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EVALUATION OF LABORATORY DOLOMITE CORE SAMPLE SIZE USING REPRESENTATIVE ELEMENTARY VOLUME CONCEPTS

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Portions of this document may be illegible in electronic image products. Images are produced from the best available original document. Abstract. The adequacy for laboratory testing of four dolomite cores from the Culebra Dolomite of the Rustler Formation at the Waste Isolation Pilot Plant near Carlsbad, New Mexico, were evaluated using representative elementary volume (REV) theory. Gamma-ray computerized tomography created three-dimensional grids of bulk density and macropore index over volumes from 1.4x10⁻⁷ to 1.6 l. Three different methods for both volume averaging and REV analysis were applied and compared. Both density and macropore index converged to single values with increasing volume, which meets the most common qualitative definition of a REV. Statistical test results for the relatively homogeneous samples indicate that volumes larger than 1 to 7 ml have constant properties. Contrarily, a highly varied sample required 250 and 373 ml to achieve invariant density and macropore characteristics, respectively. Prismatic volume averaging was found to be better than slice averaging, while a qualitative test for the REV provided similar results as a rigorous statistical method. All cores were larger than the REV, but were significantly different from one-another. This implies that multiple cores are necessary to determine the entire range of transport properties within the rock.

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1. Introduction

Transport modeling in porous media is usually based on a continuum model that assumes a representative elementary volume (REV) [*Hubbert*, 1956; *Bear*, 1972]. Figure 1 presents a conceptual representation, or "REV curve", of the change in a porous medium property as the sample volume is increased. A constant value is encountered when the sample size is between V_{min} and V_{max} . Volumes less than the V_{min} contain only small and spatially varied portions of the property and changes are rapid due to the influence of individual pores. Volumes above V_{max} include additional morphological structures allowing the property to drift to new values, which results in large field variability. When sample volumes are sized between the region of V_{min} and V_{max} they may be expected to have only minor fluctuations in the medium property as many pores are factored into the average.

The REV is defined as the range of volumes for which all averaged geometrical characteristics are single valued functions of the location of that point and time. The size of a REV, \mathcal{V}_o determined by a selected hydrological property within a given domain \Re may be written as,

$$\mathcal{V}_{p} \ll \mathcal{V}_{min} \leq \mathcal{V}_{o} \ll \mathcal{V}_{max} \tag{1}$$

where \mathcal{V}_p is the volume of a pore. The size of the REV may vary for different physical properties [*Bear and Braester*, 1972].

In a traditional sense, the REV represents the transition from the microscopic deterministic processes of traditional fluid mechanics to the macroscopic processes of porous media flow [*Hubbert*, 1956; *Corey*, 1977]. It represents the scale at which the fluid and solid components may be treated as separate phases within a unified control volume. This allows the actual distribution of properties such as velocity and concentration within the control volume to

be neglected when developing governing equations of flow and transport. Instead, each phase is characterized with single valued state variables. Thus, a seepage velocity may characterize the complex velocity distribution of the flowing fluid.

In practice, the concept has also been applied to characterize both non-homogeneous porous media and large-scale properties in fractured media [*Bear*, 1993]. In those cases, a REV was defined for both matrix and fractures. Expansion of the definition has added complications to sampling, testing and verification procedures and has produced some debate. As *Baveye and Sposito* [1984] observed, while intuitively appealing, no known data had been presented to quantify the dimension and operational significance of the REV. Only recently have *Buchter et al.* [1994] extrapolated limited 2-D, porosity data to directly describe a 3-D REV relationship, and *Clausnitzer and Hopmans* [1999] presented a simple REV plot for glass beads. Thus, while almost every advanced groundwater textbook has a hypothetical plot of the REV there is little insight on the actual shape of the REV curve, much less wisdom into how the concept may be applied.

To some extent, the concept of the REV has become obsolete due to the recent advances in characterization of spatial variability. We no longer expect to represent an aquifer or soil with a single value of porosity and conductivity, but instead model those systems with complex stochastic representations. One may rightly ask if the REV concept holds any value beyond the narrow distinction between pore scale processes and the bulk porous medium. To that, we answer the concept is still important in the design and interpretation of laboratory column testing. Experimentalists will always be faced with the difficult decision of how big a sample to collect and test. Any quantitative theory such as the REV that can provide insight into size selection will have considerable value. Computerized tomography (CT) has been intensively used for quantifying density, macropores and liquid phase content in porous media [*Grevers et al.*, 1989; *Warner et al.*, 1989; *Anderson et al.*, 1990; *Warner and Nieber*, 1991; *Brown et al.*, 1993; *Hopmans et al.*, 1994]. More recently, improvements have been proposed for the analysis of porous medium images [*Hsieh et al.*, 1998a; *Hsieh et al.*, 1998b]. With these developments it is now possible to collect data and evaluate the various REV concepts as they apply to the adequacy of laboratory core testing. The objective of this study is to utilize CT images of complex dolomite samples to demonstrate the application of REV theory. Specifically, three different volume averaging techniques and three different REV tests will be applied and evaluated, which hopefully will give experimentalists a new tool for future research.

2. Representative Elementary Volume Tests

The REV of a homogeneous porous medium is defined in *Bear and Bachmat* [1990] as the volume range for which all averaged geometrical characteristics are single valued functions of the location of that point and time only. Let $Y(x, \mathcal{H}_i)$ be the value of a hydrological property, measured on a volume of \mathcal{H}_i with a centroid at x, within a given domain \mathfrak{R} . Within the range of the REV a property will meet

$$\frac{\partial Y(x, \mathcal{V}_i)}{\partial \mathcal{V}} \bigg|_{\mathcal{V}_i = \mathcal{V}_0} = 0$$
⁽²⁾

when $\mathcal{V}_{min} < \mathcal{V}_o < \mathcal{V}_{max..}$

Bear and Bachmat noted that Equation 2 is met by domain properties that are linear functions of position. They state, "The requirement that across the REV, any macroscopic property (whether one of void space, or of a state variable) should vary linearly, or approximately so, justifies the assignment of the averaged values taken over the REV to the latter's centroid."

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Numerical approximations to these differential definitions are needed to provide useful methods for testing the REV. Three quantitative tests are used here. The first is consistent with most qualitative definitions of the REV and quantifies when a property is not a function of the sample size. A simple finite, center-difference approximation to Equation 2 yields,

$$\nabla(Y)\Big|_{\mathcal{V}_{i}} = \frac{Y(\mathcal{V}_{i+1}) - Y(\mathcal{V}_{i-1})}{\mathcal{V}_{i+1} - \mathcal{V}_{i-1}} \approx 0$$
(3)

where V(Y) is the property gradient and the subscript *i* indicates the size of the volume the averaging was performed over. The position reference on Y is dropped, since the volume averaging procedures used are not spatially stationary as described later.

Bear and Bachmat [1990] provided the second REV test. A domain \Re can be treated as approximately homogeneous if the property fluctuation is bounded within a sufficient small range, which satisfies

$$\delta(Y_{\mathcal{V}_i}) = \frac{Y_{\max}(\mathcal{V}_i) - Y_{\min}(\mathcal{V}_i)}{\overline{Y}(\mathcal{V}_i)} <<1$$
(4)

where $\delta(Y)$ is the dimensionless range of Y, and Y_{max} , \overline{Y} and Y_{min} are the maximum, mean and minimum values of Y within \Re measured on the volume \mathcal{V}_i . The limiting magnitude of $\delta(Y)$ that will pass the test is an arbitrary judgment.

Bear and Bachmat [1990] also defined the final test of the REV size. Instead of the differential form of Equation 2 a statistical equivalent is utilized. A volume size \mathcal{V}_i is said to fall within a REV region, if $Y(x, \mathcal{V}_i)$ can be treated as a stationary random function in \mathfrak{R} for any x. By statistical inference,

$$E[Y(x, \mathcal{V}_i)] = \theta = constant$$
⁽⁵⁾

where $E[Y(x, \mathcal{V}_i)]$ is the expected value of Y. In addition, the variance must be constant within the domain,

$$Var[Y(x, \mathcal{V}_i)] = E\{[Y(x, \mathcal{V}_i) - \theta]^2\} = constant$$
(6)

Equation 6 is a result of a covariance condition for Equation 2. Volumes larger than the lower bound, V_{min} , should always have a statistically invariant property until reaching the upper limit of the REV, V_{max} .

Baveye and Sposito [1984] have developed an operational definition for the REV that takes into account the spatial characteristics of the measuring device. Since CT measurements have only minor spatial dependence beyond one voxel length [*Brown et al.*, 1993], those operational definitions reduce to volume averages similar to the proceeding equations.

3. Materials and Methods

3.1. Samples

Four dolomite cores from the Culebra Dolomite member of the Rustler Formation were used in the study. Their most important parameters are listed in Table 1. VPX-25 (core section 9) was 145 mm in diameter and 100 mm in length, while-three VPX-26 cores, C1, C2 and C3, have the dimension of 38 mm in diameter and 52 mm in length. Total core volumes were 1,650 ml for VPX-25 and 59 ml for the three VPX-26 cores. They were collected by horizontal drilling at a depth of 218 m in the air intake shaft of the US Department of Energy Waste Isolation Pilot Plant (WIPP) located near Carlsbad, New Mexico [*Lucero et al.*, 1994]. All cores were relatively solid and intact, but demonstrated the fractures, gypsum infilling, vugs, and silty dolomite typical of WIPP Rustler cores. VPX-25 showed considerable gypsum and large vugs, while all VPX-26 cores were almost entirely dolomite with only small voids. A more detailed analysis of VPX-25

composition is reported in *Hsieh et al.* [1998a]. The VPX-26 cores have varying void space with C2 the least and C3 the greatest.

The VPX-26 samples were obtained by coring a larger 144 mm core. Drilling locations were selected to obtain relatively uniform regions of the larger, more-varied core, but the distance between the core centers was no more than 50 mm. Before scanning, the VPX-26 cores were sent to an outside laboratory for measurements of porosity, mineral density and permeability. Those measurements are listed in Table 1. VPX-25 was freely drained but not dry when scanned due to restrictions for its later use. All VPX-26 cores were air-dried when scanned.

3.2. CT Imaging

The pencil-beam, ¹³⁷Cs γ -ray CT scanner of *Brown et al.* [1993] was used here. Thirtyone slices at 3 mm spacing along the axis were collected for VPX-25, while 50 or 51 slices at 1 mm spacing were scanned for the VPX-26 cores. All scans were reconstructed into 120 by 120 image arrays. Image voxel volume was 6.75 µl, (1.5 x 1.5 x 3 mm) for VPX-25 and 0.14 µl, (0.375 x 0.375 x 1 mm) for VPX-26 cores. The approximate number of image voxels inside the sample was 250,000 for VPX-25 and 400,000 for each of the VPX-26 cores. Image accuracy is indicated by the single voxel density error variance of 0.006 and 0.046 g²/ml² for VPX-25 and VPX-26 cores respectively

3.3. Bulk Density Determination

Luo and Wells [1992] have shown attenuation coefficients are insensitive to mineral composition at the gamma energy used here and are linearly related to the density. It follows that the bulk density, ρ_b may be computed by

$$\rho_b(x,y) = C \ \mu(x,y) \tag{7}$$

where *C* is a calibration factor and $\mu(x,y)$ is the point attenuation value. Internal attenuation standards are preferred for ease of use when available. For VPX 25, its large gypsum intrusions provided a convenient standard with a density of 2.32 g/ml [*Weast*, 1988]. Analysis of the images provided a gypsum attenuation of 0.0171 mm⁻¹, which produces the calibration factor, *C* = 136 mm-g/ml. A different calibration was required for the smaller collimators used with the VPX-26 cores. The attenuation distribution dolomite peak [*Hsieh et al.*, 1998a] provided a calibration point with $\mu = 0.0198$ mm⁻¹. *Kelley and Saulnier* [1990] performed extensive helium-porosity measurements on 50-mm-diameter Culebra core samples, and found the median dolomite grain density to be 2.83 g/ml with a matrix porosity of 0.11. Those values yield a bulk density of 2.52 g/ml and a calibration, C = 127 mm-g/ml.

3.4. Macropore Index

In the samples examined here, the dolomite's intrinsic mineral pores constitute the microporosity, while the fractures, vugs and silty dolomite regions make up the macroporosity. CT scanning resolution commonly available will not directly identify pores smaller than 1.0 mm from scanned images [*Warner et al.*, 1989]. However, when a voxel contains a macropore, the measured voxel attenuation will be less than the surrounding region [*Brown et al.*, 1993]. Attenuation Frequency Deconvolution and Statistical Segregation Thresholding [*Hsieh et al.*, 1998a&b] were used to define voxels containing macropores. Those methods employ image statistics, Gaussian thresholds, nearest neighbor clustering, and minimum gradient thresholds to classify image voxels into either solid rock or macropore.

While a voxel with a macropore can be defined, the size of the pore will still be undefined since it may cross several adjacent voxels and not fill any one entirely. Therefore, a

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characteristic macropore index, ϕ_{macro} is defined as the number of voxels, N_{macro} with macropores divided by the total number of voxels in the volume, N_T or

$$\phi_{macro} = \frac{N_{macro}}{N_T} \tag{8}$$

Note that the macropore index may exceed the porosity. It is an indicator of the degree of large pores in a volume and not the actual size or number.

3.5. Volume Sampling Procedures

Traditionally defined volume averaging procedures used for theoretical REV discussions obtain average property values by increasing the volume around a point that is randomly selected in the domain space. Such procedures produce average properties centered on the point held constant in space. While appealing, such operations are impractical in the limited geometry of core samples scanned at moderate resolutions. The domain is too small to randomly pick a point and then expand it uniformly to an adequate size. Thus three different volume procedures, shown in Figure 2, were tested to determine the best to use. They were 1) stacked slices, 2) continuously expanding prisms, and 3) non-overlapping prisms. For the first, a simple slice-byslice method based on the total scanned core cross section was used. The smallest volume considered was one image slice. Increasing volume sizes were obtained by stacking adjacent slices together. This method provided statistically independent samples and the largest possible domain size for analysis, the entire core.

In the second method, the largest rectangular prism inside each core was defined as the sample domain. The prism for VPX-25 was $102 \times 102 \times 93$ mm, (970 ml), while for the VPX-26 cores it was $27 \times 27 \times 52$ mm, (38 ml). Increasing sample volumes were obtained by starting at an outside corner and averaging voxel values from a continuously expanding rectangular prism.

Each volume incorporated the former. On expansion, each cube increased two voxels on a side and one in height for VPX-25, and one voxel in all three directions for the VPX-26 cores. At their maximum extension, every volume filled the entire prismatic domain. Eight REV curve realizations were collected for each core by starting at each of the prism corners. The eight realizations overlap once the sampling length exceeds one-half of the domain. This method provides REV curves comparable to the traditional definition, with the exception that the volume center is not stationary.

The final procedure collected non-overlapping, statistically independent prismatic samples. Rectangular prisms of equal volume were fit into the domain in cubic arrays. This sampling procedure was only used on VPX-25 and the volumes ranged between 0.027 and 800 ml. It provides samples over a large range of sizes, but the larger sample sizes are not generally expansions of the smaller.

3.6. Statistical Test

Evaluation of Equations 5 and 6 require a specific statistical test. Following the procedure proposed in *Till* [1974], a single-factor ANOVA *F*-test was used to check that the sample property means, E(Y), are constant as volume varies. Using the eight realizations from each core obtained by the expanding prism method, the values at a constant volume, V_i , are grouped together to obtain a mean and variance. Then the REV range, V_{min} to V_{max} , may be determined as the range where mean and variance are constant. ANOVA analysis tests both mean and variance in Equations 5 and 6.

Hartley's maximum-F test, a shorter and easier version of Bartlett's variance test, was used to test variance. For each volume, V_i a sample variance, $s^2(V_i)$ was computed. The sample

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maximum F_{max} value is then defined as the ratio of the maximum sample variance, s_{max}^2 , to the minimum variance, s_{min}^2 , over the volume range \mathcal{V}_{min} to \mathcal{V}_{max} or

$$F_{max} = s_{max}^2 / s_{min}^2 \tag{9}$$

Critical values of $F_{max}(\alpha, df1, df2)$ can be interpolated from the ν - F_{max} diagram in Pearson and *Hartley* [Table 31, 1970]. The significance level, α , is set to 5% or 0.05 for all the tests. The degree of freedom, df1, is the number of REV realizations in each group minus one, or seven in this case. Since the maximum number for df^2 in Pearson and Hartley's table is 12, we defined upper, V_{max} and lower volume bounds, V_{min} and then selected 12 or fewer volume groups, V_i uniformly through the range for the test, $(V_{min} \leq V_i \leq V_{max})$. Starting at the smallest size, with each rejection of the homogeneity variance test, V_{min} is enlarged and the test repeated until the null hypothesis is accepted. At that point, the lower bound is accepted as the true \mathcal{V}_{min} . Defining the upper REV bound, V_{max} , is problematic with small cores. With the expanding prism method, once the upper bound exceeds 1/8 of the domain, the individual sample volumes will overlap at the center of the domain. The variance between samples is reduced with increasing overlap. Therefore, while applying this sampling-with-replacement procedure, the test will ultimately fail for large sample sizes. The upper REV bound will be a minimum estimate of V_{max} . If nonoverlapping prisms are used, the number of independent samples becomes small, which produces a similar problem.

4. Results

4.1. Sample Variation

Figure 3 presents representative CT images for the four cores. VPX-25 has the greatest variation with large vugs and visible gypsum intrusions. The VPX-26 cores show less variation, but distinct differences within them can be seen. Average slice bulk density and macropore

index are plotted in Figure 4. Variations in each parameter may be seen in each core. VPX-25 bulk density is the highest due to it being scanned wet. CT measurements were compared to the gravimetric analyses and are listed in Table 1. CT core bulk density was determined by taking the mean of the slice values, while total porosity may be computed for the dry cores by

$$\phi = 1 - \rho_b / \rho_p \tag{10}$$

where ρ_p is the particle density assumed as 2.83 g/ml. Gravimetric measures for VPX-25 that require oven drying were not obtained because the core was being preserved unaltered for future testing. Density estimated by CT was 0.03 to 0.05 g/ml lower than gravimetric (about 2%). The error may be attributed to loss of material during handling, miscalibration of *C*, and/or CT artifacts. Porosity is over-predicted by 0.01 to 0.02 ml/ml due to the under-prediction of ρ_b . In any case, those small errors are considered acceptable for this analysis.

4.2. REV Curves

Figure 5 presents 32 REV density curves obtained by the expanding prism method. Volume is plotted on a logarithmic scale to allow the full range to be displayed. Each core has eight realizations that converge to the total prismatic domain size. At the smallest scales, density varied from zero to that of solid dolomite. These substantiate the REV curve postulated by *Hubbert* [1956]. The large number of curves that start at or near zero is an artifact resulting from the corners being near the relatively rough core edge. None of the curves demonstrate the wild swings predicted by the traditional REV plot in Figure 1. This trend was also noted by *Buchter et al.* [1994], and we believe the traditional plot implies too much oscillation at intermediate volumes. While covering sever orders of magnitude, these plots do not approach the primary porosity pore scale. However, *Clausnitzer and Hopmans [1999]* have imaged glass beads at the pore dimension and found similar shaped REV curves.

4.3. Volume-Average Method Comparison

Three different volume-averaging methods are compared in Figure 6, which presents bulk density of VPX-25 over the range of zero to 500 ml. The three methods gave comparable results and no clear trend in the density range at various volumes can be seen.

Expanding prism results for bulk density and macropore index are plotted in Figure 7 for all four cores. Plot volume is limited to the range with the most parameter variation, zero to 30 ml. These curves should only be considered different realizations of the REV plot for the core since the sampled volumes start to overlap after about 5 ml. At large volumes, the range in density and macropore index for individual cores have narrowed, but the range over all cores is relatively large. In particular, the macropore index shows substantial variation with the total range roughly equal to the mean at 15 ml. VPX-25 shows considerably more variation than the smaller cores due to the gypsum intrusions and large vugs. Figures 8 present the gradient, $\nabla(Y)$ of bulk density and macropore index with respect to volume as computed by Equation 3. Surprisingly, all the cores demonstrate a similar relationship. By 10 ml, the bulk density gradient falls roughly within ± 0.01 g/ml² and the macropore index gradient within ± 0.01 ml⁻¹.

Figure 9 presents the variation, $\delta(Y)$ of bulk density and macropore index, as calculated by Equation 4 using the stacked slice volume method for the VPX-26 cores. The magnitude of the macropore index variation is greater than the bulk density due to the larger range compared to its mean value. For the smaller cores the bulk density variation declines rapidly to 5 ml, and then follows a roughly slow linear decline through the rest of the volume range. The macropore index variation follows a similar pattern, except for VPX-26-C2, which has a much smaller initial decrease. Figure 10 shows VPX-25 variation, $\delta(Y)$ for both parameters as calculated for by all three volume-averaging methods. The prismatic data sets are similar, but the stacked slice method provides substantially different results. This is due to their geometry contrast. VPX-25 has significant gypsum infilling and solid dolomite regions, which from surface mapping are 10 to 30 mm on a side. A slice with a diameter of 144 mm and a thickness of 3 mm will always cut across such features and average them with the surrounding material, thus it has a constant characteristic length equal to the core diameter, 144 mm. On the other hand, prisms will capture some of these features without the surrounding matrix. Their characteristic length is roughly the cube root of the volume, which provides a length scale of 10 to 90 mm for the volumes plotted. VPX-26 small cores did not demonstrate the insensitive stacked-slice variance since they were more homogeneous and their diameter to length ratio was larger.

4.4. **REV Test Comparison**

Table 2 lists the results of Hartley's test for equal variance. Homogeneous variance over a large range was found for all VPX-26 cores. Each of the minimum volumes passed the constant mean test as listed in Table 3. V_{min} for bulk density and macropore were equal for each of the small cores, with a value of 1.0, 2.2 and 7.1 ml for C1, C2 and C3 respectively. The fact that the two properties had similar V_{min} indicate the properties are related in those cores. A V_{max} for the small cores could not be found before sample overlap reduced the variance to the point that the test of constant variance failed. Thus, the upper bound should be considered a minimum value for V_{max} . For VPX-25 a V_{min} could not be found that was smaller than 1/8 of the domain. Ignoring the statistical dependence of overlapping prisms and continuing to expand the prisms indicated a lower REV size of 250 ml for density property and 373 ml for macropore index. The true V_{min} will be greater.

A direct comparison of the three methods to evaluate the REV, $\nabla(Y)$, $\delta(Y)$ and E(Y), is not possible since the first two are qualitative. The most straight forward and easiest to evaluate is $\nabla(Y)$. Its criterion of $\nabla(Y) \approx 0$ directly quantifies the variation in the parameter's units per volume. It provided $\mathcal{K}_{min} \approx 5$ to 10 ml for all cores, including VPX-25. It is clearly, the least sensitive to sample variation. The $\delta(Y)$ test is almost as easy to evaluate and gave similar \mathcal{K}_{min} ranges as $\nabla(Y)$ for the small cores, (if you consider $\delta(\phi_{macro}) = 0.5 << 1$). For VPX-25 it gave large $\mathcal{K}_{min} \approx 200$ ml. However, the $\delta(Y) << 1$ criterion, while an attractive normalization, may not be appropriate. As the ratio of the range to mean, any parameter such as ϕ_{macro} that has a large range will have a difficult time passing. A more appropriate criterion may be $\delta(Y) = constant$, which is also consistent with the E(Y) test. The statistical test, E(Y) is difficult to evaluate, but does provide quantitative results. Surprisingly, it gave the smallest estimate of \mathcal{K}_{min} on the VPX-26 cores. Of course, that estimate is a function of the confidence level used.

4.5. Evaluation of Sample Size

At this point the issue of laboratory sample size may be addressed. If density and macropore index are adequate to characterize transport processes, it can be concluded that any sample less than V_{min} would be prone to high variability. Figures 8 and 9 show that samples can have dramatic change in mean properties for volumes less than about 5 ml. Below that dimension, a transport experiment with the same piece of rock could have significantly different results if its size was changed slightly. With greater volumes mean properties will change slowly. As listed in Tables 2 and 3, for the VPX-26 cores V_{min} is no more than 5 to 10 ml, but may be as small as 1 or 2 ml for C1 and C2. Even though Figure 4 shows that properties change throughout the cores, each of the VPX-26 cores is roughly 10 to 60 times V_{min} . That implies experiments should reflect bulk core properties.

For VPX-25 \mathcal{V}_{min} is poorly defined, but it appears to be between 200 and 400 ml as shown in Figure 10 and Tables 2 and 3. That implies the core itself is only three to six times larger, and an experiment may be unduly subject to the properties of a small region. Larger cores may be more appropriate for samples as varied as VPX-25.

5. Conclusions

Computerized tomography provided small-scale density and macropore index for four dolomite cores. Integration of the small-scale density and macropore index over larger and larger volumes provided a quantitative measure of scale-dependent property variation. Sample averaging volumes spanned seven orders of magnitude from 0.14 μ l to 1.6 l. Computed REV curves show their traditional representation have too much oscillation at intermediate volumes.

Volume averaging with slices provided significantly less variation than the two prismatic volume techniques. While easy to perform, slice averaging will have a minimum characteristic length equal to of the core diameter. The two prismatic methods gave similar results, and either should be adequate.

The three REV tests presented provided different values for the REV size. The two qualitative tests, $\nabla(Y)$ and $\delta(Y)$, provided similar ranges of 5 to 10 ml for the lower REV bound on the small cores. On the highly varied VPX-25 sample, the two differed, with $\delta(Y)$ providing results similar to the constant mean test, E(Y). That more rigorous test of constant mean and variance found VPX-26 cores with regions larger than 1 to 7 ml had statistically invariant bulk density and macropore index. In contrast, the highly varied VPX-25 sample required 250 to 373 ml to achieve invariant bulk density and macropore index, respectively. Considering that the $\delta(Y)$ test gave similar results as E(Y), with much less effort, it is recommended for normal use. However, a more appropriate criterion may be $\delta(Y) = constant$, which is also consistent with the test of constant mean and variance. In all cases the upper limit of the REV, V_{max} was

conservative and the true value may be larger. Finding the true upper limit will be problematic in most cores.

Comparing the core volumes with their respective the REV size confirms all cores are appropriate for conducting column tracer experiments, but the highly varied as VPX-25 may be at the lowest acceptable size. Finally, it is apparent that each of these cores is significantly different from the rest. Thus, no single core can be expected to represent the transport properties of the entire Culebra unit. Characterization of such varied rock requires multiple laboratory experiments and appropriate modeling.

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- Anderson, S.H., R.L. Peyton and C.J. Gantzer, Evaluation of constructed and natural soil macropores using x-ray computed tomography, *Geoderma*, 46, 13-29, 1990.
- Baveye, P. and G. Sposito, The operational significance of the continuum hypothesis in the theory of water movement through soils and aquifers, *Water Resour. Res.*, 20(5), 521-530, 1984.
- Bear, J., Dynamics of Fluids in Porous Media, American Elsevier, New York, 15-26, 785 pp., 1972.
- Bear, J. and C. Braester, On the flow of two immiscible fluids in fractured porous media, Fundamentals of Transport Phenomena in Porous Media, Proc., First Symp. of, Elsevier, New York, pp. 177-202, 1972.
- Bear, J. and Y. Bachmat, Introduction to Modeling of Transport Phenomena in Porous Media, Kluwer, Boston, 16-28, 553 pp., 1990.
- Bear, J., Modeling flow and contaminant transport in fractured rock, in *Flow and Contaminant Transport in Fractured Rock*, edited by J. Bear, C.F. Tsang, and G. de Marsily, Academic Press, New York, pp. 1-37, 1993.
- Brown, G.O., M.L. Stone, and J.M. Gazin, Accuracy of gamma ray computerized tomography in porous media, *Water Resour. Res.*, 29(2), 479-486, 1993.
- Buchter, B., C. Hinz, and H. Fluhler, Sample size for determination of coarse fragment content in a stony soil, *Geoderma*, 63(3/4), 265-275, 1994.
- Clausnitzer, V., and J.W. Hopmans, Determination of phase-volume fractions from tomographic measurements in two-phase systems, *Advances in Water Res.*, 22(6), 577-584, 1999.

- Corey, A.T., Mechanics of Heterogeneous Fluids in Porous Media, Water Resources Publications, Fort Collins, Colorado, 9-10, 259 pp., 1977.
- Grevers, M.C.J., E. De Jong, and R.J. St. Arnaud, The characterization of soil macroporosity with CT scanning, *Can. J. Soil Sci.*, 69(3), 629-637, 1989.
- Hopmans, J.W., M. Cislerova, and T. Vogel, X-ray tomography of soil properties, in *Tomography of Soil-Water-Root Processes*, edited by S.H. Anderson and J.W. Hopmans, pp. 17-28, SSSA Special Publication No. 36. Am. Soc. Agronomy-Soil Sci. Soc. Am., Madison, Wisconsin, 1994.
- Hsieh, H.T., G.O. Brown, M.L. Stone, and D.A. Lucero, Measurement of porous media component content and heterogeneity using gamma ray tomography, *Water Resour. Res.*, 34(3), 365-372, 1998a.
- Hsieh, H.T., G.O. Brown, and M.L. Stone, Quantification of porous media using gamma ray tomography and statistical segregation threshold, *Trans. Amer. Soc. Agric. Eng.*, 41(6), 1697-1706, 1998b.
- Hubbert, M.K., Darcy's law and the field equations of the flow of underground fluids, *Journal of Petroleum Technology*, 8, 222-239, 1956.
- Kelley, V.A., and G.J. Saulnier, Jr., Core Analysis for Selected Samples from the Culebra Dolomite at the Waste Isolation Pilot Plant Site, Contractor Report SAND90-7011, Sandia National Laboratories, Albuquerque, New Mexico, 1990.
- Lucero, D.A., F. Gelbard, Y.K. Behl, and J.A. Romero, Test plan for laboratory column experiments for radionuclide adsorption studies of the Culebra Dolomite Member of the Rustler Formation at the WIPP site, WIPP test plan, *TP95-03*, Sandia National Laboratories, Albuquerque, New Mexico, 1994.

- Luo, X. and L.G. Wells, Evaluation of gamma ray attenuation for measuring soil bulk density: Part 1. Laboratory investigation, *Trans. Am. Soc. Ag. Eng.*, 35(1), 17-26, 1992.
- Pearson, E.S. and H.O. Hartley (eds.), *Biometrika Tables for Statisticians*, Reprinted from 3rd ed. with additions, Cambridge University Press, Cambridge, p. 202, 1970.
- Till, R., Statistical Methods for the Earth Scientist- an Introduction. p. 104-112, John Wiley & Sons, New York, 104-106, 154 pp., 1974.
- Warner, G.S., J.L. Nieber, I.D. Moore, and R.A. Geise, Characterizing macropores in soil by computed tomography, *Soil Sci. Soc. Am. J.*, 53(3), 653-660, 1989.
- Warner, G.S. and J.L. Nieber, Macropore distribution in tilled vs. grass-surfaced cores as determined by computed tomography, in *Preferential Flow*, Proc. Natl. Symp., edited by T.J. Gish and A. Shirmohammadi, pp. 192-201, Am. Soc. Ag. Eng., St. Joseph, Michigan, 1991.
- Weast, R.C. (Ed.), Handbook of Chemistry and Physics, pp. F-1, 1st Student ed., CRC Press, Inc., Boca Raton, FL, 1988.

Table 1. Measured cores properties.

| Property/Core | VPX-25 ¹ | VPX-26-C1 | VPX-26-C2 | VPX-26-C3 |
|--------------------------------------|---------------------|--------------------------|--------------------------|---------------------------------------|
| Geometry | | ······ | | • ••••• •• •• •• •• •• •• |
| Diameter (mm) | 145 | 38 | 38 | 38 |
| Length (mm) | 100 | 52 | 52 | 52 |
| Gravimetric Measures ¹ | | | | · · · · · · · · · · · · · · · · · · · |
| Permeability ² (m^2) | | 1.14 x 10 ⁻¹⁴ | 1.34 x 10 ⁻¹⁵ | 2.26 x 10 ⁻¹⁴ |
| Dry bulk density ³ (g/ml) | | 2.34 | 2.46 | 2.19 |
| Porosity (%) | | 17.3 | 13.2 | 22.4 |
| CT Measures | | | | |
| Bulk density (g/ml) | 2.46 (Wet) | 2.31 | 2.41 | 2.14 |
| Porosity (%) | 11.2 | 18.4 | 14.8 | 24.6 |

¹ Gravimetric measures for VPX-25 are not available.

² Measured by steady-state gas permeameter.

³ Computed from core volume and weight.

| Property/Core | df_2 | F _{max} | $F_{max}(\alpha, df1, df2)$ | \mathcal{V}_{min} - \mathcal{V}_{max} (ml) ^{1,2} |
|-----------------|--------|------------------|-----------------------------|---|
| Macropore index | | | | |
| VPX-25 | 7 | 9.73 | 11.8 | 373 – 729 |
| VPX-26-C1 | 12 | 10.6 | 15.8 | 1.0-15.6 |
| VPX-26-C2 | 12 | 11.6 | 15.8 | 2.2 - 15.6 |
| VPX-26-C3 | 12 | 13.6 | 15.8 | 7.1 – 15.6 |
| Density | | | <u> </u> | |
| VPX-25 | 10 | 13.2 | 14.3 | 250 - 729 |
| VPX-26-C1 | 12 | 12.7 | 15.8 | 1.0 - 15.6 |
| VPX-26-C2 | 12 | 13.6 | 15.8 | 2.2 - 15.6 |
| VPX-26-C3 | 12 | 9.9 | 15.8 | 7.1 - 15.6 |
| | | | | |

Table 2. Hartley's test for the equal variances for one VPX-25 and three VPX-26 cores.

Confidence level, α is 0.05 and the first degree of freedom, df_1 is seven.

¹Note a Ψ_{min} passing the test for VPX-25 could only be found when statistical independence of samples had been lost.

² The V_{max} listed is conservative. The true V_{max} may be larger.

| Property/Core | df_1 | df_2 | F | F(α, df1, df2) | \mathcal{V}_{min} (ml) ¹ |
|-----------------|--------|--------|---------|----------------|---------------------------------------|
| Macropore index | | | <u></u> | | |
| VPX-25 | 6 | 49 | 0.41 | 2.29 | > 373 |
| VPX-26-C1 | 29 | 210 | 0.58 | 1.52 | 1.0 |
| VPX-26-C2 | 23 | 168 | 0.12 | 1.59 | 2.2 |
| VPX-26-C3 | 11 | 84 | 0.06 | 1.90 | 7.1 |
| Density | | | | / | |
| VPX-25 | 9 | 70 | 0.24 | 2.02 | > 250 |
| VPX-26-C1 | 29 | 210 | 0.78 | 1.52 | 1.0 |
| VPX-26-C2 | 23 | 168 | 0.43 | 1.59 | 2.2 |
| VPX-26-C3 | 11 | 84 | 0.06 | 1.90 | 7.1 |
| | | | | | |

Table 3. ANOVA One-factor F-test results for constant mean properties. Confidence level, α is 0.05.

¹ Note a Ψ_{min} passing the test could only be found for VPX-25 when statistical independence of samples had been lost.

Figure Titles

Figure 1. Conceptualization of the Representative Elementary Volume.

Figure 2. Schematic of sampling procedures used.

- Figure 3. Typical CT images from four cores. a) VPX-25-9, slice 15; b) VPX-26-C1, slice 29;c) VPX-26-C2, slice 18; d) VPX-26-C3, slice 11. Note 3a is plotted at 1/4 the scale of 3 b, c, and d.
- Figure 4. Individual slice bulk density and macropore index along each core.
- Figure 5. Thirty-two REV density curves obtained by the expanding prism method on all four cores.
- Figure 6. Comparisons of three different volume-averaging methods for core VPX-25.
- Figure 7. Mean volume properties calculated by the expanding prism method for all for cores. a) Bulk density; b) Macropore index.
- Figure 8. Gradient of mean volume properties using expanding prism data for all four cores. a) Bulk density b) Macropore index.
- Figure 9. Variation in mean bulk density and macropore index using stacked slice data for VPX-26 cores.
- Figure 10. Variation in mean bulk density and macropore index using all three volumeaveraging methods for VPX-25.