# Design and testing of a device to separate tightly-bound and mobile water pools in soil

 Abstract:

Water movement, from forest soil to streams, has traditionally been modeled by translatory flow.   A hypothesis was presented by Brooks et al. (2010) suggesting that, in addition to translatory flow, there exists an immobile pool of water trapped in small pores with low matric potential. Comparisons of stable water-isotope ratios between loosely bound water and bulk soil water can provide additional evidence for this hypothesis.  In this work the beginnings of a method for separating loosely bound water from a soil core has been developed.  It was found that, by centrifuging a soil core, sufficient water for mass spectrometry can be extracted at a given matric potential and held within a porous ceramic rod.  This water can then be extracted from the ceramic using cryogenic vacuum distillation.

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## Introduction

Understanding  the complex mechanisms governing the movement of water from precipitation, through forest soil, and eventually to streams has been a goal of catchment hydrology for decades.  One of the early theories proposed by Hewlett and Hibbard (1967) concerning this flow of water is the mechanism of translatory flow, wherein water held in the soil before a storm event is displaced by newly precipitated water .  This development of translatory flow as a mechanism to explain the movement of water from the subsurface to the stream was a major step forward in hydrology, and the concept is now in common usage.  However, recent research by Brooks et. al. (2010) has put to question this model, and raised the possibility of a more complicated reality of how water moves through soil.

According to Hewlett and Hibbard (1967) translatory flow is flow produced by displacement. Each new rain infiltrating the soil displaces water from previous rain, and in this manner water at increasing soil depths will represent older and older precipitation.  At any depth it would be expected that water will be well mixed[2].  Additionally, the water transpired by plants and the water entering the stream would be expected to be similar[2].

Contrary to assumptions in this model,  water stable isotope data collected by Brooks et. al. (2010) from streams, trees, and soils in the H.J. Andrews Experimental Forest imply that soil water is not well mixed.  Rather, the data suggests the presence of a pool of water held tightly in small soil pores that does not participate in translatory flow.  While this research study represents

the beginning of a refinement of the translatory flow model, further research and testing is required to support the hypothesis.

**Two Water Worlds**

Using dry season oxygen and hydrogen stable isotope data ($\delta^{18}$O and $\delta^2$H, respectively) from water collected from a 10 ha. watershed in the H.J. Andrews Experimental Forest, Brooks et. al.(2010) have put forth the hypothesis that water exists in either a mobile or an immobile water world within forest soil. The mobile water world consists of water that, through translatory flow, eventually enters the stream. The immobile water world consists of water that, during the initial autumn rains is trapped in small pores at low matric potentials, and remains trapped therein until eventually being released through transpiration in the dry summer months. The following observations led to this conclusion.

Stable isotope data was collected for precipitation, streamflow, soil water, and tree water samples. The initial expectation was to observe soil and tree water samples exhibiting increasingly similar isotopic compositions to streamflow samples with increasing proximity to the stream. Instead, at all sampling locations the isotope ratios of soil and tree water were found to be significantly different from the isotope ratios of the streamflow and precipitation water. Whereas the streamflow and precipitation water had similar isotope ratios and plotted along the local meteoric water line (LMWL), soil and tree water samples fell below the LMWL. This type of shift is indicative of isotopic fractionation from evaporation. This fractionation is due to differences in vapor pressures between $H_2^{18}O$ and $^2HHO$ leading to disproportional enrichment of $^2$H and $^{18}$O during evaporation[3]. In addition to this, a greater variation in water isotope

values was observed with soil depth than spatially with sample location. Isotope ratios were highest for shallow soil and decreased with depth. Values at the deepest depths were isotopically depleted compared to streamwater and precipitation, and evaporation cannot explain how these depleted isotope values occurred at depth. Finally, the combined values of soil water isotope ratios were reflected in the tree water isotope ratios.

The patterns of this data can be explained by the existence of an immobile water world in H.J. Andrews soil. The ability of a soil to transmit water, or the hydraulic conductivity of a soil, is determined in large part by the pore size distribution of the soil[4]. Within soil, pores exist of varying size, and due to capillary forces these pores will hold onto water with varying tensions or matric potentials. For larger pores both matric potential and hydraulic conductivity increase which provides an easier route for water flow. The smaller pores exhibit decreased matric potentials. These small pores are the first to fill and the last to drain, and flow through these pores may be constrained to such a degree that mixing with water in larger pores is limited[2]. If the matric potentials of these pores is lower than what gravity can drain, this water may remain distinct and separate in the soil until removed via transpiration in the summer.

This region experiences a Mediterranean climate, with wet winters and dry summers. As a result, transpiration and stormflow occur at different times of the year. During the first autumn rains water begins to wet soil that dried out during the summer. The decrease in isotope values with increasing soil depth reflects the process of isotope depletion from rainout events. Rayleigh distillation of heavy isotopes causes subsequent rain through a rain event to be increasingly depleted in water isotopes. Water that first enters the smallest pores with the lowest matric

potentials remains here until  the subsequent summer when transpiration by plants pulls this water from the ground.  Therefore, water trapped in more shallow soil would represent earlier, more enriched precipitation, whereas deeper soil would contain water from later, more depleted, precipitation in those first soil wetting storms of autumn.  In order for the isotopic signatures of this water sampled in the summer to reflect rainout events from the previous autumn, matric-bound water must not be displaced by translatory flow occurring throughout the year.  Furthermore, the similar isotope ratios found in soil and tree water, and the differences between this water and streamflow, indicate that water transpired by trees is distinct from the water contributing to streamflow.  Together, these patterns suggests that there is not complete mixing of soil water.

**Testing the Concept**

In the same research study Brooks et. al. (2010) collected soil water using a low-tension lysimeter.  This represents water that is loosely bound and mobile.  Isotope ratios of this water were then compared to bulk soil water extracted using cryogenic vacuum distillation.  The bulk soil water represents a mixture of both tightly-held immobile water, and loosely-held mobile water.  Bulk soil water was consistently shown to be less enriched in comparison to lysimeter water.  This is again explained by incomplete mixing of loosely bound water and matric-bound water.

However, while this supports the hypothesis put forth concerning an immobile water pool, lysimeter water and bulk soil water are collected from different locations within the soil.  A closer investigation would seek to directly separate loosely bound water from soil, and then

compare the isotope ratios of this mobile water to those of the remaining tightly-held  soil water. This process would provide illuminating evidence to support or refute the presence of separate and distinct pools of water within forest soil.

 By separating water pools held at different soil matric potentials throughout a rainy season, and comparing the isotopic compositions of these pools to that of the season's precipitation, the degree of water mixing within the soil can be analyzed.  The results would be expected to fall between two extremes.  It may be observed that the isotopic ratios of each pool are the same, which would represent complete mixing within the soil.  On the other hand, the pools may display distinct ratios, with loosely bound water corresponding to the most recent precipitation and the bulk soil water corresponding to a mixture of this water with water from earlier precipitation held tightly in small pores.  It may also be possible that while mixing is occurring, the flow rate through these smallest pores is slow enough that the results lie in-between these two extremes.  Another possibility provided with the development of this procedure would be to compare the isotope ratios of several water pools, each being extracted at different matric potentials.  This could provide evidence for a possible matric potential cutoff point for forest soil at which water begins to show signs of incomplete mixing.

In this project, the beginnings of a procedure for separating loosely bound and tightly bound soil water was developed.  A device was designed and assembled to allow separation of soil water via centrifugation.  Preliminary feasibility tests were then performed using the device.  Once the device is proved reliable with further testing, the end goal will be to provide additional data concerning the presence of an immobile water pool in forest soil.

# Design and Approach

In the approach taken in this project, the separation of loosely bound water from a soil core is achieved via centrifugation. The use of centrifuge techniques has been well documented in the measurement of hydraulic property measurements such as water retention curves or hydraulic conductivity measurements[5-7]. In this application, the end goal is simply to collect supernatant from centrifugation to allow for isotope ratio measurements. Through the application of a centrifugal force specific matric potentials can be produced in a soil column. Here centrifugal force is used to separate mobile water from a soil column at a given matric potential and trap this water in the matrix of a porous ceramic rod. A general diagram is shown in Figure 1.
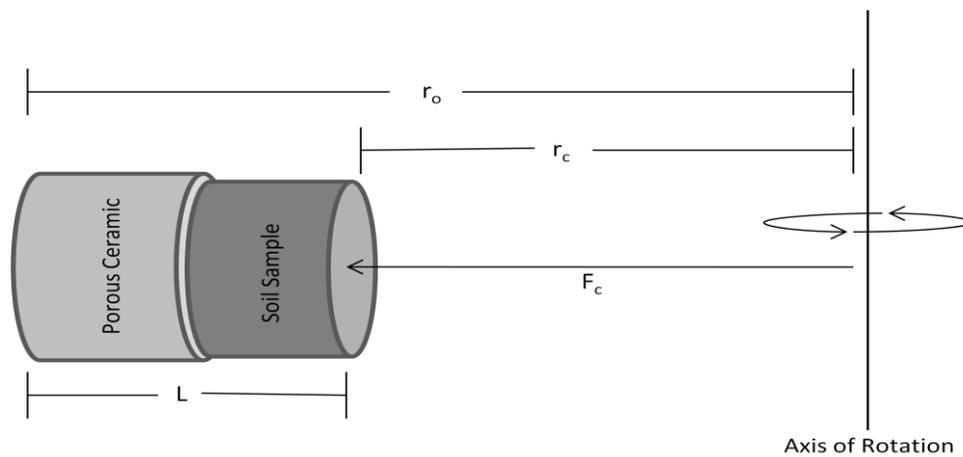


Figure 1: Simplified diagram of soil water separation scheme.

The centrifugal force, $F_c$, is dependent on the mass of the object $m$ (in this case, the column), the radial distance from the axis of rotation $r_c$, and the angular velocity at which the rotor spins $\omega$, according to Equation 1.

$$F_c = mr_c\omega^2 \qquad (1)$$

For water in a porous medium this force is often described per unit volume, $F_{cv}$, as shown in Equation 2. Here $\rho_w$ is the density of water.

$$F_{cv} = \rho_w r_c \omega^2 \tag{2}$$

Equation 2 is used to derive a form of Darcy's Law applicable for unsaturated flow in a centrifugal field at steady state. This is shown below in Equation 3 where q is water flux, K is hydraulic conductivity, and ψ is matric potential [6].

$$q/K = \rho_w r_c \omega^2 - \frac{d\psi}{dr} \tag{3}$$

For hydrostatic equilibrium, where water flux is equal to zero, Equation 3 can be integrated to describe the matric potential profile in the column. This involves the assumption that water is allowed to exit freely at the base of the column, $r_o$, and therefore ψ = 0 at this outflow point. This pressure profile is given in Equation 4.

$$\psi(r) = \frac{\rho_w \omega^2}{2} (r_c{}^2 - r_o{}^2) \tag{4}$$

At hydrostatic equilibrium the matric potential gradient is equal and opposite to the centrifugal driving force, and the matric potential distribution is non-linear along the soil sample length [6,7].

These concepts allow for water to be pulled out of a soil column at calculated matric potentials. However, for the purpose of identifying differences between isotope ratios of matric-bound and loose pools within soil, the water forced out during centrifugation needs to be collected for mass spectrometry. In addition to this, evaporation from both pools of water needs to kept to a minimum in order to prevent fractionation. To satisfy these criteria the use of a porous ceramic rod placed in contact with and below the soil column was used for water collection. This method has several advantages. First, the porous ceramic used is hydrophilic, which when placed in contact with the soil facilitates the transfer of water. Second, due to the effect of column height on the matric potentials produced, the addition of a ceramic rod to the column allows for larger

pressure profiles to be produced, and allows for variation in column height through the use of different sized rods.  Finally, water is collected in the matrix of the ceramic rod, which can be properly sealed to prevent evaporation.  This water can then be re-extracted from the rod using cryogenic vacuum distillation to provide a sample for mass spectrometry.

Using this approach allows for water to be pulled out of soil at varying matric potentials corresponding to column height and rotational speed.  The isotopic signatures of water extracted from varying matric potentials can then be analyzed.  Futhermore, the remaining water present in the bulk soil is also available for extraction, which allows for direct comparison of isotope ratios for mobile and immobile water pools.

**Centrifuge**

The sampling device was designed for use in an Avanti J-HC centrifuge with a JS-4.2A rotor. This rotor has a  radius of 25.4 cm and holds 6 swinging bucket holders with 1-L capacity.  The range of speeds for this rotor is1000-4200 rpm, which allows for a maximum RCF of 5020xg. Using this rotor six samples could run simultaneously.  For this study one sample was spun per run, with the remaining 5 buckets being properly balanced by weight.  Figure 2 shows the bucket assembly with the sampling device loaded.

**Figure 2: Avanti J-HC centrifuge with JS-4.2A rotor. The sampling device is loaded on bottom left bucket with the remaining buckets containing counterweights.**

## Ceramic Rods

The 630 series ceramic rods manufactured by Soilmoisture Equipment Corp. were purchased for use in this study. The diameter of the rods is 1 inch and the height is 2 inches. According to the manufacturer these rods are made from high-fired Alumina with an air entry value of 1 bar. The approximate porosity is 45% by volume with a maximum pore size of 1.7μm. The saturated hydraulic conductivity of the material is $8.6 \times 10^{-6}$ cm/sec. The average dry weight is 49.63g. Rods were oven dried prior to each use to remove any residual moisture.

## Device Assembly

The device was assembled to fit inside of the Multi-Disc assembly adapter for the JS-4.2A rotor. Soil samples were collected using 3/4" PVC pipe with a height of 5.92 cm. The soil core is situated on top of the ceramic rod making the total column height 11.1 cm, although soil compression during centrifugation will decrease the working height slightly over the course of a run. This configuration was held in place through the use of additional PVC fittings. The result is shown in Figure 3 below.

**Figure 3: Stepwise assembly for centrifugation. Step 1.) the ceramic rod is placed in a PVC holder.  Step 2.) the soil core is placed atop the ceramic and held in place with a PVC coupling.  Step 3.)  The entire setup is held in place with additional PVC fittings.  Step 4.)  The assembly is placed within the Multi-Disc rotor bucket holder adapter.**

This setup allows for the soil/ceramic column to be held in place for the duration of a centrifuge run.  Assembly and disassembly are quick and easy as the PVC parts are easily removable and interchangeable.

## Preliminary Testing of Device

This procedure and device was designed with an ultimate goal of providing data to further examine the hypothesis of two water worlds being present in forest soil.  However, before data can be collected in this manner, the reliability of this procedure must first be demonstrated.

**Cryogenic Extraction**

In order for this approach to be feasible, the water collected via centrifugation in the porous ceramic rods must be re-extracted using cryogenic vacuum distillation.  During this extraction the rods must not cause fractionation of water isotopes.  To demonstrate that water collected in porous ceramic rods could be re-extracted without fractionation, a cryogenic vacuum distillation run was performed using water of known isotopic composition.

Materials and Methods:

Three 630 series ceramic rods were oven dried to remove any moisture present and weighed to determine dry weights.  Two of these ceramic rods were then completely submerged in reverse osmosis water with a known isotopic signature within sealed glass vials and left to soak overnight.  The third rod was left dry. After saturating with RO water the ceramic rods were then weighed again and immediately thereafter underwent cryogenic vacuum distillation[8].  Extracted water was collected and analyzed for $\delta^2H$ and $\delta^{18}O$.  The RO water used for soaking, in addition to a fresh sample of the known RO water were also analyzed for $\delta^2H$ and $\delta^{18}O$.  The $\delta^2H$ and $\delta^{18}O$ values are expressed in relation to Vienna Standard Mean Ocean Water (VSMOW) according to Equation 5. The rods were then weighed again at the completion of the run.

$$\delta^2H \text{ or } \delta^{18}O = (\frac{R_{sample}}{R_{standard}} - 1) * 1000 \qquad (5)$$

Results and Discussion:

As shown in Table 1, the results indicate that no fractionation occurred during extraction from the saturated rods. All differences fall within the sampling range of error. However, it is important to note that the rods were fully saturated. It is unlikely that this volume of water will be collected a centrifugation run. If fractionation is occurring it is possible that the effect would be detectable with smaller volumes of water in the rod.

Table 1: Water isotope results for cryogenic vacuum distillation. Extracted water showed no signs of fractionation during cryogenic vacuum distillation. Rod 1 data is not included as no water was extracted from the dry rod.

| Water Sample | $\delta^2H$ (‰) | std. dev (‰) | $\delta^{18}O$ (‰) | std. dev (‰) |
|---|---|---|---|---|
| Rod 2 RO water | -70.77 | 0.12 | -10.24 | 0.10 |
| Rod 2 extracted | -70.32 | 0.19 | -10.07 | 0.16 |
| Rod 3 RO water | -71.19 | 0.05 | -10.26 | 0.08 |
| Rod 3 extracted | -70.66 | 0.04 | -10.18 | 0.06 |
| Fresh RO water | -71.20 | 0.12 | -10.27 | 0.12 |

Table 2: Mass data for ceramic rods. Rods 2 and 3 were saturated with RO water, whereas Rod 1 was left dry for the duration of the trial.

| Ceramic Rod | Dry Weight (g) | Saturated Weight (g) | Post-extraction Weight (g) |
|---|---|---|---|
| 1 | 50.63 | NA | 50.64 |
| 2 | 52.33 | 61.45 | 52.33 |
| 3 | 50.61 | 59.62 | 50.62 |

The data in Table 2 also shows that complete extraction of water held in the ceramic rod is possible. Dry weights from oven drying and cryogenic vacuum distillation show no evidence of water being withheld in the rod post-extraction. This data suggests that, if sufficient water for mass spectrometry can be accumulated in the ceramic rod during centrifugation, re-extraction of this water is possible and reliable for analysis.

**Water Pool Separation via Centrifugation**

The following experiment was performed with a primary goal of demonstrating the feasibility of extracting loosely bound water via centrifugation. In order to analyze any extracted water, sufficient volumes must be collected for mass spectrometry. A volume of 1 mL would be sufficient to meet this end. In addition to this, a general timeframe for a sample to reach steady-state at given rotational speeds was desired. This is important for the assumption of hydrostatic equilibrium in calculating the matric potential to be valid. Finally, the ability of the process to extract water with minimal evaporation was investigated.

Materials and Methods:

Three centrifuge trials were performed for three different rotational speeds. Soil samples were collected in the summer of 2010 adjacent to Oak Creek in Corvallis, OR. The soil was pre-wet the night prior to sampling with Oak Creek water. Samples were then taken the following morning using the PVC pipe cores. The soil samples were sealed with parafilm to reduce moisture evaporation. Ceramic rods were oven dried prior to each use. For the second and third runs, the bottom end and sides of the ceramic rods were wrapped in parafilm to reduce collected moisture evaporation during the run. The first run was performed with no parafilm barrier for the ceramic rod. Once the soil core and ceramic rods were prepared the soil/ceramic column was assembled for centrifugation.

The average matric potential, $\overline{\psi}$, generated at a given rpm was calculated using Equation 6 [5]. Here g is the acceleration of gravity (981 cm s$^{-2}$), k is equal to 0.09807 kPa cm$^{-1}$, $\omega$ is the rotational speed (rad/sec), L is the column length (cm), and r$_c$ is the centrifuge radius from the

top of the soil column to the axis of rotation (25.4 cm).  Average matric potentials between -

0.132 and -2.33 Mpa can be generated for the rotational speed range of 1000-4200 rpm.  Trials

were performed once at each of three calculated matric potentials: -0.132 MPa, -0.35 MPa, and -

1.0 MPa.

$$\bar{h} = \frac{k}{6}\frac{\omega^2 L}{g}[L - 3r_c] \tag{6}$$

The soil core and ceramic rod were weighed prior to the start of each run.  The columns were

then spun at a given rpm for the duration of a trial.  The centrifuge was stopped intermittently,

and the soil core and ceramic rod were removed and weighed.  Initially, the goal was to continue

spinning each sample until the mass transfer between the soil and rod approached a negligible

amount.  However, due to time constraints and centrifuge availability this was not possible for

each run.  The -0.13 Mpa and -0.35 Mpa runs were spun for 5 and 8 hours respectively.  The -1.0

MPa was spun over the course of two consecutive days for a total run time of 14 hours.  For this

run, the soil core and ceramic rod were properly sealed at the end of the first day to prevent

evaporation overnight.


After the completion of each run, the soil was placed in an oven to dry out any remaining

moisture.  The original moisture content of the soil was then calculated.  The moisture contents

of soil sampled were 32%, 34%, and 36% for the -0.13MPa, -0.35 MPa, and -1.0 MPa runs

respectively.


Results and Discussion:

Due to time constraints and equipment availability only the -1.0 MPa was run for a sufficient

duration to approach steady state.  However, for all three runs the ability to extract sufficient

water for mass spectrometry was demonstrated. Table 3 highlights the total amount of water

extracted for the given time frame of each run.

Table 3: Water mass collected and evaporative losses for each trial. Only the -1.0 MPa trial was run for a sufficient amount of time to reach steady state. However, for all three trials sufficient water was extracted for stable isotope ratio analysis

| Trial | Moisture Content %(wt) | Run Duration (hr) | # of Run Stops | Water Collected (g) | Evaporative Loss (g) |
|---|---|---|---|---|---|
| -0.13 MPa | 32 | 5 | 8 | 1.3 | 0.07 |
| -0.35 MPa | 34 | 8 | 10 | 1.9 | 0.09 |
| -1.0 MPa | 36 | 14 | 13 | 3.4 | 0.29 |

For each trial, the water loss from the soil sample was greater than the mass collected in the

ceramic rod. This loss is likely due to evaporative losses from both the soil core and ceramic

rod. It is unclear as to whether this loss is occurring during centrifugation or during downtime

where the soil core and rod were being weighed, but it is mostly likely that the small amount of

water loss occurs when the soils and rods are being handled during weighing and thus the final

protocol should involve as little manipulation of the soils and rods after centrifugation as

possible. For the second two trials, only the top surface of the ceramic rod was left exposed,

with the length and bottom surfaces of the rod sealed with parafilm in an attempt to minimize

evaporation. This did not noticeably affect the amount of evaporation between the first two

trials, which may suggest that the largest evaporative losses are from the soil core. For all three

runs, the soil core was sealed on top with parafilm, and the bottom was left exposed for contact

with the ceramic rod. However, to prevent fractionation a more reliable way of sealing both

sections of the column will need to be developed. The third trial was subject to additional error

which could account for the higher evaporative loss observed. After 8 hours of runtime, the

parafilm sealing the rod began to tear. Repeated removal of the rod during weight measurements

most likely caused this. The rod was resealed, but during this downtime the rod was left exposed to the surrounding air. Additionally, after 9 hours of runtime the water level had reached the bottom surface of the rod and had begun to pool beneath. This added additional error to mass measurements.

Figures 4 and 5 highlight the progress of water mass transfer over the duration of each trial. For each trial, a large transfer of water was observed during the first half hour of runtime followed by decreasing returns thereafter. The third trial was run for a sufficient amount of time to where steady state was approached. The first 7 hours of centrifugation represented 93% of the total water collected, whereas the last 7 hours of centrifugation accounted for only 7% of the total. During the last hour of centrifugation less than 1% of the total water was collected. This may become important due to the outflow of water after 9 hours of centrifugation. However, the mass transfer rates and total times will vary with soil type. This soil has not been characterized, and in general this test was meant more as a proof of concept. To be confident in the extraction of water up to a given matric potential during centrifugation, knowledge of runtimes necessary for given soil types will need to be determined.
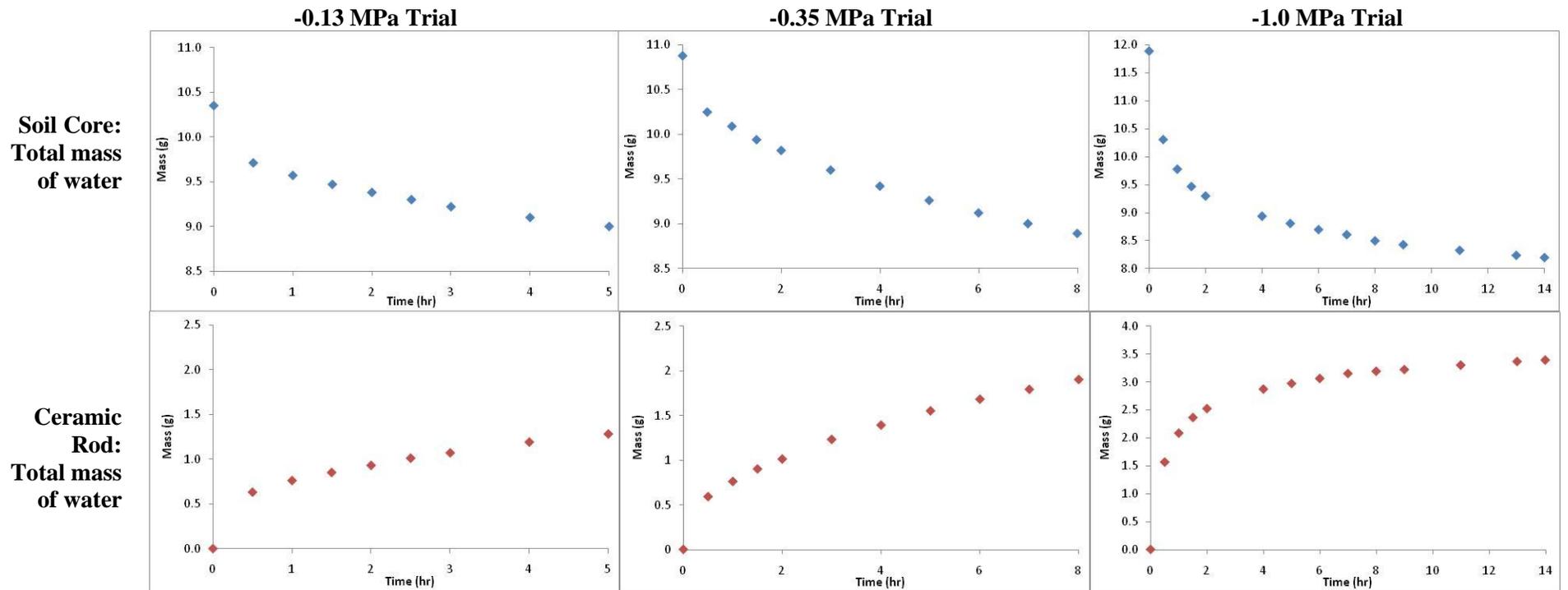
Figure 4: Total mass of water in the soil core and ceramic over the duration of each trial.
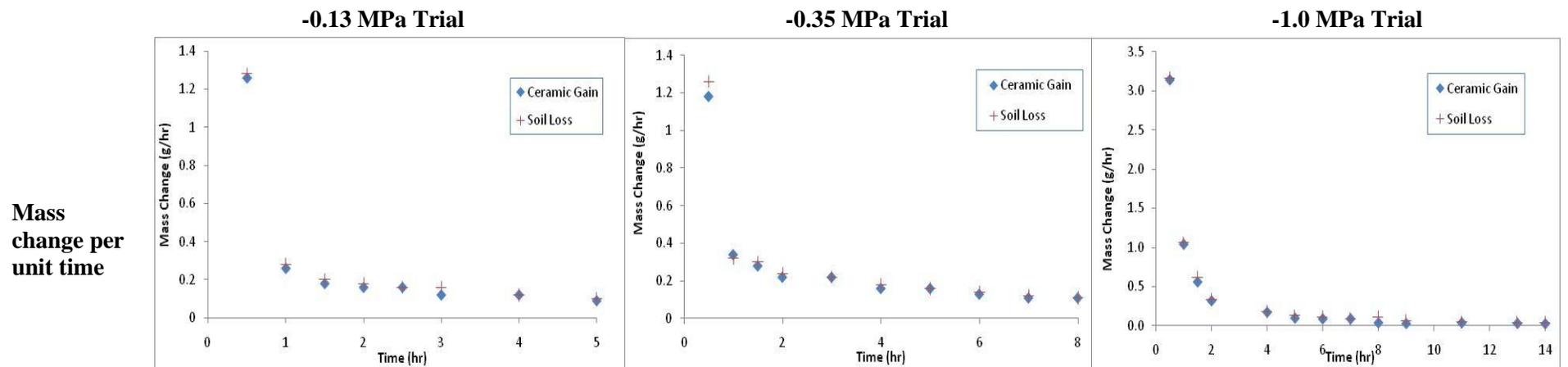


Figure 5: Mass change per unit time.  The change in water mass between time increments is given in terms of water gained by the ceramic and lost by the soil.  The difference between soil loss and ceramic gain represents moisture loss due to evaporation.

# Conclusion

This study represents the beginning stage for the design of a water pool separation procedure. Up to this point the design shows promise. Sufficient water can be extracted from soil with moisture contents ranging from 32-36% by weight. Furthermore, the water collected in porous ceramic rods can be reliably re-extracted using cryogenic vacuum distillation. However, further issues will need to be addressed before this device and procedure can be used to provide data for the two water worlds hypothesis.

Evaporation during centrifugation remains a concern. While this study indicated that evaporative losses were occurring, the timing and source of this evaporation will need to be understood. By performing an entire run without pausing for intermittent mass transfer measurements the rate of evaporation during centrifugation itself can be analyzed. If evaporation is occurring throughout centrifugation additional design consideration must be given to correct this flaw.

Only one trial was allowed to proceed to near steady-state values, and each run was conducted with an unknown soil type and variable moisture contents. As both soil make-up and moisture content will affect the required time to reach steady state, understanding the influence of each of these parameters will be important. These can be characterized through conducting additional runs where variation of these parameters is observed. In addition to this, the time required for the water level to reach the bottom of the ceramic rod will become important. The loss of water from the ceramic rod may or may not be important depending on the amount of water extracted for each run.

Once further testing provides confidence in the reliability of this water separation procedure the hypothesis developed by Brooks. et. al. (2010) can be investigated further.  With the capability of separating loosely bound water from a bulk soil sample, many insightful studies will become possible.  One of the first to be done will be to separate water from bulk soil throughout a rainy season.  The resulting isotope ratios present in each pool will help to better characterize the degree of mixing of water throughout soil pores.  During the dry summer an experiment can also be performed to test the hypothesis using H.J. Andrews  Experimental Forest soil cores.  These cores will be relatively dried out, with the water remaining being H.J. Andrews water.  The cores can then be irrigated with water from a different location and isotopic composition , and after separating the loosely bound water the resulting pools can be analyzed.  These represent a few initial ideas among many, and will be possible once the design is proved reliable.

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