

**EFFECT OF PERSULFATE FORMULATIONS ON SOIL
PERMEABILITY**

BY
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My love goes to my parents, Qian Zhang and Qinghui Yu. My love and appreciation go to my fiance Shuai Shao, for everything he did for me.

EFFECT OF PERSULFATE FORMULATIONS ON SOIL PERMEABILITY

ABSTRACT

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In situ chemical oxidation (ISCO) using persulfate is a promising technique for the remediation of soil and groundwater contaminants. Persulfate activated by iron (III)-EDTA or sodium hydroxide can accelerate the degradation of contaminants. However, in previous studies of ISCO remediation processes, the application of ISCO chemicals has been limited by low permeability zones. Three persulfate process conditions typically used in the field were used in laboratory studies to investigate the effect of unactivated and activated persulfate formulations on the permeability of a suite of subsurface solids. Column tests were conducted via a falling head permeameter for commercial silica sand and a flexible wall permeameter was used for kaolinite and two natural soils to quantify changes in hydraulic conductivity after the application of persulfate formulations. Unactivated persulfate and iron (III)-EDTA-activated persulfate had minimal effect on the permeability of all four soils. Base-activated-persulfate increased the permeability of kaolinite and the natural soils, but decreased the permeability of sand. Changes in soil dispersion, flocculation, and surface charge may have contributed to changes in soil permeability. X-ray computed tomography (XRCT) was used to investigate the effect of persulfate formulations

on soil structure. The porosity of the soil samples treated by persulfate solutions was not distributed uniformly with depth as that of dry soil samples, and minimum, nor mean porosity of the soils did not correlate with changes in hydraulic conductivity.

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1. Introduction

In situ chemical oxidation (ISCO) has been widely used for the remediation of contaminated soil and groundwater over the past two decades. ISCO processes are often capable of meeting cleanup goals in a cost effective way. Four oxidation systems are commonly used for ISCO: catalyzed H_2O_2 propagations (CHP), permanganate, ozone, and activated persulfate (Watts and Teel, 2006). CHP is effective for degrading organic contaminants, however, its use is limited by the low stability of hydrogen peroxide in the subsurface which can only last hours to days in the subsurface (Watts and Teel, 2006). Permanganate has higher stability in the subsurface compared to hydrogen peroxide but it can decrease soil permeability due to the formation of manganese oxide precipitates (Li et al., 2004). Ozone is highly reactive with contaminants, but its low stability and low rates of mass transfer from the gas phase to groundwater limit its use. ISCO can be implemented rapidly and achieve remediation goals effectively; however, it has limitations in low permeability areas. Many subsurface regions have low permeability, which can prevent the ISCO oxidants from penetrating into deeper contaminated zones, thus limiting the contact between oxidants and contaminants.

Persulfate is a strong oxidant that has been used for the destruction of a wide range of soil and groundwater contaminants. The high stability of persulfate in the subsurface provides the potential for its transport from the point of injection to contaminants in low permeability regions, which may broaden its use for ISCO. Persulfate is usually activated for use in ISCO. Activation of persulfate has been achieved most commonly through the use of chelated metals or base. The chelation of iron with EDTA maintains iron solubility at all pH regimes, providing effective activation of persulfate (Kwan and Chu, 2007). Soluble iron and iron chelates activate persulfate through the following process (Kolthoff et al., 1951):



Hydroxyl radicals (OH·) are generated by the following reaction (Watts and Teel, 2006):



Base activation of persulfate is also commonly used in ISCO. Base-activated-persulfate can produce free radicals such as sulfate radicals, hydroxyl radicals and superoxide radicals (Furman et al., 2010):



The generation of the reactive oxygen species shown in equations (3)-(6) provides the widespread reactivity of base-activated-persulfate formulations.

Persulfate formulations are highly complex solutions containing sodium, persulfate, and its decomposition products. Furthermore, the pH drops as the persulfate decomposes to sulfuric acid. The complex chemistry of persulfate may change the permeability of soils and subsurface solids by inducing dispersion and flocculation, or by changes in surface charges. X-ray computed tomography (XRCT) is a non-destructive technique with wide applications in geological engineering that can be used to evaluate soil morphology related to changes in permeability. XRCT can provide visualization and qualification of the internal structure of subsurface soil. The XRCT process involves measuring the attenuation of X-ray passing through a soil sample and developing three-dimensional CT images (Mees, et al., 2003), which are then further analyzed to

determine soil porosity (n). The general goal of this research was to investigate the effect of differing concentrations of persulfate and activated persulfate formulations on soil permeability using permeameters and XRCT analysis. The specific objectives of this study were to (1) test the soil hydraulic conductivity after treatment with different persulfate formulations using permeameters, (2) examine the effect of persulfate formulations on the internal soil structure using XRCT, and (3) model the relationship between soil hydraulic conductivity as measured by permeameters and soil structure data obtained from XRCT.

2. Materials and Methods

2.1 Materials

Sodium persulfate ($\geq 98\%$) and sodium sulfate were purchased from Sigma Aldrich (St. Louis, MO). Sodium hydroxide (98.6%) and iron (III)-EDTA were obtained from J.T. Baker Inc. (Phillipsburg, NJ). Deionized water was purified to $>18 \text{ M}\Omega\cdot\text{cm}$ with a Barnstead Nanopure II deionizing system. Four materials were used to investigate the effect of persulfate formulations on hydraulic conductivity: commercial sand (silica sand 20/30), kaolinite and two horizons of a natural soil. Commercial sand was purchased from Lane Mountain Company (WA). Kaolinite ($\text{Al}_2\text{Si}_2\text{O}_5(\text{OH})_4$) was purchased from Dry Branch Kaolinite Company (GA). Two natural surface soils, which were termed soil KB1 and soil KB2, were collected from two different horizons near Kamiak Butte in the Palouse region of Washington State. The natural soils were air dried and passed through a $300 \mu\text{m}$ sieve. Soil textural classification was analyzed by hydrometer. The physical and chemical properties of the soils are shown in Table 1 and Table 2.

2.2 Persulfate reactions

Unactivated persulfate was used at a concentration of 0.5 M. The same concentration of persulfate was used in activated persulfate formulations with the addition of 1) iron (III)-EDTA (10 mM) and 2) sodium hydroxide (1 M, for a 2:1 ratio of base to persulfate) as activators. Sodium sulfate solutions (0.5 M) were used in place of persulfate as positive control reactions for hydraulic conductivity tests. Deionized water was used in place of persulfate for control reactions for XRCT.

2.3 Hydraulic conductivity

Hydraulic conductivity was measured to evaluate potential change in soil permeability after the application of persulfate formulations. The permeameters and test methods used varied according to the physical characteristics of different soils. Permeability tests of commercial sand were conducted using a falling head permeameter as shown in Figure 1. Sand in 300 g quantities was added to the column of the permeameter, which was then filled with persulfate or control solutions and compacted in the column to a height of 13 cm. The initial head h_1 at time $t = 0$ was recorded and the solution was passed through the sample in order to obtain the final head h_2 at time t . The hydraulic conductivity k governed by Darcy's Law was calculated using equation 7:

$$k = \frac{l}{t} \ln \left(\frac{h_1}{h_2} \right), \quad (7)$$

where l is the height of sample (13 cm), t is the time interval between readings of h_1 and h_2 , h_1 is the initial height of the solution in the column, and h_2 is the final height of the solution.

Hydraulic conductivity tests for kaolinite, soil KB1 and soil KB2 were conducted with a modified flexible wall permeameter (ASTM D5856-95, D5084-03) (Hamdi et al., 2005) as shown in Figure 2. The permeameter column and sample holder ring were made of polyvinyl chloride (PVC). The sample holder ring was fitted tightly with an O-ring fitting which was set

between the sample ring and the column to prevent leakage from the inner edge of column. Kaolinite and the natural soils were mixed with 10% deionized water before compaction and saturation. Kaolinite in a mass of 90 g was compacted in the mold to a 1.5 cm layer. Similarly, 123 g of soil KB1 and 114 g of soil KB2 were compacted in the same way. Different soil masses were used because of the differences between soil densities. Compacted samples were saturated with deionized water, persulfate or sulfate solutions for 24 hr before the permeability tests were initiated. The soil samples were placed on a porous stone to hold the sample and allow transport of solutions. The solutions passed through the soil sample with head changing from h_1 to h_2 . Hydraulic conductivity k was then calculated using equation 8:

$$k = \frac{la}{tA} \ln\left(\frac{h_1}{h_2}\right), \quad (8)$$

where l is the thickness of sample (2 cm), t is the time interval between readings of h_1 and h_2 , h_1 is the initial level of solution, h_2 is the final level of solution, A is the cross-sectional area of the soil sample (45.58 cm²), and a is the cross-sectional area of the solution column (0.064 cm²). Because kaolinite and the natural soils were not very permeable, the ratio between A and a was designed to be large enough in order to conduct the tests within a reasonable time.

2.4 X-ray computed tomography

For each XRCT analysis, 74 g of sand or 68 g of the natural soil were packed in 3.4 cm-diameter PVC columns. Deionized water, unactivated persulfate, or iron (III)-EDTA-activated persulfate solutions were passed through the sand samples before X-ray scanning. Soil KB1 samples were saturated with deionized water or 0.5 M base-activated-persulfate solutions for 24 hr prior to conducting the X-ray scan. Soil KB2 samples were saturated in deionized water and

0.5 M unactivated and iron (III)-EDTA- and base-activated persulfate solutions for 24 hr prior to conducting the X-ray scan. Dry soil samples and deionized water treated soil samples served as controls. The XRCT scan apparatus has two X-ray sources that are able to generate 420 keV and 225 keV voltages, respectively. The voltage for the X-ray source used in this study was 350 keV and the source current was 1.6 amp. The X-ray sources are connected to a central work station, which is comprised of four parallel computing processors and software.

Three FlashCT programs were used to generate images of the materials. The first program, FlashCT DAQ, initiates the scanning of samples and outputs raw data. The data are processed by the second program, FlashCT DPS, which provides reconstructed cross-sectional images of the scanned slices. The third program, FlashCT VIZ, converts the cross-sectional images into three-dimensional images. Finally, these three-dimensional images are re-processed to two-dimensional (XY, YZ, XZ) format images for further analysis. Analysis of these 2-D images was carried out by Image Pro Plus software to determine soil structure data, including soil porosity (n) and mean pore radius (r). The procedure for obtaining final X-ray results is shown in Figure 3.

3. Results and Discussion

3.1 Effect of persulfate formulations on commercial sand permeability

Commercial silica sand was first used to investigate the effect of persulfate formulations on permeability. Sand permeability variations after treatment with different persulfate concentrations and formulations are shown in Figure 4. The hydraulic conductivity of the sand in control reactors after treatment with deionized water was 1.26×10^{-2} m/s. After treatment with unactivated persulfate at concentrations from 0.1 M to 0.5 M, there was no significant difference

in the hydraulic conductivity, demonstrating that unactivated persulfate did not have a significant influence on the permeability of silica sand. In contrast, the hydraulic conductivity of sand decreased from 1.28×10^{-2} to 1.17×10^{-2} m/s after treatment with sulfate and also decreased with all three persulfate formulations. Treatment with increasing concentrations of iron (III)-EDTA-activated persulfate decreased the sand hydraulic conductivity from 1.28×10^{-2} to 1.16×10^{-2} m/s, which was about the same extent of less of permeability as sulfate. Sodium sulfate and iron (III)-EDTA-persulfate solutions are acidic, which may change the surface charge on the sand and decrease its permeability. Furthermore, the addition of sodium may disperse the sand, resulting in decreased sand permeability. Treatment with increasing concentrations of base-activated-persulfate decreased the sand even more than the acidic solutions with hydraulic conductivity decreasing from 1.24×10^{-2} to 1.01×10^{-2} m/s. During the process of persulfate activation by base, small amount of gas are produced (Furman et al., 2010). If gas accumulated in the void spaces of surface sand, the result is a substantial decreased in hydraulic conductivity, which may be occurring in the base-activated-persulfate systems evaluated in this study. In addition, the silicon oxide may react with sodium hydroxide. Silicon oxide particles in the system may have been degraded to smaller size particles by the strong base. These small size particles may have then moved within the porous medium, leading to trapping in the pores and plugging (Amrhein et al., 2004). In summary, unactivated persulfate did not have a significant influence on sand permeability, while iron (III)-EDTA- and base-activated- persulfate decreased sand permeability, similar to the sodium sulfate positive control.

3.2 Effect of persulfate formulations on kaolinite permeability

Permeability tests were also conducted to study the effect of persulfate formulations on kaolinite (Figure 5). There was minimal change in the permeability of kaolinite in the presence of sulfate from 2.08×10^{-9} to 3.53×10^{-10} m/s. After treatment with iron (III)-EDTA-activated-persulfate, kaolinite hydraulic conductivity decreased slightly. The iron (III)-EDTA may have precipitated as an iron hydroxide ($\text{Fe}(\text{OH})_3$) or hydrous ferric oxide ($\text{Fe}_2\text{O}_3 \cdot n\text{H}_2\text{O}$) in these systems (Pignatello and Day, 1996; Georgi et al., 2006), possibly decreasing system permeability. Alternatively, the iron hydrolysis complexes in the system may have acted as bonding agents between kaolinite particles (Ma et al., 1997), reducing the kaolinite porosity. Kaolinite permeability increased by 18% when increasing concentrations of unactivated persulfate were applied to the columns. Because persulfate decomposition was less than 6% over 30 d and kaolinite does not promote the activation of persulfate (Ahmad, 2009), minimal change in the permeability of kaolinite would not be expected in the presence of unactivated persulfate.

In contrast to the three other systems, base-activated-persulfate significantly increased kaolinite permeability with increasing persulfate concentrations (Figure 5). The permeability of the 0.5 M base-activated-persulfate formulations was 2.8 times that of the 0.1 M base-activated-persulfate. Under basic conditions, the zeta potential of clay may have become negative, and kaolinite particles may have compacted (Ma et al., 1998) promoting the formation of cracks, resulting in increased hydraulic conductivity (Brown et al., 1987).

3.3 Effect of persulfate formulations on the permeability of natural soils

In the effect of these persulfate formulations and sulfate positive control on the permeability of two horizons of a natural soil, KB1 and KB2, which are shown in Figure 6 and Figure 7, respectively. Permeability did not change significantly with increasing concentrations

of sulfate, unactivated persulfate, or iron (III)-EDTA-activated-persulfate, although the initial (0.1 M) hydraulic conductivities in soil KB2 differed significantly. In soil KB2 (Figure 7), the trend was the same: There was no change in hydraulic conductivity with increasing persulfate concentrations when sulfate, unactivated persulfate, and iron (III)-EDTA-activated-persulfate were applied to the columns.

The trend in changes of hydraulic conductivity was quite different for base-activated-persulfate formulations in both soil KB1 and soil KB2. Both soil horizons were characterized by significant increase in hydraulic conductivity with increasing dosages of base-activated-persulfate. The hydraulic conductivity of soil KB1 increased by approximately 3.5 times from 2.2×10^{-7} m/s to 7.4×10^{-7} m/s as the persulfate concentration was increased from 0.1 M to 0.5 M. Although the hydraulic conductivity of the base-activated-persulfate systems were less than the sulfate positive control, the hydraulic conductivity in base-activated-persulfate systems increased two-fold in soil KB2. Soil minerals may undergo dissolution and precipitation under highly basic conditions (Qafoku et al., 2003), and sodium ion can facilitate the release of silicon from soil structure to promote the dissolution of soil particles (Qafoku et al., 2003), which may compact together thus reducing the permeability. In addition, soil constituents such as aluminum, silicon and iron may precipitate together to form groups of minerals such as sodalite and hematite under basic conditions (Qafoku et al., 2003; Qafoku et al., 2007), which may have reduced the KB2 soil permeability.

3.4 X-ray computed tomography tested soil porosity

The distribution of porosity of the sand with depth in persulfate-treated soil column is shown in Figure 8. The porosity of the dry sand (Figure 8a) was distributed relatively uniformly.

As shown in Figure 8b, the porosity of the sand sample treated with deionized water distributed uniformly from depth of 1 cm to 4.5 cm. The surface porosity did not distribute uniformly probably due to surface clogging in the top centimeter of the column (Manahiloh, et al., 2010). In addition, the sample surface structure was probably disturbed as the solutions passed through the sample. This phenomenon also occurred in the soil KB1 and KB2 samples. As shown in Figure 8c and d, the porosity of the sand samples treated by unactivated persulfate and iron (III)-EDTA-activated-persulfate was even less uniform with depth compared to the dry sand sample and even the deionized water treated sand sample. These results suggest that the persulfate formulation influenced the porosity of the sand samples throughout the entire depth, thus affecting the permeability throughout the entire column. The average porosity of the sand samples treated by unactivated persulfate and iron (III)-EDTA-activated-persulfate was 0.21 and 0.17, respectively, which is higher than the porosity of the sand in the presence of deionized water. There was little correlation between the mean porosity of sample after treatment and hydraulic conductivity (Figure 4). However, the minimum porosity in the control, the unactivated persulfate treated samples, and the iron (III)-EDTA treated sample was approximated 0.1, which may affect hydraulic conductivity more than the mean porosity.

The distribution of the porosity of soil KB1 with depth is shown in Figure 9. The porosity of the dry KB1 soil and deionized water treated KB1 soil samples were distributed relatively uniformly with depth compared to the distribution of the sample treated with base-activated-persulfate. This difference was likely due to changes in soil microstructure resulting from the base-activated-persulfate. The average porosity of soil KB1 after treatment with base-activated-persulfate was 0.26 while the average porosity of soil KB1 treated with deionized water was

0.16. These results correlate with increased permeability and are in agreement with the results shown in Figure 6.

The porosity distribution of soil KB2 is shown in Figure 10. Similar to soil KB1, none of the soil KB2 samples treated with persulfate solutions was characterized by uniform pore distribution compared to the dry soil sample. The average porosity of persulfate, iron (III)-EDTA-activated-persulfate and base-activated-persulfate samples were 0.21, 0.16 and 0.13 respectively. These mean porosities, nor the minimum porosities shown on Figure 10, correlate with the hydraulic conductivities shown on Figure 7. The porosity data of Figure 8-10 demonstrated that porosity is highly variable and complex in persulfate-treated samples, and that the changes in porosity do not usually correlate with changes in hydraulic conductivity. Nonetheless, the data shown in Figure 8-10 showed that all of the persulfate formulations change the porosity of all of the soils evaluated, and significantly increase the variability in porosity with depth of treatment.

4. Conclusions

The results of the research demonstrated that activated persulfate promotes changes in the permeability of different soils, and these changes are dependent on the soil type and the persulfate formulations applied. In the presence of sand, unactivated persulfate and iron (III)-EDTA-activated-persulfate had minimal effect on permeability relative to the sulfate positive control; however, the application of base-activated-persulfate resulted in significant decrease in hydraulic conductivity. However, these changes in the permeability of sand are likely not important because the hydraulic conductivity of sand is orders of magnitude higher than other subsurface soils. Changes in the hydraulic conductivity of kaolinite were only evident in base-

activated-persulfate above 0.3 M. Such a dramatic increase (3.5 times) may significantly increase the potential for treatment of contaminants in low permeability matrices of the subsurface. Similar results of increased hydraulic conductivity with increasing concentrations of base-activated-persulfate were found in soil KB1 and soil KB2.

Results of XRCT demonstrated that changes in porosity with depth were minimal in control samples, but all of the soils became highly heterogeneous with respect to porosity. Such wide ranging porosity can result in minimal potential to correlate soil porosity with hydraulic conductivity.

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Table 1 Physical properties of soil KB1 and soil KB2

Soil	% Sand	% Clay	% Silt	Texture	CEC (Cation Exchange Capacity) (Cmol(+)/kg)
KB1	39.5	11.1	49.8	Loam	19
KB2	7.77	69.15	23.08	Silt Loam	34

Table 2 Chemical properties of soil KB1 and soil KB2

Soil	Amorphous Mn ($\mu\text{g/g}$)	Total Mn ($\mu\text{g/g}$)	Amorphous Fe ($\mu\text{g/g}$)	Crystalline Fe ($\mu\text{g/g}$)	% Organic Carbon
KB1	296	510	2196	1697	0.24
KB2	194	380	4656	2789	1.61

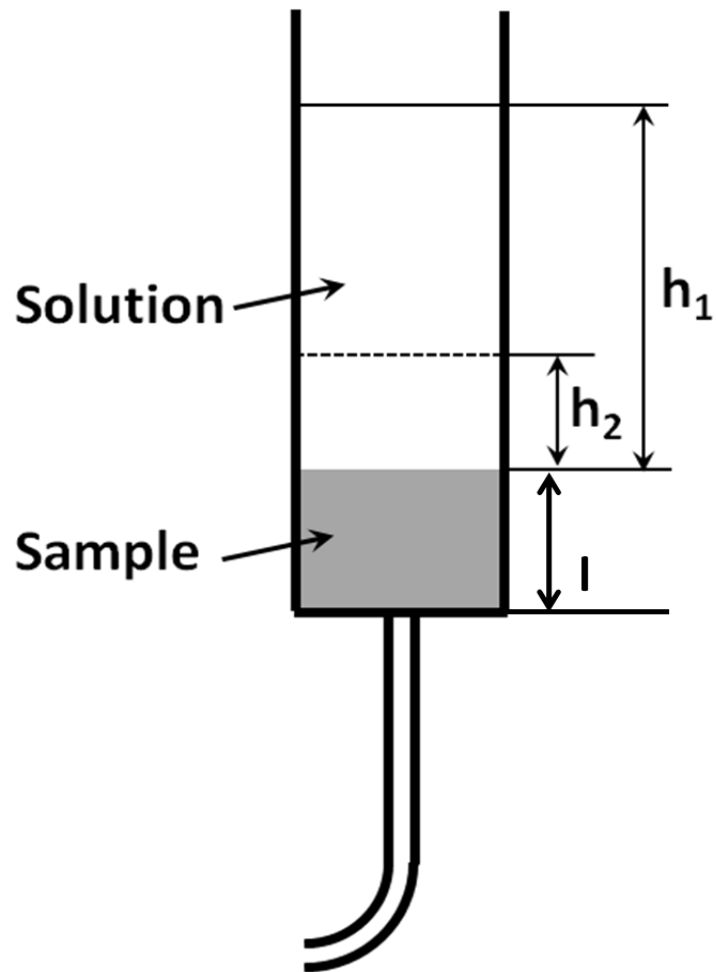


Figure 1. Falling head permeameter

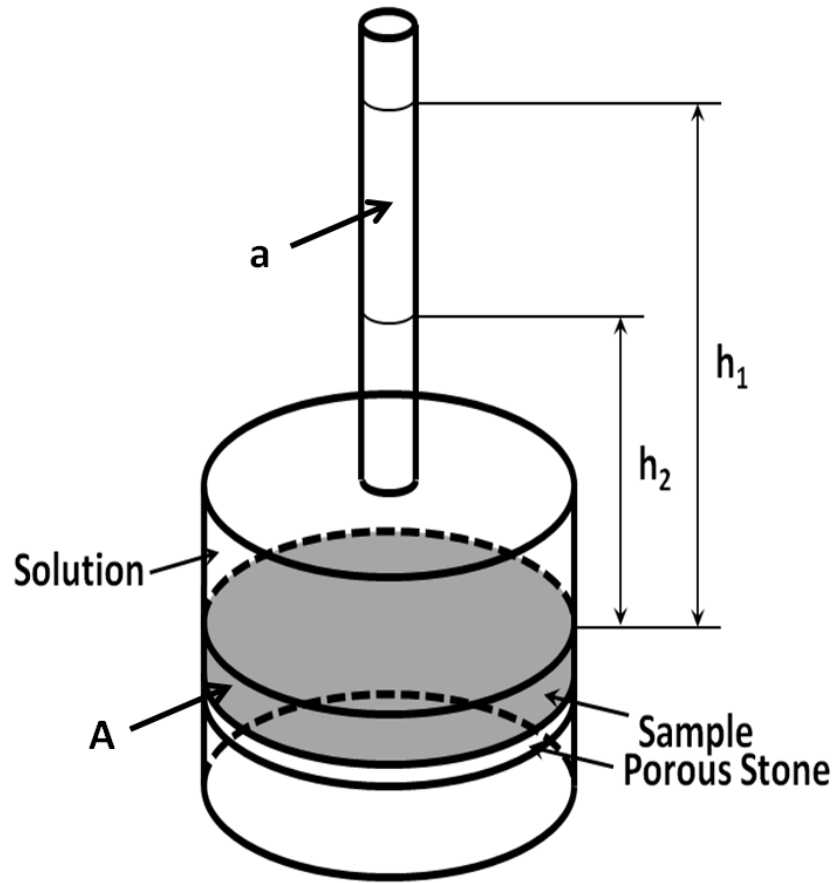


Figure 2. Modified flexible wall permeameter

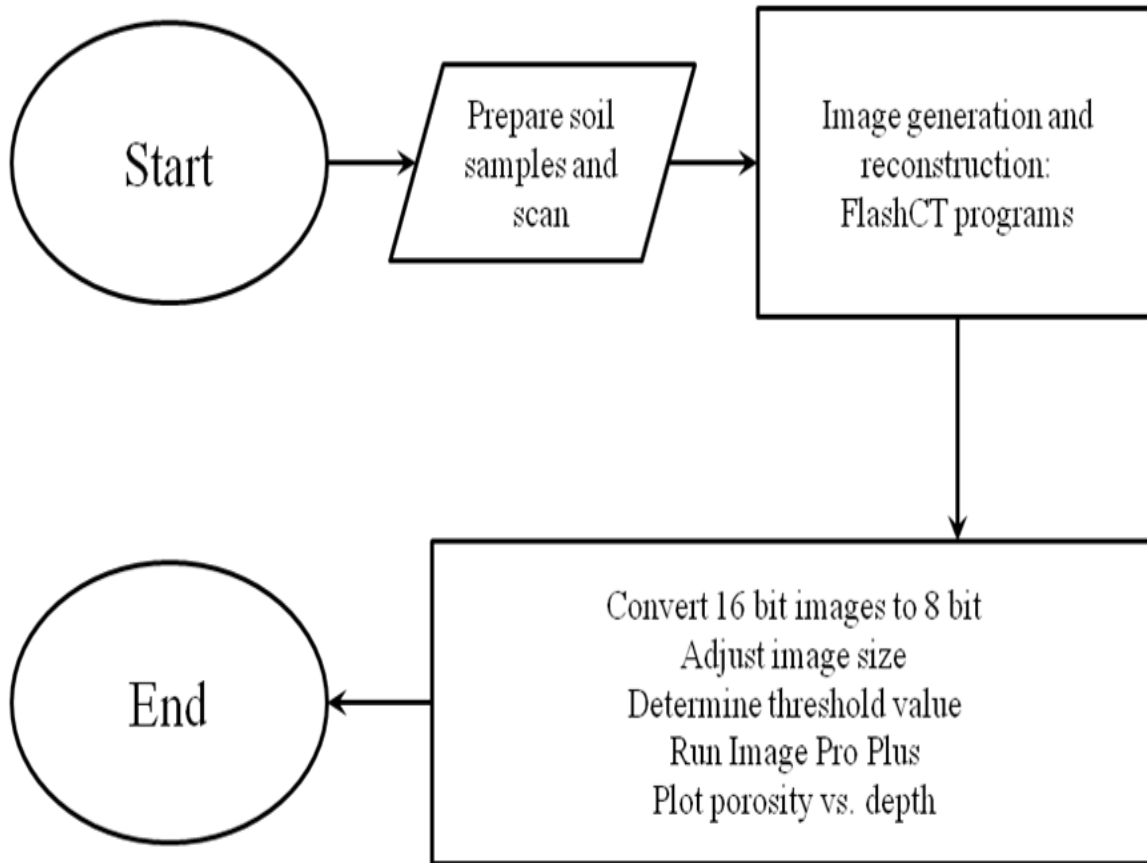


Figure 3. XRCT tests procedure

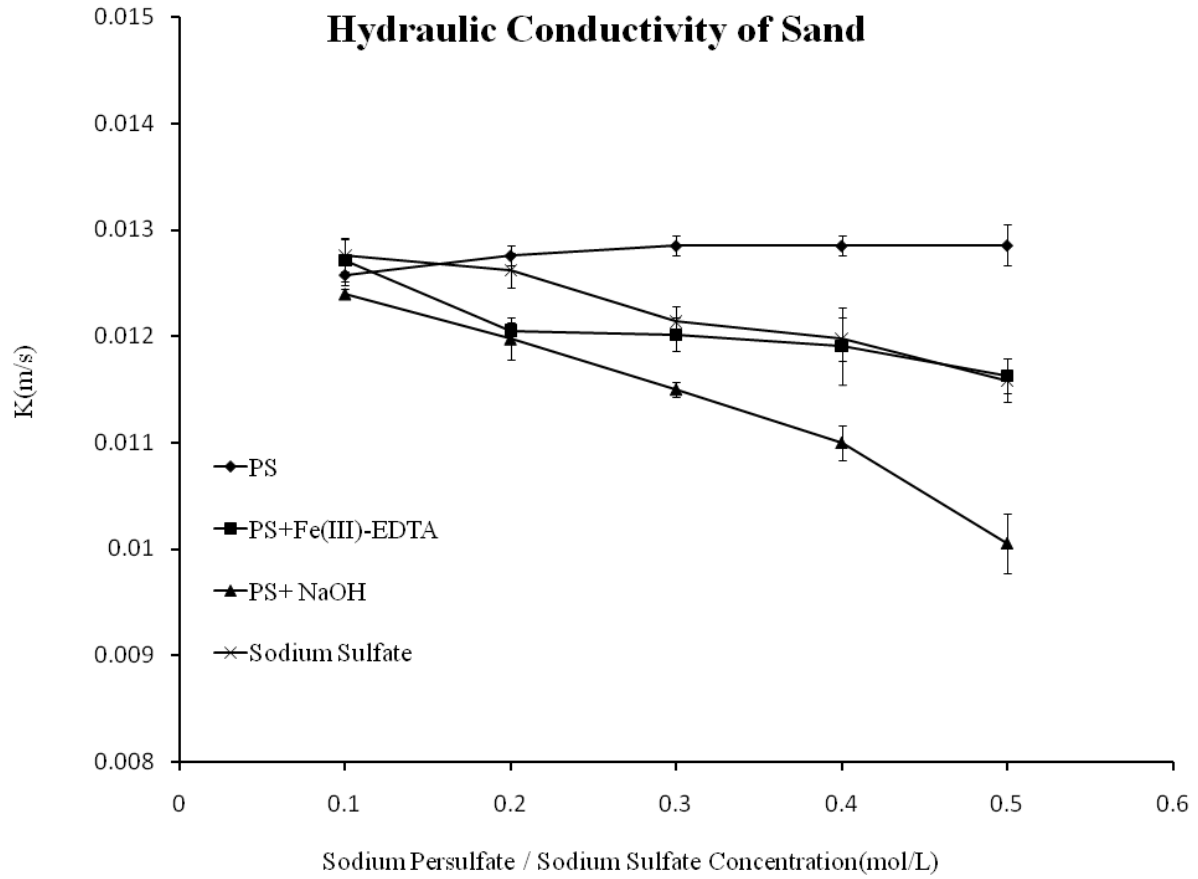


Figure 4. Effect of sodium persulfate and sulfate formulations on sand hydraulic conductivity

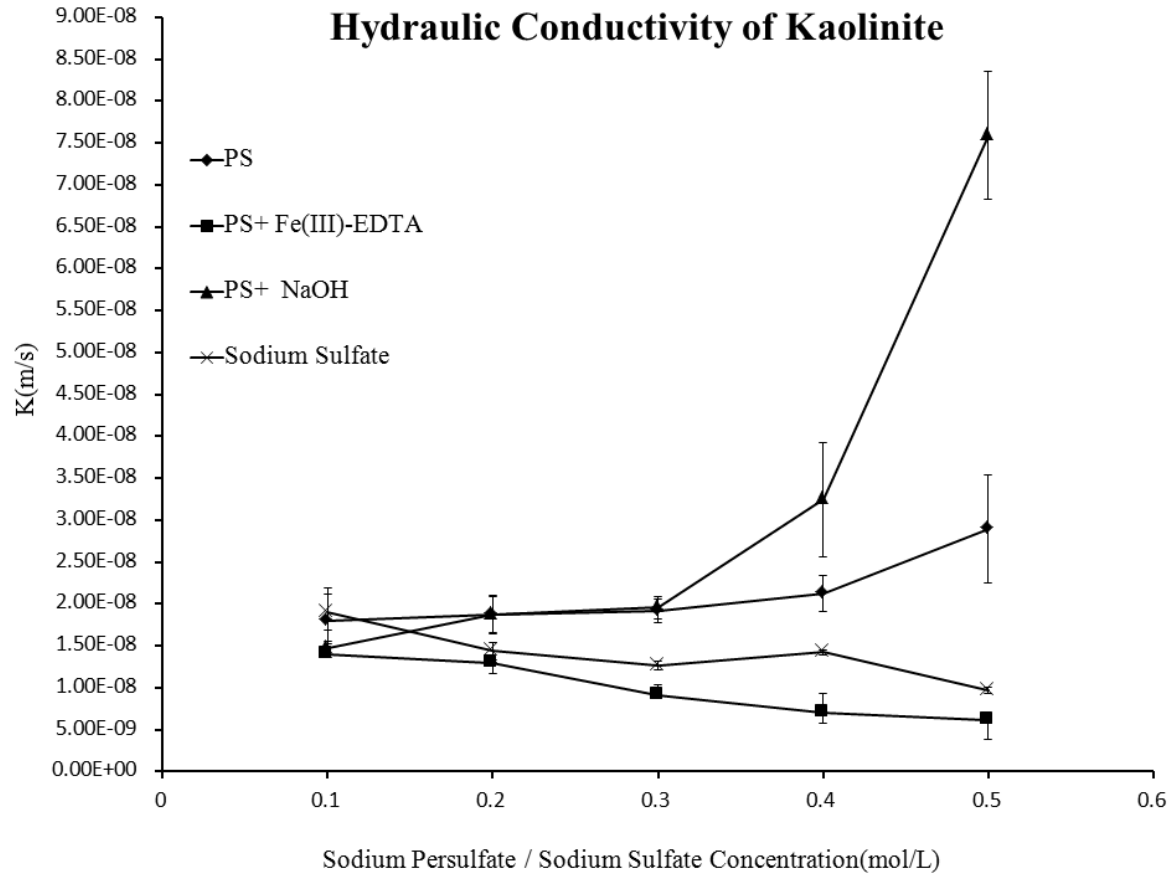


Figure 5. Effect of sodium persulfate and sulfate formulations on kaolinite hydraulic conductivity

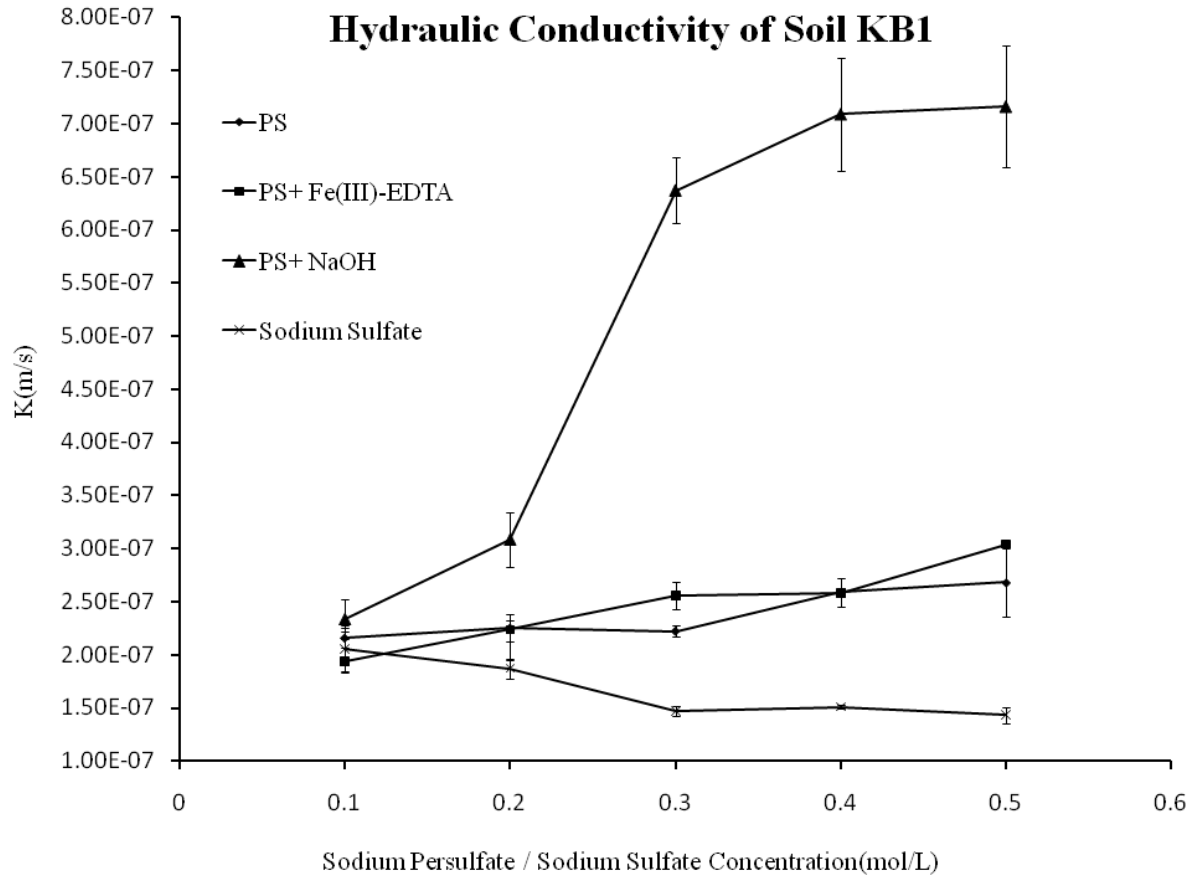


Figure 6. Effect of sodium persulfate and sulfate formulations on soil KB1 hydraulic conductivity

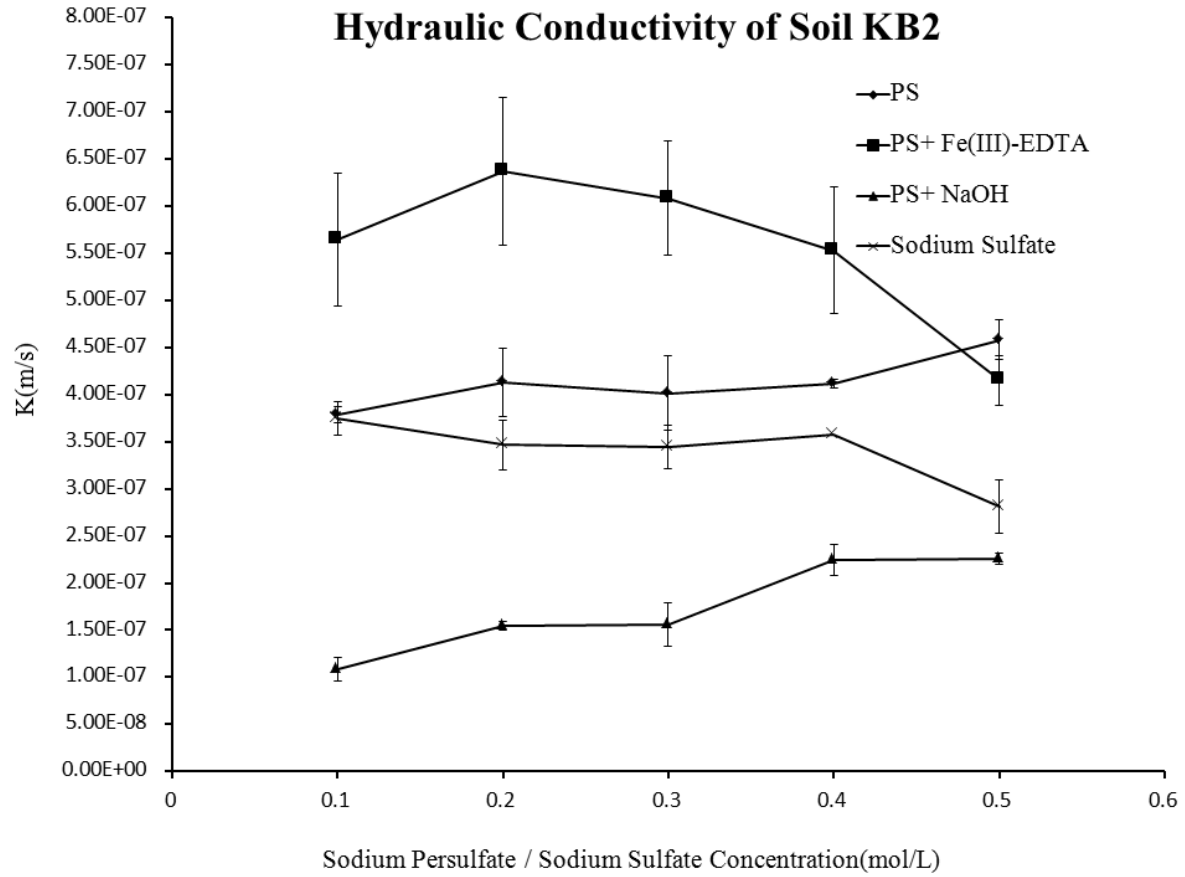


Figure 7. Effect of sodium persulfate and sulfate formulations on soil KB2 hydraulic conductivity

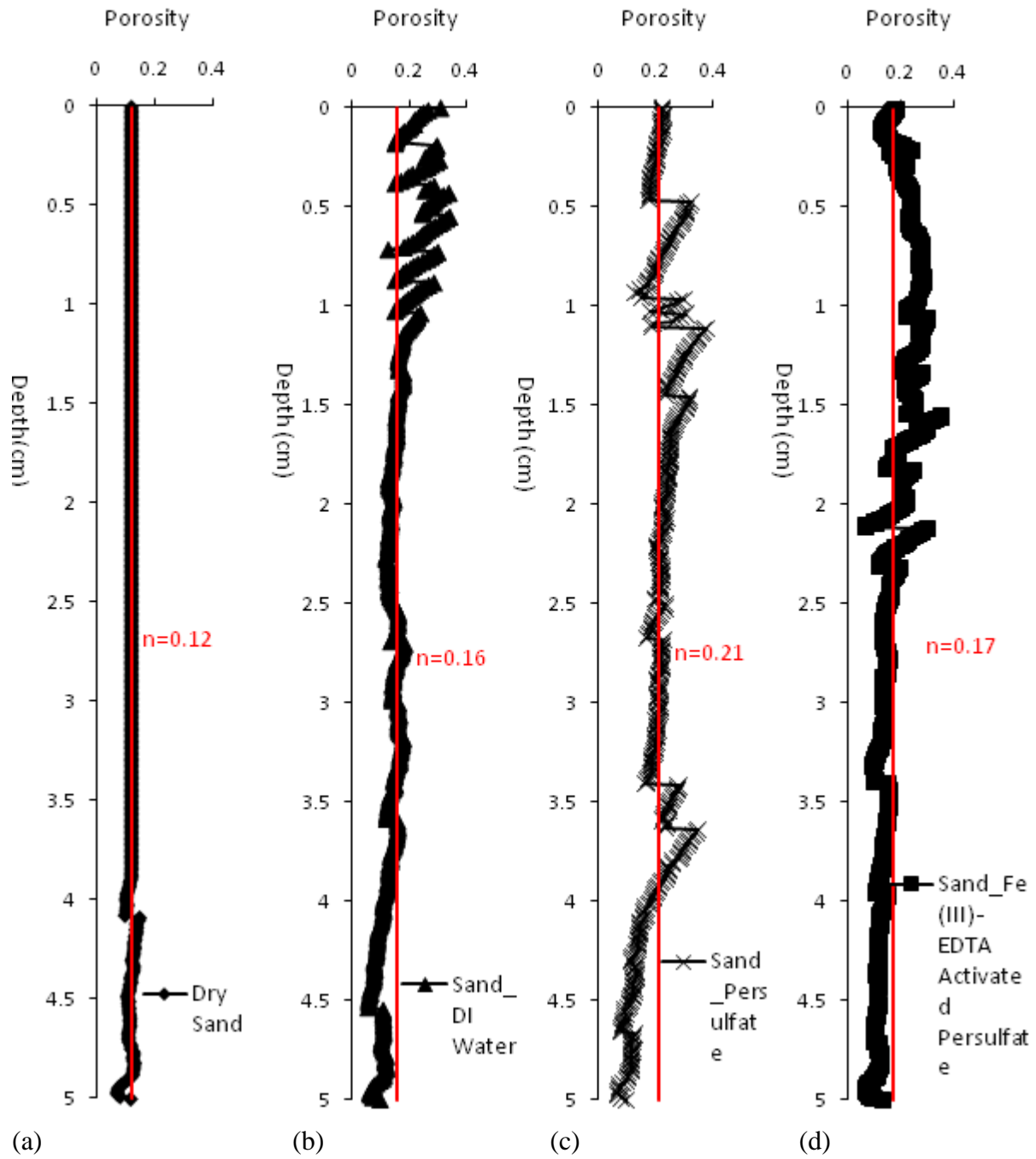


Figure 8. Porosity distribution with depth for sand and average porosity

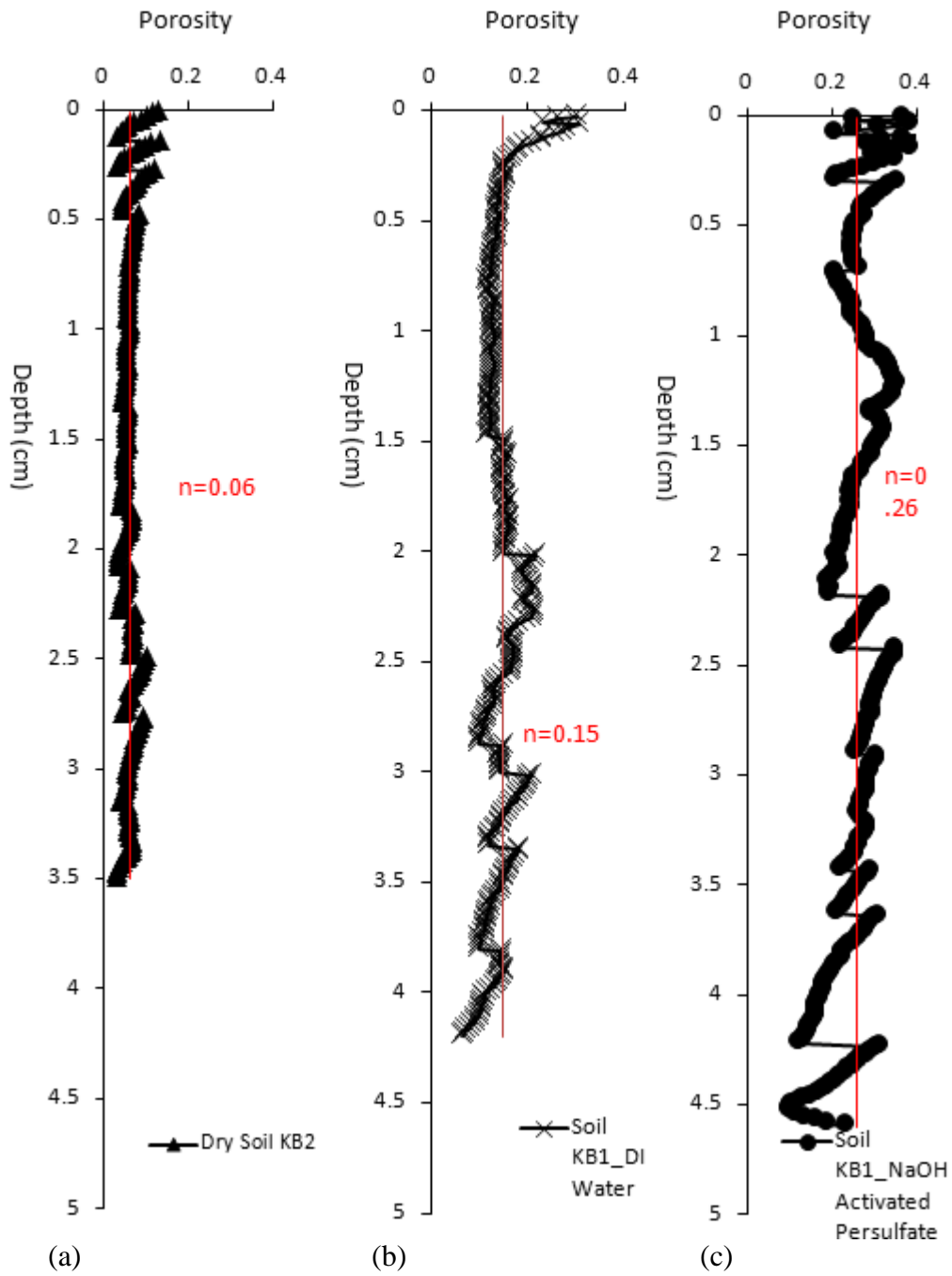


Figure 9. Porosity distribution with depth for soil KB1 and average porosity

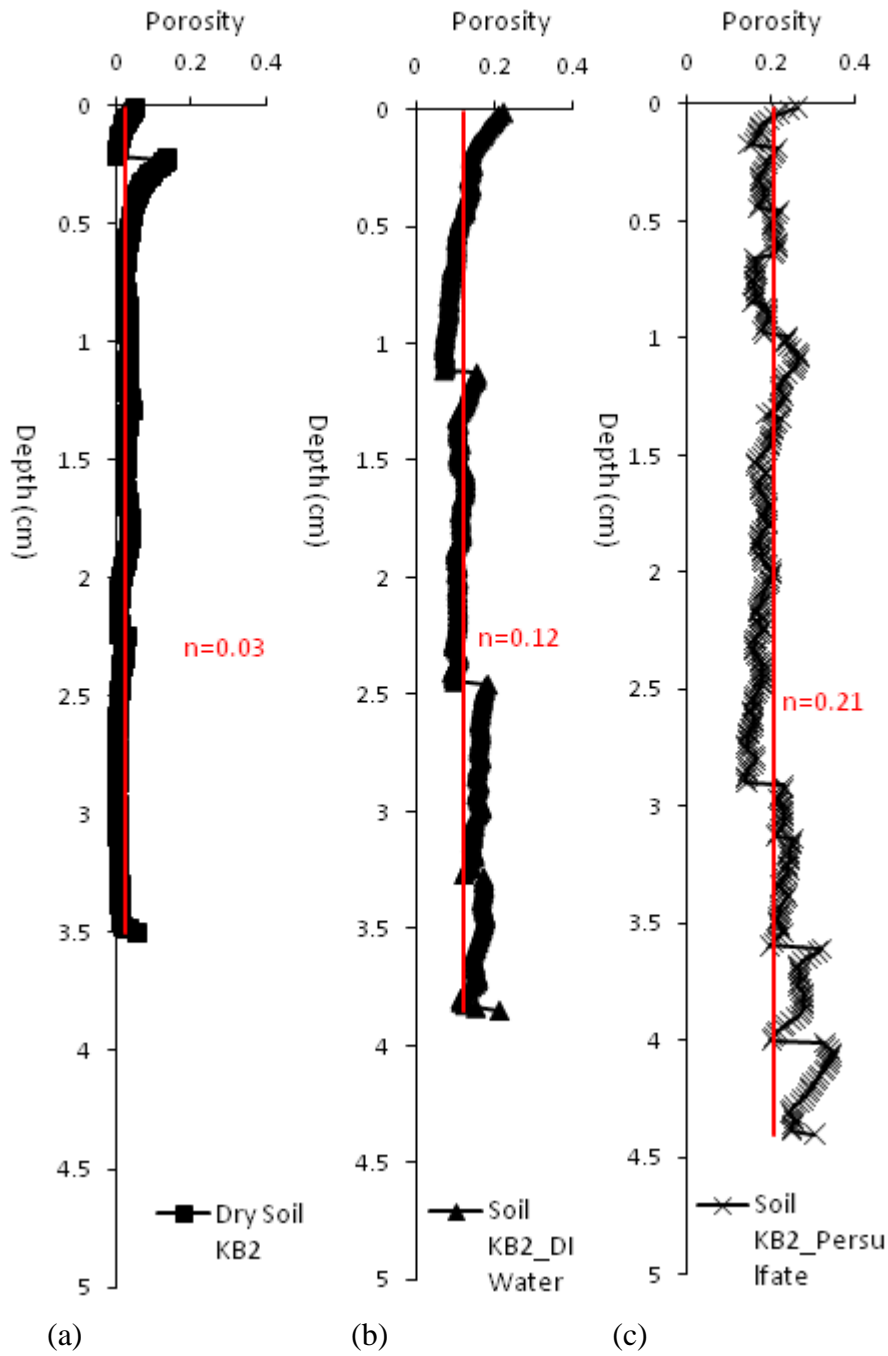


Figure 10. Porosity distribution with depth for soil KB2 and average porosity

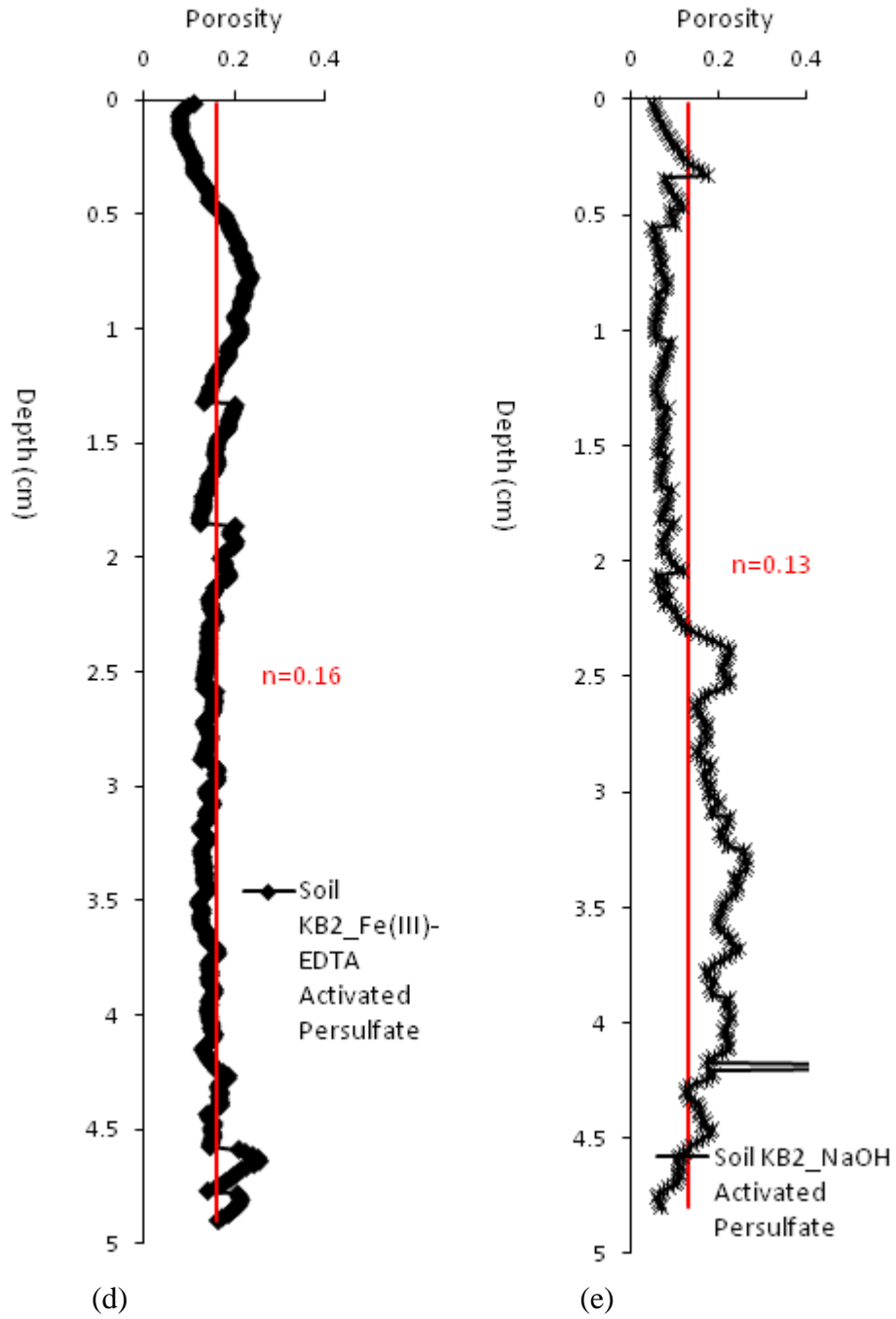


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