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Natural gas vaporization in a nanoscale throat connected model of shale: Multi-scale, Multi-component and Multi-phase

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Production of hydrocarbons from shale is a complex process that necessitates the extraction of multi-component hydrocarbons trapped in multi-scale nanopores. While advances in nanofluidics have allowed researchers to probe thermodynamics and transport in single, discrete nanochannels, these studies present a highly simplified version of shale reservoirs with homogeneous pore structures and/or single-component fluid compositions. In this study, we develop and apply a 30,000-pore nanomodel that captures the inherent heterogeneity in reservoir pore sizes (100 nm pores gated by 5 nm-pores) to study vaporization of a representative natural gas hydrocarbon mixture. The nanomodel matches major North American formations in the volumetric and number contributions of the pore sizes, porosity (10.5%), and ultra-low permeability (44 nD). Combined experimental and analytical results show 3000x slower vaporization owing to the nanoscale throat bottlenecks. At low temperatures, mixture effects reduce rates further with stochastic vaporization of light components in large pores dominating. Collectively this approach captures the coupled complexity of multicomponent, multiphase fluids in multiscale geometries that is inherent to this resource.

Introduction

Transport and thermodynamics of fluid mixtures in nanoporous media with a complex distribution of length scales is critical to a number of fields including biology and geology. In particular, the importance of the latter is highlighted by the rapid emergence of unconventional shale gas, now a mainstay of North American energy (in 2017, natural gas provided 32% of electricity generation, and 60% of natural gas was from shale sources). To produce gas from shale rock, horizontal wells over 1 km in length are drilled along the formation, and the brittle, low-permeability shale is fractured by hydraulic pressure. This process effectively opens the reservoir rock for production, and particles, or proppants, within the fracture fluid keep the fractures open as pressure is reduced. The well productivity is initially high due to rapid depletion of reservoir pressure and production from fractures and larger pores (order of meters to microns), and then declines as production shifts to the nanoporous matrix. This relatively new process has had profound impacts in global energy and the environment.

Examples of the widely varied impacts include a transition away from coal to less carbon-intensive natural gas in electricity production, establishing US as a major global oil producer, depressing global oil and gas prices, and far-reaching environmental impacts from both the development of these resources (e.g., water use, energy use, CO2 emissions, seismic activity) and the ultimate use of the produced natural gas. While the broad implications of hydraulic fracturing technology are becoming clearer, there is a lack of fundamental understanding of this complex process. Understanding the complex fluid transport and thermodynamics at the heart of this process is essential for predicting production, environmental impacts and ultimately energy security.

The fundamental complexity of shale gas production at the heart of this process is a “dual-mixture” problem, i.e., the thermodynamics of a multi-component fluid mixture, in a media with a mixture of length scales. Pore size distributions of shale reservoirs present an interesting dichotomy wherein small pores (<10 nm) dominate in terms of their number and larger pores (~100 nm) dominate in terms of volume and store most of the accessible hydrocarbons. During recovery, bubble nucleation and vaporization is likely to be favoured in the larger pores; however, the subsequent transport of fluids in the nanoporous matrix is limited by smaller conduits where nanocoalnfinement effects such as Knudsen diffusion become significant. Further complicating the process is the multicomponent nature of the fluid in these pores, and changes in the composition of liquid and gas phases as production proceeds. Lighter, volatile components vaporize first, enriching the remaining liquid with heavier fractions. Experimental tools...
are urgently needed to resolve these highly coupled complexities.

While micromodels have become an integral tool for visualizing pore scale mechanisms associated with oil sands and conventional reservoirs,18–21 physical models with nanoscopic features representing shale gas, or nanomodels, are still in their infancy.22,23 Advances in nanofabrication have enabled the direct study of nanoconfinement effects at a single length scale in discrete nanochannels,24 differentiating fluid phases in sub-10 nm deep channels using simple single-component working fluids such as propane25 and butane.26 Introducing a model fluid mixture, we recently studied dew point and bubble point in straight 8, 80 and 800-nm channels, finding significant deviation from bulk theory in the smallest channels.3 In the first work of its kind, Vincent et al. developed a system with 625 discrete micropores connected only via a nanoporous substrate to study drying dynamics of pure water. In this extreme ink-bottle geometry they found evaporation to be governed by stochastic nucleation and poroelastic mass transport, with local evaporation in large pores influencing surrounding pores.2 While these contributions have provided some insight into the fundamentals of fluid behavior in nanoconfinement, idealized pore-geometries and pure fluid systems do not sufficiently capture the dual-mixture complexity inherent to shale reservoirs.

Here we develop a nanoscale throat connected physical model of shale that incorporates the dominant pore sizes, with 100-nm large pores connected only through a 5-nm throat network. The model is loaded with a ternary hydrocarbon mixture (methane, propane and pentane) typical of liquid-rich natural gas. Dynamics of vaporization and production of natural gas are recorded by imaging the large pores over time. An analytical model is developed for vaporization in serially-connected 5-nm throats and 100-nm pores. Both the physical and analytical model show the impact of the mixed geometry, and experiments with lower vaporization driving force (lower temperature, higher pressure) resolve the additional influence of mixture dynamics in multiscale pores.

Experimental
Fabrication and characterization of the shale nanomodel
The shale nanomodel (Fig. 1) was designed to match shale nanoporous matrix properties (in terms of dominant pore sizes, porosity, and permeability), with the large internal pores connected by a smaller nanoporous matrix. Fig 1b shows the layered composition of the nanomodel with both large and small pores in the silicon substrate (full details in S1†). The 5-nm deep throat network was created using a modified Voronoi pattern with channel widths ranging from 40 nm to 200 nm. The pattern was transferred onto a silicon substrate (previously coated with a 200-nm thick silicon nitride film) using electron beam lithography and etched to a depth of 5.5 ± 0.2 nm using reactive ion etching (RIE). Voronoi patterns have been widely

Fig. 1 Shale-nanostructure inspired nanofluidic model with 100 nm deep pores connected by a network of 5 nm pores. (a) SEM image of the nanoporous matrix in shale reservoirs with a mixture or pore sizes. White arrows indicate 5-nm pore throats connecting larger 100-nm pores. SEM image obtained from Loucks et al.25 Scale bar represents 500 nm (b) Schematic of the shale nanomodel fabrication showing key steps: (i) etching of 5-nm throat network, (ii) etching of 100-nm pores and (iii) anodic bonding to a glass slide. (c) Image of the final fabricated device completely saturated with liquid (liquid filled pores are dark and isolated pores are bright). The nanomodel is connected to the inlet at the bottom and is dead-ended at the top. Scale bar represents 1 mm. (d) Characterization of shale nanomodel with SEM (top panel) and AFM (bottom panel). Scale bar represents 5 µm in the SEM images (e) Comparison of the shale nanomodel cumulative pore volume distribution to major North American shale formations (shale data obtained from Zhao et al.14)
used to introduce geometric heterogeneity in micromodels to study flow and transport.27–29 The large pores were fabricated in a regular square array, 6-μm in diameter with center-to-center spacing of 17 μm after etching. The pattern was transferred to the substrate using UV lithography and etched to a depth of 82.2 ± 2.9 nm using RIE. These large pores were created with microscale diameters to allow for visualization under an optical microscope during the experiment. Due to the heterogeneous nature of the Voronoi pattern, not all etched large pores were connected to the 5-nm network. The center-to-center spacing of the larger pores was set to ensure representative connectivity with the underlying 5-nm network, once combined. A third cycle of lithography and etching was performed to create the 20-μm deep inlet microchannel. To complete the fabrication, the substrate was anodically bonded to a glass slide to seal off the nanomodel from the atmosphere. The fabricated nanomodel was mounted onto a high-pressure manifold and connected to the external experimental system including a transfer cylinder containing the test fluid as shown in Fig. 2 (experimental details in S1†).

Fig. 1c shows a snapshot of the fabricated shale nanomodel imaged via an optical microscope. The dark circles are liquid-saturated large pores while bright circles are isolated large pores that are not connected to the small pore network, and therefore are not participating. Approximately 5,800 large pores are connected here to the 5-nm network. The number of throats in the 5-nm network was estimated to be ~23,000. The shale nanomodel was characterized using scanning electron microscopy (SEM) and atomic force microscopy (AFM) prior to anodic bonding with results shown in Fig. 1d. The average width of the small throat network channels is ~100 nm. The AFM cross-section profile illustrates the ~15-fold difference in height between the large pore and the throat network.

The areal porosity of the small pore network and the large pores are 5% and 10% respectively giving a dual-depth porosity of ~10.3% in the nanoporous matrix (see details in S3†).30 The relative volume capacity in the nanomodel is on the order of picolitres with the volume capacity of the large pores dominating that of the small pore network by 30:1. Fig. 1e compares the cumulative pore volume distribution of the shale nanomodel to major North American shale formations (adapted from literature data of N2 adsorption measurements)14 showing a relatively good match. Here, both the N2 adsorption data and our calculations include the volume contribution of the isolated, inaccessible pores. Since fluid transport in our nanomodel is expected to be governed primarily by the small nanopore network, permeability estimated by the Kozeny equation is 44 nD for the nanoporous matrix (see details in S3†). Table 1 compares the produced nanomodel to the Bakken shale formation on the basis of traditional reservoir characterization parameters, such as the volumetric and number contributions of the pore sizes, porosity and permeability, showing a reasonable match. The slight deviation in the number contributions can be attributed to the approximation method used here for the 5-nm network in the nanomodel where pore numbers were determined by estimating the total number of pore nodes. The larger pores are confined in one nanoscale dimension (depth) with pore width designed at micrometer scale to allow observation. The micrometer scale pore width influences volume, but the nanoscale dimension dominates in terms of capillarity and transport resistance. Additionally, shale formations are complex 3-D nanoporous media with a wide range of pore sizes. Although the approach here is a necessarily simplified physical model of shale, the bimodal pore size distribution included here captures the influence of nanothroat connected larger pores that is relevant to shale production generally. In addition, the model closely captures the reservoir geometry based on scale (total number pores), porosity and permeability.

Table 1 Summary of reservoir characterization parameters for the shale nanomodel as compared to the Bakken formation

<table>
<thead>
<tr>
<th>Parameters</th>
<th>Shale nanomodel</th>
<th>Bakken formation</th>
</tr>
</thead>
<tbody>
<tr>
<td>Pore size</td>
<td>5 nm, 82 nm</td>
<td>&gt; 1 nm</td>
</tr>
<tr>
<td>Volume contribution of small pores (≤ 15 nm)</td>
<td>3%</td>
<td>9%14</td>
</tr>
<tr>
<td>Volume contribution of large pores (&gt; 15 nm)</td>
<td>97%</td>
<td>91%14</td>
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<tr>
<td>Number contribution of small pores (≤ 15 nm)</td>
<td>57%</td>
<td>69%31</td>
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<tr>
<td>Number contribution of large pores (≤ 15 nm)</td>
<td>43%</td>
<td>31%31</td>
</tr>
<tr>
<td>Matrix porosity</td>
<td>10.3%</td>
<td>7.6% to 14.1%12</td>
</tr>
<tr>
<td>Matrix permeability</td>
<td>44 nD</td>
<td>1 nD to 5000 nD12</td>
</tr>
</tbody>
</table>
Results and discussion

Filling dynamics in the shale nanomodel

The hydrocarbon mixture was chosen to reflect a typical liquid-rich natural gas stream with 10% methane, 40% propane, and 50% pentane (mole fraction). This mixture was used in all the experiments discussed here. While the local mixture component concentrations will be altered during the phase change experiments at different superheats, a similar initial condition was ensured by loading all test cases at 4 MPa. The phase diagram of the prepared mixture sample is presented in Fig. 3a. After vacuuming the nanomodel for three hours, the hydrocarbon mixture was injected into the chip above the bulk bubble point pressure at room temperature as indicated by the purple line in Fig. 3a. Fig. 3b shows the nanomodel during and after the filling process. Additionally, an image following processing shows the liquid saturated pores in blue and isolated pores in grey at the end of the filling process (details of image processing in S2†).

Once injected, liquid quickly filled the supply microchannel and slowly started flowing into the nanoporous media from the inlet. At a filling pressure of 4 MPa, the total filling time was approximately 3 hours. During this process, time-lapse images were recorded every 2 minutes. The spatio-temporal map illustrating the filling dynamics at 4 MPa is shown in Fig. 4a. The filling proceeds in a fairly uniform fashion. Fig. 4b shows pore-scale observations of the filling process in a 3 x 2 set of pores midway in the filling process. Initially the pores are empty. As filling progresses, grey liquid films first coat the large nanopore boundaries before completely filling the large nanopore. These filling dynamics can be attributed to corner-flow effects characteristic of micropores.20 Fig. 4c shows the percentage of filled pores as a function of time with 4 MPa filling pressure. Additionally, Fig. 4c also plots the Hagen-Poiseuille predicted filling rate using a simplified physical model of large nanopores series-connected by singular 5-nm throats (calculation details in S5†). The time required to fill a large nanopore through a 5-nm throat is determined by relating the volume of a large nanopore to the volumetric velocity in the upstream set of liquid-filled 5-nm throats. The calculation results in a series of step-functions with a general square-root-of-time dependence that is typical of filling problems. The horizontal steps represent the time necessary to fill a large nanopore with liquid and the vertical jumps represent the relatively fast filling of 5-nm throats. In contrast to the analytically predicted case, the experimentally observed filling process was approximately four times slower and highly linear with respect to time. Anomalous, slow capillary filling has previously been reported in single-depth nanochannels due to the presence of trapped air at the nanochannel dead-end.19 In our experiments, we attribute this delayed, and more constant liquid filling rate to vaporization of lighter components, particularly at earlier times, at the liquid front that changes vapor pressure close to the nanomodel dead-end. This effect is not captured by the analytical model. To remove any potential concentration gradients resulting from the filling process, the chip was left to equilibrate for ~12 hours prior to running the vaporization experiments (the characteristic time for diffusion of the heaviest component to the volumetric velocity in the upstream set of liquid-filled 5-nm throats is determined by relating the volume of a large nanopore to the volumetric velocity in the upstream set of liquid-filled 5-nm throats).
Vaporization dynamics in the shale nanomodel

Once the entire chip was saturated with liquid and equilibrated, drawdown pressure was initiated under isothermal conditions. The system temperature was controlled by electric heating elements, at 82.5°C, 62.5 °C and 42.5 °C for cases of high, medium and low superheat. After the temperature stabilized, the pressure was lowered stepwise as shown in Fig. 3a. Time-lapse images of the process were recorded every 10 seconds. At each solid circle in Fig. 3a we set a waiting time of 15 minutes to observe potential phase change. In a control experiment, we waited for 60 minutes and observed similar results. As the pressure was lowered below the bulk bubble point, vapor bubbles started to appear in the microchannel with no detectable change in the nanomodel. This result is similar to that observed in recent experiments in single 1-D nanochannels and associated density functional theory modelling where a two-phase envelope was not observed for hydrocarbon binary mixtures at steady-state, and pressures below the bulk dew point were required for vaporization in sub-10nm channels.17

Fig. 5 shows the spatio-temporal dynamics of vaporization for the three conditions (from high superheat to low superheat) showing markedly slower and less spatially uniform vaporization fronts at reduced superheats. Superheat is defined here as the difference between the bulk dew point pressure and the vaporization pressure and is 0.76 MPa for high superheat, 0.44 MPa for medium superheat and 0.25 MPa for low superheat. For individual experimental runs, the temperature was kept constant and pressure was lowered as per Fig. 3a. Across experimental runs, the final vaporization pressure (vacuum in all cases) was the same allowing for comparison. The high superheat case (Fig 5a) exhibited a vaporization front that initiated from the inlet of the nanomodel and progressed quite uniformly into the nanomodel. In the medium superheat case (Fig 5b) vaporization events were commonly observed ahead of the vaporization front. Vaporization of porosity in the nanomodel has previously been reported in nanoporous media as a result of pre-pressurization conditions,16 and slow drying rates14 in single-component systems. Here, this effect is attributed to lighter components preferentially vaporizing ahead of the front. In contrast to the high and medium superheat cases, vaporization in the low superheat case (Fig. 5c) was slower and scattered throughout the media. After 480 minutes, approximately 760 pores (~14% of large pores), remained saturated with liquid. These were mainly grouped near the entrance of the nanomodel and are colored grey in Fig. 5c.

Fig. 5d, e and f plot the percentage of vaporized pores as a function of time for all three superheat conditions (from high to low superheat). All cases exhibit a square root of time dependence. For comparison, analytical results are plotted from a vapor transport resistance model that includes the serially connected geometry (inset it Fig. 5d). In the model, we assume a pure substance, and vaporization limited only by vapor transport resistance in the 5-nm pore throats wherein the transport is comprised of both Knudsen flow and viscous flow components acting in parallel (experimental details in S4†). The nanomodel surface is highly wetting to the hydrocarbon mixture, with a pentane contact angle approaching 0°.

The cumulative vaporization dynamics are stepwise with an overall square root of time dependence. The horizontal steps indicate the emptying time for the volume in the 100-nm pores while the jumps indicate the relatively fast transport of vapor in the 5-nm throat network. A longer time is required to empty
each successive large pore as the transport resistance through the 5-nm throat network progressively increases. The step-wise growth is indicative of the fact that production of vapor from large pores is mainly limited by the transport resistance in the small throats. As an example, for the high superheat case, the model predicts a vaporization time of ~48 minutes. However, without the gating 5-nm throat network the model predicts vaporization in a simple channel with depth matching the large pores (82-nm deep, 1.5 mm in length) to be less than 1 second at otherwise similar conditions. This 3000-fold difference is a result of the mixed geometry alone, that is, large pores gating by small throats. Likewise, the vaporization time in a simple channel of nanoscale throat depth (5.5-nm deep, 1.5 mm in length) is only 50 s (details in Fig. S41). This calculated time is of similar magnitude to that measured experimentally for evaporation in ~9 nm deep 2D nanopores. These differences highlight the strong influence of mixed geometries, and suggest that work to date in single-depth nanochannels or networks would tend to overestimate transport as compared to real shale reservoirs where multiple length scales are the norm.

The simplified vaporization model adequately describes the experimental results for the high superheat cases at 0.76 MPa and 0.44 MPa. In these cases, the relatively high vaporization driving force leads both light and heavy components to vaporize readily from the nanopore inlet as evidenced in the spatio-temporal plots in Fig. 5a and b. In these cases the dual-mixture problem is reduced to a single mixture problem, that of mixed geometry. The dynamics are thus mainly governed by vapor transport and rightly captured in the model. However, in the low superheat case (0.25 MPa), experimental results indicate additional delays in vaporization not captured by the pure-substance analytical model. Specifically, the model over-predicts the vaporization dynamics especially at later times (30% deviation between model and experimental data at ~200 min). At low superheat, we expect lighter components to preferentially cavitate throughout the nanomodel enriching liquid within the nanoscale throat network with heavier pentane that is slower to vaporize (for reference, in bulk systems and at ambient pressures, the vaporization points of methane, propane and pentane are -161.5°C, -42°C and 36.1°C,

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Fig. 5 Spatio-temporal progression of vaporization events in the nanomodel. Each panel is 2 mm in width and 1.5 mm in length and contains ~5800 connected pores. All isolated pores have been subtracted from the image. Grey pores represent pores that remained saturated with liquid and did not vaporize in the time period (a-c) Map showing dynamics in the high-superheat run ~0.76 MPa, medium superheat run ~0.44 MPa, and low-superheat run ~0.25 MPa. (d-f) Comparison of vaporization progression determined through experiment and vapor transport governed vaporization model as a function of time corresponding to high superheat, medium superheat and low superheat. Each data experiment was repeated twice (see S7† duplicate experiment). Inset image in (d) shows a simplified schematic of geometry used to calculate the vaporization dynamics (top-view). The time taken for vapor flow through the 5-nm throat network, \( t_{\text{vap}} \), is calculated by determining the volumetric flow rate through 5-nm throat network using a resistance model containing both Knudsen flow resistance and viscous flow resistance contributions. The time taken to transport vapor volume held in a 100-nm pore, \( t_{\text{vap}}^\text{pore} \), is calculated by using the 5-nm throat network volumetric flow rate and the volume of a 100-nm pore.
respectively). These compositional changes are not captured in the model where a constant composition is assumed throughout, resulting in the over-prediction of the vaporization rate.

Previous phase behavior studies with a hydrocarbon mixture confined to single-depth nanochannels showed that stochastic bubble nucleation does not significantly affect the overall mixture vaporization rate. The combination of the fluid mixture and mixed geometry here shows a much-slowed vaporization due to bubble nucleation in big pores enriching fluid composition in the pore throat, highlighting the influence of pore geometry and connectivity on the phase change dynamics of multicomponent fluids.

By resolving individual pore dynamics in the network, the nanomodel provides the opportunity to additionally assess the impact that local cavitation phase change has on surrounding pores. Vincent et al. observed such an influence by studying stochastic cavitation of pure water in a ink-bottle type system with an ordered array of very large pores (on the order of microns) above a bulk nanoporous media. Their experimental system also showed that vapor bursting behavior was periodic with 1-7 events in quick succession followed by a ~15-minute period of no activity. Inspired by this finding, we analyzed the cavitation behavior obtained in the shale-relevant nanomodel to determine if phase change in pores influenced near-by pores with results shown in Fig. 6a. In our system, vaporization is relatively continuous and does not show the dramatically periodic behavior observed by Vincent et al. We do note that incremental number of emptying pores as a function of time and the corresponding time series autocorrelation function followed an oscillatory pattern as shown in Fig. 6b, however, we conclude that in this system phase change in a given pore was largely independent of phase change in neighboring pores. Differences with the system of Vincent et al. include higher superheat levels and less extreme difference in the pore geometry in the shale-inspired cases studied here. Collectively, the results here illustrate that complex dual-mixture effects, resulting from the confinement of multi-component fluids in multi-scale nanopores, are central to the study of phase change as relevant to shale gas production.

Conclusions

We developed and fabricated a nanomodel that replicates the dominant length scales typically found in the shale nanoporous matrix. The nanomodel consists of large pores (~100 nm in depth) gated by a 5-nm throat network. The resulting nanofabricated network exhibited a pore size distribution, porosity and permeability relevant to the complex interconnected pore space characteristic of shale resources. The system was applied to quantify vaporization of a ternary hydrocarbon mixture relevant to shale gas condensates. Vaporization dynamics depended on the applied superheat, with results compared to a vapor transport model including both Knudsen flow and viscous effects. High superheats resulted in relatively uniform vaporization initiating from the model entrance. The effect of the mixture of length scales was significant, decreasing the vaporization rate by a factor of 3000 as compared to an otherwise similar system of constant cross-section. In addition, lower superheats, and slower vaporization, resulted in less coherent vaporization with isolated vaporization of pores ahead of the front. In those cases the additional effect of the hydrocarbon mixture was significant, leading to a further reduction in the vaporization rate. A spatio-temporal analysis of pore vaporization at low superheats showed no significant correlation indicating that the vaporization of large pores shale are largely independent, with each responding to local conditions.

This work shows that the small pores (10 nm and less), while insignificant with regards to their volume capacity, can greatly influence the production from larger pores by severely gating transport. Neglecting these small pores on account of their relatively small volume contribution can thus lead to erroneous modelling of gas flow during production. These geometric effects are compounded by mixture effects, as earlier vaporization concentrates heavy components in the remaining liquid.

While shale oil/gas production typically necessitates relatively low well-flow pressures at latter stages of production (typically lower than the bubble point), the very low pressures needed here to vaporize the entire nanomodel are generally not feasible in the field. Additionally, while the pressure drawdown...
in the device is relatively low compared to shale production, these results suggest that producing from the nanoporous matrix maybe difficult using current primary production strategies especially as hydrocarbons in the smallest pores become enriched with heavier fractions over time. These findings support the hypothesis that the rapid decline in production rates observed in shale gas production is the result of a shift from production in large connected features, to production from the nanoporous matrix. Enhanced recovery strategies may be necessary to extract hydrocarbons from the nanoporous matrix – another potential application of the nanostructured physical model presented here.

**Conflicts of interest**

There are no conflicts to declare.

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