Probing water structure and transport in proton exchange membranes
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SOLVAY. Aquivion® E98-05 perfluorosulfonic acid [Online].


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#pragma rtGlobals=3  // Use modern global access method and strict wave access.
#include <Global Fit 2>

variable/G V_FitTol
V_FitTol=0.00001  //the global variables to set the tolerance to break the iteration.

Function getGfit(nC, epsi, paral, name, s_Ns)
string name
variable paral
variable s_Ns //s_Ns=1, process the raw spectra; s_Ns=2, process the Normalized spectra
variable nC, epsi //nC indicates the number of components;
variable nD //nD is the number of data spectra
variable i, j;
variable k;
variable points
NVAR V_FitTol=V_FitTol
gettingData(name, s_ns)
wave/T datasets1
wave/T datasets2
wave Wvpoints
points=Wvpoints[0]
wave Int_max
Appendix A  

Igor codes for CCLS

nD=(dimsize(datasets1,0))
make/o/n=(0,((points)+nC)) Fits //save the resultant coefs
make/o/n=(0,(nD*nC+(points))) matrixInitG //save the initial guess for the coefs

//*****test wave:
FitFuncnames ******
make/o/T/n=3 FitFuncnames //a test wave containing a list of fit functions: FfunN
   FitFuncnames[0]="Fit2D1"; //fit with 2 components of which one component is deduced
   FitFuncnames[1]="Fit3D1";
   FitFuncnames[2]="Fit4D1";

//*****

fruit: wave:

Datasets

nD=(dimsize(datasets1,0))
make/T/o/n=(nD,2) datasets
string tdatasets
tdatasets="datasets"+num2str(S_NS)
wave/T w01=$(tdatasets)
Datasets[][0]= w01[p]
Datasets[][1]= "_calculated_

//*****wave

Coefdatasetlinkage: 

SetDimLabel 1,0,'f()',coefdatasetlinkage // 1=col, 0 or 1 = No of Col, string=label, wavenname
SetDimLabel 1,1,first,coefdatasetlinkage
SetDimLabel 1,2,last,coefdatasetlinkage
SetDimLabel 1,3,N,coefdatasetlinkage
Coefdatasetlinkage[][0]=(nC-2) // 0 = R0 in fitFuncnames, which is Fit2D1, points+2 coef
for (i=0;i<2;i+=1)
   for(j=0; j<nD; j+=1)
      Coefdatasetlinkage[j][i+1]=((points-1))*(i+j)+j
Igor codes for CCLS

for (i=4; i<\text{linkC}; i+=1)
    Coefdatasetlinkage[0][i] = i - 4
endfor
for (j=0; j<\text{nC}; j+=1)
    for (i=1; i<\text{nD}; i+=1)
        Coefdatasetlinkage[i][j+4] = \text{linkC} - 4 + (i - 1) \times \text{nC} + j
    endfor
endfor
for (j=(4+\text{nC}); j<\text{linkC}; j+=1)
    for (i=1; i<\text{nD}; i+=1)
        Coefdatasetlinkage[i][j] = Coefdatasetlinkage[0][j]
    endfor
endfor
Get\_initalG\_Weights(nC, nD)
wave iniGW, iniGspectrum
wave bulk
k = (nC \times nD + (\text{points}))
*****test wave: coefNames
make/o/T/n=(k) coefNames
*****test wave: constraintwave,
make/o/T/n=(0) ConstraintWave //the non-negative constraints, \text{K}>0, a TEXT wave
string strConstraint
string strSum
Appendix A

Igor codes for CCLS

string tempstr

if (nC<=4)
    for(i=0;i<nC;i+=1)
        strConstraint="K"+num2str(i)+" ">=" 0"
        insertpoints/M=0 (DimSize(constraintwave,0)),1,
    constraintwave
    constraintwave[DimSize(constraintwave,0)-1][]=strConstraint
    strConstraint="K"+num2str(i)+"
<="num2str(Int_max[0]^(abs(S_NS-2)))
        insertpoints/M=0 (DimSize(constraintwave,0)),1,
    constraintwave
    constraintwave[DimSize(constraintwave,0)-1][]=strConstraint
endfor

for(i=nC;i<((points+nC-1));i+=1)
    strConstraint="K"+num2str(i)+" ">=" 0"
    insertpoints/M=0 (DimSize(constraintwave,0)),1,
    constraintwave
    constraintwave[DimSize(constraintwave,0)-1][]=strConstraint
endfor

for (i=0;i<((nD-1));i+=1) //nD: Numbers of spectra
    for(j=0;j<nC;j+=1) //nC: numbers of components
        strConstraint="K"+num2str(nC+points+i*nC+j)+"
>=" 0"
        insertpoints/M=0 (DimSize(constraintwave,0)),1,
    constraintwave
    constraintwave[DimSize(constraintwave,0)-1][]=strConstraint
    strConstraint="K"+num2str(nC+points+i*nC+j)+"
<="+" 1"
<table>
<thead>
<tr>
<th>Appendix A</th>
<th>Igor codes for CCLS</th>
</tr>
</thead>
</table>

```plaintext
insertpoints/M=0 (DimSize(constraintwave,0)),1, constraintwave
constraintwave[DimSize(constraintwave,0)-1][]=strConstraint
endfor
endfor

elseif (nC>4)
    for (j=0;j<(nC-1);j+=1) //constraint the non-negativity: K0, K1, K2 are non-negative
        strConstraint="K"+num2str(j)+" >="+" 0"
        insertpoints/M=0
        (DimSize(constraintwave,0)),1, constraintwave
        constraintwave[DimSize(constraintwave,0)-1][]=strConstraint
        strConstraint="K"+num2str(j)+" <="+num2str(Int_max[0]^(abs(S_NS-2)))
        insertpoints/M=0
        (DimSize(constraintwave,0)),1, constraintwave
        constraintwave[DimSize(constraintwave,0)-1][]=strConstraint
    endfor
endif
make/o/T=1 Strwave
for (i=0;i<nC;i+=1) //Sum of Fit weights from spectrum 1
    Strconstraint = "K"+num2str(0)
    StrConstraint+="+ K"+num2str(i)
endfor
StrWave=Strconstraint + ">=0" //non-negative
insertpoints/M=0 (DimSize(constraintwave,0)),1, constraintwave
```

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Appendix A

Igor codes for CCLS

constraintwave[DimSize(constraintwave,0)-1][]=strwave

StrWave=StrConstraint+"<=1" //sum is smaller than 1

insertpoints/M=0 (DimSize(constraintwave,0)),1,

constraintwave

constraintwave[DimSize(constraintwave,0)-1][]=strWave

variable nn

for (i=0;i<(nD-1);i+=1)
    for(nn=0;nn<nC;nn+=1)
        Strconstraint = "K"+num2str((points)+0+i*nC)
        StrConstraint+="+
        K"+num2str(nC+(points)+nn+i*nC)
    endfor

StrWave=StrConstraint + ">=0"

//non-negative

insertpoints/M=0

(DimSize(constraintwave,0)),1, constraintwave

constraintwave[DimSize(constraintwave,0)-1][]=strWave

StrWave=StrConstraint+"<=1" //sum is smaller than 1

insertpoints/M=0

(DimSize(constraintwave,0)),1, constraintwave

constraintwave[DimSize(constraintwave,0)-1][]=strWave

endfor

//***** Coefwave, initial guesses

variable do1

do1=0;

for (do1=0;do1<paral;do1+=1)       //***** paral: # of initial guess


Appendix A

Igor codes for CCLS

make/o/n=(k,2) Coefwave

variable in1

Coefwave[0][0]=(0.6+enoise(0.1))

for (j=1;j<nC;j+=1)
    Coefwave[j][0]=(1-Coefwave[0][0])/(nC-1)
endfor

for (in1=0;in1<(nD-1);in1+=1)
    for (i=0;i<nC;i+=1)

        Coefwave[nC+(points)+in1*nC+i][0]=Coefwave[i][0]

    endfor
endfor

//****specify the initial guess of the unknown component spectrum with random values

for (i=nC;i<((points)+nC);i+=1) //227 is the spectral points
    Coefwave[i][0]=0.01+(enoise(0.01))
endfor

SetDimLabel 1,1,Epsilon,Coefwave

Coefwave[][1]=epsi

make/o/n=((points)+nD*nC) InitalGuess

duplicate/o/r=[0,((points)+nD*nC)][0] coefwave testG

InitialGuess=testG[p][0]

insertpoints/M=0 (DimSize(matrixInitG,0)),1, matrixInitG

matrixInitG[DimSize(matrixInitG,0)-1][]=initialGuess[q]

string errorName, En
En = "En";

string errorMessage, Em
Em = "Em";

variable maxIters

string resultWavePrefix, RsltP

RsltP="RsltP";

DoNewGlobalFit(FitFuncNames, DataSets, CoefDataSetLinkage, CoefWave, coefNames, ConstraintWave,31, (points),1)
string teststr="Coef_"+w01[0]
wave Coef_test=$(teststr)
endfor
killwaves testG, initalGuess
End

**Function** gettingData(name, s_Ns)

string name
variable s_Ns
String list;
String theWave;
list = Wavelist(name+"*",";","")
variable i,j,numWaves;
i=0;
j=0;
make/o/T/n=0 datasets1
make/o/T/n=0 datasets2
make/o/n=0 Wvpoints
make/o/D/n=1 Int_Max, Int_temp
Int_Max=0
do
theWave = StringFromList(i, list)
insertpoints/M=0 (DimSize(datasets1,0)),1, datasets1
datasets1[(DimSize(datasets1,0)-1)]= theWave
if (strlen(theWave) == 0)
    break
endif
wave datawave=$(thewave)
insertpoints/M=0 (DimSize(Wvpoints,0)),1, Wvpoints
Wvpoints[(DimSize(Wvpoints,0)-1)]=
(dimsize(datawave,0))
if(s.ns==2) //normalize the peak area to 1
    string wName
    wName="N"+thewave
Appendix A  

Igor codes for CCLS

insertpoints/M=0 (DimSize(datasets2,0)), 1, datasets2 

datasets2[(DimSize(datasets2,0)-1)]= wName

make/o/n=(dimsize(datawave,0)) $wName

wavestats/Q datawave 

wave w01=$wName 

w01=datawave/V_sum 

endif 

i+= 1

j+=1

while (1) 

// Loop

until break above 

killwaves Int_temp 

DeletePoints j, 1, datasets1 

End

Function Get_initialG_spec(spec1, bulk) 

//***** initial guess for spectral component: IniGSpec*****// 

wave spec1//1 spec from the batch data sets 

wave bulk// 1 known components 

duplicate/o spec1, rsd1, fit1 

Make/D/N=1/O W_coef //No. coefs 

Make/O/T/N=1 T_Constraints //No. constraints 

T_Constraints[0] = {"K0 > 0"} //the coefficient should 

be non-negative 

W_coef[0] = {0.5} 

FuncFit/Q/NTHR=0 FitwithONEwave W_coef spec1 
/X=bulk /D 

fit1=W_coef[0]*bulk[p] 

rsd1=spec1-fit1 

duplicate/o rsd1 test 

variable i 

for (i=0;i<(dimsize(rsd1,0)); i+=1) 

if (rsd1[i]>=0) 


Appendix A

Igor codes for CCLS

test[i]=0
elseif (rsd1[i]<0)
test[i]=abs(rsd1[i])
endif
endfor

variable ptest,ratio
wavestats/q test
ptest=x2pnt(test,V_maxloc)
ratio=(spec1[ptest])/(fit1[ptest])
fit1=fit1[p]*ratio
rsd1=abs(spec1-fit1)
duplicate/o rsd1 iniGSpec //IniGspec is the initial guess for the deduced components

killwaves test, rsd1, fit1

End

Function Get_initalG_Weights(nC, nD)

//*****initial guess for lstsqrfitting weights: IniGW*****

variable nC, nD //nC is the number of components, and nD is the number of data spectra
variable i,noW;
noW=(nC*nD);
make/o/n=(noW) IniGW
for (i=0;i<noW;i+=1)
    IniGW=500*abs(enoise(1,1)) //give non-negative random values to the test, 0<=test<=1
endfor

End

Function Fit2D1(w,x) : FitFunc

Wave w
variable x
wave bulk = root:blk
variable point
point = x2pnt(bulk,x)

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| Appendix A | Igor codes for CCLS |

```igor
wave Nb = root:Wvpoints
variable points
points = Nb[0]
wavestats/Q/R=[2,(points+1)] w
return w[0] * bulk[point] * v_sum + w[1] * w[point+2]
End
```

**Function** `Fit3D1(w,x) : FitFunc`

```igor
Wave w
variable x
wave bulk01 = root:bulk  //should be changed according to the know components
duplicate/o bulk01 bulk
wavestats/Q bulk
bulk/=V_sum
duplicate/o dry01 dry
wavestats/Q dry01
dry/=V_sum
variable point
wave Nb = root:Wvpoints
point = x2pnt(bulk,x)
variable points
points=Nb[0]
wavestats/Q/R=[3,229] w  //for oh region
wavestats/Q/R=[3,((points)+2)] w  //for od region
End
```

**Function** `Fit4D1(w,x) : FitFunc`

```igor
Wave w
variable x
```

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wave bulk = root:blk
wave dry = root:nonblk
wave HOD = root:mix
variable point
    bulk[0,229]=0
wavestats/Q bulk
bulk/=V_sum
    dry[219,394]=0
wavestats/Q dry
dry/=V_sum
    point = x2pnt(bulk,x)
duplicate/o/R=[4,(dimsize(bulk,0))+3] w testspec
testspec=abs(testspec)
wavestats/Q testspec
return w[0]*bulk[point]+w[1]*dry[point]+w[2]*HOD[point]+w[3]*w[point+4]/V_sum
End
A well-operated proton exchange membrane fuel cell (PEMFC) requires an optimized water management system wherein the proton is efficiently transported in the proton exchange membrane (PEM) of the FC and the flooding at the cathode of the FC is avoided. The water transport in PEM plays a crucial role in the water management that greatly affects the performance of the PEMFC. Fast water transport (driven by the electro-osmotic drag) in the PEM from the anode of the PEMFC to cathode is required because it is closely related to the proton transport in the PEM; meanwhile, fast water transport driven by the concentration gradient from the cathode to the anode of the PEMFC can suppress or even prevent the flooding at the cathode. A PEM manifesting outstandingly faster water transport property is required, and knowing how the membrane structure determines the water transport is crucial for designing such a PEM.

In Chapter 4 of my thesis, we have studied the water structure in the hydrated proton exchange membranes. Two industry-standard commercial Nafion® membranes, Nafion® 212 (N212) and Nafion® 117 (N117), were studied. We spectrally distinguished the water subspecies in the Nafion® membranes and quantified the fractional contribution of each water subspecies. There are two types of water subspecies in the PEMs: 1) bulk water (bulkW) that hydrogen bonds to other surrounding water molecules and 2) nonbulk water (nonbulkW) that interacts strongly with the membrane structure. We found that the N117 has a larger amount of nonbulkW, exhibits a larger proton conductivity, and has a larger water mobility N212. The additional amount of the nonbulkW in N117 results from a modified
nanoscale structure of the PEM where the ionic domains have a larger surface-to-volume ratio and a larger hydrophilic head group spacing compared to N212.

In Chapter 5, the diffusivity of the bulkW and the nonbulkW in Nafion® membranes were experimentally quantified in five different PEMs. We found that the diffusivity of the nonbulkW is 2.5-fold faster than the bulkW in all PEMs with different ratio between ADC(nonbulkW) and ADC(bulkW), depending both polymer chemistry. Interestingly, the overall water diffusivity in all membranes could be represented by a linear combination of the water subspecies weighted by the fractional contributions. Based on the results of Chapter 5, a clear design target for membrane manufacturing is provided: maximize the nonbulkW.

In Chapter 6, I established a model surface system to mimic the surface of the nanoscale water channels in Nafion® membranes. The concentration of the sulfonic acid groups in the surface was varied and the interactions between the water structure and these surfaces in acid solutions (HCl, pH =1) has been studied by AFM. The structure of water on the different surfaces at the molecular level was discussed.

As the final part of the thesis, Chapter 7 ideas for a modified flow cell design with which one could measure the water diffusivity in the membrane at different hydration states. Preliminary results of water diffusivity in different anion exchange membranes were shown showing that the CARS platform can be used to probe the water transport of different materials. Considering the experimental accessibility of the experimental and analytical platform, this is a powerful method to produce reliable results and compare the water transport properties of different membrane materials. Last but not least, I suggested using the vibrational Raman scattering spectroscopy to study the degradation of the PEMs in PEMFCs and provide a clear design target for making a PEM that sustain substantially long in the PEMFC.
SAMENVATTING

Een goed-functionerende protonuitwisselingsmembraanbrandstofcel (PEMFC) heeft een geoptimeerd watermanagementsysteem nodig waarin het proton efficiënt getransporteerd wordt in het protonuitwisselingsmembraan (PEM) van de brandstofcel en overstroming aan de kathode van de brandstofcel vermeden wordt. Het watertransport in het PEM speelt een cruciale rol in het watermanagement dat de prestatie van de PEMFC in grote mate beïnvloedt. Snel watertransport (aangedreven door de elektroosmotische aantrekkingskracht) in het PEM van de anode van de PEMFC tot de kathode is nodig omdat het sterk gerelateerd is aan het protontransport in het PEM; tegelijkertijd kan snel watertransport aangedreven door de concentratiegradiënt van de kathode naar de anode van de PEMFC de overstroming aan de kathode onderdrukken of zelfs tegengaan. Een PEM met een sterk snellere watertransporteigenschap is benodigd, en de wetenschap hoe de membraanstructuur het watertransport bepaalt is cruciaal om zo’n PEM te ontwerpen.

In hoofdstuk 4 van mijn proefschrift hebben we de waterstructuur in gehydrateerde protonuitwisselingsmembranen bestudeerd. Twee industriestandaard commerciële Nafion® membranen, Nafion® 212 (N212) en Nafion® 117 (N117), werden bestudeerd. We hebben de waterondersoorten in de Nafion membranen spectroscopisch onderscheiden en de fractionele contributie van elke waterondersoort gekwantificeerd. Er zijn twee soorten water ondersoorten in de protonuitwisselingsmembranen: 1) bulk water (bulkW) dat waterstofbruggen vormt met andere omliggende watermoleculen en 2)
Samenvatting
	niet-bulk water (niet-bulkW) dat sterk met de membraanstructuur interacteert. We hebben gevonden dat N117 een grotere hoeveelheid niet-bulkW heeft, een grotere protongeleidbaarheid laten zien en een grotere watermobiliteit. De toegevoegde hoeveelheid niet-bulkW in N117 is het resultaat van een gemodificeerde nanoschaalstructuur van het PEM waar de ionische domeinen een grotere oppervlakte-tot-volume verhouding en een grotere hydrofiele kopgroep tussenruimte in vergelijking met N212 hebben.

In hoofdstuk 5 werden de diffusiviteit van het bulkW en het niet-bulkW in Nafion membranen experimenteel gekwantificeerd in 5 verschillende protonuitwisselingsmembranen. We hebben gevonden dat de diffusiviteit van het niet-bulkW 2,5 keer sneller is dan het bulkW in alle protonuitwisselingsmembranen met een verschillende verhouding tussen de waargenomen diffusiecoëfficiënt (ADC) van niet-bulkW en bulkW, afhankelijk van polymeerchemie. Interessant is dat de globale waterdiffusiviteit in alle membranen gepresenteerd wordt door een lineaire combinatie van de waterondersoorten gewogen door hun fractionele contributies. Gebaseerd op de resultaten van hoofdstuk 5 wordt een duidelijk ontwerpdoel voor membraanproductie geboden: maximaliseer het niet-bulkW.

In hoofdstuk 6 heb ik een model oppervlaktesysteem tot stand gebracht om de oppervlakte van de nanoschaal waterkanalen in Nafionmembranen na te bootsen. De concentratie van sulfonzuurgroepen op de oppervlakte werd gevarieerd en de interactie tussen de waterstructuur en deze oppervlakten in zure oplossingen (HCl, pH = 1) werd bestudeerd met atoomkrachtmicroscopie (AFM). De structuur van water op de verschillende oppervlakten op de moleculaire schaal werd besproken.

In het laatste deel van het proefschrift, hoofdstuk 7, worden ideeën voor een gemodificeerd stromingscelontwerp waarmee de waterdiffusiviteit in het membraan bij verschillende hydratatiestaten gemeten kan worden. Voorlopige resultaten van waterdiffusiteit in verschillende anionuitwisselingsmembranen werden gepresenteerd die
Samenvatting

laten zien dat het CARS-platform gebruikt kan worden om het watertransport van verschillende materialen te onderzoeken. Wat de experimentele toegankelijkheid van dit experimentele en analytische platform betreft is dit een krachtige methode om betrouwbare resultaten te produceren en de watertransporteigenschappen van verschillende membraanmaterialen te vergelijken. Tenslotte stel ik voor om vibrationele Raman verstrooiingsspectroscopie te gebruiken om de degradatie van protonuitwisselingsmembranen in protonuitwisselingsmembraanbrandstofcellen te bestuderen en biedt een duidelijk ontwerpdoel om een PEM te maken dat substantieel langer standhoudt in een PEMFC.
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