Probing water structure and transport in proton exchange membranes

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APPENDIX A
IGOR CODES FOR CCLS

#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
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";
//***** test 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, wavename
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

```plaintext
endfor
endfor

Coefdatasetlinkage[][3]=(points)+nC//numbers of coef.

variable LinkC; //LinkC is the col number in the Coefdatasetlinkage.

linkC=(dimsize(Coefdatasetlinkage,1));

for(i=4;i<linkC;i+=1)
    Coefdatasetlinkage[0][i]=i-4
endfor
for(j=0;j<nC;j+=1
    for (i=1; i<nD;i+=1)
        Coefdatasetlinkage[i][j+4]=linkC-4+(i-1)*nC+j
    endfor
endfor
for (j=(4+nC); j<linkC;j+=1)
    for (i=1; i<nD; i+=1)
        Coefdatasetlinkage[i][j]=Coefdatasetlinkage[0][j]
    endfor
endfor
Get_initialG_Weights(nC, nD)
wave iniGW, iniGspec1
wave bulk
k=(nC*nD+(points))
//****test wave: coefNames
make/o/t/n=(k) coefNames
//****test wave: constraintwave,
make/o/T/n=(0) ConstraintWave //the non-negative constraints, 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]^\text{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 -1
        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"
Igor codes for CCLS

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

```plaintext
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) InitialGuess
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
    endif
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_initalG_spec(spec1, bulk)
//***** inital guess for spectral component: IniGSpec*****//
wave spec1// 1 spec from the batch data sets
wave bulk// 1 known components
duplicate/o sp
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)
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

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
    //return
w[0]*bulk[point]*v_sum+w[1]*dry[point]*v_sum+w[2]*w[point+3]
wavestats/Q/R=[3,((points)+2)] w    //for od region
return
w[0]*bulk[point]*V_sum+w[1]*dry[point]*v_sum+w[2]*w[point+3]
End

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

Wave w
variable x
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 onder soorten 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 gomodificeerd stromingscelontwerp waarmee de waterdiffusiviteit in het membraan bij verschillende hydratatiestaten gemeten kan worden. Voorlopige resultaten van waterdiffusiteit in verschillende anionuitwisselingsmembranen werden gepresenteerd die
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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