Quasi-steady centrifuge method for unsaturated hydraulic properties

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[1] We have developed the quasi-steady centrifuge (QSC) method as a variation of the steady state centrifuge method that can be implemented simply and inexpensively with greater versatility in terms of sample size and other features. It achieves these advantages by somewhat relaxing the criterion for steadiness of flow through the sample. This compromise entails an increase in measurement uncertainty but to a degree that is tolerable in most applications. We have tested this new approach with an easily constructed apparatus to establish a quasi-steady flow of water in unsaturated porous rock samples spinning in a centrifuge, obtaining measurements of unsaturated hydraulic conductivity and water retention that agree with results of other methods. The QSC method is adaptable to essentially any centrifuge suitable for hydrogeologic applications, over a wide range of sizes and operating speeds. The simplified apparatus and greater adaptability of this method expands the potential for exploring situations that are common in nature but have been the subject of few laboratory investigations.

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1. Introduction

[2] The combination of centrifugal force and steady flow has great value for the measurement of saturated and unsaturated hydraulic properties of soils and rock [*Nimmo et al.*, 2002]. The steady state centrifuge (SSC) method for hydraulic conductivity (K) has been used with two different means of applying a steady flow of water. In the internal flow control (IFC) version a constant head within the centrifuge bucket controls the inflow rate [*Nimmo et al.*, 1987, 1992]. In the unsaturated flow apparatus (UFA) version a metering pump outside the centrifuge controls the inflow rate [*Conca and Wright*, 1998]. If the centrifugal force is great enough that it constitutes the dominant driving force, measurements of the steady state flux, water content, and matric pressure can yield highly accurate values of hydraulic conductivity and water retention. The great force permits measurement of properties and conditions that are otherwise impossible or impractical. Compared to a steady state measurement using gravity-driven flow, for example, a force of 1000 g affords the possibility of decreasing the lower limit of the measurable K range by about 3 orders of magnitude. An experiment lasting a few days can measure unsaturated conductivity as low as 10^{-11} m/s. With either method of establishing steady flow, when suitable conditions develop within the sample, $K(\theta)$ can be computed using the centrifugal form of Darcy's law

$$q = -K(\theta)\left(\frac{d\psi}{dr} - \rho\omega^2 r\right), \quad (1)$$

where q is flux density, θ is volumetric water content, ψ is matric pressure, ρ is the density of water, ω is angular speed, and r is the distance from the center of rotation.

[3] The IFC SSC method can be implemented with a standard production line centrifuge, but it requires custom-built apparatus and considerable operator attention. The UFA SSC method can be implemented with commercially available apparatus and is faster and easier to operate, but it is limited to relatively small (30-mm diameter) samples. It is also prohibitively expensive for many laboratories. The quasi-steady centrifuge (QSC) method is an alternative we have developed on the basis of IFC apparatus for relatively simple and inexpensive implementation and greater versatility. It achieves these advantages by somewhat relaxing the criterion for steadiness of flow through the sample. This compromise entails an increase in measurement uncertainty but to a degree that in most applications is tolerable and well worth the gains in simplicity of apparatus, larger sample capacity, and adaptability to various machines and operating conditions.

2. Design and Testing

[4] Figures 1 and 2 show the experimental apparatus, which can be used with a sample of rock or other porous medium and which fits into a 1-L swinging centrifuge bucket that spins in a horizontal position. The upper part of the apparatus includes a flow-controlling reservoir made of a polycarbonate cylinder which is open at the top. A thin (0.5 mm), flexible, highly permeable, porous membrane (EXCO Acti-Disk, FMC Corporation; trade names are given for identification purposes and do not constitute an endorse-

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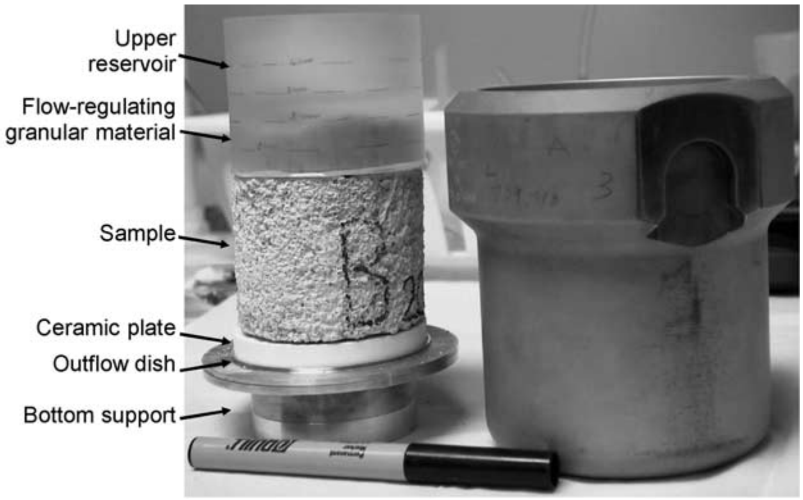

Figure 1. Photograph of quasi-steady centrifuge (QSC) apparatus next to a centrifuge bucket. The cake of flow-regulating granular material is visible through the translucent upper reservoir as darker material. See color version of this figure in the HTML.

ment by the U.S. Geological Survey) is sealed with epoxy to the bottom of the cylinder. A layer of fine granular material rests on the membrane within the cylinder. Centrifugal force acting on this layer and the membrane, both of which are flexible, maintains good contact with the sample. The conductance of this cake of fine material determines the flow rate (Figure 3). The conductance of the cake must be low enough to maintain a state of unsaturation within the sample. The lower part of the apparatus includes a ceramic plate placed on the outflow dish which rests on the bottom support (Figure 2). The ceramic plate supports the sample but does not seal to it, so outflow is not prevented. To reach a quasi-steady state normally requires a series of centrifuge runs. Between runs the sample is weighed, and water is replenished in the reservoir as necessary. The water used in the experiment in our tests was deaired Menlo Park tap water with three drops of household bleach added per liter to inhibit microbiological growth.

[5] The flow-controlling reservoir is filled to a reference level near the top at the start of each run. During centrifugation the head of the water, and hence the flow rate, declines. If the net change in head during a run is slight and the sample water content changes negligibly from one run to the next, the flow rate is taken to be quasi-steady. The average flow rate during a series of quasi-steady runs is used to calculate unsaturated hydraulic conductivity by the same formulas used with the steady state centrifuge methods [*Nimmo et al.*, 2002].

[6] This flow-controlling device permits adjustment of flux by changing the granular material, its thickness, and the speed of the run. To a first approximation, increasing the centrifuge speed causes flux to increase in proportion to the centrifugal force. In basic operation, diverse flow-regulating granular materials are selected to provide measurements at various water contents. We tested diatomaceous earth, silica flour, and bentonite in cakes of different thickness with various applied forces. These cakes were fabricated by putting the dry material on top of the membrane of the flow-controlling reservoir, mixing water into it, and centrifuging to produce a uniform, compacted cake. For most materials the dry grains can be added to approximately the height needed in the compacted cake, but for the bentonite, to allow for swelling, a much smaller amount was used, determined by trial and error. Before use in measurements each cake was compacted in several centrifuge runs with water flowing through it. To maximize

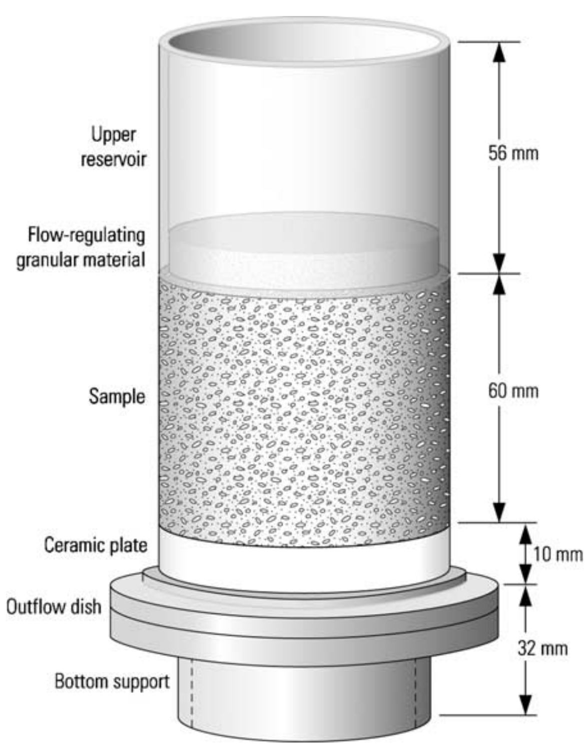

Figure 2. Diagram of QSC apparatus that fits into a 1-L centrifuge bucket. The sample shown here is a consolidated rock. The unsaturated condition inside the sample and high humidity outside prevent significant water loss through the sample walls. The centrifuge used with this apparatus has dimensions such that the lowest point of the bottom support rotates at a distance of 244.7 mm from the center of rotation. The rotating radius of other parts of the apparatus can be calculated by subtraction, using the dimensions shown here.

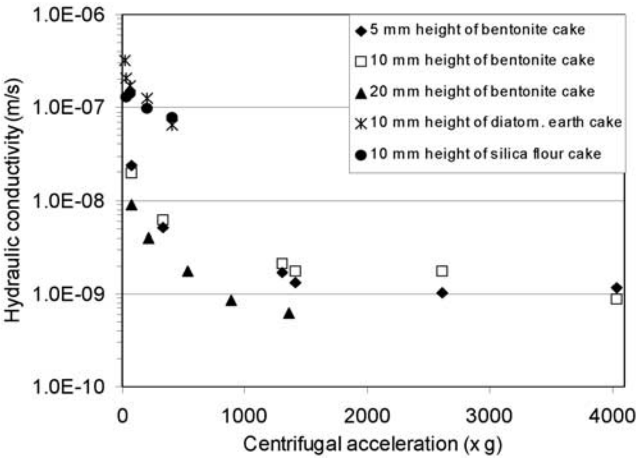

Figure 3. Variation with applied force of hydraulic conductivity (K) of flow-regulating granular material.

uniformity of the cake, between these compacting runs the sample was rotated a fraction of a turn around the vertical axis of the stationary bucket.

[7] In order to check if cracks that were sometimes observed on the top of the bentonite cake could cause nonuniformity of the flow applied to the sample we did a supplementary centrifugation test on the bentonite cake. In this test the upper reservoir with cracked bentonite rested on a layer of color indicator silica gel. After the run we observed a uniform change of color of the silica gel, indicating uniformity of flow.

[8] With increasing centrifuge speed the hydraulic conductivity decreases because of compaction, especially for cakes of bentonite or diatomaceous earth. The values of hydraulic conductivity obtained using the same cake of diatomaceous earth range from 3.2×10^{-7} m/s at 16 g to 6.5×10^{-8} m/s at 403 g. With a single cake of bentonite the hydraulic conductivity ranges from 2.0×10^{-8} m/s at 78 g to 8.9×10^{-10} m/s at 4040 g. Thus the use of a compressible granular medium permits this additional means of adjusting the flux, calibrated according to measurements as shown in Figure 3.

[9] We tested this method with consolidated rock samples without retainers because the samples alone were strong enough to support the upper reservoir. The unsaturated state of the sample prevented seepage out the sides. Sample retainers would be necessary for granular media or if saturated measurements are also needed. We chose the 80-mm-diameter sample size because it is about as large as is practical for use in a 1-L centrifuge bucket. Larger geocentrifuges could accommodate larger samples with appropriately sized experimental apparatus.

[10] A test starts with nearly saturated samples that are subjected to a series of runs of equal duration, of equal speed, and with the same configuration of the flow-controlling reservoir. Evaporation is minimized by covering the sample during the periods when it is not within the high-humidity environment of the rotating windscreen that surrounds the buckets in the centrifuge chamber. When the flow becomes quasi-steady, judged from the constancy of sample weight, the average flow rate can be used to calculate hydraulic conductivity by Darcy's law (equation (1)). The sample weight is considered constant when it varies by no more than 0.01 g from one centrifuge run to the next. This range represents a small fraction of the total weight (typically about 1000 g) of the sample. The matric potential and water content of the sample in the quasi-steady state are determined, respectively, by means of a tensiometer brought into contact with the sample's upper face after the flow-controlling reservoir has been removed and by dividing the total volume of water in the sample by the known sample volume. Under carefully selected operating conditions the matric potential is nearly uniform within the sample except near the lower face, so measurement at the upper face approximates the average. In our tests the tensiometer contact was judged to be adequate because the device correctly read zero when the sample was saturated and gave replicable, stable, negative readings when the sample was unsaturated. Because a mechanically stiff electronic transducer was used to measure the pressure within the tensiometer, the water exchange between the sample and tensiometer was negligible. Alternative ψ-measuring techniques can be used if the values are outside of tensiometer range.

[11] The values of matric potential and the sample average water content are associated with the hydraulic conductivity determined in the series of runs. Other details of procedure and interpretation, including the issue of centrifugal force being great enough to constitute the dominant driving force, are the same for the steady state centrifuge method [*Nimmo et al.*, 2002]. Repeat measurements with different fluxes, corresponding to different speeds and cake materials, give additional experimental points needed to determine the unsaturated hydraulic conductivity and water retention curves.

3. Results and Discussion

[12] The samples used to test the method were calcarenite, a sedimentary carbonatic rock of marine origin of Plio-Pleistocene age widely found in the Mediterranean basin. In the Apulia region of southern Italy, calcarenites often constitute a thick layer of the vadose zone. The unsaturated hydraulic properties of calcarenite, little known before these experiments, are critical to the prediction of fluid flow and solute transport in these regions.

[13] Calcarenite is made of a granular skeleton of lithoclasts and bioclasts in a fine, granular, carbonatic matrix, cemented to some degree. Different lithotypes from Apulia were tested: two of them, A and B, from different depths in a single quarry and the third, M, from a quarry in another location. These lithotypes vary in their proportions of lithoclasts, bioclasts, matrix, and cement. Lithotype A is characterized by medium fine grains with grainstone type and grain-supported texture; lithotype B is a biocalcarenite characterized by more bioclasts than lithoclasts of medium size with packstone type and grain-supported texture; lithotype M is made of fine grains with a wackestone type and mud-supported texture [*Dunham*, 1962]. The average porosity ranges from 0.44 for M to 0.50 for B.

[14] As a check on the results of the QSC method, we compared them with those from another experimental test performed with *Wind*'s [1968] evaporation-driven flow method for the same lithotypes. The evaporation method as described by *Arya* [2002] has been tested and evaluated by researchers, including *Minasny and Field* [2005], *Tamari*

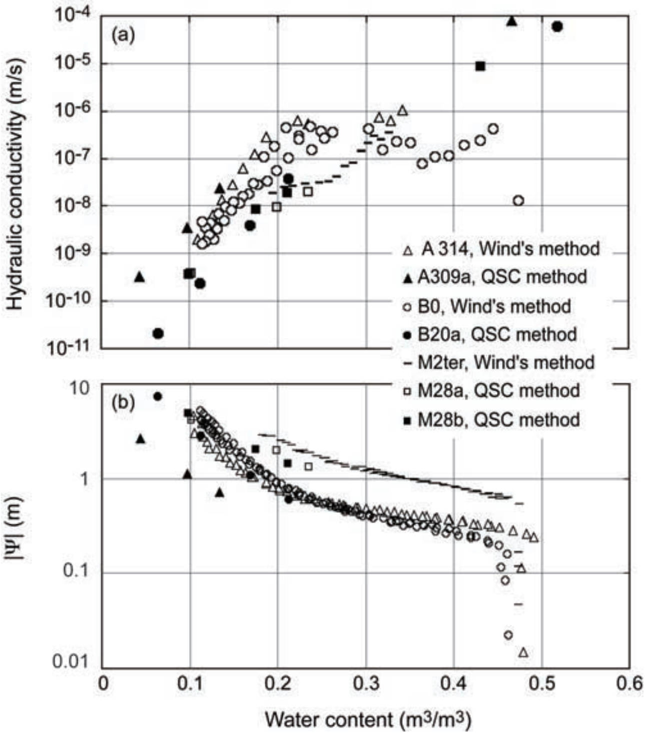

Figure 4. (a) Unsaturated *K* and (b) water retention for three lithotypes of calcarenite measured by the QSC and *Wind*'s [1968] methods. The sample names indicate the lithotype (A, B, or M) and the particular sample designation within the lithotype.

et al. [1993], and *Wendroth et al.* [1993]. It considers an initially saturated core sample, closed at the bottom, from which evaporation is allowed at the top. Water loss as a function of time is monitored by weighing. Water contents are calculated after the sample is oven-dried at the end of the experiment. Matric potential is measured using tensiometers at different depths. Calculations on the basis of this information provide both the soil water retention curve and unsaturated hydraulic conductivity.

[15] Figure 4 shows the $K(\theta)$ and $\theta(\psi)$ results. In general, the characteristics have the form expected for a medium with a sharp cutoff in pore abundance for sizes greater than a well-defined maximum. That is, there are few fractures or other large pores that would cause a more gradual decrease in θ as the medium drains from saturation. For lithotype A the results show that the two methods agree more closely for hydraulic conductivity than for water retention, although the difference is slight and may be due to small differences in the individual samples. For lithotype B, which is characterized by medium coarse bioclasts, results of the methods agree well within the range for which we have data for both methods. The water retention curves for A and B are almost the same, and the slight differences in the hydraulic conductivity curves may again be due to small differences in the individual samples. Also, for lithotype M, which is fine grained and matrix dominated, the results show that the methods agree well where there is overlap. The water retention curves are almost exactly the same, and the slight differences in the hydraulic conductivity curves may again be due to differences in the individual samples.

[16] Generally, the hydraulic conductivity results show that lithotypes A and B have greater hydraulic conductivity than lithotype M over the entire moisture range investigated. The slopes of the *K* curves are similar, and the difference for the same lithotype may be due to slight variations in the samples. An important advantage of the QSC method is that it can be used to measure values of *K* about 2 orders of magnitude lower than *Wind*'s [1968] method.

[17] The water retention curves show that for potentials of magnitude greater than 500 mm the water contents for lithotype M exceed those of A and B, which is consistent with M being finer grained and having greater water retention capacity and spread of pore size distribution. Differences between A and B are small for *Wind*'s [1968] method. These may also be better sorted, as indicated by the steeper slope in the rapidly draining portion of the curve. In general, the methods agree reasonably well.

[18] An obvious concern is whether quasi-steadiness is adequate for unsaturated hydraulic property measurements. We consider this issue in terms of the increase in measurement uncertainty caused by fluctuations in sample water content during sequential centrifuge runs. The SSC methods at best have an uncertainty of about ±8% [*Nimmo et al.*, 1987]. In general, the uncertainty is considerably more than this for low speeds, for media that are compressible, fine textured, or highly structured, or for *K* values near the high or low end of the measurable range. For the QSC method, with reasonably small decline (typically by less than a factor of 2) of head and flux during each run, the contribution of imperfect steadiness to uncertainty is negligible among these other sources of uncertainty. The usual strong sensitivity of *K* to θ works to advantage here. All of the SSC and QSC methods normally operate on the basis that for fixed speed and input flux the value of *K* is essentially determined before the measurement is begun. The purpose of carrying out the measurement is to find what value of θ corresponds to the predetermined *K* value (and normally also to find what value of ψ corresponds to this θ and this *K*). For example, during a series of QSC runs to obtain this θ value, if the head declines 50% during each run, the flow rate controlled by this head is always within ±25% of its average value. This causes at most a ±25% deviation in effective *K* during the measurement. For a typically steep $K(\theta)$ curve this would lead to an uncertainty in the measured θ of less than ±1%.

4. Conclusion

[19] On the whole, data from the quasi-steady centrifuge method are consistent with the known characteristics of the tested materials and agree well with *Wind*'s [1968] method. The observed differences between the results of the two methods may be caused by differences in terms of relative amounts of matrix, fossils, cement, or other factors between samples of the same lithotype.

[20] The QSC method expands the versatility of centrifugal unsaturated flow techniques. It reduces the cost and specialized nature of the necessary equipment, thus allowing measurement of lower hydraulic conductivity values than for gravity-driven steady methods and with higher accuracy than for other nonsteady methods of comparable practicality [*Dane and Topp*, 2002]. These advantages are achieved mainly through (1) the development of an

inexpensive, easily constructed, adjustable flow-regulating device that has no moving parts and can function in a spinning centrifuge and (2) the demonstration that the deviations from perfect steadiness achieved with this device that although somewhat greater than for apparatus of much greater complexity, in general have insignificant effect on the measured hydraulic properties.

[21] The QSC method is adaptable to essentially any centrifuge suitable for hydrogeologic applications. It can accommodate a wider range of flow rate, sample size, speed of rotation, and centrifuge radius than the UFA apparatus [*Conca and Wright*, 1998] for steady state centrifuge measurements. This adaptability to various operating conditions expands the potential for exploring unsaturated flow situations that are common in nature but have been the subject of few laboratory investigations, including macropore and fracture flow, other modes of preferential flow, and transient flow.

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