



Transport and removal of a solvent in porous media in the presence of bitumen, a highly viscous solute



Siddhant Panda, Krupal Pal, Sumeet Merzara, Murray R. Gray, Qi Liu, Phillip Choi*

Department of Chemical and Materials Engineering, University of Alberta, Edmonton, AB T6G 1H9, Canada

HIGHLIGHTS

- Drying of a solvent along with a highly viscous solute in porous media was studied.
- Duration of solvent removal stage increased with increasing solute concentration.
- Vapor pressure, capillary transport of solution and pore blockage were investigated.
- High solute concentration reduced capillary transport and thereby solvent recovery.

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ABSTRACT

Removal of solvent from the gangue generated by an oil sands solvent extraction process is essentially a process of solution transport in a porous medium with a highly viscous solute (left over bitumen of a completed extraction process or residual bitumen) at low concentrations. The solvent usually has much higher vapor pressure and lower viscosity than the residual bitumen. The concentrations of the residual bitumen are typically in the range of 0.6–2.4 wt% while those of the solvent (cyclohexane) is 8–12 wt% at the start of the drying process to remove the solvent. Drying of gangue was carried out for 2 h under ambient conditions using petri dishes with bed heights of 0.6, 1.0 and 1.4 cm. A typical drying curve (i.e., cumulative volatiles mass loss versus time) shows two distinct drying stages in which the first stage is the fast evaporation of cyclohexane (99% by weight removal) followed by a stage in which slow evaporation of water is observed. The residual bitumen was observed, unexpectedly, to migrate to the top surface of the porous media with cyclohexane via capillary transport and accumulate there in the first stage of drying. The rate of removal of cyclohexane decreased with increasing residual bitumen content. Consequently, duration of the fast cyclohexane removal stage from the gangue sample containing 2.4 wt% bitumen was 1.5 times longer than that of the 0.6 wt% bitumen gangue sample, when the initial cyclohexane concentration was 12 wt%; it was 1.2 times longer when the initial cyclohexane concentration was 8 wt%. Our studies reveal that poor capillary transport of solution in porous media at higher bitumen concentrations to be the most likely cause of slower drying rather than vapor pressure lowering of bulk solution and pores blockage. The cause is similar to previous reports in literature with the solute being salt in salt–water solution.

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1. Introduction

During the drying of a rigid porous medium, the evaporating fluid is displaced by the non-wetting gas/air (Yiotis et al., 2003; Yortsos and Stubos, 2001), so that the gas–liquid interface recedes deeper into the medium. Yiotis et al. (2004) reported that this displacement of gas–liquid interface should result in liquid film formation along the surface of the capillaries in the porous media.

The presence of liquid films results in higher evaporation rates (Prat, 2007; Sghaier and Prat, 2009; Shokri et al., 2009) since they provide hydraulic conductivity/capillary connectivity for the receding liquid thereby facilitating mass transport to the surface for evaporation (Yiotis et al., 2004). In porous media where film flow dominates, non-isothermal temperature effects are trivial (Yiotis et al., 2004). When a solution of a non-volatile solute in a solvent is dried in a porous medium, the transport and deposition of the solute alters the drying process. Past studies on drying of a solution in porous media focused on salts as the solute (Brito and Diaz Gonçalves, 2013; Camassel et al., 2005; Eloukabi et al., 2013; Sghaier and Prat, 2009; Sghaier et al., 2006, 2007; Shokri,

* Corresponding author.

E-mail address: phillip.choi@ualberta.ca (P. Choi).

2014; Zhang et al., 2007). Drying of miscible solutions of high-boiling organics or polymers has not been examined.

Alberta has large deposits of mixtures of unconsolidated sand and clay particles and a highly viscous petroleum that is called bitumen. The mixtures are referred to as oil sands or oil sands ore (Masliyah et al., 2004). Solvent based oil sands extraction, considered to be a more environmentally favorable alternative to water based process (Meadus et al., 1982; Nikakhtari et al., 2013, 2014; Pal et al., 2015) for extracting bitumen, provides an example of drying of a solvent from a miscible solution in a porous medium. In this case, solvent extraction generates a porous waste called gangue, consisting of a sand/clay porous medium with a relatively low concentration of bitumen which could not be extracted and it is referred to as residual bitumen dissolved in a considerable amount of solvent. The bitumen is a complex organic mixture that has a much higher boiling point and viscosity than the solvent, but is miscible. Water is also present in the gangue, but it is immiscible with the solvent and/or bitumen (Nikakhtari et al., 2015) and is immobile. Drying studies of oil sands gangue provide an opportunity to understand how the transport and concentration of residual bitumen (a highly viscous solute) in the porous medium affects the removal of the solvent. Initial work by Renaud (2014) demonstrated that residual bitumen migrates within the gangue during drying, analogous to salt migration in soils.

In the case of salt migration in a capillary porous medium during evaporation of water, thick films of water play a vital role (Camassel et al., 2005; Sghaier and Prat, 2009; Sghaier et al., 2006). Salt deposition primarily occurs in the fine pores at the porous bed surface when supplied by a capillary liquid flow (Shokri, 2014). The resulting crusty efflorescence of salt at the soil surface reduces the mean evaporation rate, likely due to a reduction in water activity and due to physical blockage of pores by salt crystals (Eloukabi et al., 2013). Brito and Diaz Gonçalves (2013) in their study using porous stones containing sodium salt solutions concluded that a reduction in bed sorptivity resulting from lower liquid flow at higher salt concentrations was the cause for slower drying. Here, sorptivity signifies the ability of material to absorb fluids and transmit them by capillarity (Bruto and Diaz Gonçalves, 2013; Hall and Hoff, 2011) and is dependent both on the porous material and the fluid (e.g., viscosity). Additionally, Brito and Diaz Gonçalves (2013) pointed out that pore blockage due to formation of crusts can also hinder vapor transport. Interestingly, the hypothesis of a reduction in water activity at the porous media surface due to higher solute concentration in the solution has not been confirmed.

The aim of this study was to investigate the effect of varying compositions of residual bitumen and initial cyclohexane content in the gangue on the removal of cyclohexane under ambient conditions. Through this study, we also intended to develop a deeper understanding of solution drying inside porous media, especially in the case where the solute is highly viscous such as bitumen and can dramatically affect the solution viscosity even at very low concentrations. The understanding gained can help with the modelling of the drying process better and also serve as a guideline for the effective design of a drying technique that can be used by the oil sands industry.

2. Materials and methods

2.1. Materials

A rich oil sands ore sample and an Athabasca bitumen sample were used in this work and they were provided by Syncrude Canada Ltd. Toluene (Certified ACS grade, Fischer Scientific, USA) was used for the Dean Stark extraction of the ore sample.

Dean-Stark extraction analysis with toluene as a solvent has been used previously for the determination of bitumen, mineral solids and water content in the oil sands ore (Nikakhtari et al., 2013). Dean stark extraction is a procedure of distillation extraction involving the vaporisation of water in the ore sample by the boiling extraction solvent followed by water condensation and collection in a calibrated trap (Schlumberger Oilfield Glossary). Solvent is condensed as well and allowed to flow back over the sample placed in a thimble holder to extract the oil. Extractions are continued until the extraction solvent doesn't show a change in colour i.e. is clean. The thimble containing extracted ore is dried to obtain the mineral solids, water volume can be noted from the calibrated trap and bitumen content can be determined by either mass balance (ore initial and final weights are known) or drying of the extracted solution obtained to remove the extraction solvent.

A mixture of coarse and fine solids were obtained by drying the solids collected. These solids henceforth will be referred to as soxhlet gangue. Demineralized water and cyclohexane (Certified ACS grade, Fischer Scientific, USA) were used for the reconstituted gangue preparation.

The bitumen and water contents of the oil sands ore were determined by the Dean Stark analysis to be 13.5 ± 1.1 wt% and 3.0 ± 0.9 wt%, respectively. The fines content (particles with size less than $45 \mu\text{m}$) as determined by a particle size analyzer (Mastersizer 2000, Malvern Instruments) on the Soxhlet gangue was 11.2 ± 0.7 wt%. The remainder were the coarse solids.

2.2. Reconstituted gangue drying

Given that the composition of the oil sands ore is highly variable, it is difficult to have a control on the residual bitumen and cyclohexane contents of the gangue obtained from the extraction of an oil sands ore using cyclohexane (Renaud, 2014). In general, the cyclohexane content in extracted gangue varies between 8 wt % and 18 wt% with a mean and standard deviation of 11.8 ± 2.1 wt% (Renaud, 2014). Bitumen recoveries of greater than 90% have been achieved using cyclohexane as an extraction solvent (Nikakhtari et al., 2013, 2014; Pal et al., 2015) following a lab scale protocol though a reduction can be expected when scaling up the process to industrial scale. In this paper residual bitumen is in the range of 0.6–2.4 wt% corresponding to recoveries of 84–96%. Water content of the gangue is in the range 2.1 ± 1.1 wt% (Renaud, 2014). To study the effect of residual bitumen on the removal of solvent from an extracted gangue, reconstituted gangue samples with controlled composition were needed and prepared based on the protocol described in Appendix A.

The drying runs were carried out inside a fume-hood. A constant sash height was maintained for uniform flow conditions. The designed setup is shown in Fig. 1. Images during the drying experiment were constantly recorded by a camera facing the front edge of the balance (not shown in the figure). To prevent reflections on the PYREX® CLS316060 glass petri-dish (60 mm OD, 50 mm ID, 15 mm height), a blackened light reflection cardboard was used as shown in the setup. The airflow over the sample was limited further by it. Two lamps were used at a distance of about 30 cm from the sample to obtain bright images. The temperature and humidity inside the balance chamber during a run was measured using a temperature/humidity probe (Fisher Scientific). The probe was placed about 12–14 cm above the sample location.

Prior to the start of a drying experiment, the gangue containing jar (actual or reconstituted) was taken out from a freezer and thawed under room temperature for 12 ± 2 min. Post thawing, $15.2/25.2/35.2 \pm 0.4$ g of sample was packed into the glass petri-dish to bed heights of 0.60/1.00/1.40 ± 0.05 cm, respectively. The packing was done so as to maintain a nearly constant bulk density

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