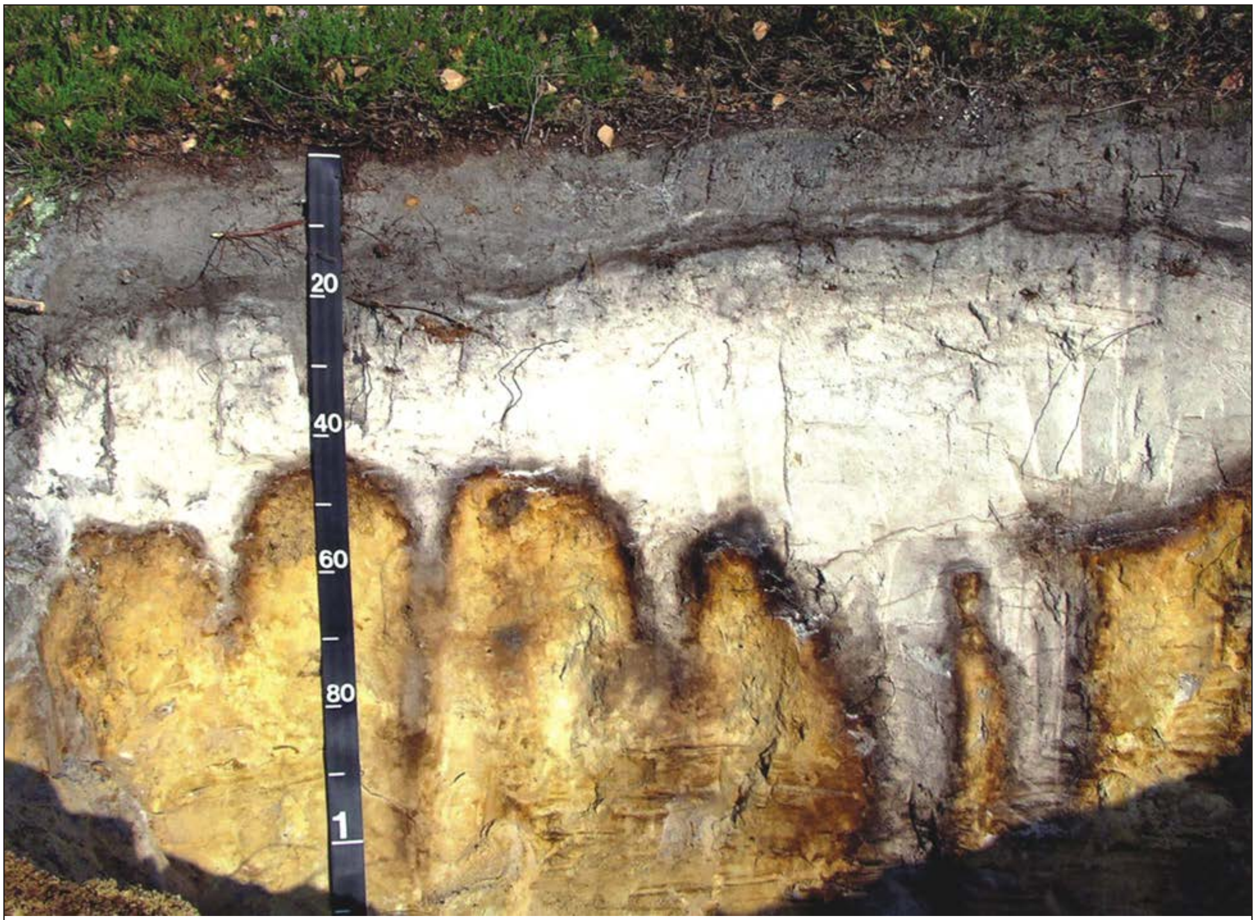

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The use of Porous Ceramic Cups for Sampling Soil Pore Water from the Unsaturated Zone

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ABSTRACT

Gaining knowledge of solute concentration and movement is one of the many keys to understanding the response of agricultural ecosystems to would-be pollutants. Monitoring the leaching of pollutants throughout the unsaturated zone is an important means of identifying the potential for groundwater contamination. Water solute monitoring and data collection, at present, can be obtained using either, *in situ* solute collection or via geophysical measurement. As data collated from the latter often requires support from knowledge gathered using *in situ* solute collection, this review evaluates the most common method for *in situ* solute extraction-the ceramic cup method. Ceramic cups are widely used for the extraction of soil pore water for monitoring of solute transport and concentration. This method offers the potential to simultaneously sample soil water at different depths in the soil profile to record the sequence of solute progress. The installation of the ceramic cup sampler into the soil profile is relatively easy when compared to other sampling techniques and causes only negligible disturbance of the soil, coupled with cost-effectiveness and the knowledge obtained makes them the most universally used technique for extracting soil water. However, the use of this system is not without problems. The spatial variability of the properties under investigation is often underestimated requiring clarification by replication. The matter of inherent bias in preferentially monitoring nutrient composition and flow of soil water in profile macropores is acknowledged and the associated possible alteration of the water sample. This study reviews the suitability of the ceramic cup as an experimental aid for the retrieval of soil solution samples for analysis and the issues associated with its use.

Key words: Ceramic cups, solute sampling, preferential flow, silica flour, sodium bentonite

INTRODUCTION

Understanding the dynamics of soil solution chemistry is a prerequisite for redressing many environmental problems (Lentz, 2006). The soil solution accommodates most chemical reactions occurring in the soil. It is relevant for studying both soil genesis and for ecosystem functions as this is the location of soil-forming processes and from which roots take up nutrients (Marques *et al.*, 1996), unlike the soil solid phase which represents the history of soil-forming processes, soil solutions provide the best information on current soil dynamics.

The collection and analysis of the *in situ* soil solution is important for studies of pedological processes, environmental quality monitoring and nutrient cycling (Zabowski and Ugolini, 1990). Soil solution measurements are relevant for plant uptake and nutrient availability concerns and estimates of solution fluxes from ecosystems are needed to balance ecosystem nutrients budgets and for research questions addressing losses of elements via leaching (Lajtha *et al.*, 1999).

Soil solution can be measured either by collecting field moist soils and extracting solutions in the laboratory or more commonly by collecting soil solution in the field, using a solution sampler such as a ceramic cup. The principle of the ceramic cup method was first described over a century ago by Briggs and McCall (1904). Since then these samplers have become well-developed and are widely used across many disciplines both practical and scientific for the collection of soil waters for analytical purposes.

The ceramic cup method is one of many systems commonly used in both agriculture and natural settings to extract soil water by means of direct contact and collection of the ambient soil solution. This sampling method permits the monitoring of solute movement with high temporal and spatial resolution (Weihermüller *et al.*, 2007). The ease of installation, low costs and the vast amount of information gathered makes this system one of the most commonly used for the *in situ* monitoring of the soil solution (Creasey and Dreiss, 1988; Lajtha *et al.*, 1999). The samplers can be installed from soil cores, therefore, large soil pits are not necessary and thus, soil disturbance is minimized. However, the use of these samplers requires some soil disturbance immediately above the sampler. It is not clear what area of the soil is being sampled where these samplers are used, although the depth at which the soil water is collected can be regulated.

In this study, the practical aspects of this method and the sampling system and its uses will be discussed. The problems arising from using this method are also described, including preferential flow and the alteration of the solution sample.

THE CERAMIC CUP METHOD

Variation in the ceramic cup sampling method has been described by many authors; however, in most cases all samplers consist of three functional units (Fig. 1): the ceramic cup and PVC pipe, the sampling bottle and the suction container (Grossman and Udluft, 1991). The most common commercially available sampler consists of PVC or other tube of inert material that is of a variable length, with a round bottomed ceramic cup at the bottom that serves as the filtering membrane for soil water (Lajtha *et al.*, 1999). The ceramic cup is cemented to the PVC pipe using an epoxy paste which bonds to both plastic and ceramic and forms a good seal between the two materials (Mitchell *et al.*, 2001). A neoprene access tube, fitted into the PVC tube by a rubber stopper, extends above the surface of the soil and is connected to the vacuum source for water collection (Lajtha *et al.*, 1999).

Ceramic cups can be purchased and samplers can be customized for specific applications (Stone and Robl, 1996). Different ceramic materials, sintered materials and membranes are used as cup materials (Dorrance *et al.*, 1991; Lajtha *et al.*, 1999). The most frequently used material is ceramic; however, other, more inert materials than ceramic have been used for samplers, cups made of aluminum oxide, glass sinter, nylon, PVC, PP and PVDF are available (Creasey and Dreiss, 1988). Teflon has also been used in construction of some cup samplers to ensure that there is no contamination from the porous medium (Mitchell *et al.*, 2001; Creasey and Dreiss, 1988). Mitchell *et al.* (2001) also mentions the use of stainless steel lysimeters employed to look for contamination within an already polluted area. The use of these materials addresses concerns about

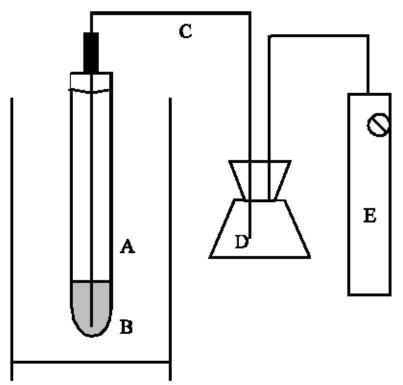

Fig. 1: The ceramic cup sampling system

the chemical inertness of porous ceramic as a material for samplers (McGuire *et al.*, 1992). The material used for the soil interface must be hydrophilic in order to maintain the capillary tension necessary to keep tension between rain events. If the sampler material is hydrophobic, samplers may be coated with hydrophilic materials such as silica flour (Lajtha *et al.*, 1999). By their very nature, these materials interact to some degree with the solutions passing through them. Litaor (1988), Grossman and Udluft (1991) and McGuire *et al.* (1992) provided a comprehensive review of chemical interactions with various types of samplers, concluding that results vary greatly depending on contact time, the ion in question and soil characteristics.

In general, installation of the cup samplers is relatively straightforward when compared with the installation of other soil water sampling systems. The literature describes four possible installation modes for the ceramic cup assembly: horizontal, vertical shaftless, vertical and vertical at 45° (Fig. 2).

With a horizontal assembly Grossman and Udluft (1991) described a hanging water column used to generate the suction. This suction is limited by the maximum length of the water column and air bubbles can hinder solute extraction. The protective piping around the sampler assists in easy installation of the unit. The sample generated is collected in an external vessel. The vertical shaftless arrangement is used for installation in arable land. To ensure the sampler is not in the path of cultivation equipment the ceramic cup component is installed without a shaft. Sampling tubes extend from the cup through the refilled soil to ground level to accommodate the extraction of the soil solution. The sample is collected in an external vessel. The suction decreases with increasing water column above the cup and increasing volume of the sample. The installation can be horizontal or vertical (Grossman and Udluft, 1991; Lajtha *et al.*, 1999). Vertically installed samplers collect the pore water in the cup and tube. The suction decreases with increasing water column above the cup and increasing volume of the sample. An alternative arrangement to the Grossman and Udluft (1991) vertically installed assembly is the vertical sampler installed at a 45° angle, as described by Mitchell *et al.* (2001). Again, the sample is collected in the cup and tube with the suction decreasing with increasing water column above the cup and increasing volume of the sample. However, the complete assembly is installed at a 45° angle to the ground to further minimize the incidence of preferential flow. With the vertically installed ceramic cup (3) a collar may be used around the sampler shaft to minimize any downward flow as a result of disturbed soil

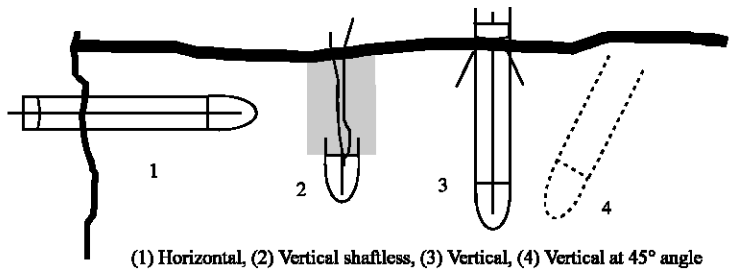

Fig. 2: Variations in ceramic cup installations

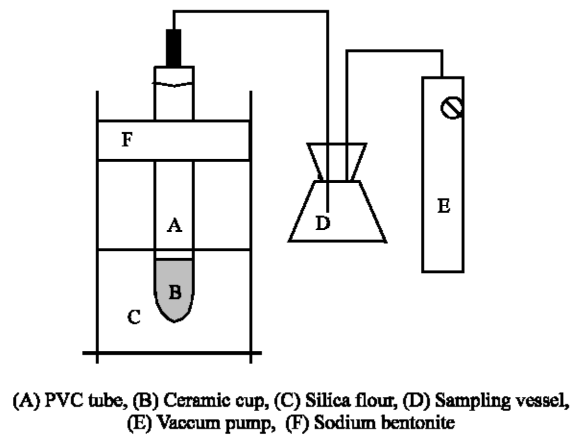

Fig. 3: The ceramic cup sampling system as installed

around the sampler. Each ceramic cup sampler has a sampling tube internally for removing extracted soil solutions. It is inserted into the sampler prior to the installation of the sampling systems into the soil. If sampling is to occur during sub-freezing conditions, it is vital that this tubing remains pliable. Therefore, for cooler temperatures, Mitchell *et al.* (2001) recommends silicone tubing rather than polyvinyl chloride (PVC) tubing.

Sampler installation always involves some degree of soil disturbance even when performed from soil pits as in plate samplers or via a soil core as in ceramic cup samplers (Fig. 3). This disturbance can result in irregularly high soil solution concentrations of nitrate and/or silica bicarbonate (Litaor, 1988; Lajtha *et al.*, 1995). Lajtha *et al.* (1999) suggested that the best way to account for this effect is to simply wait unit a large data set can be accumulated from which regular seasonal patterns can be observed, allowing irregularities to be identified. This waiting period can range from 2-4 months to 2 years. Johnson (1995) found a very large nitrate pulse lasting over a year after sampler installation in a forest soil. After installation all samplers should be emptied repeatedly before initiating solution collection for chemical analysis (Grossman and Udluft, 1991; Hendershot and Courchesne, 1991; Weihermüller *et al.*, 2005). Furthermore, the first samples should be rejected in each case and the first results of a measurement series must be examined for credibility (Curley *et al.*, 2010a).

Ceramic cups can be purchased pre assembled from a number of manufactures (e.g., SDEC France-Z.I. de la Gare-37 310-Reignac sur Indre (France)-EUROPE, (http://www.sdec-france.com), or Soilmoisture Equipment Corporation, Santa Barbara, California, USA, (http://www.soilmoisture.com)

THE PRINCIPLE OF THE CERAMIC CUP METHOD

The principle of the ceramic cup system described by Briggs and McCall (1904) has not changed significantly despite many changes in the construction materials and arrangement of the apparatus. In 1904 the authors described the apparatus as consisting of a close-grained unglazed porcelain tube, closed at one end and provided at the other end with a tubular by which it can be connected to an exhaust receiver. The principle of operation remains almost identical 100 years later. Grossman and Udluft (1991) described the ceramic cup as being constructed of hydrophilic materials with fine pores. When suction is generated within the sampling system, water is sucked inwards from the pores of the cup until a corresponding capillary pressure occurs in the pores. If the capillary pressure in the ceramic cup is lower than that in the soil, water flows from the soil into the cup until the capillary pressure in the suction cup and in the soil are equal.

For the operation of a cup sampler, a negative pressure has to be imposed by applying suction to the cup using a vacuum system (Weihermüller *et al.*, 2007; Curley *et al.*, 2010a). Using simple pumps, it is possible to generate suctions down to -0.09 MPa (Grossman and Udluft, 1991). In order to achieve such suction in the sampling system, no air must pass through the pores of the cup. For this, the diameter of the largest pore must not exceed a certain size. A ceramic suction cup with a pore size of 3 μm and a low air entry value (0.1 MPa) has a high sampling flow making it a suitable constituent for sampling (http://www.sdec-france.com).

The suction generated in the sampling system creates a potential gradient around the ceramic cup. As a result, the seepage water flows from a specific space into the sampler (Grossman and Udluft, 1991). This potential field around the ceramic cup has been measured and determined to extend to over 1 m in inhomogeneous soils. However, the actual recharge area (the area from which the water flows towards the cup) is much smaller and is dependant on a number of factors including, capillary pressure in the soil, suction in the cup, cup diameter and pore size along with installation depth and the position of the groundwater surface (Grossman and Udluft, 1991). The suction to be applied to the ceramic cup, in general, is dependant on a number of factors, such as, soil type, the specific amount of water required for analysis, the actual soil water content and the time of applied suction (Warrick and Amoozegar-Fard, 1977; Weihermüller *et al.*, 2005). It should be remembered that different suction levels will draw on different sources of soil water with potential repercussions for chemical analysis (Lajtha *et al.*, 1999). Because suction is applied, it cannot be assumed that the water collected by ceramic cup samplers is chemically equivalent to water that leaches through the soil profile.

OPERATION OF THE CERAMIC CUP SAMPLER

For soil solution extraction, using the ceramic cup method two procedures are available; continuous extraction or discontinuous extraction.

With continuous extraction, a potential gradient is applied, which depends on the actual pressure head in the undisturbed soil measured by a tensiometer (Weihermüller *et al.*, 2007). In general, the continuous extraction method is used to determine concentration changes. The advantage of this mode of operation is the permanent collection of soil water and subsequently a reasonably accurate assessment of the drainage pattern (Magid *et al.*, 1992; McGuire and Lowery, 1994). In a comprehensive study on the extraction of soil water by the ceramic-cup method, Grossman and Udluft (1991) stated that the withdrawal of water from the soil system can cause significant disturbance. However, when using a continuous operating mode the small volume of water withdrawn over unit time will have a minimal affect on the natural flow pattern of the soil water and thus, reduced disturbance allowing data reliability. Weihermüller *et al.* (2005), in a review of soil water extraction techniques, reported that the continuous water flow also reduces sorption processes in the cup material and low potential gradients are necessary to collect sufficient amounts of water for chemical analysis. The authors also identify the main disadvantages of this system; in that it can initiate the creation of preferential flow paths to the cup. Moreover, considerable maintenance of the system is necessary and most importantly the composition of the extracted water sample can change during storage in the sampler prior to removal.

Using discontinuous extraction, water collection is performed during selected short-time intervals, typically to determine the status of a soil during a specific time or event (Weihermüller *et al.*, 2007). That is, this procedure can be used to indicate the presence of solutes at a particular time (Linden, 1977). The main benefit of this mode of operation is the minimal temporal disturbance of the natural flow field (Jury and Fluhler, 1992). Also, there is little maintenance associated with this system. The main disadvantage of this method is that the non-permanent flow through the cup material can result in high sorption, making it advisable to discard the first water sample. Furthermore, temporary actions such as rapidly changing solute concentrations caused by heavy rainfall and preferential flow may be inadequately recorded (Jury and Fluhler, 1992).

Lajtha *et al.* (1999) noted that with samplers set at a constant vacuum; there is almost always either an underestimation or an overestimation of soil water flux because the vacuum at the sampler usually differs from that of the soil, which varies. This is sometimes referred to as coning. If vacuum is too low, water will move around the sampler and flow through the soil as unsaturated flow in the soil matrix.

PREPARATION AND INSTALLATION OF SAMPLERS

Preparation of samplers: A thorough cleaning is necessary to remove contaminants left over from the production process when preparing new samplers (Grossman and Udluft, 1991). Thus, it is recommended that new samplers be cleaned before use by flushing them with dilute acid (Litaor, 1988) and repeatedly rinsed with deionized water (Hendershot and Courchesne, 1991; Curley *et al.*, 2010a).

To circumvent the potential sorption effect that new samplers can display during the first sampling after installation, particularly in the case of trace substances, Grossman and Udluft (1991) stated that the surface of the ceramic cup should be preconditioned, i.e., equilibrated with a solution of more or less the composition of the soil solution. The authors discussed two possible methods of conditioning:

- Prior to installation the ceramic cup can be rinsed with a solution similar to the expected soil solution
- Post installation during the necessary stabilization phase conditioning can be achieved by repeated water sampling

It is apparent from the literature that after installation all samplers be left in the field and emptied repeatedly before initiating solution collection for chemical analysis (Grossman and Udluft, 1991; Hendershot and Courchesne, 1991; Weihermüller *et al.*, 2005). Furthermore, the first samples should be rejected in each case and the first results of a measurement series must be examined for credibility.

Installation of samplers: When installing the ceramic cup sampler, good hydraulic contact between the sampler and the ambient soil is critical (Lord and Shepherd, 2006; Weihermüller *et al.*, 2007). To ensure this, on installation, the sampler should be inserted into a hole drilled by means of a soil auger that has a diameter similar to that of the sampler, taking care to prevent soil material from the upper horizons from falling into the hole (Grossman and Udluft, 1991). To further ensure good soil-sampler contact, Barbee and Brown (1986) recommend making a slurry of the soil removed from the hole and pouring it back into the hole prior to installation to ensure good soil contact with the sampler. The coarse sand and gravel fraction are removed from the material used as the slurry by sieving. When the sampler is put in place the slurry begins to move upwards between the sampler and the augured hole thus filling any gaps.

Many researchers have discussed the use of Silica Flour (fine quartz silt), a selected silica sand with a SiO_2 content of over 99 %, used to improve contact between the ceramic cup and the soil (Barbee and Brown, 1986; Smith and Carsel, 1986; Weihermüller *et al.*, 2007; Curley *et al.*, 2010a). An aqueous suspension of the silica flour is injected into the borehole created for the installation of the sampler. Care should be taken during installation to ensure that water cannot leach from the surface down through the augured hole, thereby creating a pathway for preferential flow (Lord and Shepherd, 2006). To minimize this risk the sampler can either be sunken completely into the soil (Grossman and Udluft, 1991) or a collar can be created around the shaft of the sampler. Rhoades and Oster (1986) described the use of wet bentonite to prevent excessive percolation through the back-filled hole.

Sodium bentonite: Surface modifications of clay minerals have received much attention as this process allows the creation of new materials and new applications (DePaiva *et al.*, 2008). The main focus of surface modification of clays is material science with uses such as absorbents for organic pollutants in soil, water and air (Beall and Goss, 2004).

The suitability of a clay for use in this manner depends on its properties, such as, plasticity and cohesion, swelling, shrinkage, dispersion and flocculation (Brady and Weil, 2002). Certain clay groups with different types of clay minerals swell in volume when wetted. The physical and chemical properties of these swelling-type clays, particularly the smectites (bentonite), make them useful tools in preventing unwanted movement of water and water contaminants due to a high cation exchange capacity, swelling behavior, adsorption properties and large surface area (Vougaris and Petridis, 2002). Such clays are widely used in the construction of liners to seal ponds, sewage lagoons, industrial waste lagoons and landfills. A layer of the smectite placed on the bottom and sides of the pond or lagoon expands when wetted and forms a relatively impenetrable barrier to the movement not only of water but of organic and inorganic contaminants contained in the water (Brady and Weil, 2002). These contaminants are held in the containment area and are prevented from moving downward into the groundwater.

Bentonite works by acting as a sealant to prevent preferential flow in the re-packed borehole. During back filling of the augered hole 10-15 cm of pelleted air-dried bentonite is tapped into place, then wetted. When the wetted clay expands, it fits tightly around the PVC pipe and the borehole wall (Rhoades and Oster, 1986; Curley *et al.*, 2010a). The bentonite plug protects the sampler from the possibility of chemicals washing down from the soil surface and therefore being mistaken for groundwater contamination.

SOLUTE RESPONSE-COMPOSITION OF THE SAMPLE

A number of researchers have investigated the effect of ceramic cup samplers on extracted water chemistry. In relation to nutrients, specifically nitrate, the anion nitrate (NO_3^-) is characterized by low interaction with most materials (Weihermüller *et al.*, 2007). Studies indicate good agreement of nitrate fluxes determined with ceramic cups for well drained loamy sand and sandy loam (Webster *et al.*, 1993; Curley *et al.*, 2009). Ammonia, however, is subject to cation exchange processes. Ammonia and nitrate concentration are easily altered by biological processes (e.g., nitrification). Wagner (1962), Hansen and Harris (1975), Nagpal (1982) and Poss *et al.* (1995) noted during a laboratory study that NO_3-N absorbed by porous ceramic cups placed in solution was minimal.

Although, several studies have found that soil solutions collected with ceramic cup samplers had higher concentrations, as one would expect since they should collect a more tightly bound fraction of soil water, this varied a great deal depending on the ion examined and the site (Lajtha *et al.*, 1999). In a study undertaken by Marques *et al.* (1996), on the chemical composition of soil solutions collected by zero-tension plate lysimeters and ceramic-cup samplers, the authors concluded that organic matter greatly influenced the solution chemistry in both samplers. Also, in relation to ammonia, tension lysimeter solutions/ceramic cup samplers were more concentrated for this parameter. Thus, short sampling intervals (<2 weeks) combined with cool, dark storage of samples is necessary (Weihermüller *et al.*, 2007).

Sample solutions obtained via the ceramic cup method can also be stabilized with the addition of acids (HCl) to the extracted soil water provided this action does not disturb the detection and analysis of other target data, e.g., pH (Weihermüller *et al.*, 2007).

MACROPORES AND PREFERENTIAL FLOW

Macropores by definition are large soil pores, typically having a diameter greater than 0.06 mm, from which water drains readily by gravity (Brady and Weil, 2002). These pores accommodate not only the movement of air and water but they are also large enough to facilitate plant roots and the wide range of organisms that inhabit the soil. Macropores can occur in many places, between individual sand grains in coarse textured soils or in well-structured soils between peds (interped pores). Macropores created by roots, earthworms and other organism constitute a very important type of pore termed biopore (Brady and Weil, 2002).

The relationship between macropores and ceramic cup samplers is not easily represented. In incidents of heavy rainfall, well-structured soils channel water quickly via macropores or sections of soil with high permeability, meaning seepage water may by-pass the ceramic cup if it is not in direct contact with the macropore (Grossman and Udluft, 1991). Essentially, while ceramic cup samplers can collect soil solution at a wide range of soil tensions with relative ease they cannot measure macropore flow (Barzegar *et al.*, 2004). This phenomenon has been discussed by many authors and was summarized by Shaffer *et al.* (1979), who stated that in structured soils, ceramic cup samplers may be completely circumvented by the channeling of water and chemicals through interped pores. Ceramic cup samplers may be unreliable in soils with a high incidence of preferential flow, whereby recording of bypass flow might be a fundamental weakness of the sampler or as a result of inadequate numbers of samplers to sample the flow system (Jury and Fluhler, 1992).

Brady and Weil (2002) described preferential flow as non-uniform movement of water and its solutes through a soil along certain pathways, which are often macropores. The authors continue by stating that any process that stimulates the formation of macropores with continuity downwards through the profile will encourage preferential flow. Burrowing animals as well as decaying plant roots leave tubular channels through which water can flow rapidly, accommodating hydraulic short-circuiting of the ceramic cup.

SAMPLER FAILURE

A number of authors have documented incidence of sampler failure despite appropriate procedures being carried out during the preparation and installation. The most noteworthy cause of failure after installation is damage caused by small animals (Mitchell *et al.*, 2001). Samplers need to be routinely checked for damage by animals as they have a tendency to chew on the tubing and any component of the apparatus above ground. It has been this author's experience that damage to samplers by small animals e.g., rabbits and hares can be hugely destructive rendering them inoperative.

CONCLUSIONS

The ceramic cup method is widely used to extract soil solution for monitoring and analysis of solute transport (Curley *et al.*, 2010b). The acknowledged benefits of this method are the ease of installation with minimal soil disturbance and as a consequence the negligible changes in natural percolation activity. However, it has also been identified that this sampling method shows a bias in preferentially monitoring the chemical composition of solutes from larger soil pores to the detriment of finer pore solutions. Also, a serious limitation identified in the literature is the fact that the imposed changes in matric potential of the natural flow during sampling are not well known. Likewise, the affect of the sampling system on soil solution chemistry is unclear.

In the absence of absolute evidence that the ceramic cup method has an effect on solution chemistry, the researcher when planning experiments and monitoring programs is free to select the type of apparatus that best fits the requirements and limitations of the study (Hendershot and Courchesne, 1991). However, it is clear from the literature that soil solution samples derived from the ceramic cup method are more useful when studying the relationship between plant nutrient uptake and soil solution chemistry.

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