Development of an IR-MEMS Sensor for Aqueous CO_2 in Beverages

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Abstract

One of the main parameters for the quality control of beer and other beverages is the amount of dissolved carbon dioxide. At present a so called “volume expansion method” is used where the volume above the sample is expanded and the pressure of the gas is measured. One of the major disadvantages of this method is the dependency of the calibration on the beverage.

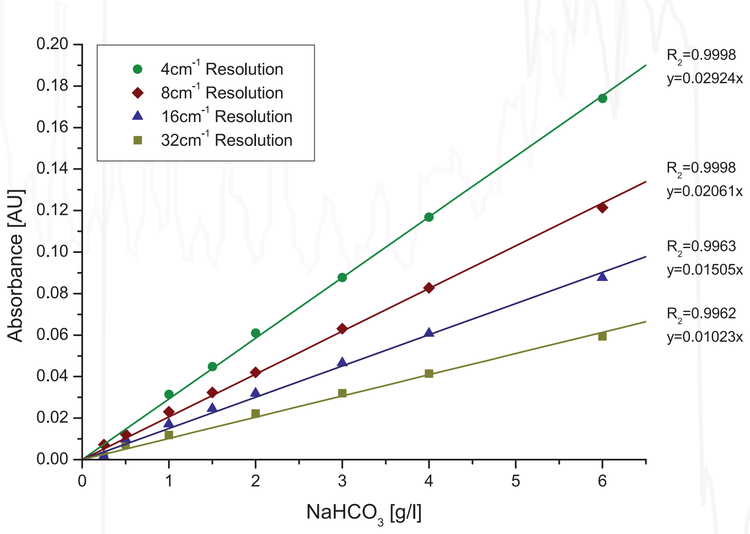
With the IR measurement a matrix independent measurement of CO_{2aq} will be achievable. Another advantage of the IR measurement is the possibility for the simultaneous measurement of sugar concentrations.

To build an affordable small analyzer for CO_{2aq} a new spectrometer, which is developed by the Carinthian Tech Research Center, is being tested.

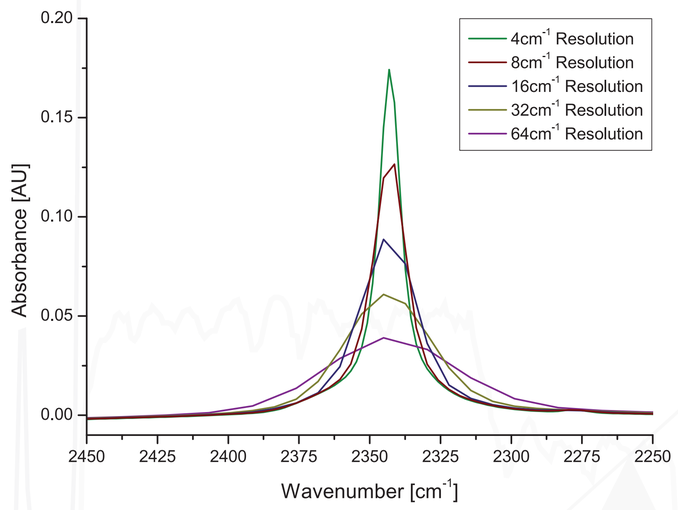
FTIR Measurement of CO_{2aq}

The absorption band of CO_{2aq} is located at 2343 wavenumbers.

Different concentrations of CO_{2aq} were prepared by mixing different $NaHCO_3$ concentrations with a citric acid puffer at pH 3 and calibration lines were recorded.



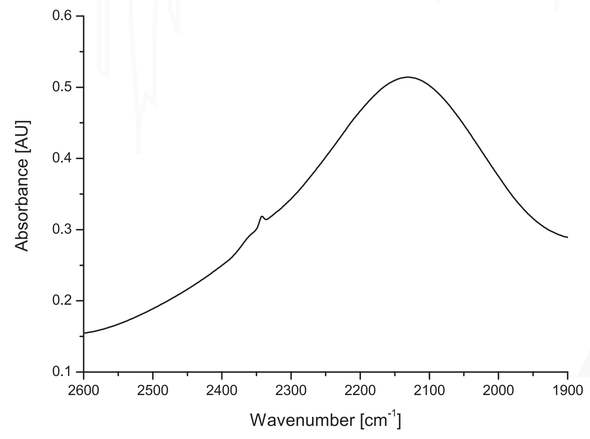
To find the best spectral resolution to measure CO_{2aq} calibration lines were recorded at different spectral resolutions which are plotted below.



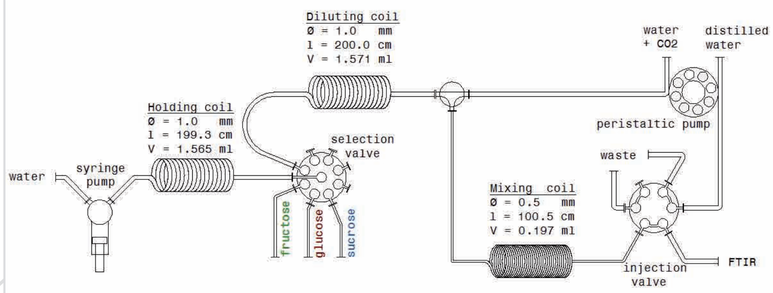
The optimal resolution resulted from the ratio of the absorbance and the peak to peak noise at the given spectral resolution.
Using a Tensor 27 spectrometer from Bruker the optimum is at a resolution of 16 cm^{-1}.

Matrix Effects

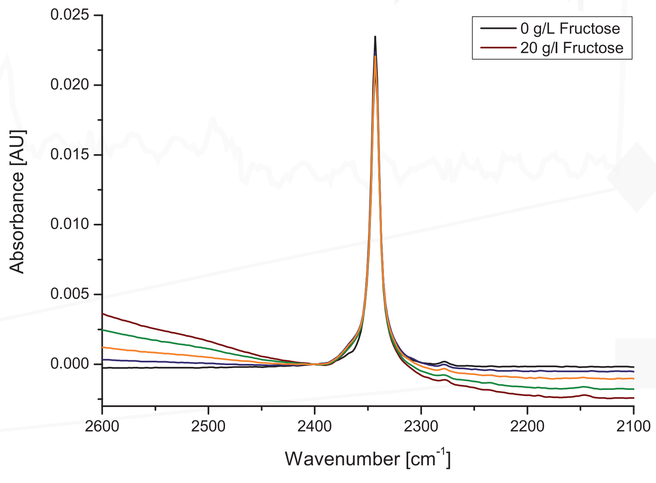
The absorbance band of CO_{2aq} is overlapping with the combination band of water and any changes in the structure of water result in a change of the background spectrum for the CO_2 measurement.



Especially the three sugars glucose, fructose and saccharose are of interest because they are ingredients in many carbonized softdrinks.
To study the influence of different sugar concentrations it was necessary to measure many different concentrations in a short time because the CO_2 in the atmosphere is also interfering with the measurement of aqueous CO_2. To accomplish this fact a FIA system was used. The CO_{2aq} solution was prepared by saturating water with CO_2.



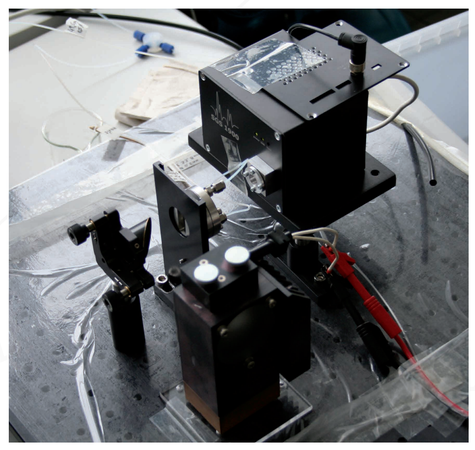
The effect of fructose on the water spectrum can clearly be seen in the plotted spectra. Increasing the fructose concentration from zero to the FIA peak maximum results in a tilting baseline.



MEMS Spectrometer

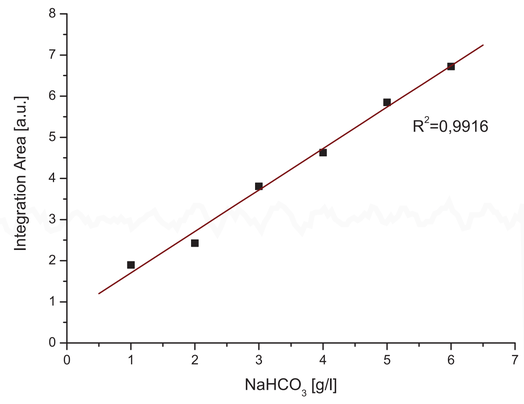
The used spectrometer is based on the Czerny-Turner Monochromator principle and was first developed for the near infrared region by the Carinthian Tech Research Center[1].
The spectrometer uses a micro-electro-mechanical mirror device with a reflection grating surface to disperse the light and the individual wavelengths are scanned over a detector.
To adapt the spectrometer for the middle infrared the grating and the detector had to be changed.
The key advantages of a MEMS based spectrometer are the small size of 100 × 80 × 75 mm^3 and the high scanning speed which enables the coaddition of many scans to compensate the reduced inherent sensitivity.

For the first measurements with this spectrometer a globar from a Vector 22 spectrometer from Bruker was used. The light was collimated with an off axis mirror and passed a flow cell with a 25 micron spacer. Behind the flow cell the light was focused into the spectrometer by a ZnSe lens.

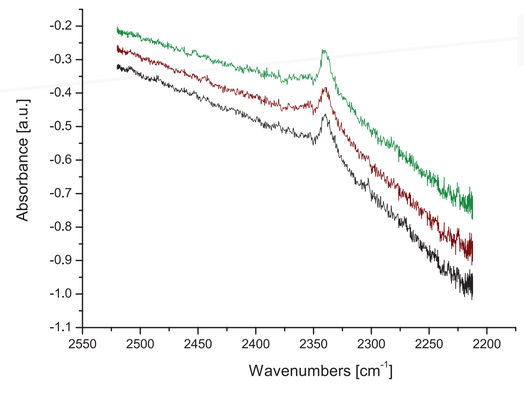


MEMS Measurement of CO_{2aq}

The sample preparation for the calibration of the MEMS spectrometer was done with the same solutions as the calibration of the FTIR spectrometer before. To obtain one spectrum 3000 scans were averaged which took approximatly 40 seconds.



To demonstrate the capability of the MEMS spectrometer a sample of mineral water was measured. As you can see below the absorbance of CO_{2aq} can clearly be seen altough the sampling technique was far from ideal.
The spectra also show the most critical problem of the first prototyp. Due to the high power of the globar the spectrometer heats up and the detector signal is drifting.



[1] M. Kraft, A. Kenda, A. Frank, W. Scherf, A. Heberer, T. Sandner, H. Schenk and F. Zimmer
Single-detector micro-electro-mechanical scanning grating spectrometer.
Anal Bioanal Chem 386:1259–1266, 2006