



PEMION® APPLICATION NOTE: **Handling, Dispersion, and Ink Formulation**

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PROTON EXCHANGE MEMBRANES: HANDLING, STORAGE AND PRE-TREATMENT INSTRUCTIONS

GENERAL

Ionomr Proton Exchange membranes are the only non-fluorinated ion exchange membranes with the required chemical stability for operation in fuel cells. They have low ionic resistance, ultra-low hydrogen crossover, high electrical resistance, and offer strong chemical durability demonstrating over 1000 h of lifetime at Open Circuit Voltage (OCV) when subjected to the Department of Energy's in-situ chemical durability accelerated stress test.

BEFORE YOU START

- Refer to the Pemion® Safety Data Sheet (SDS), and follow appropriate safety practices.
- Store, handle and process the membrane in a clean, dust-free environment.
- All surfaces that come in contact with the membrane during handling, inspection, treatment, storage, and installation should be smooth and clean.
- Pemion® membranes are sensitive to moisture content, and may expand or contract in response to changes in relative humidity. Prior to handling of Pemion® membranes, Ionomr recommends opening the packaging and allowing the membranes to equilibrate to ambient conditions inside for 1 h.
- For best results, handle membranes in a humidity and temperature-controlled environment between 35-50% RH and 20-25 °C.

HANDLING

- Gloves should be worn at all times when handling Pemion® membranes.
- The membrane should be handled with care: do not puncture, crease, abrade, or tear the membrane. Any punctures, creases, lacerations, or abrasions of any kind may reduce membrane performance, or increase the likelihood of leaks (e.g., gas crossover).
- When cutting membranes to desired dimensions, best results are achieved by cutting membranes dry (prior to wetting/soaking, if applicable) using a new, sharp blade, or high-quality sharpened die.
- Membranes may be processed at temperatures up to 160 °C for short durations (< 5 min), but exposure to temperatures exceeding 90 °C for extended durations is not recommended.
- Pemion® membranes are incompatible with soaking in polar solvents such as alcohols for extended durations; Pemion® is readily soluble in polar solvents (see List of Suitable Solvents for Pemion®), but is insoluble in water.
- While on the clear backing sheet, Pemion® membranes are not compatible with temperatures exceeding 65 °C (PF1-HLE9-15-X) or 80 °C (PF1-HLF9-15-X), or coating with solvents containing more than 50% w/w water.
- Free-standing Pemion® membranes (i.e., once the backer has been removed) are not compatible with temperatures exceeding 110 °C (PF1-HLE9-15-X) or 160 °C (PF1-HLF9-15-X).

STORAGE

- Long-term storage of Pemion® membranes is best done in the native, dry form, in original packaging with minimum exposure to heat, light, and changes to relative humidity. Storage between 35-50% RH is recommended.
- Wet storage may be achieved in inert containers containing deionized water with minimum exposure to heat and light.
- Membranes may also be stored in aqueous electrolytes (e.g., NaCl), however, cation exchange from the membrane's acid form to the chosen electrolyte will occur (e.g., H⁺ to Na⁺).



REMOVAL OF PEMION® MEMBRANES FROM THE BACKING LAYER:

Primary method

1. With clean gloved hands, hold the membrane on its backing layer.
2. Using a thumb or a finger, rub against the corner edge of the membrane to produce separation from the backing layer.
3. Once corner separation from the backing layer is achieved, carefully and gently, begin to pull the membrane from the backing layer whilst holding the backer down on a clean dry surface.
4. As the backing layer is released, support the membrane as it is removed from the backing layer, until all of the membrane has been removed.

Secondary method

1. With clean gloves, wet a portion of the membrane/backing layer edge with de-ionized water; this should aid in separation of the membrane from the backing layer. Repeat the steps 3 to 4 in the primary method
2. If the membrane does not separate upon initial wetting, apply a small amount of water across an area close to the edge of the membrane and repeat the primary method. Do not subsequently use the wetted portion of the membrane in the device active area.

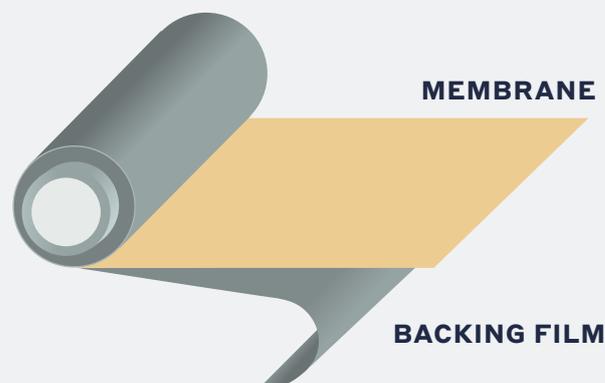
FOR COATING PEMION® MEMBRANE AFTER REMOVING FROM THE BACKING LAYER

Ensure that the membrane remains flat.

A powder coating masking tape (e.g. McMaster-Carr: <https://www.mcmaster.com/7630A24/>), or generally low bonding strength tape is recommended to overlap the edges of the membrane prior to coating procedures, to stabilize the membrane in place. This will help maintain the membrane positioning, and eliminate stress lines that may develop.

Use caution when removing tape from the membrane after the coating process, as tape may strongly bind to Pemion®.

Roll Unwind Orientation (Base Film Facing Out)



PROTON EXCHANGE IONOMERS: DISPERSION AND INK FORMULATION

DISPERSION AND INK FORMULATION

The following is a starting point for preparing catalyst inks based on an ink formulation designed for fuel cell catalyst layers. Please note that different ionomer content may be required depending on the application and operational conditions required. Similarly, depending on the application and performance requirements, further optimization may be necessary, such as solvent composition and weight percent of solids in solution.

1. Calculate the mass of polymer, catalyst powder, and solvents required for electrode composition. This is heavily application-dependent. The recommended solvent ratio is 1:1 alcohol to water (e.g., 1:1 IPA/water). The primary alcohol can be adjusted after the polymer is dissolved in step 2. The volume of solvents required should be dictated by a final weight percent of total solids (catalyst powder + ionomer), with 1-2 wt% suggested for electrode application by spray-coating, or higher as necessary for direct coating catalyst layer deposition methods. One method of controlling drying characteristics (e.g., membrane swelling causing wrinkled catalyst layers, catalyst layer porosity, etc.) is to alter the alcohol ratio; it is strongly advisable not to increase this ratio above 3:1 (alcohol/water).

Note: When adapting existing ink formulations incorporating polymers of other densities, an initial approximation based on polymer density and vol% is suggested. For instance, the density of Pemion is ~ 1.2 g/mL, so an ink based on 30 wt% of a ~ 2.0 g/mL polymer such as a typical PFSA-based ionomer would be approximately equivalent to 18 wt% Pemion.

Ionomr recommends starting with an ink solids composition of 15 wt% ionomer powder and 85 wt% catalyst powder. For example, if utilizing a Pt/C catalyst powder which contains 45% Pt by weight, this recommended formulation would comprise 15 wt% ionomer, 38 wt% Pt catalyst, and 47 wt% carbon, corresponding to an ionomer-to-platinum ratio of 0.39, and ionomer-to-carbon ratio of 0.32.

Disclaimer

Ionomr Innovations Inc. is not responsible for any damages or loss of materials when preparing catalyst inks. Always use caution when mixing any finely divided metal catalyst particles with flammable solvents as spontaneous combustion may occur. Use proper containment procedures and wear personal protective equipment at all times.

2. On a magnetic stirring plate, prepare a 3-5 wt% solution of Pemion and suitable solvent or solvent mixture from below (e.g., methanol) to dissolve the polymer. Reserve a small portion (e.g., 5 mL) of the chosen alcohol to capture the concentrated ionomer coating the glassware after dropwise addition (mentioned later).

Ionomr strongly recommends slow addition of its ionomer powder to stirring solvent (e.g., 400-600 rpm), rather than addition of solvent to the ionomer powder, to ensure uniform dispersion and faster dissolution times. Mild heating during dissolution is recommended (e.g., 60 °C). Typically, heating and stirring for 24-48 hours is sufficient when preparing a Pemion ionomer solution. If particles are noted, the ionomer solution can be passed through a glass fiber filter to eliminate any insoluble and contaminants. Di

3. Preferably in a narrow-necked glass bottle and on a magnetic stirring plate, add the catalyst (e.g., Pt/C) powder followed by a stir bar, and all of the calculated water. Stir gently (e.g., 100 RPM) until catalyst powder is fully wetted and dispersed. Increase stir rate until a vortex begins to form but before cavitation (e.g., 400-600 RPM, heavily dependent on ink volume and stir bar size).

4. Into the fully wetted catalyst powder mixture, slowly pour in the calculated alcohol, excluding the 5 mL reserve solvent (from step 2) or the alcohol which is contained in the ionomer solution.

5. Maintain vigorous stirring and add the alcohol/ionomer solution dropwise, visually ensuring surface accumulation of polymer is kept to a minimum. Occasional swishing of the solution may help to integrate catalyst particles that accumulate at the ink-bottle interface. Using the remaining 5 mL reserve of alcohol, rinse the ionomer glassware (i.e., capture the residual polymer so calculated values for ionomer are realized) and dislodge any remaining catalyst powder from the sides of the ink bottle. Stir at moderate rate (e.g., 300 RPM) until use; a minimum of four hours is recommended. Treatment in a low-power sonication bath for 15-30 minutes after an initial 30-60 minutes of stirring may help homogeneity.

LIST OF SUITABLE SOLVENTS FOR PEMION™ POLYMER PP1-HNN9-00

Dissolution of Pemion® ionomer is typically achieved following stirring and gentle heating (e.g., 250-500 rpm at 60 °C) in a chosen solvent for 24-48 h. Filtration of the polymer solution following dissolution is recommended. A sample procedure is given below the dissolution table.

Solvent Type	Comments	Solubility wt%
Methanol	Low-boiling solvent for spray coating, electrode fabrication, etc.	1% – 10%
Reagent Alcohol (85% EtOH/ 5% MeOH/ 5% isopropanol)	Low-boiling solvent for spray coating, electrode fabrication, etc.	1% - 10%
Ethanol	Low-boiling solvent for spray coating, electrode fabrication, etc.	1% - 7%
Ethanol/IPA (50:50 by weight)	High boiling point solvents, can cause complications with gas permeability	1% - 5%
IPA/water (50:50 by volume)	Low-boiling solvent for spray coating, electrode fabrication, etc	1% – 10%
DMSO	Recommended high boiling point solvent to achieve high viscosity, can cause complications with gas permeability	1% - 10%
NMP, DMF	High boiling point solvents, can cause complications with gas permeability	1% - 10%

PREPARING A 5 WT% SOLUTION OF PEMION® IONOMER IN METHANOL:

To a 20 mL glass scintillation vial with a stir bar was added 9.5 g of suitable alcohol or solvent mixture (e.g. methanol). The vial was placed on a magnetic stirring hot plate and stirred at 300 rpm. Pemion® ionomer (0.5 g) was slowly added to the solvent over the course of 10 seconds. The resulting 5 wt% polymer suspension was stirred at 300 rpm with heating at 60 °C for 48 hours, giving a viscous and uniform polymer solution. Slow addition of the ionomer to the solvent helps prevent clumping and improves dissolution characteristics. After cooling to room temperature, the solution was filtered through a 55 mm diameter 11 µm pore size Grade 1 Whatman™ (CAT no. 1001-055) filter paper to remove any insoluble materials before further use.

DOCUMENT CHANGE HISTORY

Document ID	Document ID
FM-7005-F	Pemion® Application Note: Handling, Dispersion, and Ink Formulation

Revision	Prepared By	Approved By	Effective Date
F	Omid Toussi	Ben Britton	June 2, 2021

This document is reviewed to ensure its continuing relevance to the systems and process that it describes.

REVISION HISTORY:

Revision	Date	Description of Changes	Approved By
A	Jan. 27, 2020	Initial Draft	Ben Britton
B	Oct. 16, 2020	Updated backer removal, ink formulation, solvents	Ben Britton
C	Nov. 16, 2020	Additional solvents	Ben Britton
D	Feb 26, 2021	Document design and name updated	Ben Britton
E	May 31, 2021	Pemion-on-backer incompatibilities added; precautionary statement regarding tape added; general revisions	Ben Britton
F	Aug. 10, 2021	Added new sections: before you start, handling & storage	