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Melt-front propagation and velocity profiles in packed beds of phase-change materials measured by magnetic resonance imaging

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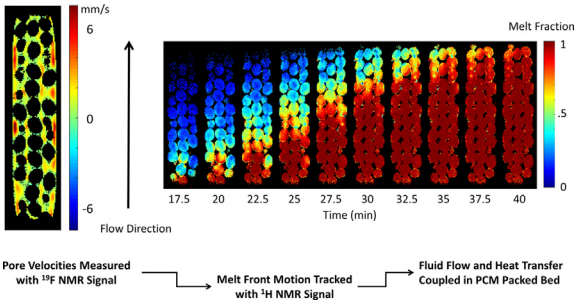
HIGHLIGHTS

- High resolution coupling of local velocity and PCM melt front via NMR images.
- Phase change material (PCM) packed bed melted by heated internal fluid flow.
- Robust form of temperature tracking by NMR T_2 signal change during phase transition.
- Pore space velocity maps captured with ^{19}F frequency NMR.
- Spatially and temporally resolved melt front monitoring of PCM with ^{1}H frequency NMR.

GRAPHICAL ABSTRACT



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ABSTRACT

Fluid flow and heat transfer characteristics in packed beds are of importance in many fields of study. However, experimental characterization and model validation of the numerical methods used to study packed beds is difficult. Utilizing nuclear magnetic resonance (NMR), we present a novel approach to track the melt front (and thus melt temperature) in a non-isothermal packed bed. The approach tracks the melt front through packed beds of wax encapsulated phase change material (PCM) by taking advantage of the change in T_2 associated with phase change. Additionally, velocity maps of the flow in the pore space of the same packed bed sample are obtained. The approach uses the ^{1}H signal from the PCM packing and the ^{19}F signal from the flowing fluid to allow monitoring of both particle bed structure/phase and flow velocities in the same sample with the use of a dual tune coil. Together, this information can be used to probe the heat transfer characteristics of packed beds experimentally.

1. Introduction

Packed beds are widely used in operations such as thermal energy storage from solar-power facilities [1,2], industrial waste heat recovery [3], and fixed bed catalytic reactors [4]. While current design approaches for these systems are well guided by principles at the input-output macroscale, further improvements to design next-generation versions of these units will have to rely on better understanding of transport processes at the pore microscale. A key challenge to the development and use of effective microscale models is the lack of detailed experimental measurements of temperature and velocity fields at the same scale. Such measurements are critical for assessing the performance of models at the microscale. Current experimental approaches are limited to temperature and flow measurements at the input and output of the

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units [4,5] or via thermocouple measurement of temperature at fixed locations within the bed [6–8]. The relatively low spatial density of these measurements does not provide enough information at the spatial resolution needed for pore scale model validation and cannot correlate the results to the local velocity. As a consequence, the use of only macroscale measurements for testing predictions of the microscale has led to wide scatter in correlations for key design parameters such as interphase heat transfer coefficients [9] as well as larger-than-desired uncertainties in the sizes of final unit designs.

Nuclear magnetic resonance and magnetic resonance imaging (MRI) methods have proven to be powerful techniques for measuring features of transport in porous media [10–21]. For example, NMR and MRI have been used to measure dispersion in packed-bed flows, porosity and density maps, pore surface to volume ratios and pore size distributions at length scales from 10 μm to 100 mm. Importantly, the non-invasive nature of NMR allows for these measurements to be made *in situ*, and real-time observations are possible. Hence, NMR and MRI methods are uniquely suited for observation and measurement of transport processes at the length scales needed to assess and validate proposed microscale models that can improve designs of packed-bed devices.

Many NMR parameters are sensitive to temperature [22]. These include active nuclei density, T_1 and T_2 relaxation rates, diffusion coefficients, magnetization transfer rates, and changes in chemical shifts [22–32]. While these parameters provide ample means for temperature mapping, many of them interact and can depend on sample heterogeneity. The approach here provides a robust alternative method for the study of energy transport processes. In the current study, bed particles containing ^{1}H and a pore fluid containing ^{19}F enables monitoring of both the solid phase and the pore space separately in the same sample, akin to the approach of Boyce et al. applied to fluidized beds [33]. Porous media structure was composed of wax encapsulated in a plastic shell, PCM particles. Pore space was filled with fluorinated oil heated to a specific temperature by a heat bath, and pumped through an insulated flow loop. By tuning to the ^{1}H frequency (300 MHz), one-dimensional (1D) and two-dimensional (2D) imaging of the melt front propagating through the bed was possible. In addition to this, velocities in the pore space were recorded from the ^{19}F frequency (282.4 MHz), allowing for visual comparison of melt dynamics to velocity distribution *in situ*, with no signal degradation due to signal in the pore space and particle bed interacting.

The work reported here entails three main points. First, we present and discuss aspects of the experiment, particularly the materials used for the study, and how the changes in T_2 for the material can be interpreted. Second, we summarize imaging and flow measurements of the transport dynamics of a packed bed whose solid phase contains a phase-change material. Finally, we assess the results with a view toward what can currently be measured and the connections of this approach to detailed quantification of heat transfer processes in PCM units.

2. Experimental setup and methods

The key elements of the experiment are a continuous-flow loop for circulating a fluorinated heat transfer fluid through a packed bed inside the magnet system. The packed bed was constructed from a high-performance liquid chromatography column (Kinesis USA, OmniFit Benchmark HIT 10-150-AF) with a 10 mm inner diameter. The outlet fitting could be adjusted to allow bed lengths of 50–150 mm. A flow system was built to allow for continuous flow and heating of the pore-phase fluid through the bed, shown in Fig. 1. Flow rates were varied from 200 mL/h to 400 mL/h with inlet temperatures of approximately 39 °C. The solid phase of a

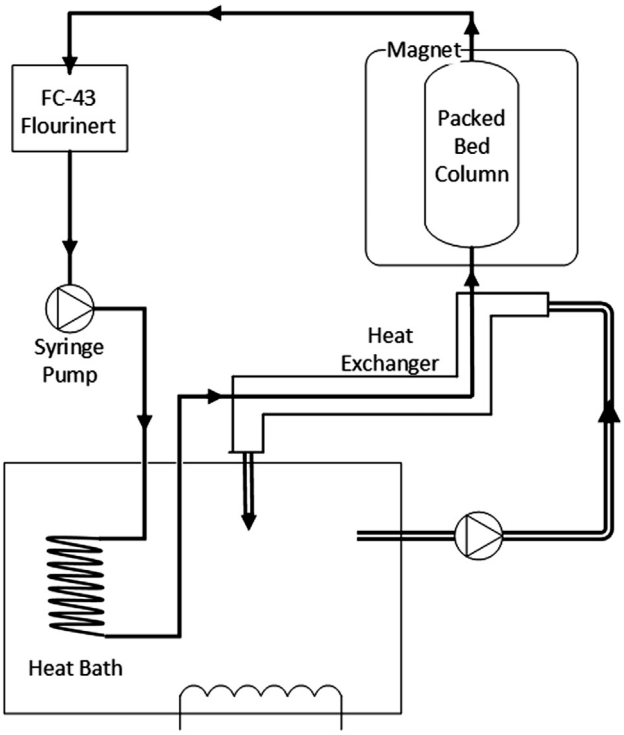

Fig. 1. Schematic of the heated Fluorinert flow loop. Fluid was heated after the pump with a heat bath, and the temperature was maintained to the base of the probe with a counter-flow heat exchanger. Relationship between sample inlet and bath set temperatures was determined in a separate experiment.

packed bed was composed of 5–15 μm encapsulated eicosane wax capsules agglomerated into 3.7–5 mm diameter spheres (Microtek Encapsulation Technologies). Two waxes of different melting points are shown to have NMR relaxation properties useful for application here, octadecane filled macroPCM28 (T_m = 28 °C) and the eicosane filled macroPCM37 (T_m = 37 °C). The packed bed of macroPCM37 is studied in detail. The fluid phase was Fluorinert FC-43 (3M Corporation), a heat transfer fluid, chosen for its heat transfer characteristics and high fluorine density. The fluid system used a dual syringe pump (Pharmacia P-500) to pump the fluid through a temperature-controlled bath. On exit from the bath, the fluid line temperature was maintained by using a counter-current double-pipe heat exchanger that spanned the distance from the bath to the base of the magnet. Calibration experiments were performed to obtain the actual probe-inlet temperature from the settings on the temperature-controlled bath.

NMR and MRI measurements were performed on a vertical wide-bore Bruker Avance III 300 Spectrometer at 7 Tesla (300 MHz nominal proton frequency, 282.4 MHz nominal fluorine frequency) controlled by ParaVision 5.1 software on a Linux operating system. The receive/transmit coil was a ^{1}H/^{19}F dual tuned birdcage with an internal working diameter of 25 mm and an active imaging length of about 40 mm. Shielded gradient coils (Micro 2.5, Bruker) for imaging and flow measurements had maximum gradient strengths of 1.5 T/m at 60 A.

Images were formed using both the ^{1}H and ^{19}F resonances. The ^{1}H images of the PCM material were made using a multi-echo spin-warp imaging sequence. Imaging methods included one-dimensional frequency-encoded profiles in the direction of flow through the bed, as well as two-dimensional (phase- and frequency-encoded) images of either the bed cross-section (transverse images) or the bed profile (longitudinal images). 1D profiles could be acquired rapidly (<15 s needed for image acquisition)