IONOMR INNOVATIONS

PEMION® APPLICATION NOTE:

Handling, Dispersion, and Ink Formulation

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Document ID: FM-7013-J

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

GENERAL

Ionomr's Pemion® membranes are the only hydrocarbon-based cation 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 excellent chemical and mechanical durability.

This document serves as a general guide for the handling, use, and storage of Pemion® membranes.

BEFORE YOU START

- Refer to the Pemion® Safety Data Sheet (SDS), and follow appropriate safety practices. Although Pemion® membranes are supplied in a dry state, the packaged product may contain residual water or trace solvents.

- Store, handle and process the membrane in a clean, dust-free, and well-ventilated environment, preferably in a relative humidity (RH) and temperature-controlled environment between 50-60% RH and 20-25 °C.

- 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. Hence, proper control of ambient environmental conditions is strongly 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 60 °C.

  In addition, dry conditions (e.g., < 40% RH) are not recommended, because they may cause difficulty with removal of the membrane from the backing sheet.

- Free-standing Pemion® membranes (i.e., once the backer has been removed) are not compatible with temperatures exceeding 160 °C.

- Pemion® membranes are best used as-received. No pre-treatment is necessary, and soaking in aqueous solutions is not recommended. Pemion® will readily ion-exchange with aqueous electrolytes (cations) in solutions containing them.

- For applications requiring radical scavengers or antioxidants, it is recommended that their integration is achieved by incorporation into cell electrodes during cell assembly, rather than by soaking/exchange of the membrane.

HANDLING

Gloves should be worn at all times when handling Pemion® membranes.

- The membrane should be handled with care: do not puncture, crease, abrase, 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. Care should be taken when cutting membranes and related membrane electrode assembly components via laser, because incorrect laser settings or operation may result in burred edges & negatively impact materials integrity.

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 in original packaging or humidity barrier bags between 50-60% RH is recommended; original packaging materials are resealable using a heat sealer.

- Wet storage is not recommended due to swelling of the material in aqueous environments.

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 small corner or edge of the membrane/backing layer with de-ionized water; this should aid in separation of the membrane from the backing layer. Use of precision tweezers may additionally help with this separation. Repeat the steps 1 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.

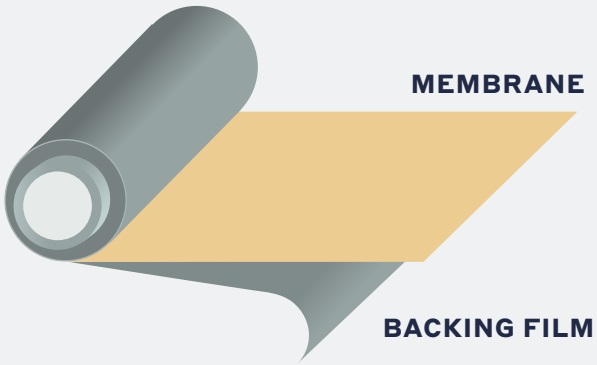
FOR COATING PEMION® MEMBRANES AFTER REMOVING FROM THE BACKING LAYER

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)



LIST OF SUITABLE SOLVENTS FOR PEMION® POLYMER PP1-HNN8-00

Dissolution of Pemion® ionomer is typically achieved following stirring and gentle heating (e.g., 300-600 rpm at 60 °C) in a chosen solvent for 24-72 h, depending on vessel size, concentration, and amount of ionomer being dissolved. 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% EtOHI/ 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 powder PP1-HNN8-00 (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 WhatmanTM (CAT no. 1001-055) filter paper to remove any insoluble materials before further use.

PROTON EXCHANGE IONOMERS: 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.

GENERAL CONSIDERATIONS

a. The recommended solvents for initial dissolution of Pemion® ionomer are provided further in this document. The primary alcohol and alcohol/water ratio can be adjusted after the polymer is dissolved or dispersed per step 3.

The volume of solvents required should be dictated by a final weight percent of total solids required by the application. For example, 1-2 wt% (of ionomer + catalyst powder) is suggested for electrode application by spray-coating. Higher may be necessary for direct coating catalyst layer deposition methods.

The recommended solvent composition for fuel cell catalyst inks is 2:1 water to alcohol (e.g., 2:1 H2O/IPA).

b. Drying characteristics of catalyst inks (e.g., in the event of wrinkled catalyst layers, to adjust catalyst layer porosity, etc.) can be modulated by altering the alcohol/water ratio; it is however strongly advisable not to decrease this ratio below 1:1 (water/alcohol). Use of higher boiling point co-solvents (e.g., isopropanol instead of methanol), or non-solvents, may also be suitable.

c. The density of Pemion is 1.2 g/mL. 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 typical PFSA-based ionomers is approx. 2.0 g/mL. Hence, an ink based on 30 wt% of a ~2.0 g/mL polymer such as a PFSA would be approximately equivalent to 18 wt% Pemion.

d. An ink solids composition of 15 wt% Pemion® ionomer powder and 85 wt% catalyst powder is recommended as a starting point when working with Pemion® ionomer. 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-Pt ratio of 0.39, and ionomer-to-carbon ratio of 0.32.

PROTON EXCHANGE IONOMERS: DISPERSION AND INK FORMULATION

PREPARATION

Calculate the mass of polymer, catalyst powder, and solvents required for the desired electrode composition. This is heavily application-dependent.

IONOMER DISSOLUTION

a. Measure out the calculated amount of suitable solvent or solvent mixture (e.g., methanol; see previous page) and begin stirring on a magnetic stirring plate at medium speed (e.g., 300-600 rpm) using appropriate laboratory glassware.

Reserve a small portion (~10 mL) of the chosen alcohol to capture the concentrated ionomer coating the glassware after dropwise addition (mentioned later).

b. Weigh the appropriate amount of Pemion® ionomer necessary to achieve a 3-5 wt% solution. Slowly add it to the stirring solvent to dissolve the polymer. Slow addition of polymer to solvent (rather than addition of solvent to the ionomer powder) is strongly recommended 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 insoluble particles are noted after dissolution, the ionomer solution can be passed through a paper or glass fiber filter.

Disclaimer

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

CATALYST INK PREPARATION

a. Preferably in a narrow-necked glass bottle and on a magnetic stirring plate, add the desired 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., 300-600 RPM, heavily dependent on ink volume and stir bar size).

b. Into the fully wetted catalyst powder mixture, slowly pour in the calculated alcohol, **excluding** the ~10 mL reserve solvent (from step 3a) or the alcohol which is contained in the ionomer solution.

c. 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 ~10 mL reserve of alcohol (from step 3a), rinse the glassware which contained the ionomer solution (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 and dispersion.

DOCUMENT CHANGE HISTORY

Document ID	Document ID		
FM-7013-J	Pemion® Application Note: Handling, Dispersion, and Ink Formulation		
Revision	**Prepared By**	**Approved By**	**Effective Date**
J	Mike Adamski	Andrew Belletti	Apr. 3, 2023

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	Andrew Belletti
G	Mar. 22, 2022	General Updates	Andrew Belletti
H	Apr. 7, 2022	General Updates	Andrew Belletti
I	Sept. 16, 2022	Catalyst ink solvent recommendations, general updates	Andrew Belletti
J	Mar. 27, 2023	General handling and storage guidelines updates	Andrew Belletti