Chemical Engineering Science 102 (2013) 309–314

Contents lists available at ScienceDirect

Chemical Engineering Science

journal homepage: www.elsevier.com/locate/ces

Microcapsule flow behaviour in porous media

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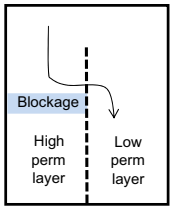
HIGHLIGHTS

- We demonstrate the triggering of temperature sensitive micro-capsules in a porous media.
- The flow-paths in the porous media can be affected by introducing micro-capsules.
- Un-triggered capsules can affect the flow but triggered capsules affect the flow to a larger degree.
- Increased flow through low permeability regions is demonstrated.

GRAPHICAL ABSTRACT



Flow in porous media can be altered by introducing blockages

ARTICLE INFO

Article history:
Received 22 May 2013
Received in revised form
28 July 2013
Accepted 11 August 2013
Available online 19 August 2013

Keywords:
Encapsulation
Enhanced oil recovery
Triggered release
Porous media

ABSTRACT

This article investigates the possibility of using micro-capsules, with a gelling agent, hydroxypropyl cellulose, in the core, during enhanced oil recovery. The aim is to block off high permeability regions of a formation known as thief zones, thereby diverting the chase injection water into adjacent unswept low permeability regions. Temperature triggered micro-capsules were made by polymer precipitation through a solvent evaporation method, with poly(lactic-co-glycolic) acid as the polymer shell. Release studies with methylene blue demonstrated the temperature induced release from these micro-capsules. A customised tank was made to allow porous media flow and single permeability experiments were conducted. Even without the gelling agent, the micro-capsules gradually blocked the ballotini pore network. However, by varying the tank temperature, a drop in permeability was observed when the capsules released their core. Experiments with two permeability regions showed that the micro-capsules clogged up the high permeability layer more than the low permeability region.

1. Introduction

Micro-capsules are micron sized particles, encapsulating an active ingredient with an organic or inorganic shell. Encapsulation is used to protect the core from harsh thermal, chemical or mechanical degradation environments and have therefore been researched extensively in the food, medical and pharmaceutical industries (Bouchemal et al., 2004; Bouillot et al., 1999; Madene et al., 2006; Persico et al., 2005; Yamakawa et al., 1992; Yow and Routh, 2006). Encapsulation is widely used in the food industry to mask odours and tastes by controlling the release from food matrices. (Gibbs, S.K. 1999; Madene et al., 2006). A further example of a harsh chemical environment is the stomach. The presence of gastric juices means that it is difficult to ensure survival of probiotic bacteria until they reach the gut. A viable solution to this problem is to encapsulate the bacteria through various techniques such as colloidosomes, extrusion or spray drying (Chávarri et al., 2010; Islam et al., 2010; Keen et al., 2012a, 2012b).

The use of micro-capsules also allows control over the core release. This is not surprising as the shell provides the release barrier. Depending on the active ingredient and shell, one can manipulate the release characteristic by changing the shell polymer, porosity, size and thickness (Dowding et al., 2005, 2004; Romero-Cano and Vincent, 2002; Yow et al., 2009). An example where

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http://dx.doi.org/10.1016/j.ces.2013.08.028

controlled release is vital, is in the pharmaceutical industry where pulsatile drug release is often desired for hormone deficiencies or chronic pain treatment (Richards Grayson et al., 2003).

Although the use of encapsulation in the pharmaceutical and food industries is common, the idea of using encapsulation in the oil industry seems to be relatively novel. No journal articles could be found that document the use of micro-capsules in oil reservoirs, during enhanced recovery. This is likely due to the lack of an appropriate time response, cost, the required particle size of low porosity reservoirs, and harsh reservoir conditions with high salinity and temperature. Only patents have been found studying this area (Bertkau et al., 2012; Montanaro, 2012). The concept is to flow micro-capsules containing gelling polymer into an oil reservoir, and to trigger release in high permeability regions, which are already swept of mobile oil. The released polymer then forms a gelled network, causing a marked drop in permeability or better still, completely blocking off that particular region (Fig. 1a). Consequently, during the next water flood, more of the reservoir is swept (Fig. 1b). Flow in porous media had been extensively studied both theoretically and experimentally using imaging techniques such as MRI, with fingering instabilities often observed (Zimmerman and Homsy, 1991).

By knowing the release profile and mechanism, one can trigger the release of gelling agents at specific locations in the oil reservoir. This is advantageous when compared to simply flowing large amounts of viscous polymer solutions, such as polyacrylamide, into oil reservoirs and then crosslinking to attain a block, which could be expensive. In addition, encapsulation is likely to save material costs, since less gelling agents are needed when it is targeted to specific regions. Hence, an understanding of how micro-capsules behave in porous media is sought in this paper.

2. Materials and methods

2.1. Microcapsule formation and release profile

For this work, micro-capsules that trigger in response to temperature are used. The shell polymer is made of poly(lactic-co-glycolic) acid with a 50:50 M ratio between monomers. This polymer is biodegradable and temperature responsive because of ester bond hydrolysis at high temperatures (Dunne et al., 2000; Grayson et al., 2005) It is also important to note that the degradation rate can be controlled by changing the lactic/glycolic acid ratio (Makadia and Siegel, 2011).

The micro-capsules were made using a polymer precipitation method, induced by evaporating a good solvent for the shell polymer. The method is described fully in a previous publication and so is only briefly discussed here (Gun and Routh, 2013). First, 1.5 g of poly(lactic-co-glycolic) acid (M_w 60,000) (PLGA) from Sigma-Aldrich, was completely dissolved in 70.54 g of dichloromethane (Acros Organic, stabilised with ca. 0.2% Ethanol) and 0.8 g SPAN 80 (Fluka), emulsion stabiliser. The solution is then added to 1.5 g of an aqueous solution of 10 wt% Hydroxypropyl cellulose (M_w 370,000, Sigma-Aldrich), and sheared for 3 min, using a high shear mixer (Silverson Model SL2), to form a water-in-oil emulsion. This mixture is then poured into 200 mL of an aqueous solution containing 2 wt% polyvinylalcohol (M_w 85,000–124,000, Sigma-Aldrich), to form a water-in-oil-in-water double emulsion. The dichloromethane is then evaporated slowly at 20 °C for 24 h to precipitate the PLGA, forming the shell, encapsulating the hydroxypropyl cellulose in the core.

An alternative core material is methylene blue, and this is encapsulated to allow measurement of the capsule release profile. In this case, 15 mL of the microcapsule solution was placed into a dialysis bag, which was then placed in 500 mL of continuously stirred deionised water (Purelab Ultra from ELGA process water). Concentration measurements were made by placing 4 mL of the release medium into a UV-grade cuvette and analysing the adsorption spectrum. After obtaining the UV spectrum, the sample was returned back into the release medium to maintain a constant volume of water. The methylene blue dye has a distinctive UV peak at 668 nm.

2.2. Porous media

2.2.1. Single permeability experiment

A Hele Shaw cell, with dimensions 330 mm × 40 mm × 2 mm, was used to conduct porous media flow experiments. Fig. 2 gives a sketch of the cell used and photos can be seen in Fig. 7.

An aluminium frame was used because of its high thermal conductivity. This facilitated heating of the tank using a circulating water bath. To assemble the experiment, the tank was initially flooded with deionised water. Ballotini with diameter 0.5 mm, were then added gradually whilst gently stirring, to break up any ballotini clusters. The outflow from the tank was recorded every second, using a mass balance.

For a typical experiment, the temperature in the tank was set by circulating water at 63 °C into the tank shell. The initial permeability of the tank was measured by simply flowing deionised water for a few pore volumes. This was then followed with 1 pore volume of dispersion containing micro-capsules at 10 vol% and then a further three pore volumes of deionised water. This step was repeated several times to determine whether a gradual decrease in permeability was seen from the accumulation of multiple microcapsule flushes. The experiment was also repeated at room temperature, to investigate whether untriggered capsules were capable of blocking the flow.

2.2.2. Dual permeability experiment

Having performed single permeability experiments, dual permeability experiments with both low and high permeability regions were performed in the same tank, at temperatures of 63 °C. To fill

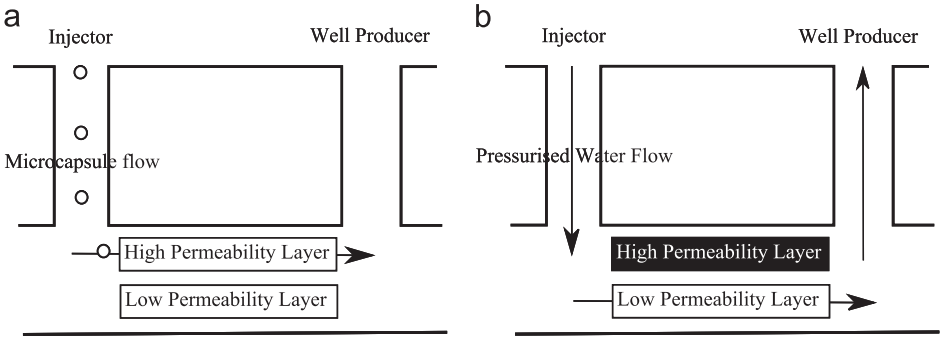

Fig. 1. Schematic of oil reservoir (a) Microcapsule flow in high permeability layer (b) Flow path after triggered release of gelling agents.