New Experimental Approaches to Measuring Plant-Extracted Waters

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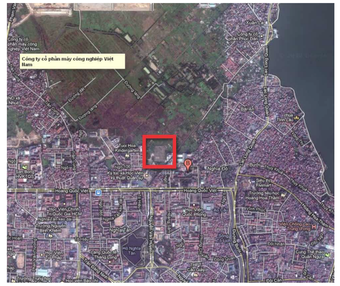
Introduction

With the increasing demands on fresh water supplies globally and especially in the developing world, proper management of irrigation water for crop productivity is critical given that agriculture accounts for approximately 80 % of global water use. Measurements of the isotopic composition of the water vapor above the crop canopy are used to identify sources of evapotranspiration (ET) ratio when coupled with ET flux measurements can be used to determine the proportions of evaporation and transpiration fluxes of the crop. Until recently isotopic measurement of water vapor in the atmosphere could only be achieved by cryogenic trapping of the water vapor and subsequent laboratory analysis, usually by isotope ratio mass spectrometry. The recent development of a field deployable water isotope analyzer using CRDS technology was used to directly measure the vapor isotopes in real time and at multiple heights above the crop canopy. This technique was coupled with additional samples gathered using conventional techniques and a new xylem water extracting apparatus developed by the IAEA.



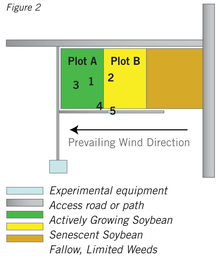
Northwestern portion of Hanoi with experimental field area boxed in red. The bubble indicates the location of the Institute for Ecological and Biological Resources

Field Sampling

Measurements were carried out in an experimental field (Figure 1) at the outskirts of Hanoi Vietnam operated by the Institute for Ecological and Biological Resources in plots of actively growing and senescent soybean. The plot sizes were approximately 30 meters along the wind direction, and both plots received irrigation from the same source. Conditions were partly cloudy with light winds of variable strength but constant direction and temperatures were 10 to 15 °C.

Vapor was measured in real time for both plots and at multiple heights in the actively growing plot. Vapor was also trapped cryogenically in the actively growing plot. Entire soybeans were uprooted from various locations in the actively growing plot (Plot A). A sample of the irrigation water was taken from the ditch adjacent to both plots. Figure 2 shows the relative locations of the different sampling points. Figure 3a is a photograph of the experimental field showing the location of some of the sampling points as well as the CRDS analyzer. Figure 3b shows a photograph of the vapor sampling posts.

Figure 2



1 vapor by CRDS, two heights
2 vapor by CRDS, one height
3 vapor by cryotrap at canopy
4 irrigation water samping
5 CRDS analyzer location

Figure 3a

Figure 3a. The CRDS analyzer shown in the foreground. Point labeled in red show the location of vapor intakes with 1 corresponding to Plot A and 2 immediately downwind of Plot B.

Figure 3b

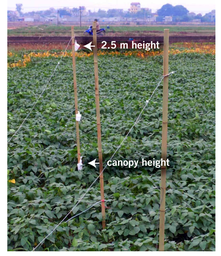


Figure 3b. Shows the bamboo poles used to support the vapor sampling tubes at different heights

Methods

Vapor sampling points were supported by bamboo pole driven into the soil. Bev-A-Line IV ¼" tubing was mounted at the specified heights and run back to the CRDS analyzer and/or cryogenic trap. Mechanical vacuum pumps were used to draw vapor, in the case of the CRDS a manifold was used which allowed the analyzer to switch between vapor sources with minimal delay.

The cryogenically trapped vapor turned ice was scrapped from the cold finger and collected in a sample vial for liquid analysis by CRDS. The irrigation water was collected directly into a serum vial, capped, allowed to settle, and then transferred to 2 mL sample vial for liquid analysis by CRDS.

Direct analysis of vapor was performed using a Picarro L1115-*i* CRDS based water isotope analyzer. The automatic valve sequencer was used to alternate vapor sources between the different plots and heights on a 5 minute basis. The analyzer was calibrated against VSMOW2, GISP and SLAP2.

Whole (including roots) soybean plants were collected around point 3 in Figure 2 and were placed roots down in a serum vial. The seal point around the stem allowed the application of a vacuum using a hand pump. The pressure differential between atmosphere and the vacuum present in the serum vial drives water back out of the roots for collection in the serum vial. This water (approximately 100-200 microliters from several plants) was allowed to settle and then transferred to 2 mL sample vial for liquid analysis by CRDS. A larger scale example of the technique using a branch and vacuum flask is shown in Figure 4.

Results of Two Plot Measurement

Figure 4

The comparison measurement at canopy height between two plots was taken between 12:55 to 14:15 Dec 8 2010 Hanoi time under sunny conditions with a light breeze.

Plot B showed an average vapor concentration of approximately 12519 ppm during an hour long period with light winds. During the same time period Plot A showed an average vapor concentration of approximately 13582 ppm. A comparison of the temporal variation in vapor concentration is shown in Figure 5. A comparison of the temporal variation in vapor isotope ratio is shown in Figure 6a and 6b for the two isotopes. A summary of the data is presented in Table 1 including the results from the cryogenically trapped vapor collected. The cryogenically trapped vapor collected from two locations downwind of the CRDS measurements for a 15 minutes at 12:00 Dec 8 2010 Hanoi time.

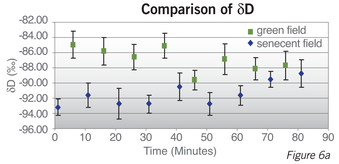


Figure 6a

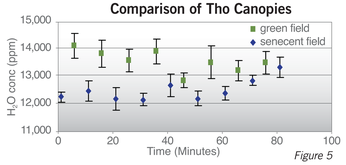


Figure 5

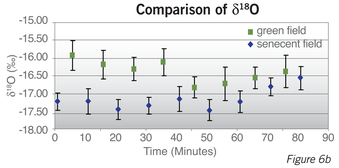


Figure 6b

Field	Conc. (ppm)	$\delta^{18}O$	δD
Plot B Vapor (senecsent)	12519	-17.16 ±0.29 ‰	-91.50 ±1.49 ‰
Plot A Vapor (senecsent)	13582	-16.38 ±0.38 ‰	-86.86 ±1.69 ‰
Plot A Vapor Trap		-17.12 ±0.21 ‰	-83.01 ±0.51 ‰

Table 1

Results of Two Height Measurement

The alternating measurements at canopy level and 2.5 meter height were taken in plot A between 15:00 to 16:50 on 08 Dec 2010 Hanoi time under sunny conditions with a light breeze. The average of the vapor concentration and isotope ratio for the 5 minute cycle taken at each height was calculated.

Data is presented in Keeling plot format plotting the isotopic value against the inverse vapor concentration. Figure 7a and 7b are for deuterium and oxygen respectively. A linear least square fit was applied to the data to determine the Keeling plot intercept. A summary of the data is presented in Table 2 including the results from the analysis of liquid samples obtained from soybean xylem water using the hand vacuum pump method and irrigation water collected at the same approximate time.

Discussion of Two Plot Measurement

Vapor measurements by CRDS showed clear differentiation in concentration and isotope ratio for the actively growing and senescent fields at the canopy level. Furthermore there was good agreement with overall isotope values when compared liquid sample obtained by cryogenic trapping. The differences in isotope values between the two plots is outside of the standard deviation of the measurement. The full time sequence with the summary data in Figure 6b is shown in Figure 8, here we see the $\delta^{18}O$ value alternating at the five minute intervals associated with switching vapor sources between the two plots. Clearly the differences in measured values between the different vapor sources are a combination of true differences and, in the case of cyrogenically trapped samples, differences in sampling mechanisms and timing.

Discussion of Two Height Measurement

The experimental sampling density, particularly the limited fetch and non-ideal location are clearly visible from Figures 1 and 2. Ideally a larger fetch, of at least 250 meters, and sampling from additional heights for a longer duration would have been used to generate the data for the Keeling plot. As this was a demonstration experiment the available location, time and equipment were limited. However it is still interesting to note the close agreement between the Keeling plot intercepts and the values obtained from the soybean water.

The limited driving force (< 1 atm) associated with the vacuum technique is unlikely to rupture cell walls and is thus most likely a direct sampling of the xylem water. Assuming there is no fractionation is associated with water uptake by the roots the water obtained in this manner should reflect the soil water composition. The vacuum technique is easily deployed in the field with a minimum amount of equipment, indeed no electricity is required! A comparison between this method and cryogenic distillation have not yet been undertaken. Differences are possible since this method does not quantitative remove all water from the sample as is the case for cryogenic distillation.

Experimentally care must be taken to filter the obtained water of sediments since traces of the soil adhering to the roots is inevitably entrained in the sample and causes mechanical problems in syringes used for the liquid handling portion of the analysis.

The irrigation water isotopic values were positive relative to the soybean water and Keeling intercepts. This is fully consistent with fractionation effects which are to be expected from an open ditch with ongoing evaporation.

Conclusions/Acknowledgement

In this half day experiment in situ analysis of vapor concentration and isotopic composition was performed, along with analysis of water from liquid and plant sources under challenging field conditions. Clear differences in vapor concentration and isotopic composition between different experimental plots was observed. Keeling plot data generated from in situ vapor measurements agreed well with water samples obtained from the irrigation source and plant material. Unfortunately the experimental conditions, particularly insufficient fetch, limited the scope of interpretation of the experimental data.

A new hand vacuum pump method, developed by the IAEA, was demonstrated to be effect and robust for easily obtaining samples in the field. The CRDS water isotope analyzer, developed by Picarro, was demonstrated to be sensitive and robust for performing both vapor and liquid measurements in the field.

This study was performed as part of the 3rd Research Coordination Meeting: Managing Irrigation Water for Crop Productivity, organized by FAO/IAEA Joint Division, held on Dec 6-10 2010, and hosted by the Institute for Water and Environment, Hanoi Vietnam. The authors express their gratitude to the Institute of Water and Environment and the Institute for Ecological and Biological Resources as well as Dr. Nhanh for their hospitality and scientific cooperation.

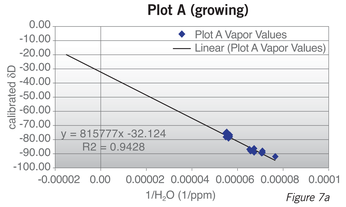


Figure 7a

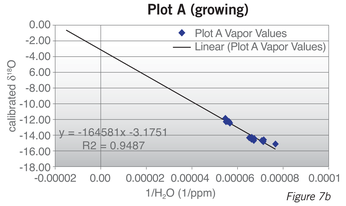


Figure 7b

Sample	$\delta^{18}O$	δD
Soybean Water	-4.27 ‰	-28.59 ‰
Irrigation Water	-3.88 ‰	-25.53 ‰
Keeling Plot Intercepts	-3.18 ‰	-32.12 ‰

Table 2

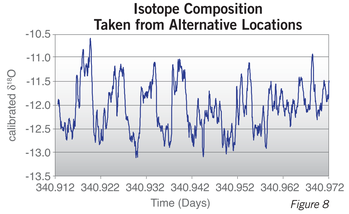


Figure 8