DEVELOPMENT OF THE CALIBRATION MODEL FOR REAL-TIME MEASUREMENT OF GLYCINE CONCENTRATION IN GLYCINE-WATER SYSTEM

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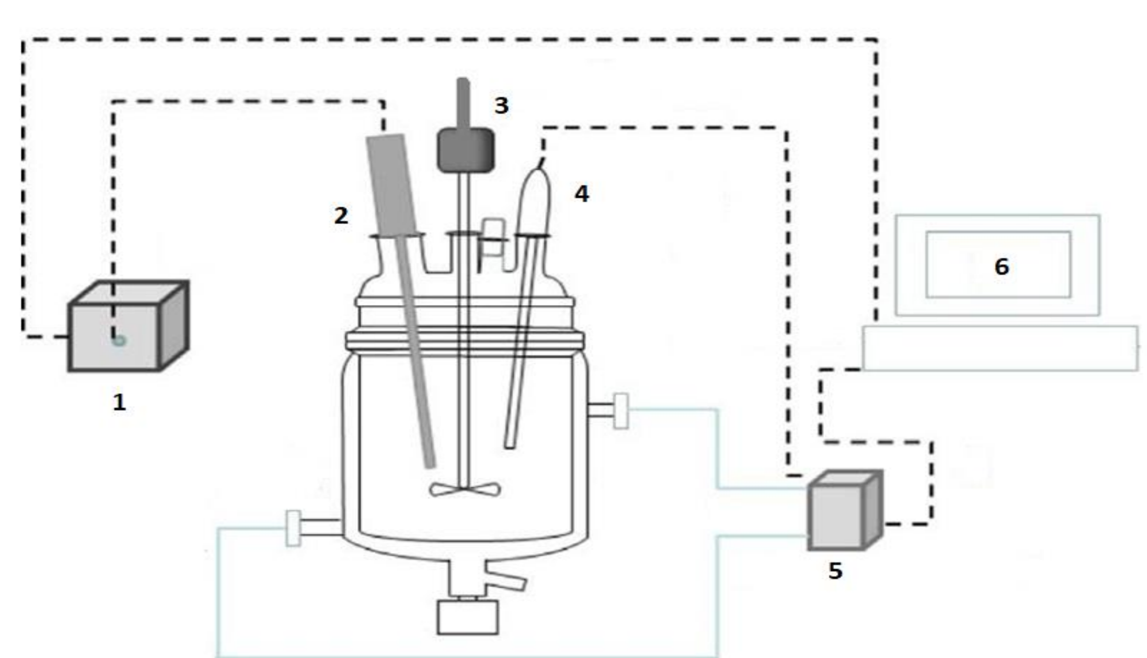
Introduction

The subject of this research is the development of a calibration model based on in-situ ATR-FTIR measurements combined with artificial neural network (ANN) for monitoring concentration of glycine in glycine-water system in a batch crystallizer. Attenuated total reflectance - Fourier transform infrared spectroscopy (ATR-FTIR) is used for on-line measurement of solute concentration. The output from the FTIR is spectral data. To obtain useful information from the spectral data, a calibration model is needed. The application of the developed model for monitoring the concentration in real-time combined with solubility curve and metastable zone width provides information about supersaturation in real-time. That allows the application of a process control method for maintaining the desired degree of supersaturation and thus, obtaining desired uniform product properties.

Experiment

Ten solutions of different concentrations were prepared. Solutions were heated to the temperature above solubility to ensure complete dissolution of glycine in water, after which they were cooled at constant cooling rate before crystals appeared. During the cooling process solution temperature and ATR-FTIR spectra were collected.



Experimental set-up: (1) FTIR; (2) ATR-FTIR probe; (3) Stirrer; (4) Temperature sensor; (5) Thermostat; (6) Computer for data aquisition

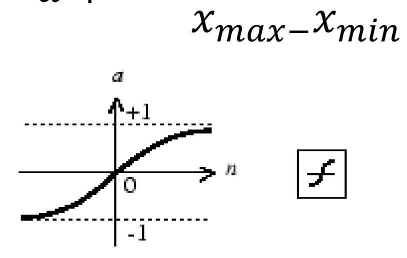
The neural network was trained using spectral data with the corresponding temperature values. Models were developed using several different datasets shown in the table. The characteristic absorbtion bands were marked from the wavenumber 1800 to 1200 cm^{-1}. Levenberg-Marquardt(LM) and Scaled conjugate gradient (SCG) backpropagation were compared. Hyperbolic tangent sigmoid transfer function was and the data was scaled using the following methods:

Z-score normalization, std:

$$x' = \frac{x - \bar{x}}{\sigma}$$

Min-max normalization, norm:

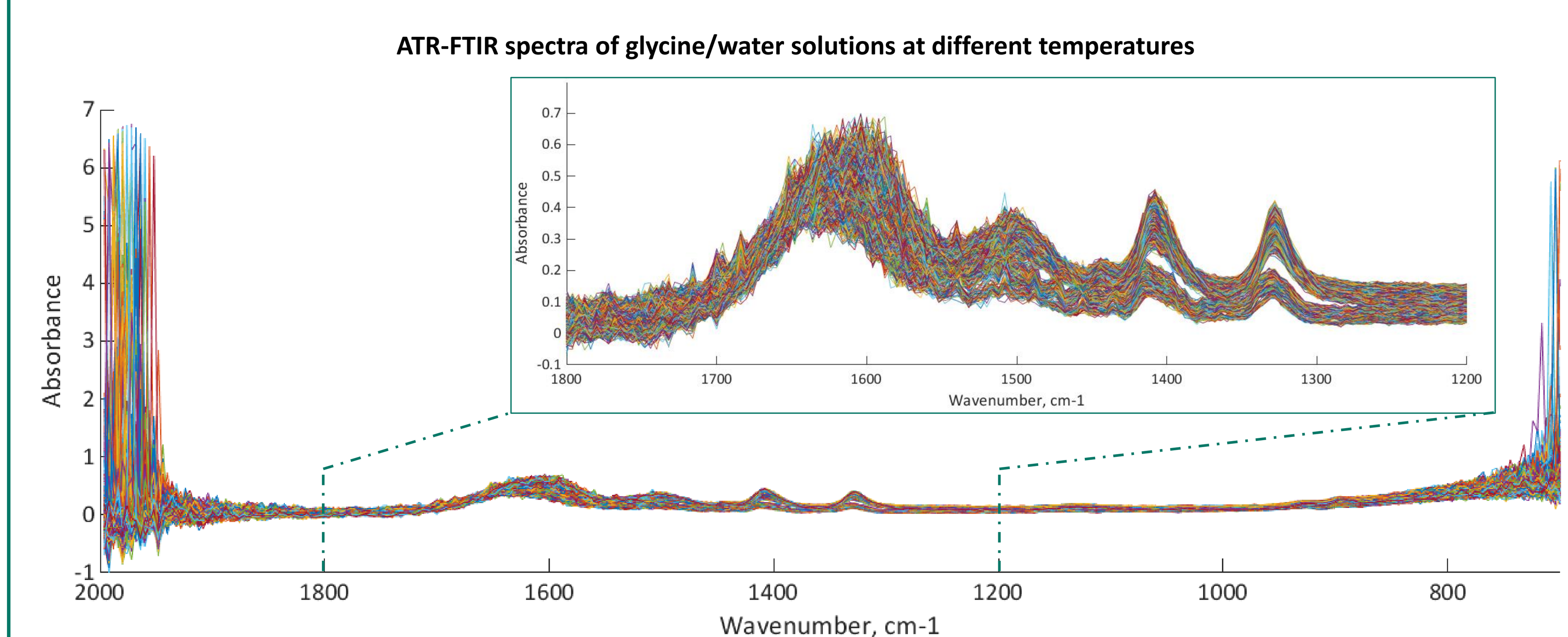
$$x' = a + \frac{(x - x_{min})(b - a)}{x_{max} - x_{min}}$$

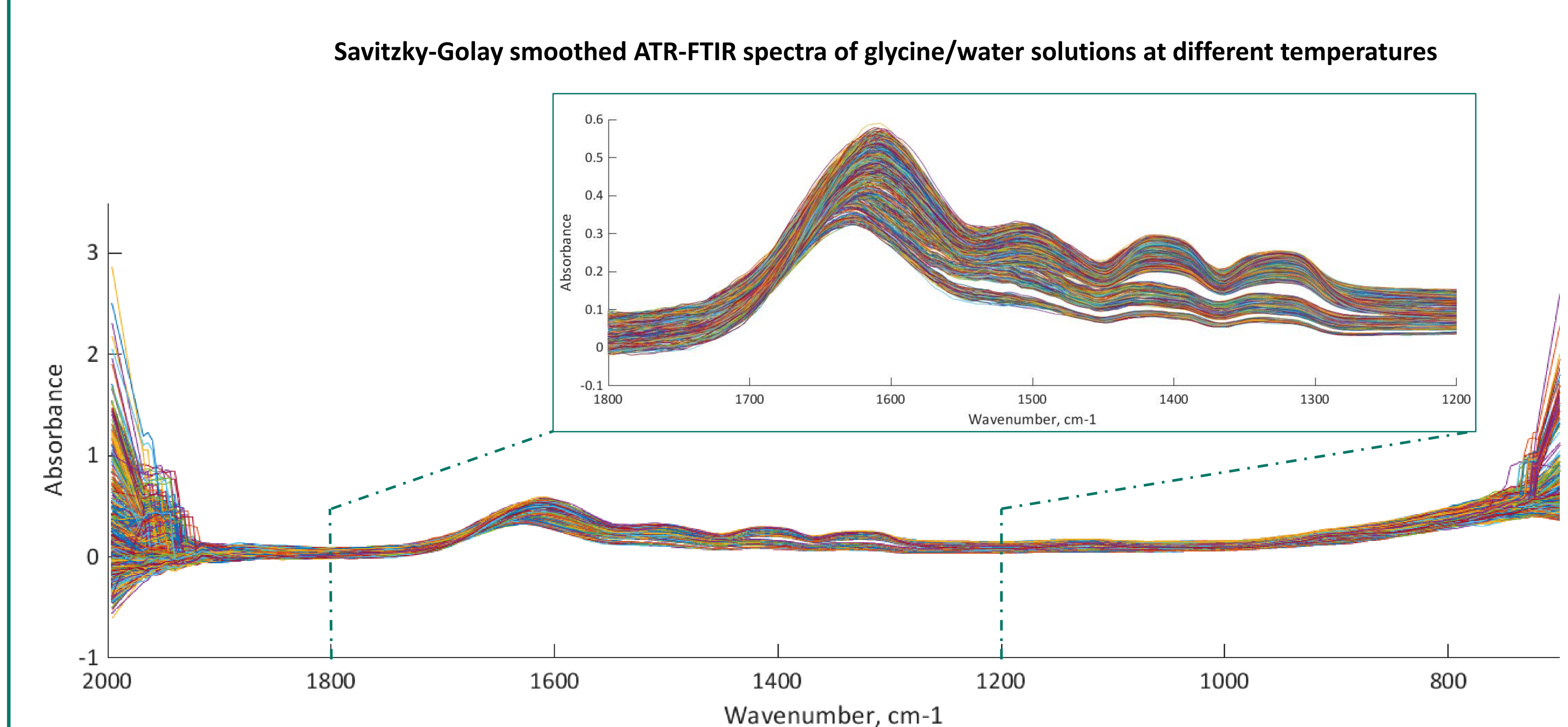


Tan-Sigmoid Transfer Function

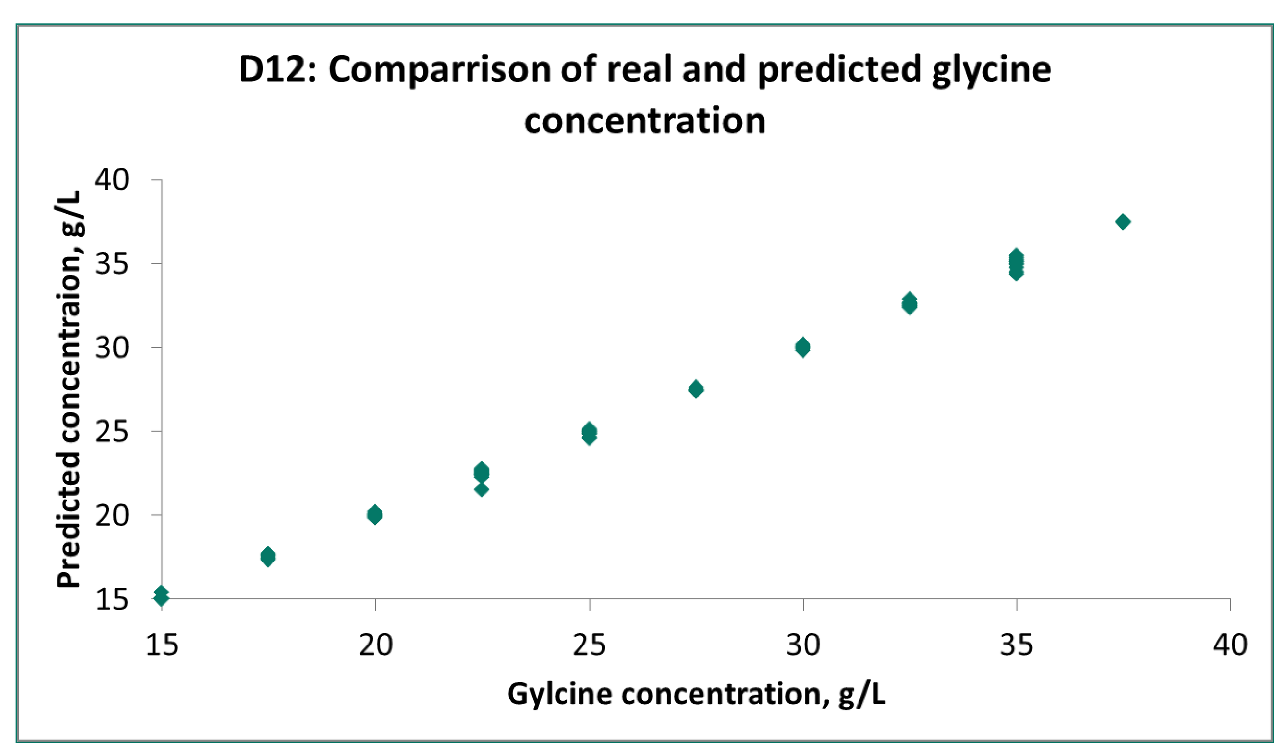
DATASET	PRE-PROCESSING	TRAINING FUNCTION
D1	/	TRAINLM
D2	/	TRAINSCG
D3	1800-1200 cm^{-1}	TRAINLM
D4	1800-1200 cm^{-1}	TRAINSCG
D5	Savitzky-Golay smooting	TRAINLM
D6	Savitzky-Golay smooting	TRAINSCG
D7	Savitzky-Golay smooting; 1800-1200 cm^{-1}	TRAINLM
D9	Savitzky-Golay smooting; 1800-1200 cm^{-1}	TRAINSCG
D9	Savitzky-Golay smooting; 1800-1200 cm^{-1} ; ystd	TRAINSCG
D10	Savitzky-Golay smooting; 1800-1200 cm^{-1}; ystd; ynorm	TRAINSCG
D11	Savitzky-Golay smooting; 1800-1200 cm^{-1}; xnorm; ystd; ynorm	TRAINSCG
D12	Savitzky-Golay smooting; 1800-1200 cm^{-1}; xstd; xnorm; ystd; ynorm	TRAINLM
D13	Savitzky-Golay smooting; 1800-1200 cm^{-1}; xstd; xnorm; ystd; ynorm	TRAINSCG

Results





DATASET	R^2	MEAN ERROR
D1	0,9963	0,0064
D2	0,9941	0,0152
D3	0,9961	0,0086
D4	0,9923	0,0170
D5	0,9966	0,0085
D6	0,9907	0,0197
D7	0,9959	0,0116
D9	0,9918	0,0190
D9	0,9932	0,0175
D10	0,9935	0,0178
D11	0,9913	0,0204
D12	0,9993	0,0047
D13	0,989	0,0228



Conclusion

Developed neural network-based calibration models for monitoring the concentration in real-time combined with solubility curve and metastable zone width provides information about supersaturation in real-time. That allows the application of a process control method for maintaining the desired degree of supersaturation and thus, obtaining desired uniform product properties.

References

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