HANDLING AND GENERAL CONSIDERATIONS FOR PEMION® PP1-HNN8-00 IONOMER

- Refer to the Pemion® Safety Data Sheet (SDS), and follow appropriate safety practices. Pemion® ionomers are supplied in a dry, powder state.
- Handle and process ionomers in a clean, dust-free, and well-ventilated environment such as a fume hood.
- Store ionomers sealed in clean, inert containers (e.g., glass jars).
- Gloves should be worn at all times when handling Pemion® ionomers.
- Pemion® ionomers are sensitive to moisture content, and will uptake or lose water content in response to changes in relative humidity. Hence, proper control of ambient environmental conditions is strongly recommended to ensure consistent, accurate weighing and handling.

LIST OF SUITABLE SOLVENTS

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% EtOHl/ 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.

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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.

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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.

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.

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.

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