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SURFACE PERMEABILITY OF NATURAL AND ENGINEERED POROUS BUILDING MATERIALS

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31 Abstract

32 Characterization of surface gas permeability measurements on a variety of natural and 33 engineered building materials using two relatively new, non-destructive surface permeameters is 34 presented. Surface gas permeability measurements were consistent for both laboratory and field 35 applications and correlated well with bulk gas permeability measurements. This research 36 indicates that surface permeability measurements could provide reliable estimates of bulk gas 37 permeability; and due to the non-destructive nature and relative sampling ease of both surface 38 gas permeability tools, it is possible to quantify the range of the spatial autocorrelation, 39 heterogeneity, and anisotropy in porous building materials and their degree of degradation from 40 weathering.

41

42 <u>Key words</u>: building materials; porous media; surface permeability; non-destructive techniques;

43 weathering; autocorrelation; geostatistics

45 **1. INTRODUCTION**

46 Fluid transport through porous materials is an area of study relevant to many scientific 47 and engineering disciplines such as hydrogeology, geoenvironmental engineering, petroleum 48 engineering, chemical engineering, physics, biology and medicine (e.g. Dandekar, 2006; Dullien, 49 1992; Gladden et al., 2003; Jang et al., 2003; Steele and Heinzel, 2001). Understanding of 50 permeability and its spatial variability is critical for reliable characterization and prediction of 51 fluid transport. As a result, there is increasing interest in quantifying the aqueous and gaseous 52 permeabilities of natural and engineered porous materials for many practical applications 53 including the durability of porous building materials.

54 Surface permeability measurements of porous building materials are useful in many 55 scenarios. For example, moisture and air movement throughout a building is dependent on the 56 pore structures, porosities and permeabilities of the building materials used in the structure. If the 57 material bulk permeability of an existing structure is needed, coring the material would be required to determine its bulk permeability in a laboratory setting. In such instances, 58 59 nondestructive surface permeability measurements, already correlated to bulk permeability 60 measurements, would be more convenient. Another example is the exposure to contaminating 61 agents such as acid rain, toxic spills, and possible chemical and biological agent release, to name a few. After being exposed to such contaminating agents, the demolition or removal of structures 62 may not be viable options, especially for those of historic and cultural significance and 63 64 emergency facilities. In situations where a building material/structure cannot be removed or 65 destroyed, it is highly likely that only the surface of the materials will be available for nondestructive rapid response measurements and characterization. Therefore, understanding how
measured surface permeability correlates to bulk permeability, fluid transport, and the durability
of building materials is instrumental to the development of effective decontamination strategies.
This research evaluated the surface and bulk permeabilities of typical porous building materials,
including natural stones (e.g., sandstones and limestones) and engineered materials (e.g. bricks
and concrete).

72 Non-destructive and cost-efficient mini or probe permeametry has become an important 73 tool, which can quickly provide data for both ex situ laboratory and in situ field permeability 74 measurements (Chandler et al., 1989; Davis et al., 1994; Dreyer et al., 1990; Dutton and Willis, 75 1998; Eijpe and Weber, 1971; Fossen et al., 2011; Goggin, 1988; Goggin et al., 1993; Hornung and Aigner, 2002; Huysmansa et al., 2008; Iversen et al., 2003; Rogiers et al., 2011; Sharp Jr. et 76 77 al., 1994). Valek et al. (2000) developed a surface permeability device to examine the difference 78 in permeability of weathered versus cleaned historic sandstone masonry. Filomena et al. (2013) 79 studied sandstone using two permeameter cells suitable for measuring bulk gas permeability in 80 the laboratory and using two mini permeameters designed for measuring surface gas 81 permeability in the field, and found the two to be strongly correlated. Studies that evaluate 82 correlations between surface and bulk permeabilities across a wide range of materials are not 83 available in the literature. Similarly, literature is lacking that correlates data collected using 84 laboratory surface permeameters and those available for field measurements across a wide range of materials. 85

86 Most building materials are porous to some degree and have inherent heterogeneities and 87 anisotropy. For example, in natural materials such as sandstone, stratification often results from 88 the depositional processes that occur during formation producing strong directional anisotropy. 89 Whereas, concretes, the most frequently used engineered building materials (Lomborg, 2000), 90 are typically made of similar constituents; however, the variations in mix proportions and curing 91 times result in more heterogeneous pore structures. Many building materials are regularly 92 exposed to weathering and other degradation processes that are initiated along the surface; and 93 the permeating properties have been recognized as critical for their durability (e.g. Figg, 1972; 94 Zaharieva et al., 2003).

95 In this paper, we investigate surface gas permeability for a broad range of building 96 materials using an AutoScan II surface gas permeameter, which is suitable for laboratory 97 surface permeability measurements at the sub-millimeter scale. Surface gas permeability was 98 measured over a uniform grid on about 20 different building materials. A majority of these 99 datasets were then compared to those collected with a different permeameter more suitable for 100 field applications, TinyPerm II. These two permeameters are unique in that they are non-101 destructive and capable of measuring a wide range of surface gas permeabilities. Subsequently, 102 we examined the correlation between surface permeability and bulk gas permeability. To 103 assess the effectiveness of these techniques in characterizing the surface permeability 104 measurements, sample data were analyzed geostatistically to extract the spatial autocorrelation, 105 anisotropy and heterogeneous features inherent to many building materials. Furthermore, 106 surface permeabilities for a subset of the materials studied were measured before and after 107 weathering (simulated freeze-thaw in laboratory), and their applicability for assessing the108 extent of weathering and degradation was evaluated.

In summary, the specific study objectives were to generate a data set of surface and bulk gas permeability measurements on a variety of natural and engineered porous building materials, and assess whether (1) the surface gas permeability measurements of the two permeameters are comparable, (2) surface gas permeability can reliably estimate bulk gas permeability, and (3) the two devices can be used to characterize the building material structure (e.g. spatial autocorrelation, heterogeneity, and anisotropy) and the degree of degradation from weathering (e.g., freeze-thaw).

116

117 2. STUDY MATERIALS

118 This study evaluated both natural (i.e., granite, sandstones, and limestones) and 119 engineered (i.e., concretes, cement, asphalt, and bricks) porous building materials. The majority 120 of the concretes and cementitious mixtures were hand mixed until the ingredients appeared 121 uniformly mixed, subsequently poured into cylindrical molds (70-78 mm in diameter) or small 122 slabs, and moist cured for a minimum of 28 days and in many cases much longer. All concrete 123 surfaces were 'finished' by hand screeding (removing defects and creating a smooth, finished 124 surface), as is typically done in practice. Cylindrical specimens of natural stone were cored from 125 larger pieces. Cylindrical brick, paver, and in some cases, concrete specimens, were cored from 126 commercially available slabs of these materials. The cylindrical specimens were generally either 127 70 mm or 78 mm in diameter with heights ranging from 40 to 100 mm.

Initial results revealed that both natural weathering and the concrete screeding process affected the surface permeability. Therefore, several centimeters (between 1 and 4 cm) of material were removed from the top and bottom screeded or weathered portions of specimens to retrieve the interior material as the test specimen and create specimens of equal height. A water saw (i.e., table saw fitted with a constant stream of water) was used to help avoid overheating the specimen during cutting. Interior specimens extracted from cores are explicitly identified in the text.

135 2.1 Natural Materials

136 The *natural* materials examined in this study include: (1) Ohio Sandstone, (2) Arkose 137 Sandstone, (3) Portland Brownstone, (4) Indiana Limestone of differing colors, (5) Indiana 138 Limestone, (6) Bluestone Sandstone from a local landscaping company, and (7) Granite of 139 unknown origin. Specimens of materials 1 through 4 were acquired from Granite Importers, Inc., 140 and specimens of materials 5 and 6 were acquired from Indiana Limestone Company and a local 141 landscaping company, respectively. In some cases, materials of the same type but from different 142 sources were tested; they are denoted as Specimen 1, Specimen 2, etc. In addition, surface 143 permeability measurements on a slab specimen of (8) Berea Sandstone are also reported here. 144 These raw Berea Sandstone data were collected by New England Research (2013).

145

2.2 Engineered Materials

146The *engineered* materials examined in this study include: (1) Quickrete Ready Mix147Concrete, (2) 3,000 psi Concrete, (3) 5,000 psi Concrete, (4) Sakrete High Strength Concrete, (5)

Portland Cement (with no added aggregate), (6) premade D04 Concrete, , (7) Red Clay Brick, (8) Red Colored Concrete Paver, (9) Tan Colored Concrete Paver, (10) Concrete Paver, (11) Asphalt recovered from a road excavation, and (12) Concrete of Unknown Origin. Specimens of materials 1 through 5 were prepared in laboratory, specimens of D04 concrete were supplied by the Idaho National Laboratory, specimens of Red Clay Brick were obtained from a brick yard in Vermont, USA, and specimens of materials 8 through 10 were purchased from a hardware store.

The specific compositions of some materials are unknown, although they were selected because they represent commonly used porous building materials. When materials of the same nominal type or composition, but from different batches or sources, were tested, they are denoted as Specimen 1, Specimen 2, etc. Concretes of specified compressive strengths were prepared using recommended recipes, but the strengths were not confirmed through compression testing.

160 **3. METHODS**

Surface gas permeability was measured using two relatively new devices, AutoScan II and TinyPerm II. Relevant ASTM standards do not yet exist for these devices. The testing procedures for the two permeameters and for bulk gas permeability measurements are described below. In addition, a subset of the specimens was subjected to 30 water-saturated, freeze-thaw cycles to evaluate the effects of weathering on surface permeability.

166 **3.1** Surface gas permeability using AutoScan II

167 Fine-scale gas permeability was measured on specimen surfaces in a laboratory setting 168 using the automated surface gas permeameter apparatus AutoScan II (Figure 1) developed by 169 New England Research, Inc., White River Junction, VT, USA (New England Research, 2014). 170 The entire process is computer-controlled via a connected work station; the user defines the 171 measurement locations along a high-precision, 2-D grid as well as the target pressure and flow 172 rates. Measurement data are stored on the work station, and can be processed and plotted with 173 little user interaction. The device is capable of measuring permeability ranging from 0.1 milliDarcy (mD) (9.87 x 10⁻¹⁷ m²) to 3 Darcy (D) (2.96 x 10⁻¹² m²) (New England Research, Inc., 174 175 2008). Multiple specimens can be tested in a single run and a different measurement grid can be 176 specified for different specimens. The measurement spacing can be as small as 0.5 mm. The 177 testing presented here employed grid spacings ranging between 1 mm and 5 mm. The specific 178 interval for a specimen depended upon the size of the measurement surface area. The 179 permeability probe (Figure 1b) has a tip seal made of soft rubber that is pressed against the 180 specimen at the specified sampling location to prevent leakage between the probe and the 181 specimen surface as pressurized gas flows down through the specimen to the atmosphere in a 182 roughly hemispherical path as depicted in Figure 1c. Nitrogen gas was used in this work per the 183 manufacturer's recommendation. Once steady-state flow through the specimen is achieved, 184 Darcy's law is employed to determine the surface gas permeability using the following equation 185 (neglecting gas slippage and high velocity flow effects):

186
$$K_{apparent} = \frac{2Q\,\mu P_{atm}}{a\,G_o\left(P^2 - P_{atm}^2\right)},\tag{1}$$

187 where $K_{apparent}$ is the apparent permeability (L²), Q is the flow rate of gas at P_{atm} (L³/T), μ is the 188 gas viscosity (M/LT), P is the injection pressure of the gas (M/LT²), P_{atm} is the atmospheric 189 pressure (M/LT²), a is the internal tip-seal radius (L), and G_o is a geometrical factor 190 (dimensionless).

191 For this work, the apparent permeability was determined using the manufacturer's default settings for gas viscosity (1.78 x 10^{-5} Pa·s), internal tip-seal radius (0.005 m), and a geometrical 192 193 factor of 0.0059. AutoScan II varies the gas injection pressure (P) and the flow rate (Q) for each reading until steady-state conditions are achieved before calculating the $K_{apparent}$. The initial P 194 195 and Q can be adjusted to achieve steady-state conditions more quickly and the maximum time 196 limit for a sample reading can be specified such that the device will not record a measurement 197 unless steady-state conditions have been reached in the allotted time. In cases where permeability 198 varied greatly across a single specimen and measurements could not be obtained in the time 199 allotted, the specimens were rerun with different initial P and Q values.

200 The measured apparent permeability is then corrected (K_k) for gas slippage effects at low 201 gas injection pressures:

202
$$K_{k} = \frac{K_{apparent}}{1 + \left(\frac{B}{P_{mean}}\right)},$$
 (2)

where *B* is the Klinkenberg slip factor and P_{mean} is the mean pressure measurement $P_{mean} = (P + P_{atm})/2$ (Klinkenberg, 1941).

206

The permeability computed using equation (2) is further corrected (K_o) for high velocity flow effects (turbulence and inertial) using a Forchheimer factor (F_h) (Goggin et al., 1988):

207
$$\frac{1.0}{K_o} = \frac{1.0}{K_k} - F_h \cdot Q , \qquad (3)$$

208 AutoScan II determines the Klinkenberg and Forchheimer factors at each sample location.

209 **3.2** Surface gas permeability using TinyPerm II

210 The surface gas permeability was also measured using TinyPerm II (New England 211 Research, 2015) developed by New England Research, Inc. This is a handheld (~1.2 kg, $38 \text{cm} \times$ 212 12.5cm \times 5cm), portable device (Figure 2a) that measures surface permeability data in the field 213 (Figure 2b) as well as in a laboratory setting. This device has been used by other researchers, e.g. 214 Rogiers et al. (2013) on soils and Filomena et al. (2013) on sandstone. The rubber nozzle at the 215 end of the device is pressed against the specimen to form an airtight seal. The operator then 216 pushes the syringe toward the specimen, which creates a vacuum by removing air from the 217 specimen. By monitoring the syringe volume and the vacuum pulse at the specimen surface, the 218 TinyPerm II calculates a characteristic value (T), which is related to the gas permeability (K in 219 mD) per the following equation (New England Research, Inc., 2008):

220

$$K = 10^{\left(\frac{12.8737 - T}{0.8206}\right)}.$$
 (4)

Typical *T* values range between 12.5 and 9.5 yielding permeability measurements between 2 mD (1.97 x 10^{-15} m²) and 10 D (9.87 x 10^{-12} m²), respectively (New England Research, Inc., 2008). A permeability reading of 10 mD (9.85 x 10^{-15} m²), which is the manufacturer's suggested lower limit of the measurement capability, takes about five minutes. Materials with greater permeabilities typically require shorter measurement times. However, some of the materials studied had surface permeabilities below 10 mD, which required 30 minutes or longer to achieve a steady-state flow. For these specimens, TinyPerm II was mechanically supported to avoid operator fatigue and maintain the conditions required for the correct operation of the device.

230 Of the 17 interior (note that interior refers to the samples with the top and bottom 231 surfaces removed to sample interior surfaces) specimens tested with AutoScan II, 16 were re-232 sampled using TinyPerm II, which is well suited for field use. In contrast to the 1,296 points 233 measured with AutoScan II on a 35×35 grid with 1 mm grid spacing, TinyPerm II 234 measurements were typically taken at 23 locations on the same specimen surface within the 35 235 $mm \times 35$ mm area with the exception of Granite and Bluestone which had exceptionally low 236 permeability and thus needed only 12 readings. Statistical analysis was performed on both the 237 raw (rather than log₁₀-transformed) measurements and the geometric means (i.e., arithmetic 238 means on the log scale) of AutoScan II and TinyPerm II measurements.

3.3 3.3

Bulk gas permeability

The bulk gas permeability was measured in accordance with ASTM D4525-90: Standard Test Method for Permeability of Rocks by Flowing Air (ASTM International, 2002). The Wykeham Farrance permeability cell was used with two identical pressure transducers to measure the pressure drop across the specimen. A high confining pressure (~275 kPa) was applied to the cell to ensure that the air would pass through, and not between, the specimen and the latex membrane encasing it. A regulated supply of compressed air was applied to the specimen, while the exiting airflow was measured with a calibrated bubble-flow meter. This test was repeated five times on each specimen, with the average of the five measurements reported as the measured bulk gas permeability for that specimen. The gas permeability was calculated as:

250
$$K = \frac{2Q_e P_e \mu L}{\left(P_i^2 - P_e^2\right)A},$$
 (5)

where, *K* is the coefficient of permeability (L²), Q_e is the exit flow rate of air (L³/T), P_e is the exit air pressure (M/LT²), μ is the viscosity of air at temperature of test (*M/LT*), *L* is the length of specimen, P_i is the entrance pressure of air (M/LT²), and *A* is the cross-sectional area of specimen (L²).

255

256 **3.4 Weathering Effects**

257 Five specimens were cored from nine select materials (Ready Mix, 3,000 psi, 4,000 psi, 258 5,000 psi, and High Strength Concretes, Portland Cement, Red Brick, Indiana Limestone, and 259 Arkose Sandstone) for a total of 45 specimens. All specimens were approximately 75 mm in 260 diameter and 65-100 mm in height and each was subjected to accelerated weathering of 30 261 simulated freeze-thaw cycles (-24°C to 20°C) while submerged in water within a mechanical 262 refrigeration chamber. The specimens were placed at random locations within the chamber and 263 relocated between cycles to reduce any placement effects within the freeze-thaw chamber. The 264 surface gas permeability of each specimen was measured along a uniform 3 mm grid spacing 265 using AutoScan II before and after the simulated weathering. These results allow an evaluation of the potential for using surface gas permeability technique to quantitatively characterize theeffects of weathering.

268

3.5 Geospatial Statistical Analysis

Surface gas permeability data, especially the large datasets generated by AutoScan II. can 269 270 be used to assess the heterogeneity and anisotropy of porous materials. In this study, we 271 determine the spatial auto-correlation along the surface of each specimen, using a geospatial 272 semi-variogram analysis developed and coded in MATLAB (Release 2010a, The Mathworks, 273 Natick, Massachusetts, USA). Semi-variance, $\gamma(h)$, in the geostatistical literature, describes 274 spatial patterns between measured observations as a function of the separation distance (i.e. two 275 points closer to each other in space should be more similar). An example of a semi-variogram 276 (for the specimen of Berea Sandstone) can be found in Figure 11, which will be discussed later in 277 greater detail. These patterns are usually described in terms of dissimilarity rather than similarity 278 or correlation. Thus, the spatial dissimilarity between observations separated by a distance h may 279 be defined as:

280

$$\gamma(h) = \frac{1}{2N(h)} \sum_{i=1}^{N(h)} [u(a)_i - u(a+h)_i]^2$$

281

where N(h) is the number of data pairs separated by the distance, h, and u(a) and u(a+h)are the parameter values (i.e., surface permeability) at locations (a) and some distance (a+h) away (Issaks and Srivastava, 1989; Journel and Huijbregts, 1978). 285 A semi-variogram plots the variance between surface permeability measurements against 286 the distance between paired measured values. These paired data are assembled into bins defined 287 by ranges of separation distances. The horizontal axis represents the separation distance between 288 binned, paired data (e.g., all pairs of surface permeability separated by distances between 0 and 3 289 mm are included in the first bin of Figure 11 discussed later in detail); and the average variance 290 for all paired data in each bin is plotted as a single point along the vertical axis. The resulting 291 plot is known as the experimental semi-variogram and can be best fit by a model semi-variogram 292 that describes the spatial structure of the data characterized by three model parameters – the 293 nugget, sill, and range of spatial auto-correlation. The projected discontinuity near the origin of 294 the plot, known as the *nugget*, represents both the measured parameter error (in our study, the 295 error associated with both the collection and measurement of surface permeability using 296 AutoScan II) as well as the spatial sources of variation at distances smaller than the shortest 297 sampling interval (Journel and Huijbregts, 1978). If two surface permeability measurements 298 taken from the same location along the surface of a specimen have no sampling or laboratory 299 error, the values should be the same (i.e., result in a nugget = 0). The range (also referred to as 300 the decorrelation distance) is the distance at which the measured variable is no longer spatially 301 correlated. The value of semi-variance associated with the model plateau is defined as the *sill*.

302

4

RESULTS OF PERMEABILITY CHARACTERIZATION

303 Surface gas permeability measurements were collected at varying spatial resolutions to 304 demonstrate the versatility and comparability of the AutoScan II and TinyPerm II in 305 characterizing a broad range of natural and engineered porous building materials. Surface 306 permeability data for three representative specimens, Ohio Sandstone, Red Clay Brick, and 3,000 307 psi Concrete, are presented first to highlight the notable trends. Subsequently, the surface gas 308 permeability data collected at 1 mm grid spacing on 17 internal specimens are presented to 309 facilitate comparisons across a variety of materials.

- 310
- 311

4.1 AutoScan II Surface Permeability Measurements on Select Materials

312 An example of measured surface gas permeability on the 70 mm diameter Ohio 313 Sandstone specimen 2 (Figure 3a) at 2 mm grid spacing over a 50 mm diameter circular area 314 using AutoScan II is presented in Figure 3b. The same data are plotted in Figure 3c, where the 315 permeability measurements at given y-coordinates are distributed along the horizontal axis. 316 Figure 3d shows the probability density function (PDF) of the specimen's permeability. The peak 317 of the PDF is the "most observed" value (63.4 mD) and is often used to "globally" characterize 318 the overall permeability. The arithmetic mean, 76.3 mD, geometric mean, 74.3 mD, and the 319 median, 76.5 mD are provided for comparison. The lower permeabilities (i.e., 40s and 50s mD) 320 are located in the mid to lower right portion of the core surface, but most measurements are 321 distributed between 60 and 90 mD. The maximum, minimum, and standard deviation are 140.7 322 mD, 28.2 mD, and 17.3 mD, respectively. Table 1 presents a summary of these measurements.

The Red Clay Brick specimen 2 (Figure 4a), an example store-bought engineered material, is less homogeneous than the Ohio Sandstone. The permeability values measured at 5 mm interval over the surface area of 170 mm by 65 mm span more than three orders of magnitude, so permeability readings have been log₁₀-transformed. The most observed permeability value is 10^{3.87} or 7,414mD, which is closer to the arithmetic mean, 7,102 mD, than
the geometric mean, 4,564 mD, and the median, 4,800 mD. The maximum, minimum, and
standard deviation are 79,090 mD, 71 mD, and 8,088 mD, respectively (Table 1).

330 Surface gas permeability was measured along one side of a screeded slab of 3,000 psi 331 Concrete approximately 260 mm \times 180 mm \times 75 mm (Figure 5a). The permeability was 332 measured at 4 mm grid spacing over a 240 mm by 152 mm area, resulting in 2,331 surface 333 permeability measurements (Figure 5b, where white squares indicate no reading). The surface 334 permeability data, replotted in Figure 5c, generally range from 10 to 80 mD, with a few points 335 outside this range. Figure 5d shows the most observed value of permeability at approximately 336 41.5 mD, which is close to the arithmetic mean, 42.89 mD, geometric mean, 40.2 mD, and the 337 median, 42.9 mD. The maximum, minimum, and standard deviation are 167 mD, 7.6 mD, and 338 15.2 mD, respectively (Table 1).

339 While the surface of the 3,000 psi Concrete specimen of Figure 5 was smoothed and 340 finished with the screeding process, the interior is expected to be more representative of a typical 341 concrete mixture that includes cement paste and fine and coarse aggregates. The permeability 342 measurements made on the screeded surface, in all likelihood, involve only mortar as a result of 343 the screeding process. A comparison between the permeability of a screeded concrete surface 344 (Figure 6a) and the interior surface ~ 2 mm below the screeded top (Figure 6b) was therefore 345 investigated using a 70 mm core of 3,000 psi Concrete (a different specimen from the slab shown 346 in Figure 5). The surface permeability was measured on the screeded exterior surface, and on the 347 exposed surface after cutting a 2mm slice off of the core using AutoScan II. The $\log_{10}(mD)$

348 permeability fields of each surface (Figures 6c and 6d, respectively) were measured over a 349 $35\text{mm} \times 35\text{ mm}$ area with 0.5 mm grid spacing.

350 Both the screeded surface and the interior surface show similar spatial patterns in the 351 surface permeability. These patterns exhibit the expected less permeable "islands" where 352 aggregates are surrounded by thinner, more permeable borders of mortar. While the emerging 353 shapes suggest similar spatial patterns of permeability, the magnitudes differ. Most notable is 354 that the screeded surface permeability measurements are overall approximately one order of 355 magnitude greater than the surface permeability measurements of the interior surface. The 356 presence of aggregates near the measurement surface probably limited gas flow from entry to 357 exit point along the assumed hemispherical flow path, resulting in smaller permeability.

4.2 Comparison of AutoScan II Gas Permeability of Different Porous Building Materials

359 Using AutoScan II, we characterized 17 interior core specimens using a consistent 35 mm 360 \times 35 mm area with 1 mm grid spacing. All cores were extracted from central portions of the 361 specimens, which reduced surface alterations due to screeding or weathering. The specimens discussed in this section are different than those discussed in the previous section (Figures 3-5), 362 363 even when the specimens share the same parent material. Table 2 summarizes the results of the 364 AutoScan II surface permeability testing along with global statistics (i.e., arithmetic mean (mD), 365 geometric mean (mD), most observed (PDF peak in mD), maximum (mD), minimum (mD), 366 standard deviation (mD)) used to characterize the materials. Photographs of each specimen with 367 its measured 35 mm \times 35 mm surface permeability field are presented in Figure 7. The data 368 range over more than five orders of magnitude. Therefore, all surface permeability maps use the

same $\log_{10}(mD)$ color scale (bottom of Figure 7) for easier visual comparison, where dark blue is less than 1 mD and dark red is greater than 100,000 mD. Grid locations at which a steady-state permeability measurement was not produced are shown in white.

Surface permeabilities of the studied materials ranged from less than 1 mD to over 140,000 mD. Granite is the least permeable with a geometric mean of 0.76 mD, and the Red Colored Brick Paver is the most permeable with a geometric mean of 23,689 mD. Asphalt has the largest number of locations where a steady-state permeability measurement could not be achieved, likely due to the many surficial air pockets.

The global surface permeability characterization was observed to be affected by whether the material is natural or engineered. Most of the natural materials have very low permeabilities, as do the two concretes cured for specified strengths. The four most permeable materials were engineered materials not designed for a specific strength. The 5,000 psi concrete specimen includes a small area of high permeability measurements. This may be indicative of an indentation or imperfection such as a crack along the surface.

383

4.3 Comparison of AutoScan II and TinyPerm II Measurements

To investigate how well TinyPerm II may characterize a specimen in the field compared to AutoScan II laboratory measurements, each specimen's averaged (geometric mean) permeabilities, measured using TinyPerm II and AutoScan II, are plotted in Figure 8. The geometric mean is less susceptible to outliers or erroneous data. The two geometric means are highly correlated ($\rho = 0.97$). The 1:1 line is provided for comparison. Overall, the global specimen permeabilities using each of the devices are very similar. 390 As noted in Section 3.2, TinyPerm II is typically recommended for specimens with a 391 surface permeability greater than 10 mD, yet many of the measurements were below that 392 threshold and required measurement times beyond five minutes. Our results show that overall 393 the specimen characterization appeared to be accurate even below the manufacturer's 394 recommended 10 mD. Hence the limiting factor, when characterizing low permeability 395 materials, is the allotted maximum time required for sampling and not the accuracy of the 396 device itself. It is worth noting that the ranking (either ascending or descending order) of the 397 results with TinyPerm II is similar to that of AutoScan II (Fig. 8), as also indicated by a very 398 high Spearman correlation coefficient which was computed to be 0.97. This suggests 399 that regardless of differences in values, TinyPerm II may be useful for field characterization 400 and selection of sampling points.

401

402

Surface versus Bulk Permeability 4.4

403 The average surface permeability (geometric mean, n=4 specimens) for each material 404 measured with AutoScan II is plotted against the average bulk permeability in Figure 9 with a 405 one-to-one line and a least-squared regression model with an adjusted R^2 of 0.61 (n= 60). The 406 solid, horizontal lines represent the range of the four most observed \log_{10} -transformed surface 407 gas permeability values for each material, while the vertical dashed lines indicate the range of the 408 bulk gas permeability measurements for that material. The latter are within one order of 409 magnitude of each other, with the exception of 3,000 psi Concrete, which spans almost two

410 orders of magnitude. Natural materials are plotted with darker symbols while engineered411 materials are plotted in light gray.

412 The natural materials (Ohio Sandstone, Arkose Sandstone, Indiana limestone), Red Clay 413 Brick and Portland Cement, are relatively homogeneous and are located close to the one-to-one 414 line in Figure 9, indicating that differences between the surface and bulk gas permeability 415 measurements are relatively small. The remaining materials are fairly heterogeneous (at least for 416 the size of the specimens) engineered materials (Ready Mix Concrete, 3,000 psi Concrete, 5,000 417 psi Concrete, D04 Concrete, and Red Colored Brick Paver) and contain aggregates. 418 Measurements deviate further from the one-to-one line in Figure 9, suggesting that the bulk 419 permeability of the entire specimen is somewhat different than that of the specimen surface. 420 Given that the concrete specimen surfaces were smoothed and finished via screeding while the 421 interior core is more representative of the heterogeneous mixture, the interior aggregates likely 422 create a longer and more tortuous flow path in bulk permeability measurements, resulting in the 423 smaller observed values of bulk permeability. With the exception of the Arkose Sandstone 2 and 424 Clay Brick, all other materials had greater surface gas permeability measurements than bulk 425 permeability measurements. This bias is likely due to the more tortuous flow path through the 426 entire specimen.

427

5 EFECTS OF WEATHERING ON PERMEABILITY

In general, surface permeability measured with AutoScan II and/or Tiny Perm II is more
similar to bulk permeability for the relatively homogenous materials in this study (natural stones,
Red Clay Brick, and Portland Cement) compared to the more heterogeneous engineered

431 materials (i.e., concretes and pavers). The latter is not surprising; and as a result, for applications 432 involving surface contamination or surficial weathering, the use of bulk permeability might not 433 be appropriate, and the surface permeability is probably more suitable.

434 The surface permeabilities of nine select building materials were tested using AutoScan II 435 before and after weathering simulated using freeze-thaw cycles. The selection of the specific 436 materials was such that there were both natural and engineered materials represented. After 437 weathering, however, specimens from three materials (3,000 psi and 4,000 psi Concretes and 438 Portland Cement) were degraded to the point where they could not be tested. Thus, only 439 specimens from the six fairly intact materials (Ready Mix, 5,000 psi and High Strength 440 Concretes, Red Brick, Indiana Limestone and Arkose Sandstone) were tested and presented 441 here. Figure 10 plots the geometric mean permeability of unweathered specimens on the x-axis 442 and weathered specimens on the y-axis; a 1:1 line is shown for comparison. The natural materials 443 and the ready mix fall on or close to the 1:1 line; however, the latter does so to a slightly lesser 444 degree than the natural materials. All other engineered specimens are substantially farther away 445 from and lie above the 1:1 line, indicating that their surface permeability had increased with 446 weathering. The weathering process produced notable cracks and possibly increased the size of 447 the pores or fractures/openings, facilitating air flow through the specimen. Consequently, the 448 materials have higher surface permeability after weathering, and the use of AutoScan II enables 449 characterization of the weathering effects at high spatial resolution.

450

The natural materials were considerably less affected by weathering than the engineered 451 materials examined in this study. One possible explanation may be related to the extended period 452 of time that natural materials took to form compared to the relatively rapid curing time allowed 453 for engineered materials. Further studies are necessary to improve our understanding of the 454 underlying mechanism as many factors can have significant influence on the weathering process 455 (e.g., Goudie, 1999; Elert et a;, 2003; Benavente et al., 2004; Scherer, 2004; Flatt et al., 2014). 456 The less homogeneous materials might be more susceptible to weathering damage since tensile 457 stresses are more likely to develop as a result of non-uniform volume expansion/shrinkage. It is 458 important to note that the 5,000 psi Concrete had similar surface permeability to the natural 459 materials before weathering, so the original surface permeability before weathering takes place is 460 probably not a good indicator of resistance to weathering or long-term preservation. However, 461 the increase in permeability with weathering may provide a reliable means to quantify the degree 462 of weathering.

463

6 GEO-STATISTICAL ANALYSIS OF SURFACE PERMEABILITY

464 AutoScan II is well suited for acquiring surface permeability data at high resolution and with high precision when evaluating spatial autocorrelation and anisotropy, which is relevant in 465 466 identifying preferential flow paths inherent in natural materials or modeling flow and transport in 467 building materials. A detailed geo-statistical analysis of the surface gas permeability (mD) 468 measured at 1 mm spacing using AutoScan II along the surface of a slab ($306 \text{ mm} \times 114 \text{ mm}$) of 469 Berea Sandstone is shown in Figure 11. Figure 11b shows the corresponding omnidirectional 470 semi-variogram. Variance values associated with paired data have been grouped into 62 equally 471 spaced bins and a typical semi-variogram is produced by plotting the average variance for each 472 bin (small black dots). The semi-variogram shows the measured surface permeability to be

473 spatially auto-correlated at 25 mm. It is possible for the variable in question to become spatially 474 auto-correlated again at larger distances (i.e. where data begin to increase consistently above the 475 sill), resulting in a semi-variogram with multiple decorrelation distances. One important 476 advantage of acquiring data at such high spatial resolution is that we are able to characterize the 477 material's anisotropy. The latter is very important if one wishes to model or predict preferential 478 flow pathways. Figure 11(c) shows the direction of maximum anisotropy to be at 0 degrees (i.e. 479 horizontal direction). It is to be noted that because this was a laboratory specimen, we were able 480 to align the maximum direction with our horizontal x-axis. The directional semivariograms show 481 that the maximum and minimum ranges of spatial autocorrelation to be ~ 275 mm and ~ 17 mm in 482 Figure 11c and Figure 11d, respectively.

483 Since we did not find this type of material characterization in the literature for the breadth 484 of materials reported here, a similar analysis was performed on all 17 materials; the 485 corresponding values for sill, range, and nugget are listed in Table 2 (see Grover (2014) for 486 further analysis). The omnidirectional range of spatial autocorrelation varied between 5 and 29 487 mm for the materials tested. However, we were unable to discern any particular trend across 488 materials. The sill on the other hand reveals a significantly larger variance in the engineered materials $(0.02 - 1.95 \times 10^9, -11)$ orders of magnitude) compared to the natural materials $(0.003 - 1.05 \times 10^9, -11)$ 489 8.5 x 10^3 , ~6 orders of magnitude); again, this is an important parameter for modeling flow and 490 491 transport through the materials' surface as it allows one to quantify the error variance (i.e., 492 uncertainty) associated with the model results.

494 7 CONCLUSIONS

495 Surface permeability measurement has been shown to be an effective and reliable non-496 destructive method for characterizing porous building materials both in the laboratory and in the 497 field. Automated collection and high-resolution measurements render this technique useful for 498 detailed, quantitative characterization of specimen surfaces (e.g. geometric mean, most observed, 499 maximum, and minimum values) and comparisons across specimens.

500 In general, the measured permeabilities (surface and bulk) compared better to each other 501 for the relatively homogeneous materials of this study (natural stones, Clay Brick and Portland 502 Cement Mortar) than the less homogeneous engineered materials such as concretes. Surface permeability may be easier to measure *in situ*, but it may not be an appropriate surrogate for bulk 503 504 gas permeability for all materials (e.g. concrete).

505 Our results indicate that the surface permeability measurements made by TinyPerm II 506 correlate well to those made using AutoScan II. TinyPerm II is compact, portable and easy to use 507 compared to AutoScan II; it is well suited to field use, and it may provide a way to rapidly 508 characterize materials in situ. However, it does not allow grid spacing of less than about 3 mm 509 and the measurement point cannot be precisely automated or controlled since it is human 510 operated. In contrast, AutoScan II is well suited when surface permeability data at high 511 resolution and precision are needed. Such high resolution data can enable characterization of the 512 spatial autocorrelation, anisotropy or heterogeneity inherent in building materials.

513

The high-resolution surface permeability characterization may be necessary for modeling 514 and prediction of preferential flow and transport, as well as quantifying relative changes on the 515 surfaces of porous building materials exposed to effects such as weathering. If the weathering 516 effects related to reduction in material strength, characterizing changes in surface permeability 517 might be used as an indicator of a material's strength/durability over time, especially in harsh 518 climates. These measurements illustrate the operational effectiveness of the surface permeability 519 measurement techniques, which is particularly relevant to investigations involving surface 520 effects.

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Material	Measurement	Surface Permeability (mD)								
	Details	Arithmetic	Geometric	Most	Median	Maximum	Minimum	Standard		
		Mean	Mean	Observed				Deviation		
Ohio	2 mm grid	76.3	74.3	63.4	76.5	140.7	28.2	17.3		
Sandstone	spacing over									
(specimen 2)	50 mm									
	diameter									
	circular area									
Red Clay	5 mm grid	7,102	4,564	7,414	4,800	79,090	71	8,088		
Brick	spacing over									
(specimen 2)	170 mm x 65									
	mm area									
3,000 psi	4 mm grid	42.9	40.2	41.5	42.9	167	7.6	15.2		
Concrete	spacing over									
	240 mm x									
	152 mm area									

Table 1. Summary of surface permeability measurements made on Ohio Sandstone, Red Clay Brick, and 3,000 psi concrete specimens (Figures 3, 4, and 5)

Table 2. Summary of surface permeability measurements made on porous building materials (internal specimens with ends discarded)at 1 mm grid spacing using AutoScan II

		Surface Permeability (mD)								
			Geometric	Most						
		Arithmetic	Mean	Observed			Standard	Range	Sill	Nugget
Material Type	Origin	Mean			Maximum	Minimum	Deviation	(mm)	(\mathbf{mD}^2)	(mD)
Arkose Sandstone	Natural	3.21	2.94	2.05	9.23	1.39	1.50	14	2.12	0.19
Ohio Sandstone	Natural	4.74	4.44	6.82	8.63	2.23	1.68	13	2.99	0.970
Portland Brownstone	Natural	3.84	3.80	3.55	6.00	2.59	0.54	9	0.26	0.016
Bluestone	Natural	0.89	0.87	0.74	1.90	0.68	0.21	4	0.43	0.43
Granite	Natural	0.76	0.76	0.75	1.23	0.64	0.06	11	0.0029	0.0012
Buff Indiana Limestone	Natural	177	160	138	575	39.47	82.32	29	8497	73
Gray Indiana Limestone 1	Natural	5.79	5.72	6.20	8.38	2.68	0.85	10	0.79	0.18
Gray Indiana Limestone 2	Natural	3.64	3.57	3.21	10.47	2.25	0.80	9	0.48	0.037
Silver Indiana Limestone	Natural	5.49	5.46	5.53	7.66	2.55	0.56	9	0.26	0.034
Red Clay Brick	Engineered	3.35	3.26	2.76	5.67	1.95	0.79	27	0.92	0.036
3,000 psi Concrete	Engineered	0.98	0.95	1.08	6.08	0.62	0.35	12	0.14	0.0039
5,000 psi Concrete	Engineered	800	1.66	2,344	140,583	0.95	7,964	5	0.020	0.020
Concrete Paver 1	Engineered	8,376	5,974	5,738	31,181	26.43	5,605	10	38,256,867	316,172
Red Colored Brick Paver	Engineered	29,320	23,689	33,228	86,017	413	14,698	11	194,910,243	4,502,877
Tan Colored Brick Paver	Engineered	6,654	2,664	637	38,151	1.20	7,575	9	68,141,303	568,792
Concrete Paver 2	Engineered	9,988	7,758	4,227	36,225	1.17	6,635	19	54,143,304	622,337
Asphalt	Engineered	317	34.71	140	8,325	0.98	585	16	344,270	181,494



Figure 1. (a) A laboratory surface permeameter AutoScan II measuring surface gas permeability on multiple specimens, (b) permeability probe on a Red Clay Brick specimen 2, and (c) assumed flow path of injected gas (source: New England Research, 2008).



Figure 2. (a) Components of a portable surface permeameter (TinyPerm II) used in this study (source: New England Research, 2008), and (b) an example of how the device can be used in the field.



Figure 3. Measured surface gas permeability on a 70 mm diameter Ohio Sandstone specimen 2 at 2 mm grid spacing within the 50 mm diameter circular area shown as a dashed circle, (a) a photograph of the tested surface of the specimen, (b) map of gas permeability, (c) distribution of gas permeability along each y-coordinate, (d) probability density function of gas permeability, most observed value is indicated.



Figure 4. Measured surface gas permeability on a store-bought Red Clay Brick specimen 2 at 5 mm grid spacing over the surface area of 170 mm x 65 mm, (a) a photograph of the tested surface of the specimen, (b) map of surface gas permeability, (c) distribution of surface gas permeability along each y-coordinate, (d) probability density function of gas permeability.



Figure 5. Measured surface gas permeability on a screeded 3,000 psi Concrete specimen at 4 mm grid spacing over the surface area of 240 mm x 152 mm, (a) a photograph of specimen surface, (b) map of gas permeability field, (c) distribution of gas permeability along each y-coordinate, (d) gas permeability probability density function.



Figure 6. Core (70 mm diameter) of 3,000 psi Concrete specimen (a) Picture of screeded top, (b) picture of interior about 2 mm below screeded top, (c) surface gas permeability map of the screeded top, (d) surface gas permeability map of on the interior surface. White areas did not return a measurement



Figure 7. Photographs and surface permeability of the building materials specimens. The natural materials are in the left two columns, and the engineered materials are to the right.



Figure 8. TinyPerm II averages (geometric mean) versus AutoScan II averages (geometric mean). Natural materials are shown as black or dark gray while engineered materials are show as light gray. Both data sets were log_{10} transformed, and the correlation coefficient $\rho = 0.94$.



Figure 9. Bulk gas permeability plotted against geometric mean of surface gas permeability obtained using AutoScan II. The natural materials (e.g. Indiana, Ohio and Arkose Sandstone) are depicted with black symbols, while the engineered materials (all Concretes, Brick, and Pavers) are depicted in lighter gray.



Figure 10. Comparison of specimen geometric mean permeabilities obtained using AutoScan II under unweathered and weathered conditions. The natural materials (Indiana Limestone and Arkose Sandstone are depicted with black symbols, while the engineered materials (all Concretes and Brick) are depicted in lighter gray.



Figure 11. (a) The surface gas permeability (mD) measured along the surface of a Berea Sandstone slab (1 mm spacing); the corresponding (b) omnidirectional semi-variogram analysis (model: spherical, range: 25 mm, sill: 6,063, nugget: 1,600); (c) horizontal semivariogram at 0° (model: linear, range: 275 mm, sill: NA, nugget: 200); and (d) vertical semivariogram at 90° (model: spherical, range: 17 mm, sill: 7,200, nugget: 2000).

Graphical Abstract

