

Dataset to The Effect of Sulfate Electrolytes on the Liquid-Liquid Equilibrium of 2-MTHF/Water/5-HMF: Experimental Study and Thermodynamic Modeling

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Abstract

The presented experimental data are associated with the investigations and results published in the following article: "The Effect of Sulfate Electrolytes on the Liquid-Liquid Equilibrium of 2-MTHF/Water/5-HMF: Experimental Study and Thermodynamic Modeling. Daniel Matthias Roth, Moritz Haas, Alexander Echtermeyer, Sebastian Kaminski, Jörn Viell, and Andreas Jupke, *Journal of Chemical & Engineering Data*, 2023, **68**(6), 1397–1410, DOI: 10.1021/acs.jced.2c00698." We investigate the influence of sulfate salts and sulfuric acid on the equilibrium behavior of 2-methyltetrahydrofuran (2-MTHF)/H₂O/5-hydroxymethylfurfural (5-HMF). Liquid–liquid equilibrium measurements are performed at atmospheric pressure and in a temperature range of T = (293–333) K. The compositions of the aqueous and organic phases, together with the dissociation state of the sulfate species, are determined with infrared spectroscopy and Indirect Hard Modeling. The data set comprises mid infrared (MIR) spectra in original file format as acquired by the employed spectrometer with the associated composition data from sample preparation that is used for calibration of the chemometric models based on Indirect Hard Modeling for the organic and aqueous phases. Moreover, MIR spectra for the equilibrium experiments are provided at the respective temperature with initial sample composition and phase composition analyzed with the calibrated chemometric models.

Keywords

Biorefinery, Multiphase reaction system, Liquid-liquid equilibrium, ePC-SAFT, Salt, Acid, Dissociation, Infrared spectroscopy, Indirect Hard Modeling, 5-Hydroxymethylfurfural, 2-Methyltetrahydrofuran

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D.M.R.: Experimental setup; Modeling ePC-SAFT; Writing manuscript. M.H.: Experimental setup; Experimental calibration of KF and HPLC; Composition data; Modeling ePCSAFT; Review and editing of manuscript. A.E.: Construction of PCMs and IHM; Calibration of IHM; Evaluation of spectral data with IHM; Discussion and analysis of measurement results; Writing manuscript. S.K.: Scientific support, guidance, and discussion on method and results; Advice on structure and presentation of this work; Reviewing and editing the manuscript. J.V.: Scientific support, guidance, and discussion on method and results; Advice on structure and presentation of this work; Reviewing and editing the manuscript. A.J.: Design of the project, Scientific support; Advice on structure and presentation of this work; Reviewing and editing the manuscript.

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Notes:

The authors declare no competing financial interest.

Table of Content for Data Set:

The data set comprises mid infrared (MIR) spectra in OPUS-format as acquired by the employed spectrometer (Matrix MF+ FT-MIR, Bruker Optics GmbH) and operational software (OPUS ver. 7.5, Bruker Optics GmbH) with the associated composition data from sample preparation (summarized and assigned to the associated spectrum in Excel tables) that is used for calibration of the chemometric models based on Indirect Hard Modeling for the organic and aqueous phases.

Moreover, MIR spectra in OPUS-format (see above) for the equilibrium experiments are provided at the respective temperature with initial sample composition and phase composition analyzed with the calibrated chemometric models (summarized and assigned to the associated spectrum in Excel table described below).

1) MIR calibration data

a. Aqueous (lower) phase

- i. Calibration set of 2-MTHF (2-methyltetrahydrofuran) in water (MIR spectra in triplicates and sample composition assigned to the spectra in Excel table)
- ii. Calibration set of sulfuric acid in water (MIR spectra in triplicates and sample composition assigned to the spectra in Excel table)
- iii. Calibration set of sodium sulfate in water (MIR spectra in triplicates and sample composition assigned to the spectra in Excel table)
- iv. Calibration set of lithium sulfate in water (MIR spectra in triplicates and sample composition assigned to the spectra in Excel table)
- v. Calibration set of 5-HMF (5-hydroxymethylfurfural) in water (MIR spectra in triplicates and sample composition assigned to the spectra in Excel table)

b. Organic (upper) phase

- i. Calibration set of water in 2-MTHF (MIR spectra in triplicates and sample composition assigned to the spectra in Excel table)
- ii. Calibration set of 5-HMF in 2-MTHF (MIR spectra in triplicates and sample composition assigned to the spectra in Excel table)

2) MIR equilibrium data

The numerical values of the respective temperature, initial sample composition and phase composition can be taken from the Excel table: *Spectrum Assignment_Total.xlsx*

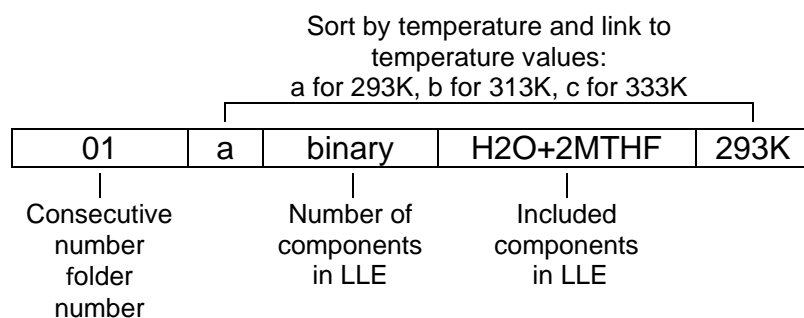
In table sheet *file_name_assignment*, the spectra are assigned to the corresponding experiments via their exact file names. In table sheet *data_table*, all the associated

numerical values are listed. The dilution factor $\xi_{\text{aq/org}}$ [1] is also included for evaluation. $\xi_{\text{aq/org}}$ is defined as:

$$\xi_{\text{aq}} = \frac{m_{\text{H}_2\text{O for dilution}}}{m_{\text{original LLE sample, aqueous phase}}} \quad (1)$$

$$\xi_{\text{org}} = \frac{m_{\text{2-MTHF for dilution}}}{m_{\text{original LLE sample, organic phase}}} \quad (2)$$

Naming scheme for folder structure:



Assignment of equilibrium data to paper tables [1]:

Consecutive number folder number	Included components in LLE	Corresponding Table in [1]
01	H ₂ O+2MTHF	Table 4
02	H ₂ O+2MTHF+Na ₂ SO ₄	Table 5
03	H ₂ O+2MTHF+Li ₂ SO ₄	Table 6
04	H ₂ O+2MTHF+H ₂ SO ₄	Table 7
05	H ₂ O+2MTHF+5HMF	Table 8
06	H ₂ O+2MTHF+5HMF+Na ₂ SO ₄	Table 9
07	H ₂ O+2MTHF+5HMF+H ₂ SO ₄	Table 10

[1] The Effect of Sulfate Electrolytes on the Liquid-Liquid Equilibrium of 2-MTHF/Water/5-HMF: Experimental Study and Thermodynamic Modeling. Daniel Matthias Roth, Moritz Haas, Alexander Echtermeyer, Sebastian Kaminski, Jörn Viell, and Andreas Jupke, *Journal of Chemical & Engineering Data*, 2023, **68**(6), 1397–1410, DOI: 10.1021/acs.jced.2c00698.