carbon chain lengths but varying degrees of saturation.

Experimental

Materials

N,N-Dimethylformamide (DMF), dichloromethane (DCM), dicyclopentadiene (DCPD), Grubbs first-generation catalyst, disulfur dichloride, *p*-nitrobenzaldehyde (PNB), triphenylmethane (TPM), butylated hydroxytoluene (BHT), methyl stearate, and methyl linoleate were purchased from Acros, Sigma-Aldrich, and VWR and used at their highest purity.

Characterization

^1H nuclear magnetic resonance (NMR) spectra were collected using a Bruker DRX-400 at 400 MHz at room temperature. NMR samples were referenced to deuterated chloroform solvent or internally referenced to methyl stearate or PNB for quantitative analysis.

Synthesis of PDCPD membranes at a 5000:1 DCPD: Grubbs Catalyst ratio

A 20 mg mL^{-1} solution of Grubbs first-generation catalyst was prepared in DCM. 0.31 mL of this solution was added to 5 g of DCPD in 0.05 mL of DCM at room temperature and was stirred for 3 seconds. This solution was then immediately poured onto a clean silanized glass plate with 2 parallel strips of 100 μm polytetrafluoroethylene tape as spacers at 45º C. A second plate was quickly placed over the first and was pressed for 20 seconds. The sample was left for 2

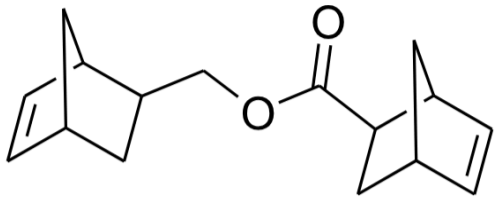
hours and then removed from the glass plates. Circles a little larger than an O-ring were cut out from the samples and stored in a freezer maintained at -18º C.

Use of the dinorbornene crosslinker in PDCPD membranes

The dinorbornene ester crosslinker used in this study, as shown in Figure 1, was synthesized before the present experiments by a member of the Bowden Laboratory using a previously established procedure (Ivanoff et al., 2020). The subsequent use of the crosslinker in PDCPD membranes and all permeation experiments reported in this study were performed by the author.

Figure 1

Dinorbornene Crosslinker



Methods of crosslinking PDCPD with S_2Cl_2 in DMF

The method of crosslinking a PDCPD membrane with S_2Cl_2 in DMF shifted throughout the experiments. Agitation was implemented in methods B and C to ensure replicability. A shorter swelling time in method C was used due to the ability of the membrane to swell effectively in a DCM/DMF mixture. Crosslinking conditions are shown in Table 1.

Method A: 100% DMF Series

A PDCPD membrane was submerged in DMF, and S_2Cl_2 was immediately added at varying equivalence ratios (0.5, 0.8, 1.0, and 10.0 mol/mol DCPD) to the solution. The membrane was allowed to swell and undergo crosslinking for 30 minutes without agitation before being removed, stored in a plastic bag, and placed in the freezer maintained at -18º C.

Method B: DMF Variation Series

A PDCPD membrane was placed in a glass separation apparatus. A crosslinking solution containing S_2Cl_2 (1.0 mol/mol DCPD) in the appropriate DCM/DMF mixture was added to both sides of the apparatus. The apparatus was stirred for approximately 17 hours on a stir plate at room temperature. The membrane was then removed, stored in a plastic bag, and placed in the freezer maintained at -18º C.

Method C: Standard Method

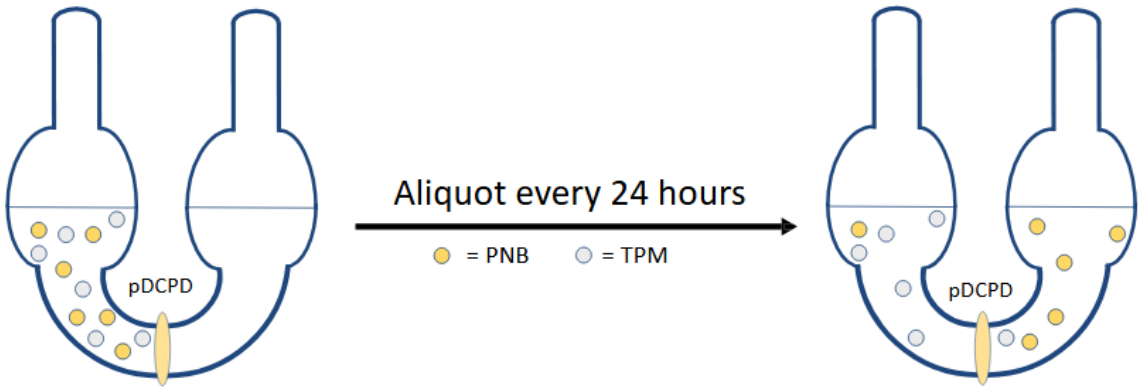
A PDCPD membrane was submerged in DCM for 30 minutes without agitation to allow swelling. A crosslinking solution containing S_2Cl_2 (1:1 molar equivalence relative to DCPD monomer units) was prepared in either 19 mL DCM with 1 mL DMF (5% DMF) for proposed monosulfur crosslinking, or in 20 mL DCM for dinorbornene-crosslinked membranes. After swelling, the membrane was transferred to the S_2Cl_2 solution and allowed to crosslink for 30 minutes on an orbital shaker. The membrane was then removed, stored in a plastic bag, and placed in the freezer.

Permeation of PNB and TPM through crosslinked PDCPD membranes

30 mL of a 0.088 M PNB and TPM solution with 0.006 M BHT was prepared along with 30 mL of DCM. BHT was added to capture free radicals to prevent oxidative degradation of the target molecules. A crosslinked PDCPD membrane was placed into the glass apparatus, and the solutions were poured in simultaneously. The apparatus is shown in Figure 2. The solutions were left to stir, and 1 mL aliquots from each side were extracted every 24 hours. 1 mL of methyl stearate standard was added to each aliquot, and the solvent was dried with air. The ^{1}H NMR spectra were obtained.

Figure 2

Separation Apparatus detailing PNB-TPM Separation



Permeation of methyl stearate and methyl linoleate through a PDCPD membrane crosslinked with a dinorbornene crosslinker and disulfide bonds

30 mL of a solution with equimolar amounts of methyl stearate and methyl linoleate, along with trace amounts of BHT, and 30 mL of DCM were prepared. The same procedure as described previously was used with 1 mL of PNB internal standard instead of methyl stearate.

Table 1

Crosslinking Conditions Tested for PDCPD Membranes

Membrane Type	S_2Cl_2 Equivalence (mol/ mol DCPD)	DMF (mL)	DCM (mL)
100% DMF S_2Cl_2 (0.5 eq)	0.5	20	0
100% DMF S_2Cl_2 (0.8 eq)	0.8	20	0
100% DMF S_2Cl_2 (1.0 eq)	1.0	20	0
100% DMF S_2Cl_2 (10.0 eq)	10.0	20	0
80% DMF S_2Cl_2 (1.0 eq)	1.0	16	4
66% DMF S_2Cl_2 (1.0 eq)	1.0	13.3	6.7
50% DMF S_2Cl_2 (1.0 eq)	1.0	10	10
5% DMF S_2Cl_2 (0.5 eq)	0.5	1	19
5% DMF S_2Cl_2 (0.8 eq)	0.8	1	19
5% DMF S_2Cl_2 (1.0 eq)	1.0	1	19
Dinorbornene and S_2Cl_2	1.0	0	20

Selectivity was determined by dividing the ratio of the concentration of molecules in the permeate and the retentate, termed the P/R ratio. In the case of the separation with PNB and TPM, the selectivity was determined by dividing the P/R ratio of PNB by the P/R ratio of TPM. The selectivity between methyl stearate and methyl linoleate was determined in a similar manner.

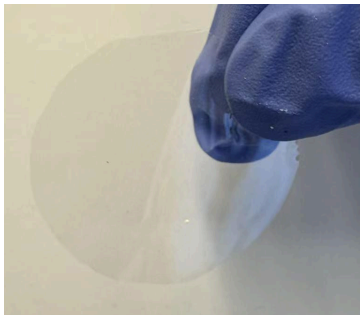
Results and Discussion

Synthesis of PDCPD membranes

PDCPD membranes were synthesized by combining DCPD monomer and Grubbs catalyst in a 5000:1 ratio and pressing between two glass plates. The membranes used in this study were approximately 50 – 80 μm thick. The membrane was transparent and flexible, as shown in Figure 3. Based on unpublished data from the Bowden Lab, an uncrosslinked PDCPD membrane would achieve a maximum selectivity of 1.73.

Figure 3

Pure PDCPD Membrane before Crosslinking with S_2Cl_2



Permeation of PNB and TPM through PDCPD membranes crosslinked with S_2Cl_2 in varying amounts of DMF

PDCPD membranes were crosslinked with 1.0 mol/mol S_2Cl_2 to DCPD in varying ratios of DMF and DCM. The selectivities between PNB and TPM for these experiments are shown in Table 2. The selectivity of the PDCPD membrane increased from 2.21 to 2.56 when changing the DCM/DMF ratio from 80% DMF to 66% DMF, and we found that a 5% DMF solution provided the best selectivity of 5.23. Based on the upward trend of selectivity from 80% DMF to 66% DMF to 5% DMF, one would expect that the 50% DCM/DMF PDCPD membrane would then offer the second-best selectivity, but only a selectivity of 1.23 was achieved. The unexpectedly low selectivity observed could have resulted from physical membrane defects, but this explanation cannot be confirmed. Alternatively, the non-monotonic trend suggests that DMF may influence swelling, crosslinking kinetics, or the structure of the resulting membrane in addition to its proposed effect on sulfur bonding. Further membrane characterization would be required to distinguish between these possibilities. The hypothesis that increasing DMF composition would increase selectivity was rejected, as the smallest DMF composition produced the largest selectivity.

We also found that the highest observed selectivity always occurred at the first measured data point. This trend suggests a reduction in the concentration driving force of PNB as its concentration in the permeate side increases. As the PNB concentration gradient decreases, its rate of permeation would be expected to decrease as well. Meanwhile, since TPM remains at a significantly lower P/R ratio than PNB due to its larger size, its concentration on the permeate side still increases linearly. The changing P/R ratios of the two solutes may cause the overall decrease in selectivity.

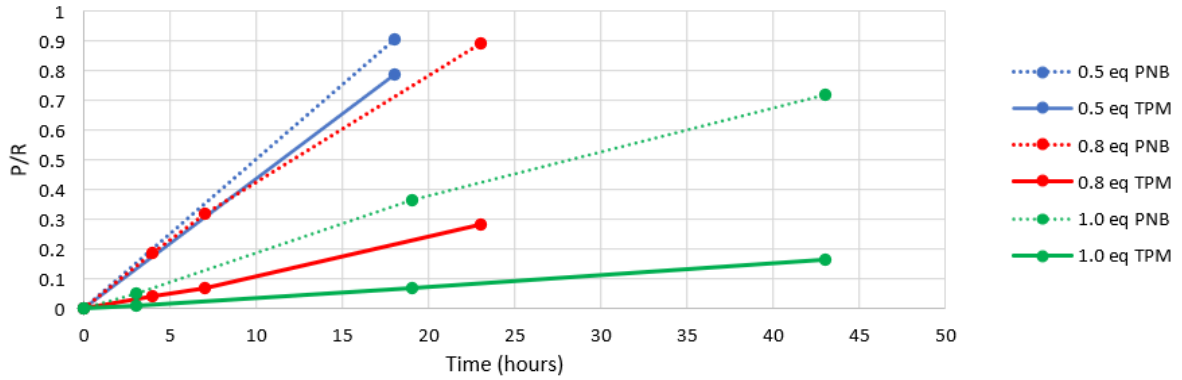
Permeation of PNB and TPM through PDCPD membranes crosslinked with varying equivalents of S_2Cl_2

PDCPD membranes were crosslinked with varying equivalents of S_2Cl_2 in 5% DMF by volume. The selectivities between PNB and TPM for these experiments are shown in Table 2, and the P/R ratios over time are shown in Figure 4. We found that as the molar equivalence of

S_2Cl_2 to DCPD approached 1.0, selectivity increased and flux decreased. The maximum observed selectivity was 5.23 at 1.0 eq. Equivalents beyond 1.0 were not tested. As before, the P/R ratio decreased over time due to decreased PNB permeability, while TPM growth remained constant.

Figure 4

Selectivity of PNB and TPM in PDCPD Membranes with Varying S_2Cl_2 Equivalence in 5% DMF



Permeation of PNB and TPM through a PDCPD membrane crosslinked with a dinorbornene crosslinker and S_2Cl_2

A PDCPD membrane crosslinked with a dinorbornene crosslinker and S_2Cl_2 was synthesized and achieved a maximum observed selectivity of 21.7 between PNB and TPM. The P/R ratio with respect to time is shown in Figure 5. As in previous experiments, the selectivity decreased over time due to the decreasing flux of PNB. Based on unpublished data from previous studies conducted by the Bowden Lab, a PDCPD membrane crosslinked with only the dinorbornene crosslinker achieved a selectivity of approximately 5.7, and a PDCPD membrane crosslinked with only S_2Cl_2 in DCM achieved a selectivity of 13.8. The combined-crosslinker membrane showed a higher observed selectivity than previously reported laboratory measurements for membranes containing either crosslinker individually, suggesting that combining the two crosslinking strategies may enhance selectivity. We hypothesize that the dinorbornene crosslinker and the disulfide links both contribute to crosslinking PDCPD chains together, significantly reducing free volume and increasing selectivity. However, we could not verify this hypothesis using analytical methods such as Raman spectroscopy and X-ray photoelectron spectroscopy.

Figure 5

P/R Ratio of PNB and TPM through a PDCPD Membrane Crosslinked with Dinorbornene Crosslinker and S_2Cl_2

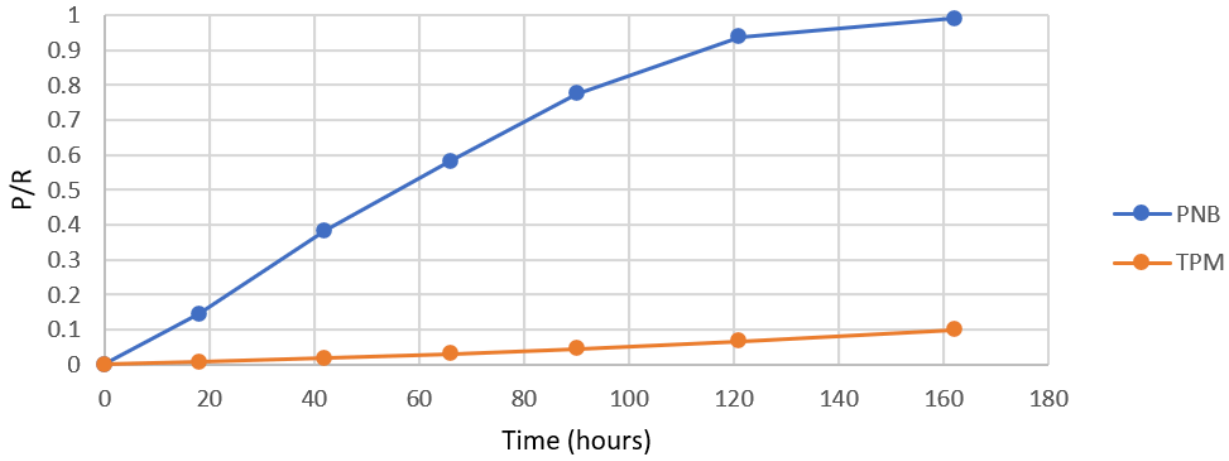


Table 2

Maximum Selectivity of Various Crosslinked PDCPD Membranes

Membrane type	Time (hours)	P/R of PNB	P/R of TPM	Maximum Selectivity
100 % DMF S_2Cl_2(0.5 eq)	17	1.07	0.965	1.37
100 % DMF S_2Cl_2 (0.8 eq)	17	0.752	0.427	1.76
100 % DMF S_2Cl_2 (1.0 eq)	3	0.127	0.0677	1.88
100 % DMF S_2Cl_2 (10.0 eq)	17	0.759	0.324	2.34
80 % DMF S_2Cl_2 (1.0 eq)	3	0.182	0.0824	2.21
66% DMF S_2Cl_2 (1.0 eq)	3	0.152	0.0593	2.56
50% DMF S_2Cl_2 (1.0 eq)	3	0.283	0.229	1.23
5 % DMF S_2Cl_2 (0.5 eq)	18	0.908	0.789	1.15
5 % DMF S_2Cl_2 (0.8 eq)	4	0.186	0.0408	4.56
5 % DMF S_2Cl_2 (1.0 eq)	3	0.0528	0.0101	5.23
Dinorbornene and S_2Cl_2 in DCM	18	0.145	0.00667	21.7

Because the reported maximum selectivities were measured at different time points between experiments, direct quantitative comparison among the maximum selectivities is limited. These values should therefore be interpreted as approximate comparative indicators rather than measures of intrinsic membrane selectivity. In the experiments reported here, the highest observed selectivities generally occurred at early stages of separation, before substantial equilibration between the retentate and permeate solutions.

Permeation of methyl stearate and methyl linoleate through a PDCPD membrane crosslinked with dinorbornene crosslinker and S_2Cl_2

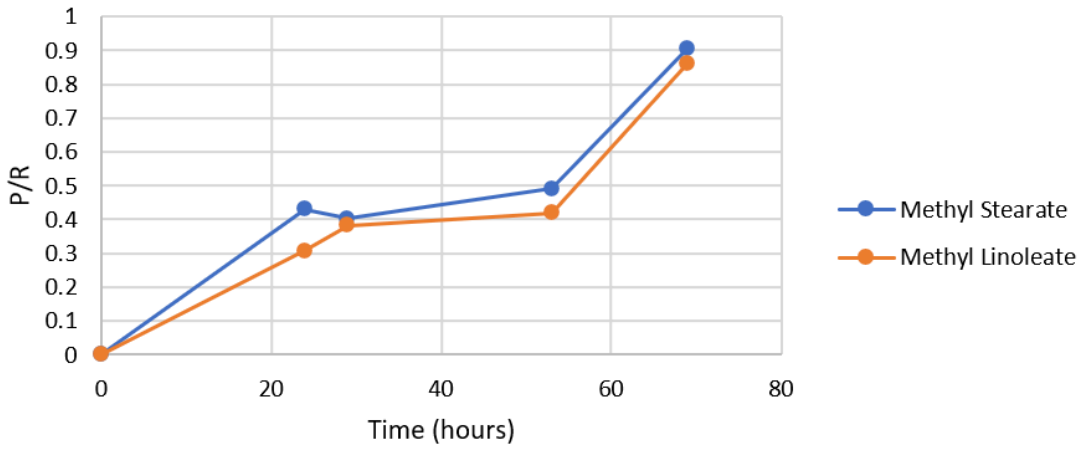
After identifying the dinorbornene-S_2Cl_2 crosslinked PDCPD membrane as the membrane exhibiting the highest observed PNB/TPM selectivity, we conducted an experiment to separate C18 FAMEs, methyl stearate and methyl linoleate, using the double-crosslinked membrane. We achieved a maximum observed selectivity of 1.4 at 24 hours, as shown in Figure 6, meaning that the membrane had a small separation between the two C18s. However, the selectivity decreased after this data point, suggesting that the membrane was only effective in the initial period of separation.

PDCPD membranes may have inherently different separation behavior toward different types of molecules. For instance, the double crosslinker PDCPD membrane was highly effective in separating PNB and TPM, with a selectivity of 21.7, but did not have good separation for the C18 FAMEs. The structure of the pores in PDCPD may be suitable for separating molecules with significantly differing surface areas, but less efficient at separating molecules with similar surface areas.

Future work should determine whether the crosslinking conditions that maximize selectivity for a model system also maximize selectivity for FAMEs by testing varying membrane compositions directly against methyl stearate and methyl linoleate.

Figure 6

P/R ratio of C18 FAMEs through PDCPD membrane crosslinked with dinorbornene crosslinker and S_2Cl_2



Conclusions

This study demonstrates that crosslinking PDCPD membranes with S_2Cl_2 and a dinorbornene crosslinker improves selectivity for molecules with differing sizes. The membrane with the highest observed PNB/TPM selectivity was the dinorbornene-S_2Cl_2 crosslinked PDCPD membrane, with a selectivity of 21.7, representing a large improvement over the previously observed selectivity of the uncrosslinked PDCPD membrane under similar experimental conditions. However, the same membrane also showed a selectivity of 1.4 for methyl stearate and methyl linoleate, indicating that C18 FAMEs were difficult to separate.

These results demonstrate the potential of crosslinked PDCPD membranes for organic solvent nanofiltration while also revealing their limitations. Future work should focus on alternative crosslinker chemicals or increased crosslinking density to achieve selectivity for molecules with similar cross-sectional areas. This research provides a foundation for the development of more selective membranes for FAME separation and contributes to the broader goal of sustainable biofuel production.

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