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Porous Ceramic Cups: Preparation and Installation of Samplers for Measuring Nitrate Leaching

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Abstract: Ceramic cup samplers are widely used in agriculture to obtain samples of soil pore water for nitrate analysis. This method has become well developed over the past 30 years; however, it is not fully clear what preparation and installation procedure gives the most reliable results or what factor could limit the validity of their use. This study describes the design and use of these samplers including modifications made during large-scale experimental work, on heavy soils in the East of Ireland. Ceramic cup samplers must be installed carefully to avoid preferential flow through the disturbed soil around them and to maximize contact between cup and soil. Furthermore, if samplers are not prepared adequately prior to installation, the cup material may release contaminants into the collected samples resulting in prejudice study results. Newly installed ceramic cups were used at University College Dublin (UCD), Ireland, in 2008 to determine the degree of nitrate leaching from various crops post-fertilization with organic manures. The ceramic cups were cleaned using a dilute acid and repeatedly rinsed with deionized water. During the installation process sodium bentonite and silica flour were used to minimize preferential flow and ensure good hydraulic contact between the sampler and the soil.

Key words: Ceramic cup, nitrate, sodium bentonite, silica flour

INTRODUCTION

Leaching of Nitrogen (N) from agricultural and urban plant-soil systems is typically quantified using ceramic cup samplers (Bowman *et al.*, 2002). Their use, first described by Briggs and McCall (1904) has evoked interest in the past 30 years through the need to measure nitrate leaching from light soils in order to develop appropriate strategies for reducing nitrate in groundwater (Council Directive 80/778/EEC, 1980). The main advantages of these samplers are their ease of installation and low cost (Bowman *et al.*, 2002; Weihermüller *et al.*, 2007). Hansen and Harris (1975) showed that nitrate is not absorbed by the ceramic cup at concentrations typically found under agricultural land while Webster *et al.* (1993) conclude that ceramic cups gave an accurate measure of nitrate leaching losses on sandy soils, therefore, making them suitable for use in nitrate analysis. Various studies exist on the arrangement and preparation of samplers for

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installation, with many authors describing the sampling system (Grossman and Udluft, 1991; Hendershot and Courchesne, 1991; Weihermüller *et al.*, 2005). The main objectives of this study are to describe modifications made to the ceramic cup sampling system while detailing the design and use of these samplers in Irish soils and also to describe the preparation and installation of the samplers. The samplers were used in a field-scale study at UCD Lyons Research Farm, Ireland, in 2008/09 to investigate the potential for nitrate leaching post fertilization from short rotation coppice willow and miscanthus crops.

THE CERAMIC CUP METHOD

Numerous publications exist on the arrangement and installation of the various types of ceramic cup samplers (Barbee and Brown, 1986; Litaor, 1988). In most cases all samplers consist of a cylindrical porous ceramic cup sealed to a tube, with a smaller sampling tube inside to collect the extracted water (Dorrance *et al.*, 1991). In practice the complete sampling system consists of three functional units: the ceramic cup and a PVC solution pipe, the sampling bottle and the suction container/pump (Grossman and Udluft, 1991), a common arrangement of these components is shown in Fig. 1.

Mitchell *et al.* (2001) described the assembly; the ceramic cup is cemented to a PVC pipe using an epoxy paste which bonds to both plastic and ceramic and is easily applied to the outer rim of the cup and the edge of the pipe to form a good seal. A rubber plug with a 10 mm (outside diameter, OD) plastic tube attached was fitted to the open end of the PVC pipe, thus giving the basic assembly.

This arrangement was used in trials at UCD in 2008, with the following modifications outlined below:

 A long tube of small diameter (5 mm, OD) was inserted into the PVC pipe inside the 10 mm plastic tube to remove the sample gathered

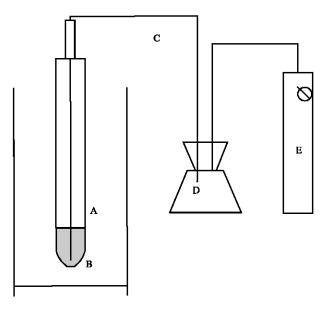


Fig. 1: The ceramic cup sampling system. A: PVC pipe, B: Ceramic cup, C: Sampling tube, D: Sampling vessel and E: Vacuum pump

- The inner sampling tube was inserted into the assembly until it touched the bottom of the cup and then withdrawn slightly so as it did not adhere to the bottom of the cup
- This 5 mm inner sampling tube was secured and its position marked (where it protrudes from the outer 10 mm tube) with luminous tape to alert the user if it was accidentally withdrawn of the cup during the sampling process
- Finally, an extension to the outer 10 mm plastic tube was fitted over the protruding 5 mm tube and secured with a 10 mm push fitting and clamped

To apply vacuum, the outer 10 mm tube was unclamped and the vacuum applied. For sampling, the extension to the outer tube was removed and the vacuum pump was fitted directly to the inner 5mm sampling tube again with a push fitting (Fig. 2).

Installation of the ceramic cup samplers was relatively straightforward when compared with other soil water sampling systems. In general, the literature describes four possible installation modes for the cup assembly: horizontal, vertical shaftless, vertical and vertical at 45° (Fig. 3).



Fig. 2: Ceramic cup assembly used at UCD, 2008. 1: V acuum pump, 2: 10 mm Outer vacuum tube, 3: Sampling container, 4: 5 mm Sampling tube, 5: V acuum tube extension, 6: PVC pipe and 7: Ceramic cup

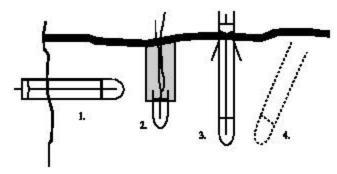


Fig. 3: Variations in ceramic cup installations. 1: Horizontal, 2: Vertical shaftless, 3: Vertical with collar and 4: Vertical at 45°

With a horizontal assembly the sampler is installed from a pit/hole dug beside the proposed location of the sampler; the sampler is installed horizontally into the pit face. Vertical shaftless samplers, for use in arable land, are used to ensure the sampler is not in the path of cultivation equipment. The ceramic cup component is installed without a shaft/PVC pipe with the sample tubing extending from the cup through the refilled soil (Grossman and Udluft, 1991).

An alternative arrangement to the vertically installed assembly is the vertical sampler installed at a 45° angle, as described by Mitchell *et al.* (2001). The complete assembly is installed at a 45° angle to the ground to further minimize the incidence of preferential flow. With the vertically installed samplers a collar may be used around the PVC pipe to minimize any downward flow as a result of disturbed soil around the sampler. Each sampler has a sampling tube internally for removing extracted soil solutions. All samplers used in the UCD trial were installed vertically with PVC sample tubing fitted.

Preparation of Samplers

When preparing new soil solution samplers a thorough cleaning is necessary to remove contaminants left over from the production process (Grossman and Udluft, 1991). If the samplers are not prepared adequately before installation, the ceramic cup material may release contaminants into collected samples and potentially bias experimental results (Creasey and Dreiss, 1988). 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). A study undertaken by Creasey and Dreiss (1988) into the cleaning procedure for ceramic cup samplers concluded that the level of some trace elements and major cations leached from the cups can be reduced significantly if the samplers are first cleaned. A study by Grover and Lamborn (1970) described the use of 50-60 pore volumes of 1 N HCl for cleaning new samplers followed by repeated flushing with distilled water (Wolff, 1967). To circumvent the potential sorption effect that new samplers can display during the first sampling after installation, Grossman and Udluft (1991) stated that the surface of the ceramic cup should be preconditioned. The authors discussed two possible methods of conditioning; (1) prior to installation, the ceramic cup can be rinsed with a solution similar to the expected soil solution, or (2) post installation during the necessary stabilization phase, conditioning can be achieved by repeated water sampling, as was the procedure used in the course of this study.

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.

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, take care to prevent soil material from the upper horizons 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 the hole and pouring it back into the hole prior to sampler installation to ensure good soil contact with the sampler. In trials at UCD in 2008, Silica Flour (fine quartz silt), a selected silica sand with a SiO₂ content of over 99%, was used to improve contact

between the ceramic cup and the soil. An aqueous suspension of the silica flour was injected into the borehole created for the sampler installation. This method has been discussed by Barbee and Brown (1986), Smith and Carsel (1986) and Weihermüller *et al.* (2007).

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. In the afore-mentioned trials at UCD, sodium bentonite was used as a plug to seal the augured hole, thus eliminating flow around the sampler and possible hydraulic short circuiting (Fig. 4).

Sodium Bentonite

Surface modifications of clay minerals have received much attention as this process allows the creation of new materials and new applications (De Paiva *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). Certain clay groups with different types of clay minerals swell in volume when wetted. This particular property greatly enhances the usefulness of such soils for both engineering and agricultural purposes. 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). In Fig. 4, the 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 palette air-dried bentonite was tapped into place and then wetted. When the wetted clay expands, it fits tightly around the PVC pipe and

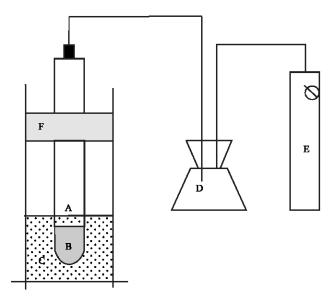


Fig. 4: The ceramic cup sampling system as installed. A: PVC pipe, B: Ceramic cup C: Silica flour, D: Sampling vessel, E: Vacuum pump and F: Sodium bentonite

against the borehole wall. The plug protects the sampler from the possibility of chemicals washing down from the soil surface and therefore being mistaken for groundwater contamination.

CONCLUSIONS

This study identifies many studies concerning the optimal set-up of *in situ* pore water samplers. It seems that it is almost impossible to establish samplers and obtain pore water samples which have not been altered or biased by the sampling process. Nonetheless, this article should advise researchers on how to minimize potential alteration to samples. The recognized advantages of these samplers are their cost effectiveness and ease of installation which has made them the most commonly used devices for collecting water from the vadose zone (Creasey and Dreiss, 1988).

It is imperative to exactly define the goals of planned experiments to make choices in which bias or alterations can be accepted (Weihermüller *et al.*, 2007). Furthermore, all possible alterations and known measurement errors should be well documented during the course of the experiment to identify possible biased data. These limitations should not prevent researchers from using these samplers but rather identify potential methods of enhancing their use such as accompanied non-invasive geophysical procedures or advanced modeling techniques. From experimental work carried out at UCD in 2008, it became apparent that the samplers were cost-effective and both preparation and installation of these samplers was relatively easy, when compared with samplers used previously.

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