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Characterization of volcanic tuff pores pre- and post-underground nuclear detonation using ultra-small and small angle neutron scattering

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The ability to accurately model the subsurface transport of radionuclides is fundamental to the remote detection and characterization of underground nuclear explosion (UNE) events. Developing more sophisticated transport models presents a significant opportunity to enhance monitoring capabilities, particularly in the reliable prediction of signature migration. Experimentally determined characterization of geologic materials associated with transport properties is the pertinent base information for such robust model development and calibration. Here, we report results from an unprecedented study demonstrating changes to the pore and fracture network structures in geological materials in response to UNEs over nanometer to micrometer length scales. Volcanic tuffs of five different lithological formations from pre- and post-UNE environments were collected from the Nevada National Security Site. Combined ultra-small and small-angle neutron scattering techniques were used to characterize the tuff pore structure. The results demonstrate measurable differences in the specific surface area and porosity of samples pre- and post-shot from texturally similar lithological formations, indicating that pore properties can serve as a direct physical signature of a UNE. The results also provide experimentally determined transport parameters in support of advanced model development through the integration of gas migration, hydrodynamic simulations, and geologic framework models.

Keywords Neutron scattering, Tuff, Porosity, Pore structure, Underground nuclear explosion

Global nuclear explosion monitoring relies heavily on capturing both prompt and delayed multi-phenomenological signatures, such as the seismic signals and short-lived noble gas radioisotopes produced by an explosion^{1–4}. Improved monitoring and verification are critical needs that require the development and calibration of advanced models, with accurate, multi-scale geologic characterization, gas migration modeling, and hydrodynamic simulations. Robust models enabling a reliable, temporally constrained prediction of gas migration (i.e., the window of opportunity for detection), as well as the concentration of radionuclides gases that result from an underground nuclear explosion (UNE), are crucial in advancing UNE detection, location, and identification^{5–8}. A major step forward in developing high-fidelity, field predictive models is to calibrate and verify modeling efforts using experimentally-derived rock property data^{9–13}. Previous modeling studies suggest that rock properties, including fracture aperture, permeability, porosity, and saturation, have a significant influence on subsurface gas migration pathways^{2,3,6,8}. These properties are generally associated with the microstructure of geologic materials including pore and fracture morphology, pore size and volume distribution, pore and fracture connectivity, and pore surface area. Recent studies have unveiled the importance of pore scale (i.e., nanometers to micrometers) processes on macroscopic (i.e., centimeters to meters) transport properties that consequently affect flow migration and geophysical signal propagation on the field scale^{14–17}. Furthermore, the evolution of

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pore microstructure plays a decisive role in governing interaction of fluids with reactive surfaces. For example, previous studies show the impact of chemical processes, such as dissolution and precipitation of minerals, on pore-fracture opening, cementation, and healing, which results in altered flow paths and reaction rates^{18–22}.

During and following a UNE, microstructural property variation (i.e., deformation), such as enhanced or reduced pore-fracture networks and pore-fracture opening, closing and healing, may occur due to the rock's exposure to high pressure and temperatures²³, similar to observed changes in pore characteristics of condensed material from high explosive events^{24–29}. Many studies have observed the alteration of rock pore structure in response to chemical and mechanical stresses such as geochemical weathering²², energy exploration and repository science^{21,30–32}, and engineered barriers for waste isolation^{33–36}. Measurable changes in pore scale characteristics, particularly the generation of unique microfracturing such as the induction of trans-granular and grain-boundary microfractures and unique fracture network patterns, can be a direct physical signature of a UNE³⁷. Furthermore and most critically, pore structure alteration influences macroscopic transport properties that control gas migration and geophysical signal propagation^{38,39}. Therefore, pre- and post-shot experimentally determined pore scale properties should be integrated into models to more accurately simulate radionuclide gas transport through subsurface fractured rock to the ground surface, and subsequently into the atmosphere⁴⁰. Improving current gas transport models will provide better constraints on radionuclide and geophysical signature modeling for UNE detection, location, and identification⁴¹.

While conventional measurement of permeability using flow/diffusion experiments can provide important information on the transport properties of UNE-impacted lithological formations at macro scale⁴², these permeability measurements alone do not address the characteristics and sensitivity of the pores and pore networks to an explosive shock wave. Determining these factors is necessary to extrapolate findings and apply them to additional lithologies, such as those that occur at foreign test sites⁴³. As outlined above, pores and their network characteristics over very small scales are important affectors of rock transport properties. However, porosity and pore size distribution estimates from traditional electron imaging, including scanning electron microscopy (SEM), Focus Ion Beam-SEM (FIBS), and optical microscopy (OM) are limited to micro/millimeter scales and are subject to the effects of sample preparation for such measurements^{44–46}. Constraints in length scales, limited fields of view and the resulting difficulty in quantifying pore morphology with reliable statistical accuracy are additional problems that restrict the usefulness of these imaging techniques in producing data for predictive modeling of transport properties. By contrast, combined ultra-small and small angle neutron scattering (USANS/SANS) has been recognized as an effective technique to determine the statistical properties of rock pore structure over broad length scales from micrometers down to nanometers^{47–52}. Additionally, the penetrability of neutrons allows interrogating “bulk” and “intact” samples a few hundred micrometers thick. Thus, while the length scales probed by neutrons are small, the areas interrogated are relatively large, thereby providing a statistically significant sampling of the structure.

With this in mind, the goal of the project reported herein was to employ USANS/SANS to characterize and compare the pre- and post UNE pore network structure of volcanic tuffs from five different lithological formations, with the aim of determining how rock surrounding a UNE deforms upon explosion. Specific research objectives include (i) quantification of microporosity changes; (ii) comparison of deformation extent within different lithologies; and (iii) connection to larger-scale signature transport. Samples were collected from rock core retrieved from a stratigraphic column of variably welded and altered tuff from pre- and post-detonation environments at the Nevada National Security Site (NNSS)^{53,54}. Core samples were collected as part of the acknowledged project and no special permissions were required or obtained for their collection or use.

Sampling sites are located within the Miocene Southwest Nevada Volcanic Field, which is characterized by silicic ash-flow tuffs, bedded tuffs, and rhyolitic lava derived from the Calico Hills Formation (Silent Canyon Caldera, 13–14 Ma), the Paintbrush Group (Claim Canyon Caldera, 12.7–12.8 Ma), and the Timber Mountain Group (Timber Mountain Caldera, 11.5–12.5 Ma)³⁷. These ash-flow tuffs are generally laterally heterogeneous pyroclastic flow deposits and are composed of a mixture of ash, pumice, phenocrysts, and lithic fragments with a ranging degree of welding and post-depositional crystallization. Pre-shot cores were sampled from four core holes (UE-19b, UE-19f, UE-20c, and UE-20f, Table 1) on the Pahute Mesa near the Barnwell UNE source hole, UE-20az (Fig. 1a). Samples from UE-19b, UE-19f, UE-20c, and UE-20f are from boreholes drilled prior to Barnwell or other close UNEs, and are therefore pre-shot. Post-shot samples were taken from core hole UE-20az-NG-4 (Fig. 1b; Table 1). This suite of samples provides a unique opportunity to better describe nuclear signature transport related to historical testing in NNSS-relevant lithologies.

Based on a suite of lithologic properties, pre- and post-shot samples were categorized into five lithologies (Table 1): (1) zeolitic nonwelded bedded tuff; (2) welded tuff; (3) vitric nonwelded vitrophyric tuff; (4) vitric

Lithological Unit	Pre-shot Sample	Post-shot Sample
Zeolitic nonwelded tuff	UE-20c-2552	UE-20az-NG4-128
Welded tuff	UE-20c-654	UE-20az-NG4-240
Vitrophyric tuff	UE-19b-1022	UE-20az-NG4-470
Vitric nonwelded tuff	UE-19b-349	UE-20az-NG4-519
Rhyolitic lava flow	UE-19f-1987	UE-20az-NG4-900

Table 1. Samples measured using USANS/SANS. The core depth for each sample in feet is given as the number after the last hyphen of the core designations.

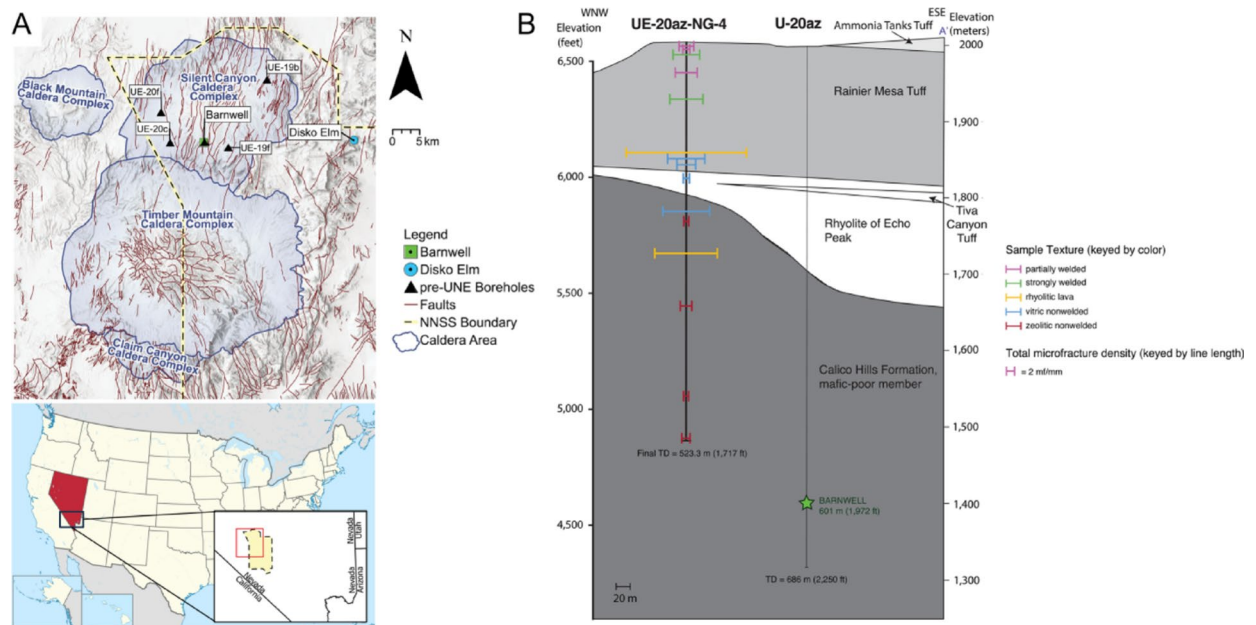


Fig. 1. (a) Map of the NNSS showing the proximal coreholes used for pre-shot analysis. Post-shot coreholes were taken from UE-20az-NG-4 at the Barnwell site. (b) Cross section of Barnwell and the UE-20az-NG-4 corehole. Reprinted with permission from Swanson et al.³⁷.

nonwelded bedded tuff; and (5) rhyolitic lava. We aimed to match pre-shot sample lithologies to the Barnwell post-shot samples as closely as possible using observations of mineralogy and texture. There is however some geologic variability and therefore not all the differences between pre-shot and post-shot samples can be attributed to effects from the UNE, as outlined in the discussion section below.

Results

Dimensional model description of pores in tuffs

The fractal model fit tool in Irena v.2.32, was used to carry out the best fit calculation of the particle/aggregate structures from power law exponents of the combined USANS/SANS data⁵⁵. This model fits the USANS/SANS data into separate domains with different power law scatter, separated by inflections or knees that delimit the power law domains. This analysis is based on the dimensional considerations outline in the Methods section but works well regardless of the interpretation of the power law. More information on this fitting can be found in Section S2 of the Supporting Information (SI). Scattering intensities calculated based on these models are plotted as the fitted curves in Figs. 2, 3, 4, 5 and 6 directly under the scattering data. Parameters derived from fitting the data to the model can be found in Table 2. These derived values provide a quantitative measure of particle and aggregate properties (i.e., pore properties) of tuff samples.

Most model parameters show little change between pre- and post-shot samples among lithologies that have good geologic matches: welded tuff, vitric nonwelded tuff, and vitrophyric tuff. There is a large difference in S , the surface to volume ratios, between pre-shot and post-shot samples for the zeolitic nonwelded tuff and rhyolitic lava lithologies. These samples are the two lithologies with the greatest mismatch in geologic context for pre-shot and post-shot samples, as described in the Discussion section. Thus, this parameter may be strongly affected by other geological histories such as alteration, whereby secondary minerals reduce pore volume but maintain pore surface area.

Given the nature of these samples, it is difficult to distinguish UNE-induced changes from diagenetic variability. This challenge has been described in more detail in Swanson et al.³⁷. One reason for this challenge is that similar microtextures can be produced by multiple processes, and thin sections sample only a small window of a rock's history. Quantification is additionally limited by the lack of a true "pre-event baseline" for that exact rock volume and by natural heterogeneity. For this reason, while we can point out where a clear mismatch appears to be present, this is not a characteristic which can be assessed quantitatively. Additionally, recommendations are made in the Conclusions for developing a future framework for improved discrimination of these effects.

The three lithologies with good matches in geologic context all show a modest reduction in S . This is unexpected, since pore collapse may be expected to reduce volume and maintain surface area, which would increase S . Of the three lithologies with good matches in geologic context, there is a mixed response of r , the size of individual scatterers, with one sample decreasing, one sample increasing, and one sample remaining the same. This suggests a minimal change in size of individual scatterers between pre- and post-shot samples.

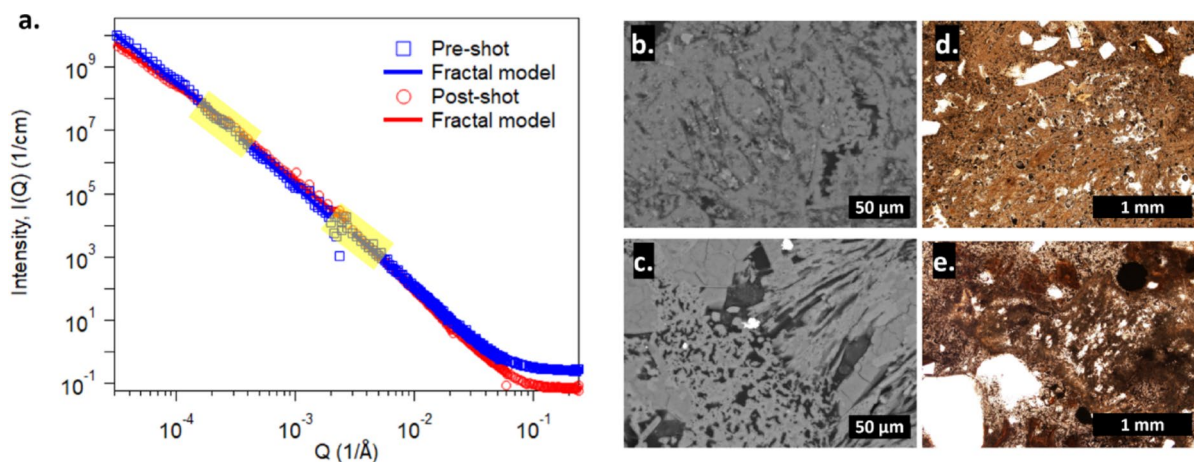


Fig. 2. Zeolitic nonwelded tuff pre- and post-shot samples, (a) combined USANS/SANS scattering curves, (b) pre-shot SEM image, (c) post-shot SEM image, (d) pre-shot thin section image, (e) post-shot thin section image.

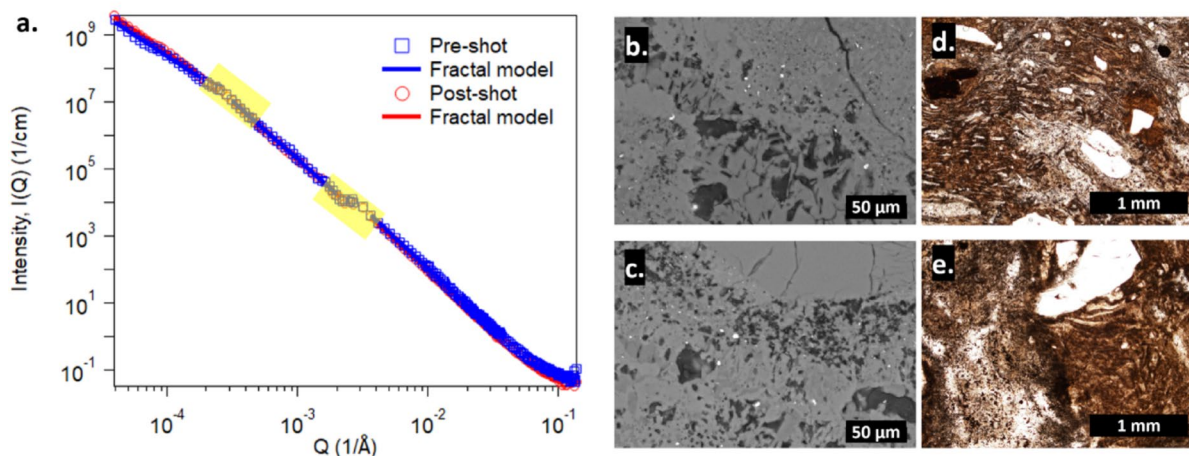


Fig. 3. Welded tuff pre- and post-shot samples, (a) combined USANS/SANS scattering curves, (b) pre-shot SEM image, (c) post-shot SEM image, (d) pre-shot thin section image, (e) post-shot thin section image.

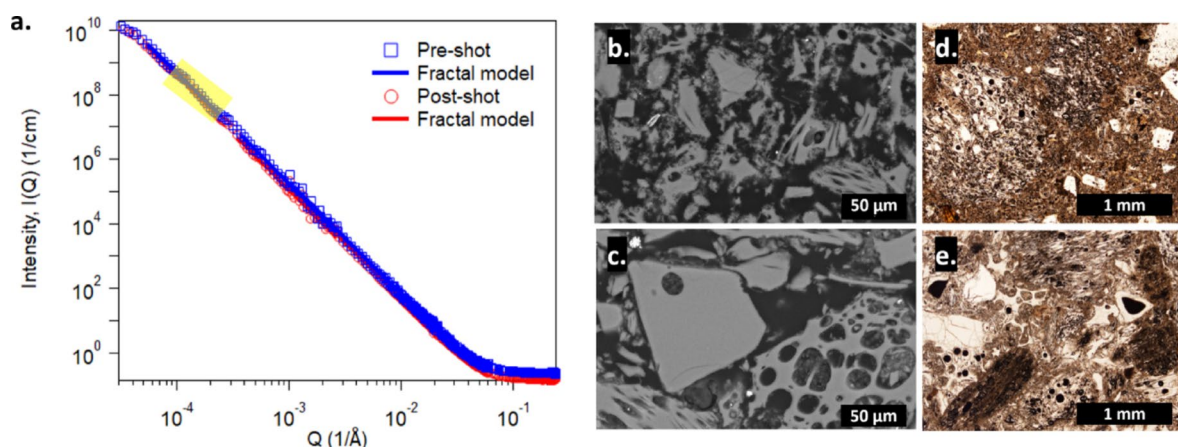


Fig. 4. Vitric nonwelded tuff pre- and post-shot samples, (a) combined USANS/SANS scattering curves, (b) pre-shot SEM image, (c) post-shot SEM image, (d) pre-shot thin section image, (e) post-shot thin section image.

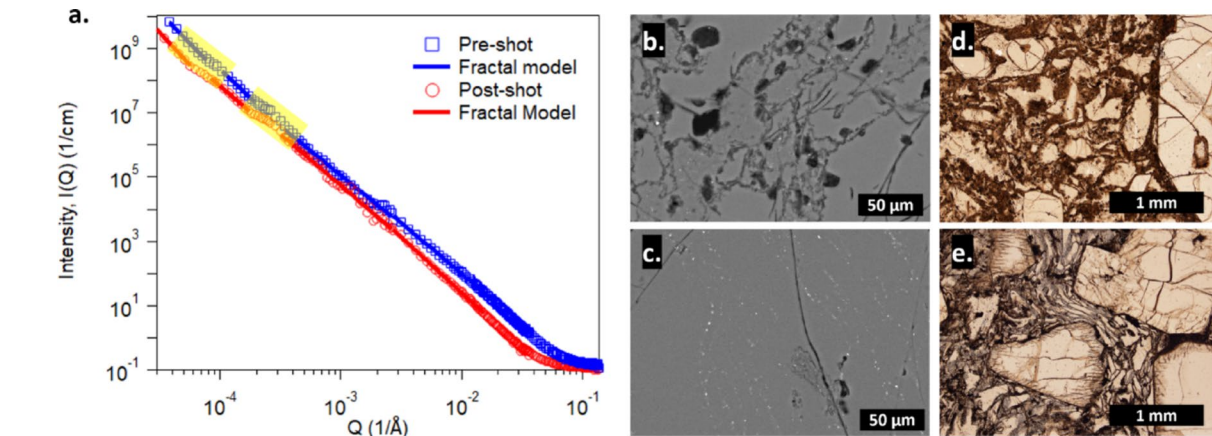


Fig. 5. Vitrophyric tuff pre- and post-shot samples, (a) combined USANS/SANS scattering curves, (b) pre-shot SEM image, (c) post-shot SEM image, (d) pre-shot thin section image, (e) post-shot thin section image.

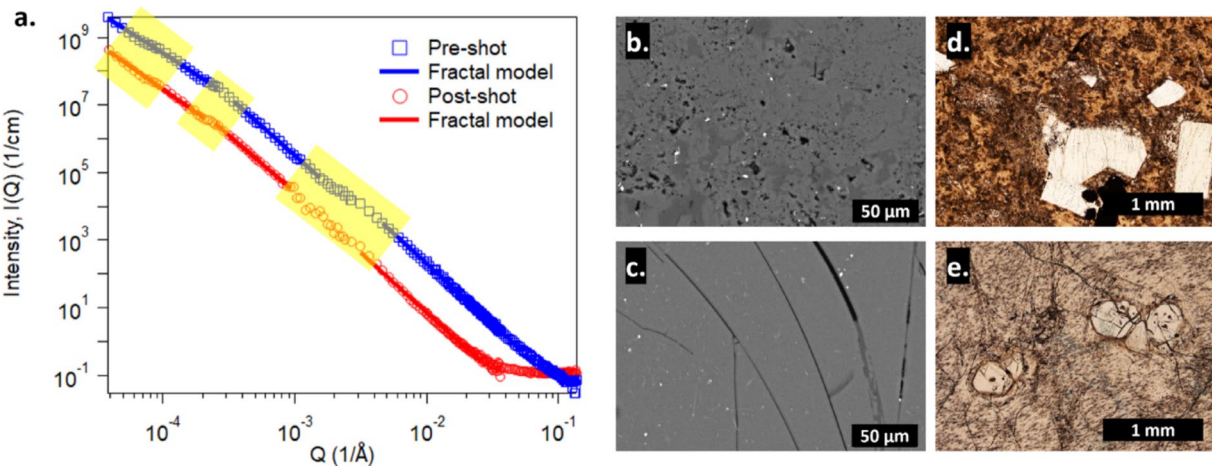


Fig. 6. Rhyolitic lava flow pre- and post-shot samples, (a) combined USANS/SANS scattering curves, (b) pre-shot SEM image, (c) post-shot SEM image, (d) pre-shot thin section image, (e) post-shot thin section image.

Lithological Unit	Pre- or post-shot	Mass fractal fit				Surface fractal fit			Physical interpretation
		D_m^a	P_r^b (Å)	P_v^c (cm ³)	R^d (Å)	D_s^e	S^f (cm ² /cm ³)	r^g (Å)	
Zeolitic nonwelded tuff	Pre-shot	NA ^h	NA	NA	NA	2.73	45	100,000	Decreases in scatterer size suggest pore collapse, while increases in S suggest the formation of new scattering surfaces such as microfractures.
	Post-shot	2.50	16,000	0.003	60,000	2.50	290	9000	
Welded tuff	Pre-shot	2.40	20,000	0.003	60,000	2.50	350	8000	Scatterers were bigger, yet have a smaller S post-shot, suggesting deformation.
	Post-shot	2.45	20,000	0.005	60,000	2.50	250	11,000	
Vitrophyric tuff	Pre-shot	2.40	40,000	0.003	100,000	2.80	60	50,000	Scatterer were smaller and had a smaller S, suggesting pore collapse.
	Post-shot	2.80	60,000	0.005	100,000	2.70	28	30,000	
Vitric nonwelded tuff	Pre-shot	NA	NA	NA	NA	2.55	80	50,000	Minimal changes in pore structure observed.
	Post-shot	NA	NA	NA	NA	2.55	68	50,000	
Rhyolitic lava flow	Pre-shot	2.50	20,000	0.006	60,000	2.55	550	8000	Large decrease in S despite similar scatterer size, suggesting significant porosity loss.
	Post-shot	2.40	20,000	0.005	60,000	2.40	30	8000	

Table 2. Model parameters fitted to scattering curves. ^a D_m : Mass fractal dimension. ^b P_r : Size of the fractal particle. ^c P_v : Volume of particles. ^d R : Distance between particles, which gives the size of the fractal aggregates. ^e D_s : Surface fractal dimension. ^f S : Surface area to volume ratio. ^g r : Size of the individual scatterers. ^hNA: not applicable. *Italic indicates poorer match in geologic context of pre- and post-shot samples. Thus, non-italic lithologies are more likely to show changes due to the UNE.*

Porosity of tuffs derived from neutron scattering data

Neutron scattering is a well-established method to provide porosity for condensed materials including rocks and high explosives^{26,27,44,50}. Numerical computation used to derive porosity of a sample from its scattering data is based on standard results of small angle x-ray scattering (SAS) theory, known as the Kratky transform⁵⁰. The integral of this transformation, assuming a two-phase (empty pores/voids and rock matrix) isotropic material, is the invariant, INV, and the porosity ϕ is obtained from the invariant as:

$$INV = \int_0^\infty Q^2 I(Q) dQ = 2\pi^2 (\Delta\rho)^2 \phi (1 - \phi) \quad (1)$$

where $\Delta\rho$, the scattering contrast, is the difference in the scattering length density between the solid phase and the pore. The use of the invariant removes the ambiguity of the dimensional model, as the integral measures the volume fraction of the pore phase, to the extent that is measured in the combined SANS/USANS vignette, regardless of the source of the scattering power law.

The neutron scattering length density for each phase of pseudo-molar mass M is:

$$\rho = \frac{N_A d}{M} \sum_j p_j \left(\sum_i s_i b_i \right)_j \quad (2)$$

where $N_A = 6.022 \times 10^{23}$ is Avogadro's number, d is density (in g/cm^3), s_i is the proportion by number of nucleus i in the compound j , p_j is the proportion by molecular number of the compound j in the mixture, and b_i is the coherent scattering amplitude for nucleus i .

To calculate ρ for tuffs from Eq. 2, we assume the matrix of tuff is composed of mixtures of oxides, $\sum M_n O_x$. Instead of atomic proportion, p_j here is the oxide molecule proportion. The chemical composition as oxide forms for each tuff sample were measured using XRF and given in wt% in Table S1 in the SI. For 100 g molar weight (M) of the mixture, $M = \sum_j (\text{oxide proportion})_j \times (\text{oxide mass})_j$. The scattering length density, ρ , can be calculated from Eq. 3 by substituting $N_A = 6.022 \times 10^{23}$, d from Table 3 (obtained from direct measurement on core samples), $M=100$ g, and the $\sum_i s_i b_i$ value in Table S2 in the SI, which was obtained for every oxide using data reported from Sears⁵⁶. The invariant (INV) values of every scattering curve were obtained from analysis of the results of fits using the unified model (Eq. S4) in Figs. 2, 3, 4, 5 and 6⁵⁷. Table 3 lists calculated ρ from Eq. 2, invariant values obtained from each curve fitting using the unified model, and porosity calculated from Eq. 1. Parameters fitted to scattering curves are provided in Table 2.

As seen from Table 3, porosity of zeolitic tuff post-shot increased by about 49% (UE-20c-2552 vs. NG4-128), whereas the porosity of rhyolitic lava post-shot decreased by more than 80% (UE-19f-1987 vs. NG4-900). It is important to note here that interpretation of the difference between pre- and post-shot porosity must consider the fitness of the sample geologic match, as outlined in the Discussion section below. The overall trend of porosity decreasing post-shot did match previous observations from core-scale porosity measurements for all samples other than zeolitic tuff (Table 3). While the magnitude of the measured porosity differed for our USANS/SANS derived values, this can be attributed to the fact that USANS/SANS is able to measure both open and closed porosity, unlike traditional porosimetry approaches which can only measure fluid-accessible pores⁵⁸. As a result, USANS/SANS porosity can be higher for samples with abundant nanopores. Additionally, measured porosity using USANS/SANS is limited to void structures smaller than the upper measurement limit of 3 μm , meaning some porosity contributions from larger voids are not full accounted for.

Lithology	Pre- or post-shot	SLD ρ ($\cdot 10^{10} \text{cm}^{-2}$)	Density d (g/cm^3)	Invariant ($\cdot 10^{21} \text{cm}^{-4}$)	SANS/USANS Measured Porosity, ϕ	Reported Porosity ^a	Reported Permeability (mD) ^b
Zeolitic nonwelded tuff	Pre-	2.14	1.53	1.13	0.147	0.362	0.15
	Post-	3.17	2.10	3.84	0.199	0.128	1.24 from 2 ft away
Welded tuff	Pre-	3.35	2.29	4.37	0.272	0.094	0.47
	Post-	3.38	2.25	3.53	0.195	0.088	1.1, 0.63
Vitrophyric tuff	Pre-	3.50	2.33	3.53	0.177	0.033	0.000075
	Post-	2.34	1.60	0.59	0.058	0.011	N/A
Vitric nonwelded tuff	Pre-	1.74	1.20	0.59	0.113	0.485	62
	Post-	1.75	1.20	0.59	0.111	0.405	57.6, 83
Rhyolitic lava flow	Pre-	3.46	2.31	4.52	0.260	0.086	0.00033
	Post-	3.32	2.30	0.98	0.047	0.026	0.18

Table 3. Calculated scattering length density, ρ , from Eq. 3, invariant values obtained from each curve fitting using a unified model, and porosity calculated from Eq. 2. The SLD of the pore, which is used to calculate the scattering contrast, $\Delta\rho$, is assumed to be 0. Italic lithologies have poor matches in geologic context between pre- and post-shot samples and may reflect differences in alteration as well as UNE-induced changes. ^afrom SAND report and USGS ds297; ^bfrom SAND2018-9134 Report (Broome et al. 2018), SAND2019-14378 Report (Heath et al. 2019), and "From ng4+6_Permability summary_12-7-2015 by Broome".

Discussion

Alteration of NNSS-relevant lithologies

Zeolitic Nonwelded Tuff

Inspection of the USANS/SANS data shows that post-shot zeolitic nonwelded tuff (Fig. 2) has a smaller scattering intensity relative to the pre-shot equivalent, by a factor of 2 to 3, for Q -values below an inflection point around $Q = 2.3 \times 10^{-4} \text{ \AA}^{-1}$. If this difference is due to the UNE, it would suggest some degree of pore or void collapse over the USANS accessible length scales between 0.43 and 3.3 μm . The pre-shot sample is best fit by a single power law with $n = 3.25$ over the Q range of 4×10^{-5} to $4.6 \times 10^{-2} \text{ \AA}^{-1}$, comprising ten orders of magnitude in $I(Q)$. On the other hand, the post-shot sample appears to have 3 distinct power law domains. For Q less than $2.1 \times 10^{-4} \text{ \AA}^{-1}$, $n = 2.61 \pm 0.01$. Over the intermediate Q of 3.1×10^{-4} to $2.7 \times 10^{-3} \text{ \AA}^{-1}$, $n = 3.1$. Over larger Q , 2.7×10^{-3} to $0.029 \text{ \AA}^{-1}$, the power law slope steepens to $n = 3.7$.

Welded Tuff

Similarly, for the welded tuff samples, we divide the USANS/SANS data into power law domains defined by inflection points (Fig. 3). For Q less than $2.1 \times 10^{-4} \text{ \AA}^{-1}$ in welded tuff, the value of n indicated a slight difference in dimension of 2.54 ± 0.01 for the pre-shot sample compared to 2.71 ± 0.01 for the post-shot sample. Over an intermediate Q -domain, 3.1×10^{-4} to $2.1 \times 10^{-3} \text{ \AA}^{-1}$, the fits gave a slight difference in the exponent of 3.45 ± 0.02 for pre-shot and 3.56 ± 0.02 for post-shot samples. Over the largest Q -domain, 3.8×10^{-3} to 3.4×10^{-2} the values are essentially identical, with $n = 3.67 \pm 0.01$ and 3.69 ± 0.01 for pre- and post-UNE samples, respectively.

Vitric Non-welded Tuff

Vitric nonwelded bedded tuff also showed fairly consistent power law scattering over the entire domain (Fig. 4). From Q equal to 5.5×10^{-5} to $2.5 \times 10^{-4} \text{ \AA}^{-1}$, where there is a slight inflection, the n values were 3.34 ± 0.01 and 3.34 ± 0.01 pre- and post-shot; for 3.1×10^{-4} to $2.3 \times 10^{-3} \text{ \AA}^{-1}$, the exponents are 3.64 ± 0.02 and 3.68 ± 0.03 pre- and post-shot; and finally for Q -values from 1×10^{-3} to $2.7 \times 10^{-2} \text{ \AA}^{-1}$ $n = 3.38 \pm 0.01$ and 3.39 ± 0.01 for the pre- and post-shot samples.

Vitrophyric Tuff

The vitrophyric tuff lithology shows a distinct difference between pre-shot and post-shot samples (Fig. 5). The largest and smallest values of Q show a factor of 4 difference in intensity, with the pre-shot sample having higher Q values than the post-shot sample. This factor of 4 difference applies over the Q -domain below the inflection at $3.4 \times 10^{-5} \text{ \AA}^{-1}$ and for Q above $3.3 \times 10^{-3} \text{ \AA}^{-1}$. In the intermediate Q -domain, the difference was about a factor of 2 between the pre- and post-shot samples. Detailed features of the combined SANS and USANS data are sufficiently subtle that it could result in over fitting the data in domains with different values of n ; thus, these data are best fit through the linear double-log region between 3.9×10^{-5} and $2.7 \times 10^{-2} \text{ \AA}^{-1}$, giving $n = 3.14$ and 3.22 for the pre- and post-shot samples, respectively, over ten orders of magnitude in intensity. The region in the pre-shot sample that is lower in intensity for Q between $\sim 4 \times 10^{-4}$ and $\sim 2 \times 10^{-3} \text{ \AA}^{-1}$ shows $n = 3.1$, a value not significantly different from that determined over the large Q domain. These differences in the USANS/SANS scattering curves indicate pore and void collapse with a stronger length scale dependence relative to other lithologies.

Rhyolitic lava

Lastly, rhyolitic lava flow showed the most significant difference in intensity, with pre-shot samples having intensity values a factor of ~ 14 higher than post-shot samples over most of the measured Q -domain (Fig. 6). The pre-shot data show inflections at 8×10^{-5} , 2.1×10^{-4} and $2.2 \times 10^{-3} \text{ \AA}^{-1}$, with the power law n being 3 in the lower Q -domain and 3.5 over larger Q . At the low- Q portion of the USANS post-shot data, there are two inflection points at 9×10^{-5} and $2.1 \times 10^{-4} \text{ \AA}^{-1}$, suggesting a crossover in the scatter from components with characteristic sizes of about 1 μm and about 0.47 μm , respectively. The value of n over each of these limited domains is 3.0. Over larger Q , the scatter falls off with a single power law over six orders of magnitude in intensity between $Q = 2.9 \times 10^{-4}$ and $0.018 \text{ \AA}^{-1}$, with $n = 3.6 \pm 0.01$ for both the pre- and post-shot samples.

It is important to note in our interpretation that the differences in characteristics as seen in the USANS/SANS data may not be entirely attributable to the UNE. In particular, while welded tuff, vitric non-welded tuff, and vitrophyric tuff showed close texture match between pre-shot and post-shot samples, this was not the case for the zeolitic tuff and rhyolitic lava. The post-shot zeolitic nonwelded sample came from a sample depth of only 128 feet and has a substantially lower zeolite content, in contrast to the pre-shot sample coming from a depth of 2552 ft and being highly zeolitic. Because of secondary alteration, it would be expected that the deeper sample (pre-shot) has lower porosity, and perhaps smaller pores, than the shallower sample (post-shot). Thus, for zeolitic nonwelded samples, it is ambiguous whether these differences relate to the geologic environment, to the UNE effects, or a combination. For rhyolitic lava, the pre-shot sample shows significantly more secondary alteration than the post-shot sample. Thus, for these two lithologies, it is ambiguous how much of the difference in USANS/SANS curves is due to geologic alteration, and how much may result from the UNE.

Based on the interpretation of dimensional models as described in the Methods section below and in Section S2 of the SI, we conclude that over length scales smaller than 0.36 μm , the scatter from rhyolitic lava, welded tuff and zeolitic nonwelded bedded tuff results from rough interfaces or power law-like void and pore size distributions. Above this length scale, voids and pores show scaling consistent with networks of channels. On the other hand, scatter from vitric nonwelded vitrophyric and vitric nonwelded bedded tuffs appears to result from surfaces or from pore void size distributions with no obvious contribution from mass fractals. Rhyolitic lava, vitric nonwelded vitrophyric and zeolitic nonwelded bedded tuffs showed different degrees of length scale dependent susceptibility to UNE shock, with rhyolitic lava having the most substantial evidence of pore crush,

while welded and vitric nonwelded bedded tuffs showed no evidence of pore crush. However, it is important to note that for rhyolitic lava and zeolitic tuff, changes in power law may also be attributed to other geological factors such as alteration.

Changes in porosity derived from neutron scattering

The relationship between changes in specific surface area (SSA) and porosity from USANS/SANS methods as a function of depth is shown in Fig. 7. These changes are calculated as the difference between natural (i.e., pre-shot) and shocked (i.e., post-shot) formations. The loss in surface area and porosity is largest for rhyolitic tuff, which is located at a depth of approximately 900 feet below the surface and is therefore closest to the explosive source. Meanwhile, zeolitic tuff, the only sample to demonstrate a gain in surface area and porosity, is farthest from the explosive source and is located at a shallower depth. It is possible that the closer proximity to the explosive source and a higher depth (i.e., higher overburden pressure) contributed to the more significant reduction of porosity and surface area for rhyolitic tuff, although pre-existing textural differences, including substantial differences in zeolite content, may also have played a role for this lithology⁵⁹.

Porosity is related to tortuosity according to the Millington-Quirk (MQ) empirical relationship⁶⁰:

$$\tau = \frac{\theta_a^{7/3}}{\varphi^2} \quad (3)$$

where θ_a is the air-filled porosity (m^3/m^3), defined as the volume of air divided by the total volume of rock (alternatively, $\theta_a = (1 - S_w)\theta$, where S_w is liquid saturation (m^3/m^3)), and φ is the porosity (m^3/m^3). Tortuosity is further related to effective diffusivity according to:

$$D_{\text{eff}} = \tau D_{\text{free}} \quad (4)$$

where D_{eff} is the effective diffusion coefficient and D_{free} is the free-air diffusion coefficient. While these relationships can give some insight into the expected gas transport through UNE-impacted tuff samples, it is important to note that core- to field-scale transport is much more complicated and will be controlled by fracture-matrix interactions, rather than matrix transport alone⁶¹.

Comparisons with larger-scale observations

The samples discussed here have had larger-scale observations previously published for permeability and porosity⁶², mechanical properties⁴², and microfracture density³⁷. A comparison of these observations at different scales can give greater insight into the specific deformation mechanisms that occur within each lithology.

Figure 8 presents the measured changes in pore scale properties (porosity and specific surface area), as well as the macroscopic transport property of permeability, which was obtained from direct measurement on the core sample as part of the Underground Nuclear Explosion Signatures Experiment (UNESE)⁴². Zeolitic tuff and rhyolitic lava represent end members in terms of their individual responses to a UNE, with zeolitic tuff demonstrating the only increase in porosity and SSA, where rhyolitic lava had the largest decrease in both parameters. However, as mentioned previously, these two lithologies have the poorest textural correlation between pre-shot and post-shot samples.

Porosity values calculated from USANS/SANS techniques are fairly different from porosities measured in the lab, as reported in Table 3. Since they are measuring at different scales, this is not unexpected. The USANS/SANS technique is only sensitive to pores with sizes smaller than about $3 \mu\text{m}$, whereas the lab-based porosity measurements are only sensitive to pores that are sufficiently large and connected for mercury or nitrogen to reach them⁶³. There is also an apparent difference in the direction of change between pre- and post-shot zeolitic nonwelded samples between SANS-derived porosities (increase) and lab-derived porosities (decrease). This

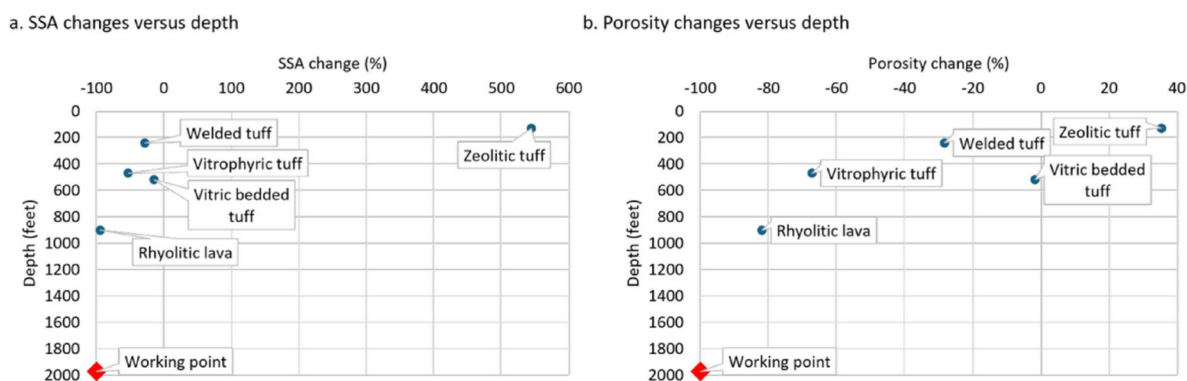


Fig. 7. The change between pre- and post-shot specific surface area (a) and porosity (b) in percent as a function of depth to explosive source (~2000 ft BLS). Zero indicates no change, negative values indicate a decrease, and positive values indicate an increase. Note that rhyolitic tuff at a depth of 900 ft and zeolitic tuff at a depth of 128 ft span the sampling depth range.

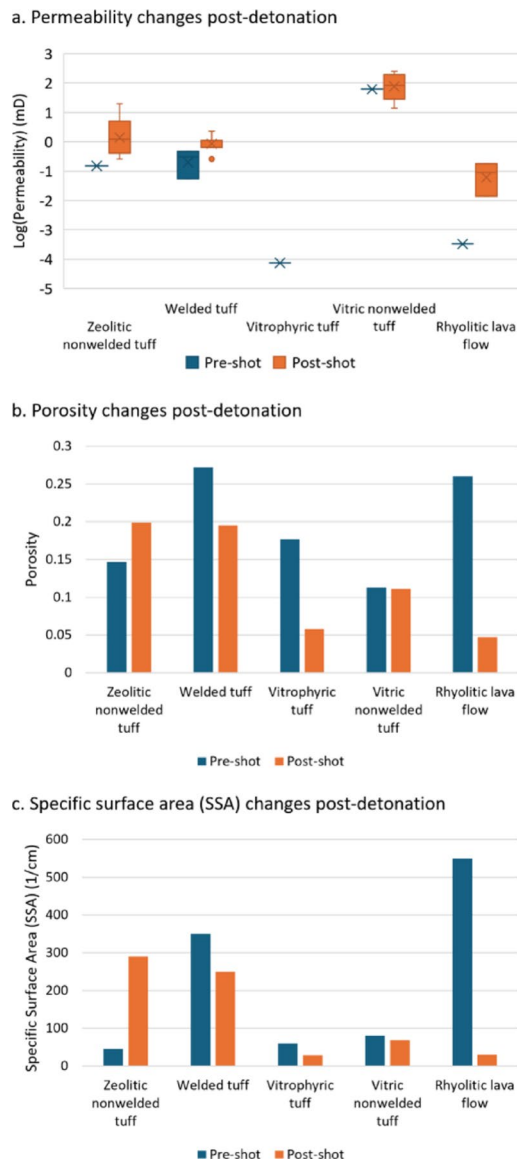


Fig. 8. Comparison between pre- (blue bar) and post-shot (orange bar) formation properties for (a) permeability, (b) porosity, and (c) specific surface area. Permeability was experimentally measured using a number of core samples from the same lithology at 500 psi confining pressure. Pre-shot permeability was not measured for a comparable vitrophyric tuff sample. The porosity (b) and specific surface area (c) represent small angle neutron scattering results from this study.

indicates that the nano- and micro-scale behaviors may be different for this rock type. That being said, there are substantial differences in alteration between these samples, with the post-shot sample having a much lower zeolite content as compared to the pre-shot sample. The other four lithologies show either a decrease, or no change, in porosity from both measurement techniques.

Despite these differences, all formations showed a higher permeability in the post-shot samples relative to the pre-shot equivalents. Most lithologies (except zeolitic tuff) showed a decrease in both SANS-derived and core-scale porosities for post-shot samples relative to pre-shot samples, as well as SSA. Thus, porosity and SSA do not necessarily directly scale with permeability. Core-scale permeability increase despite decreases in porosity and SSA indicates that the differences in these samples are scale-dependent⁶⁴. One possibility is that the smaller pores are crushed in a way that connects larger scale pores and/or fractures. In particular, crushing of grains and collapse of fine pores can concentrate stress along grain boundaries and within grains, promoting the development of transgranular microfractures. These microfractures can bridge otherwise isolated pores, effectively short-circuiting the pore network and increasing hydraulic connectivity despite an overall reduction in porosity. Swanson et al.³⁷ found an increase in transgranular microfractures across all examined lithologies in post-shot samples relative to pre-shot equivalents, supporting the interpretation that UNE-induced damage increases the connectivity of pores and fractures. At the core scale, permeability is strongly controlled by the presence and connectivity of the most conductive pathways rather than by total pore volume. Thus, even a

modest increase in the density or connectivity of microfractures can disproportionately increase permeability, negating the effect of porosity loss at the matrix scale.

SANS and USANS data reported here have the potential to better understand one of the enigmas reported by Swanson et al.³⁷ that the vitric nonwelded tuff, which has, by far, the lowest strength of all the lithologies⁴², has either a minimal increase in microfractures, or no increase in microfractures for post-shot samples over pre-shot equivalents. There are no deformation bands, the type of deformation expected from tectonic processes⁶⁵, either. This observation begs the question: if this rock is so weak, why is the deformation so minimal? The SANS and USANS data show the same pattern as the larger-scale observations: very little change in either porosity or SSA between pre-shot and post-shot samples in the very weak, vitric nonwelded bedded lithology. This may lend support to the possibility that the vitric nonwelded bedded rock samples happen to be located between the near-source, high strain region, and the near-surface spalled region that experienced tensile stresses. The post-shot vitric nonwelded bedded sample thus encountered lower stress than other lithologies. However, it is still possible that this lithology deforms via a mechanism that does not lead to measurable changes in any of these observations.

Conclusions

In the current study, we demonstrate that there are differences in rock properties between post-UNE samples and pre-UNE equivalents at very small scales ($< 5 \mu\text{m}$) measurable from SANS and USANS techniques. For all samples other than zeolitic non-welded tuff, nanoporosity and specific surface area decreased in post-UNE samples. While this may have the potential to hinder gas transport within rock matrices, on the larger scale, UNE detonation increased core-scale permeability likely due to larger fracture formation. However, changes in both permeability and porosity were much smaller for vitric nonwelded tuff compared to other lithologies, suggesting that this rock type is more resistant to explosive deformation.

The development of a decision framework is needed that can explicitly separate UNE-induced changes from pre-existing diagenetic or alteration variability. This approach should first define a set of measurable attributes expected to vary with UNE occurrence (e.g., nanoporosity/SSA as observed in this study, microfractures, mineral phase changes, etc.). Next, control samples must be attained to determine consistency of these attribution changes across samples with known diagenetic histories. Finally, a transparent classifier should be employed to determine the probabilities that observed changes are explosion-induced or a result of pre-existing variability. Such an approach will require detailed physical and chemical analyses of many more samples but will be an important undertaking and is supported by our preliminary observations.

Discerning how rock surrounding a UNE deforms upon explosion is important for detecting and characterizing a UNE⁶⁶. Previous models of post-UNE signature transport often ignore damage zone extent or assume simplistic geometries without accounting for lithologic variability, undermining realistic modeling of gas migration⁵. The novel observations presented here of nanoscale pore response to UNEs provide additional insight into rock matrix deformation by subsurface explosive stresses. More work is needed to understand how these microporosity changes contribute to field-scale signature transport, particularly through the integration of these responses into larger scale models.

Methods

Sample preparation

Additional core and thin section images of samples can be found in Figure S1 in the SI. To prepare samples for USANS/SANS measurement, an intact, uniformly cut, 0.3 mm thick, cylindrical, Ø25 mm slice was cut across the core axis from each core section. These slices were mounted with epoxy on quartz plates for USANS/SANS measurements. To further place the USANS/SANS samples in context, SEM and optical micrographs were taken and are presented with the scattering data in Figs. 2, 3, 4, 5 and 6.

USANS/SANS Measurements

SANS measurements were conducted on the NGB spectrometer at the National Institute of Standards and Technology (NIST) Center for Neutron Research (NCNR) in Gaithersburg, MD, USA⁶⁷. An incident neutron wavelength (λ) of 8.4 Å was used with a wavelength spread $\Delta\lambda/\lambda$ of 0.13. The scattering intensity, $I(Q)$, of a sample was measured at three different sample-to-detector distances (1.33, 5.0, and 13.17 m), with MgF_2 lenses employed at 13.17 m distance to extend the measured Q -domain down to 0.001 Å^{-1} . This provided sufficient overlap with the USANS data. $I(Q)$ is a function of the scattering wave number, $Q (\text{Å}^{-1})$, which is defined as $Q = 4\pi\lambda^{-1}\sin(\theta)$, where θ is half of the scattering angle. The Q -range of the measurements was from 0.001 to 0.4 Å^{-1} . This measurement domain corresponds to length scales from approximately 100 nm to 0.25 nm⁶⁸.

USANS measurements were performed on the same samples using the BT5 instrument at NCNR⁶⁹. This instrument uses a pair of three bounce, channel cut, perfect silicon (2 2 0) crystals, one as monochromator and the other as an analyzer. The incident neutron wavelength was 2.38 Å with a wavelength spread, $\Delta\lambda/\lambda$, of 0.059. The usable Q -range of the measurements was from 3×10^{-5} to $3 \times 10^{-3} \text{ Å}^{-1}$, which corresponds to length scales from approximately 3.3 μm to 33 nm, respectively. USANS data were corrected for slit smearing using the Lake algorithm⁷⁰ incorporated in the NCNR data reduction software⁷¹. It is important to note that this desmearing method can amplify noise, leading to artifacts such as oscillations in USANS spectra. As described in Section S1 of the SI, these artifacts did not affect our interpretation. SANS and USANS were merged after being placed on an absolute scale of differential scattering cross section per unit volume, $I(Q) (\text{cm}^{-1})$, using the direct beam protocol in the NCNR data reduction package, after subtracting the reduced background from a quartz slide coated with the epoxy adhesive used to mount the samples. The SANS for all samples showed isotropic scattering; thus, the

SANS data is presented as the azimuthal average and results from the Lake algorithm desmeared USANS data were used without rescaling⁷².

Neutron transmissions for the SANS measurement were measured to be above 0.93, indicating the absence of multiple scattering. Transmission values for the USANS measurements were between 0.64 and 0.88, within a range where some multiple scattering is present over the lowest Q -values. Multiple scattering may distort the scattering data, complicating data analysis⁷³. However, transmission values measured for these samples indicate that USANS interpretation is not significantly affected²⁶. To confirm this assessment, additional measurements were carried out for a few samples with thickness 100 μm . The reduced USANS data were identical to those of the thicker samples, indicating that multiple scattering did not significantly affect the data. The advantages of using thicker samples include: ease of sample preparation, higher scattering intensity, and a more representative sample volume.

Neutron scattering curve interpretation

Combined USANS and SANS curves for each pre- and post-shot lithological unit and their correspondent SEM images (b: pre-shot, c: post-shot) and thin section images (d: pre-shot, e: post-shot) are presented in Figs. 2, 3, 4, 5 and 6 in the Discussion Section. Model fits, described below, are plotted with the scattering data.

The scattering curves, $I(Q)$, show power-law behavior over specific Q -domains as:

$$I(Q) = \frac{A}{Q^n} + B \quad (5)$$

Here, A is the log-space intercept, $I(Q = 0)$, and B is the Q -independent incoherent background scattering intensity. Scattering data may contain more than one power law domain. If present, each domain is delimited in the USANS/SANS data by inflections or “knees” at specific Q -values. The changes in slope represent transitions in the scattering profiles from different structural levels of the sample, typically in a hierarchy. The presence of crossovers implies that these structural components have very different characteristic sizes; thus, they contribute to different domains in Q . The Q -value of the crossover corresponds to the inverse of the characteristic size of the smaller structural level⁵⁷. An assessment of the exponent, n , describing any given Q -domain in the scattering curve, therefore gives information on the rock pore and void morphology and connectivity in different lithographic formations, as well as indicates any changes that may occur due to a UNE.

Previous studies have shown that systems comprised of particles (i.e., herein voids and pores) typically arrange into hierarchical geometries of structures^{26,49,74,75}. In these cases the exponent defines a dimensionality of the features leading to the scatter: the shape or mass dimension is defined as $D_m = n$, which is used to quantify the network dimension of the scatterer; the surface dimension, D_s , is equal to $6 - n$, which is used to quantify roughness of the scattering surface. In this interpretation, power law scatter with exponent $n < 3$ is due to the overall shapes, sizes and connectivity of the voids or pores. However, in geological samples, the dimensional interpretation of power law scattering is ambiguous, as they can also be attributed to size distributions, such as log-normal distributions, of pore and void sizes, and this is often taken to be the case^{26,68,76}. The interpretation of power-law relationships in Eq. 1 with n values greater than 4 is less so. Such values are consistent with scattering from surface structures, in this case the surfaces of pores, cracks and fractures, and voids. While values greater than 4 are typically associated with surface geometry^{77,78}, those between 3 and 4 are associated with surface roughness or polydispersity^{49,79}. From these considerations, inflections and “knees” (i.e., visibly bends in the USANS/SANS spectra), which delimit the different n -values, define the length scales where scattering reflects changes in the scatter source from the surface to pore size distributions and/or mass geometries.

Data availability

The datasets generated and/or analyzed during the current study are available in the Zenodo repository, <https://doi.org/10.5281/zenodo.17613298>.

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References

1. Bowyer, T. W. et al. Detection and analysis of xenon isotopes for the comprehensive nuclear-test-ban treaty international monitoring system. *J. Environ. Radioact.* **59**(2), 139–151 (2002).
2. Carrigan, C. R., Heinle, R. A., Hudson, G. B., Nitao, J. J. & Zucca, J. J. Trace gas emissions on geological faults as indicators of underground nuclear testing. *Nature* **382** (6591), 528–531 (1996).
3. Carrigan, C. R. et al. Delayed signatures of underground nuclear explosions. *Sci. Rep.* **6**(1), 23032 (2016).
4. Schulze, J., Auer, M. & Werzi, R. Low level radioactivity measurement in support of the CTBTO. *Appl. Radiat. Isot.* **53** (1–2), 23–30 (2000).
5. Jordan, A. B., Stauffer, P. H., Knight, E. E., Rougier, E. & Anderson, D. N. Radionuclide gas transport through nuclear explosion-generated fracture networks. *Sci. Rep.* **5**(1), 18383 (2015).
6. Jordan, A. B. et al. Uncertainty in prediction of radionuclide gas migration from underground nuclear explosions. *Vadose Zone J.* **13**(10), vzj2014-06 (2014).
7. Lowrey, J. D., Biegalski, S. R. & Deinert, M. R. UTEX modeling of radionuclide isotopic fractionation resulting from subsurface transport. *J. Radioanal. Nucl. Chem.* **296**(1), 129–134 (2013).
8. Sun, Y. & Carrigan, C. R. Modeling noble gas transport and detection for the Comprehensive Nuclear-Test-Ban Treaty. *Pure Appl. Geophys.* **171**(3), 735–750 (2014).
9. Agbotui, P. Y., Firouzebehi, F. & Medici, G. Review of effective porosity in sandstone aquifers: Insights for representation of contaminant transport. *Sustainability* **17**(14), 6469 (2025).

10. Dai, Z. et al. Identification of sorption processes and parameters for radionuclide transport in fractured rock. *J. Hydrol.* **414**, 220–230 (2012).
11. De Paolo, D. J. & Orr, F. M. Geoscience research for our energy future. *Phys. Today* **61**(8), 46–51 (2008).
12. Quinn, P., Cherry, J. A. & Parker, B. L. Hydraulic testing using a versatile straddle packer system for improved transmissivity estimation in fractured-rock boreholes. *Hydrogeol. J.* **20** (8), 1529–1547 (2012).
13. Samper, J. et al. Interpretation of solute transport experiments in compacted Ca-bentonites using inverse modeling. Migration 2005 Conference. Report no. INIS-FR-5478. (2005).
14. Binley, A. et al. The emergence of hydrogeophysics for improved understanding of subsurface processes over multiple scales. *Water Resour. Res.* **51**(6), 3837–3866 (2015).
15. Ding, M. Self-sealing/healing isolation and immobilization caused by chemical discontinuities in porous media. ISBN: 90-5744-022-9, Ponsen en Looijen by Wageningen, 151p. (1998).
16. Ding, M., Geusebroek, M. & Van der Sloot, H. A. Interface precipitation affects the resistance to transport in layered jarosite/fly ash. *J. Geochem. Explor.* **62**(1–3), 319–323 (1998).
17. Verberne, B. A., Plümper, O., de Winter, M., Spiers, C. J. & D.A. and Superplastic nanofibrous slip zones control seismogenic fault friction. *Science* **346** (6215), 1342–1344 (2014).
18. Allen, A. J. Time-resolved phenomena in cements, clays and porous rocks. *J. Appl. Crystallogr.* **24**(5), 624–634 (1991).
19. Emmanuel, S., Ague, J. J. & Walderhaug, O. Interfacial energy effects and the evolution of pore size distributions during quartz precipitation in sandstone. *Geochim. Cosmochim. Acta* **74**(12), 3539–3552 (2010).
20. Emmanuel, S. & Berkowitz, B. Effects of pore-size controlled solubility on reactive transport in heterogeneous rock. *Geophys. Res. Lett.* **34**, L06404. <https://doi.org/10.1029/2006GL028962> (2007).
21. Liu, C. J., Wang, G. X., Sang, S. X. & Rudolph, V. Changes in pore structure of anthracite coal associated with CO₂ sequestration process. *Fuel* **89** (10), 2665–2672 (2010).
22. Navarre-Sitchler, A. K. et al. Porosity and surface area evolution during weathering of two igneous rocks. *Geochim. Cosmochim. Acta* **109**, 400–413 (2013).
23. Peach, C. J. & Spiers, C. J. Influence of crystal plastic deformation on dilatancy and permeability development in synthetic salt rock. *Tectonophysics* **256** (1–4), 101–128 (1996).
24. Mang, J. T. & Hjelm, R. P. July. SANS and contrast variation measurement of the different contributions to the total surface area in PBX 9501 as a function of pressing intensity. In *AIP Conference Proceedings* (Vol. 845, No. 1, pp. 1271–1274). American Institute of Physics. (2006).
25. Mang, J. T. & Hjelm, R. P. Small-angle neutron scattering and contrast variation measurement of the interfacial surface area in PBX 9501 as a function of pressing intensity. *Propellants Explos. Pyrotech.* **36**(5), 439–445 (2011).
26. Mang, J. T. & Hjelm, R. P. Fractal networks of inter-granular voids in pressed TATB. *Propellants Explos. Pyrotech.* **38**(6), 831–840 (2013).
27. Mang, J. T., Hjelm, R. P. & Francois, E. G. Measurement of porosity in a composite high explosive as a function of pressing conditions by ultra-small-angle neutron scattering with contrast variation. *Propellants Explos. Pyrotech.* **35**(1), 7–14 (2010).
28. Mang, J. T., Skidmore, C. B., Hjelm, R. P. & Howe, P. M. Application of small-angle neutron scattering to the study of porosity in energetic materials. *J. Mater. Res.* **15** (5), 1199–1208 (2000).
29. Willey, T. M. et al. Changes in pore size distribution upon thermal cycling of TATB-based explosives measured by ultra-small angle X-ray scattering. *Propellants Explos. Pyrotech.* **31**(6), 466–471 (2006).
30. Geng, Y., Liang, W., Liu, J., Cao, M. & Kang, Z. Evolution of pore and fracture structure of oil shale under high temperature and high pressure. *Energy Fuels* **31** (10), 10404–10413 (2017).
31. King, H. E. Jr et al. Pore architecture and connectivity in gas shale. *Energy Fuels* **29** (3), 1375–1390 (2015).
32. Tiwari, P., Deo, M., Lin, C. L. & Miller, J. D. Characterization of oil shale pore structure before and after pyrolysis by using X-ray micro CT. *Fuel* **107**, 547–554 (2013).
33. Chaudhuri, A., Rajaram, H., Viswanathan, H., Zyvoloski, G. & Stauffer, P. Buoyant convection resulting from dissolution and permeability growth in vertical limestone fractures. *Geophys. Res. Lett.* **36**, L03401. <https://doi.org/10.1029/2008GL036533> (2009).
34. Dewers, T. A. et al. Oedometric small-angle neutron scattering: In situ observation of nanopore structure during bentonite consolidation and swelling in dry and hydrous CO₂ environments. *Environ. Sci. Technol.* **52**(6), 3758–3768 (2018).
35. Ding, M. et al. Pore characteristics and their evolution in clays using small angle neutron scattering. In *14th International High-Level Radioactive Waste Management Conference: Integrating Storage, Transportation, and Disposal, IHLRWMC 2013* (pp. 241–246). (2013).
36. Johnson, P. J., Zyvoloski, G. A. & Stauffer, P. H. Impact of a porosity-dependent retention function on simulations of porous flow. *Transp. Porous Media* **127**(1), 211–232 (2019).
37. Swanson, E. M., Wilson, J. E. & Sussman, A. J. Lithologic controls on microfracturing from legacy underground nuclear explosions. *GSA Bull.* **135**(7–8), 1635–1651 (2023).
38. Mehmani, Y. & Balhoff, M. T. Mesoscale and hybrid models of fluid flow and solute transport. *Rev. Mineral. Geochem.* **80**(1), 433–459 (2015).
39. Røyne, A. & Jamtveit, B. Pore-scale controls on reaction-driven fracturing. *Rev. Mineral. Geochem.* **80** (1), 25–44 (2015).
40. Chipman, V., Mellors, R., Milbrath, B., Sussman, A. & Vigil, S. *Underground Nuclear Explosion Signatures Experiment (UNESE) Science Plan* (DOE/NV/25946–2264, 2015).
41. Williams, B. J. et al. Multiphenomenology explosion monitoring (MultiPEM): A general framework for data interpretation and yield estimation. *Geophys. J. Int.* **226**(1), 14–32 (2021).
42. Broome, S. T., Ingraham, M. D. & Barrow, P. C. *Permeability and direct shear test determinations of Barnwell core in support of UNESE (No. SAND-2018-9134)* (Sandia National Lab. (SNL-NM), 2018). Albuquerque, NM (United States).
43. Pohlmann, K., Hassan, A. & Chapman, J. Description of hydrogeologic heterogeneity and evaluation of radionuclide transport at an underground nuclear test. *J. Contam. Hydrol.* **44** (3–4), 353–386 (2000).
44. Anovitz, L. M. et al. Effect of quartz overgrowth precipitation on the multiscale porosity of sandstone: A (U) SANS and imaging analysis. *Geochim. Cosmochim. Acta* **158**, 199–222 (2015).
45. Anovitz, L. M. et al. Diagenetic changes in macro- to nano-scale porosity in the St. Peter Sandstone: An (ultra) small angle neutron scattering and backscattered electron imaging analysis. *Geochim. Cosmochim. Acta* **102**, 280–305 (2013).
46. Wang, H. W. et al. Multi-scale characterization of pore evolution in a combustion metamorphic complex, Hatrurim basin, Israel: Combining (ultra) small-angle neutron scattering and image analysis. *Geochim. Cosmochim. Acta* **121**, 339–362 (2013).
47. Neil, C. W. et al. Probing oil recovery in shale nanopores with small-angle and ultra-small-angle neutron scattering. *Int. J. Coal Geol.* **253**, 103950 (2022).
48. Neil, C. W. et al. Small-angle neutron scattering investigation of oil recovery in mineralogically distinct Wolfcamp shale strata. *Energy Fuels* **37** (7), 4937–4947 (2023).
49. Radlinski, A. P. Small-angle neutron scattering and the microstructure of rocks. *Rev. Mineral. Geochem.* **63**(1), 363–397 (2006).
50. Radlinski, A. P. et al. Angstrom-to-millimeter characterization of sedimentary rock microstructure. *J. Colloid Interface Sci.* **274**(2), 607–612 (2004).
51. Triolo, R. & Agamalian, M. The combined ultra-small-and small-angle neutron scattering (USANS/SANS) technique for earth sciences. In *Neutron Applications in Earth, Energy and Environmental Sciences* (eds Liang, L. et al.) 571–594 (Springer US, 2009).

52. Xu, H. Probing nanopore structure and confined fluid behavior in shale matrix: A review on small-angle neutron scattering studies. *Int. J. Coal Geol.* **217**, 103325 (2020).
53. Carle, S. F. *Interpretation of Mineralogical Diagenesis for Assessment of Radionuclide Transport at Pahute Mesa, Nevada National Security Site* (Lawrence Livermore National Lab.(LLNL), 2020). No. LLNL-TR-810225(United States).
54. Huckins-Gang, H. B., Larotonda, J. M., Eleogram, B. R., McKenna, I. J. & Burt, C. B. Reconstruction of Pre-and Post-Test Surfaces at the U-20az BARNWELL Site, Nevada National Security Site (No. DOE/NV/03624 – 1068). 1–23 (Nevada National Security Site/ Mission Support and Test Services LLC (NNSS/MSTS), North Las Vegas, NV, United States, 2021).
55. Ilavsky, J. & Jemian, P. R. Irena: Tool suite for modeling and analysis of small-angle scattering. *J. Appl. Crystallogr.* **42**(2), 347–353 (2009).
56. Sears, V. F. Neutron scattering lengths and cross sections. *Neutron News*. **3** (3), 26–37 (1992).
57. Beaucage, G. Approximations leading to a unified exponential/power-law approach to small-angle scattering. *Appl. Crystallogr.* **28** (6), 717–728 (1995).
58. Neil, C. W. et al. Small-angle neutron scattering (SANS) characterization of clay-and carbonate-rich shale at elevated pressures. *Energy Fuels*. **34** (7), 8178–8185 (2020).
59. Eaton, B. A. The effect of overburden stress on geopressure prediction from well logs. *J. Pet. Technol.* **24**(08), 929–934 (1972).
60. Millington, R. J. & Quirk, J. P. Permeability of porous solids. *Trans. Faraday Soc.* **57**, 1200–1207 (1961).
61. Neil, C. W., Swager, K. C., Bourret, S. M., Ortiz, J. P. & Stauffer, P. H. Rethinking porosity-based diffusivity estimates for sorptive gas transport at variable temperatures. *Environ. Sci. Technol.* **58** (42), 18903–18914 (2024).
62. Heath, J. E., Kuhlman, K. L., Broome, S. T., Wilson, J. E. & Malama, B. Heterogeneous multiphase flow properties of volcanic rocks and implications for noble gas transport from underground nuclear explosions. *Vadose Zone J.* **20**(3), e20123 (2021).
63. Neil, C. W. et al. Unravelling the origins of shale nanoporosity using small-angle neutron scattering (SANS). *Fuel* **389**, 134578 (2025).
64. Pal, A. K., Garia, S., Ravi, K. & Nair, A. M. Pore scale image analysis for petrophysical modelling. *Micron* **154**, 103195 (2022).
65. Wilson, J. E., Goodwin, L. B. & Lewis, C. J. Deformation bands in nonwelded ignimbrites: Petrophysical controls on fault-zone deformation and evidence of preferential fluid flow. *Geology* **31**(10), 837–840 (2003).
66. Carrigan, C. R. & Sun, Y. Detection of noble gas radionuclides from an underground nuclear explosion during a CTBT on-site inspection. *Pure Appl. Geophys.* **171**(3), 717–734 (2014).
67. Glinka, C. J. et al. The 30 m small-angle neutron scattering instruments at the National Institute of Standards and Technology. *J. Appl. Crystallogr.* **31**(3), 430–445 (1998).
68. Debye, P. A. J. H., Anderson, H. R. Jr. & Brumberger, H. Scattering by an inhomogeneous solid. II. The correlation function and its application. *J. Appl. Phys.* **28**(6), 679–683 (1957).
69. Barker, J. G. et al. Design and performance of a thermal-neutron double-crystal diffractometer for USANS at NIST. *J. Appl. Crystallogr.* **38**(6), 1004–1011 (2005).
70. Lake, J. A. An iterative method of slit-correcting small angle X-ray data. *Acta Crystallogr.* **23**(2), 191–194 (1967).
71. Kline, S. R. Reduction and analysis of SANS and USANS data using IGOR Pro. *J. Appl. Crystallogr.* **39**(6), 895–900 (2006).
72. Gu, X. & Mildner, D. F. R. Ultra-small-angle neutron scattering with azimuthal asymmetry. *J. Appl. Crystallogr.* **49**(3), 934–943 (2016).
73. Mazumder, S. & Sequeira, A. Multiple small-angle scattering—A review. *Pramana* **38** (2), 95–159 (1992).
74. Anovitz, L. M. & Cole, D. R. Characterization and analysis of porosity and pore structures. *Rev. Mineral. Geochem.* **80**(1), 61–164 (2015).
75. Teixeira, J. Small-angle scattering by fractal systems. *Appl. Crystallogr.* **21** (6), 781–785 (1988).
76. Pfeifer, P. & Avnir, D. Chemistry in noninteger dimensions between two and three. I. Fractal theory of heterogeneous surfaces. *J. Chem. Phys.* **79** (7), 3558–3565 (1983).
77. Martin, J. E. Scattering exponents for polydisperse surface and mass fractals. *J. Appl. Crystallogr.* **19**(1), 25–27 (1986).
78. Martin, J. E. & Hurd, A. J. Scattering from fractals. *J. Appl. Crystallogr.* **20**(2), 61–78 (1987).
79. Wong, P. Z., Howard, J. & Lin, J. S. Surface roughening and the fractal nature of rocks. *Phys. Rev. Lett.* **57**(5), 637 (1986).

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Author contributions

M.D. and R.P.H. conducted SANS experiments and analysis and wrote the first draft of the manuscript. M.E.H. conducted SANS experiments. A.J.S., S.T.B., and J.E.W. conducted supporting sample analysis. J.G.B. supported SANS experiments. E.M.S. contributed to writing the manuscript. C.W.N. finalized the manuscript and prepared Figs. 2, 3, 4, 5, 6, 7 and 8. All authors reviewed the manuscript.

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Declarations

Competing interests

The authors declare no competing interests.

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