Separation and Purification Technology xxx (2017) xxx–xxx

Contents lists available at ScienceDirect

Separation and Purification Technology

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

The use of fluid dynamic gauging in investigating the thickness and cohesive strength of cake fouling layers formed during cross-flow microfiltration

Tuve Mattsson [a,b,*], William J.T. Lewis [c], Y.M. John Chew [c], Michael R. Bird [c]

[a] Department of Chemistry and Chemical Engineering, Chalmers University of Technology, SE-412 96 Gothenburg, Sweden
[b] Wallenberg Wood Science Center, The Royal Institute of Technology, Chalmers University of Technology, SE-100 44 Stockholm, Sweden
[c] Department of Chemical Engineering, University of Bath, Claverton Down, Bath BA2 7AY, UK

ARTICLE INFO

Article history:
Received 29 September 2016
Received in revised form 7 December 2016
Accepted 21 January 2017
Available online xxxx

Keywords:
Microfiltration
Fouling
Cross-flow filtration
In-situ measurement
Cake thickness

ABSTRACT

A common challenge during membrane filtration is cake fouling, whereby the build-up of material on the membrane surface reduces the permeate flux. Such fouling layers can also alter the selectivity of the separation. In this study, fluid dynamic gauging (FDG) is used *in situ* to investigate the cake fouling formed during cross-flow filtration of a model material: softwood Kraft lignin. FDG was used to estimate (i) the thickness of the cake layers (in the μm scale) and (ii) the local cohesive strength at different depths in the cake layer. Fouling layers formed at different transmembrane pressure (TMP) values were investigated. The estimated thickness of the cake layers increased with increasing TMP. However, it was difficult to capture the full cake thickness for the more loosely formed cakes layers. An increase in the cohesive strength of the cake was found to occur with increasing TMP values.

1. Introduction

Membrane operations are often energy-efficient and cost-effective ways of achieving separation and fractionation. They can be used to treat feeds with components over a very wide range of sizes: from micro-filtration for particle separation, down to reverse osmosis for desalination. Membrane operations are widely used today in a range of sectors that includes water purification/effluent treatment, pharmaceuticals, food and beverages. Membrane separation and fractionation is also expected to be an important operation within the biorefinery concept. A wood-based biorefinery has been proposed as the counterpart of a fossil-based petro refinery [12], and can be expected to be central in the production of more sustainable alternatives to the fossil-based materials and chemicals currently being used.

It is well known that fouling is a factor that can often impede the performance of membrane separation [1]. In the case of cake fouling, the build-up of material on the membrane surface reduces its performance by increasing the effective flow resistance; such a surface layer may also affect the selectivity of the separation. While it is difficult to investigate (often very thin) cake layers, a fundamental understanding of the properties of fouling layers is crucial for developing a mechanistic model that can provide guidelines for the design and improvement of the unit operation.

Several methods for the non-intrusive investigation of surface fouling thickness can be found in the literature. These methods include laser triangulometry (e.g. [13]), ultrasonic time-domain reflectometry (e.g. [9,14]) and nuclear magnetic resonance (NMR) imaging (e.g. [2]). A relatively new technique is Fluid Dynamic Gauging (FDG), which uses a physical probe to investigate the thickness of thin cake fouling layers *in situ* during membrane operation [4]. FDG can also be used in a destructive mode to scan through the cake layers that are formed to investigate cohesive strength properties throughout the cake [6]. These local measurements can be used to estimate the hydrodynamic forces necessary to remove the cake layer during crossflow operations and washing cycles. Several foulants have been investigated by FDG techniques using different experimental set-ups with varying precision during cross flow filtration. These foulants include glass beads [8], yeast [6], molasses [5,4], Kraft lignin [10] and mixtures of glass beads and lignin [7]. While it has been shown earlier that FDG can be used to identify a build-up of a cake fouling layer during cross flow filtration, more in-depth investigations into the challenges in the estimation of the fouling layer thickness for challenging organic foulants are needed.

* Corresponding author at: Department of Chemistry and Chemical Engineering, Chalmers University of Technology, SE-412 96 Gothenburg, Sweden.
E-mail address: tuve.mattsson@chalmers.se (T. Mattsson).

http://dx.doi.org/10.1016/j.seppur.2017.01.040
1383-5866/

Nomenclature

Roman

d_t	inner diameter of the nozzle throat [m]
D	particle diameter [m]
h	gauge height above a deposit [m]
m_g	mass flow through the gauge [kg s^{-1}]

Greek

δ_c	thickness of the cake fouling layer [m]
ϕ	solidosity [–]
μ	viscosity of the fluid [Pa s]
ρ	density of the fluid [kg m^{-3}]
$\tau_{w,max}$	maximal fluid shear stress [N m^{-2}]

Acronyms

FDG	Fluid Dynamic Gauging
TMP	Transmembrane pressure
NMR	Nuclear Magnetic Resonance

In this study, an newly developed FDG unit designed for high precision measurements is used to investigate, *in situ*, the thickness and cohesive strength of soft cake fouling layers formed during cross-flow microfiltration at different transmembrane pressures (TMPs). The estimated thickness using the FDG technique is compared to cake heights calculated based on gravimetric analyses, and the FDG technique is evaluated over the investigated TMP range. A softwood Kraft lignin suspension is used as a model foulant, as this material presents a range of interesting and important phenomena relating to soft organic deposits, including stickiness and a mild degree of compressibility.

2. Experimental

2.1. Materials

A softwood (mainly spruce, with some pine) Kraft lignin produced using the Lignoboost™ technology [11] was used as a model material. This lignin not only forms soft organic cake fouling layers that cause considerable flow resistance but also displays an interesting range of phenomena that include stickiness, agglomeration and a mild degree of layer compressibility. The Lignoboost™ lignin is a by-product from the Kraft paper pulp process and is modified chemically to be alkali-soluble. Dissolution of the particles was avoided by maintaining the pH of the suspension and rinsing water below pH 4 by the addition of sulphuric acid, unless otherwise stated. Likewise, the water used to prepare all slurries and rinse liquid was purified by reverse osmosis to limit the presence of additional ions. The solid density of the lignin particles was determined at 1350 kg m^{-3} using a gas pycnometer (AccuPycII 1340, *Micromeritics*). The size distribution of the lignin particles/ agglomerates was investigated for heavily diluted samples, at a pH below 4, in conjunction with each filtration experiment by laser diffraction (Mastersizer X, *Malvern*).

The membrane used for the microfiltration was a regenerated cellulose membrane (hydrophilic, with no wetting agents) of 0.2 μm nominal pore size (RC58, *Whatman*). The membrane was soaked for at least 30 min before use in order to wet the membrane pores.

2.2. Filtration equipment

The filtrations were performed in a pressurised filtration cell with a 250 mm flow channel of 15 mm square cross-section. A horizontal, flat, sheet membrane 150 × 15 mm in size was fitted to the bottom of the cell, set 1 mm lower than the rest of the flow cell and placed equidistant from the inlet and outlet. The suspension was circulated from a stirred and baffled feed tank through the test section by a regenerative pump (HPR6/11, *Totton*). The TMP was measured using a differential pressure transducer, (PX26-005DV, *Omega Engineering*) and regulated by a needle valve downstream from the filtration cell, while the cross-flow velocity was measured using a variable area flowmeter (1100-series Rotameter, *KDG*). The permeate was collected on an electronic balance (FX-3000i, *A&D*) connected to a data-logging PC. A schematic representation of the set-up can be found in Fig. 1 and a representation of the filtration cell can be found in Fig. 2.

The FDG probe, with a diameter (d_t) of 0.5 mm at the nozzle opening, was positioned at the centre of the membrane; all thickness and cohesive strength measurements were performed at this location. The small opening diameter enhances the precision of the measurements while allowing for local measurements to be made on a small area of the cake layer, this small footprint along with the central location of the probe allows for the capturing of thickness and strength data at a position where possible rand effects close to the edges of the membrane are minimised. The vertical position of the probe was measured by a linear variable differential transformer (SM-series LVDT, *RS Components*) with an accuracy of 0.5 μm. The vertical movement of the probe was monitored using a stepper motor attached to a linear guide rail (KR1501AM, *THK*). A constant mass flow through the gauge probe was generated using a syringe pump (Touchscreen 100 series, *Cole-Parmer*), whilst the pressure drop across the probe was measured using a differential pressure transducer (PX26-001DV, *Omega Engineering*). The distance between the probe and a surface (e.g. membrane or fouling layer) can then be related to the pressure drop across the probe. Pressure drop data for different probe distances from the membrane surface, experimentally determined using the filtration equipment, can be found in Fig. 3. A more detailed description of the apparatus and the FDG probe and technique can be found elsewhere [7], wherein a description of the calibration procedure is also provided.

2.3. Filtration experiments

Each fouling experiment was performed using 15 L of a 0.02 vol% pH-adjusted Kraft lignin suspension at ambient temperature (16–19 °C). This feed suspension was prepared from stock slurries with a concentration of 2 vol% that had, in turn, been prepared from lignin powder and pH-adjusted reverse osmosis water mixed together using a homogeniser (*IKA*, T25 Ultra Turrax) at 9000 RPM for 2 h. Prior to its first use, the stock slurry had been kept agitated using a magnetic stirrer for no less than 24 h; it was kept this way between uses. The stock slurry was diluted to a total volume of 1 L with pH-adjusted water before being added to the 14 L pH-adjusted reverse osmosis water in the stirred tank: this was done to facilitate fast mixing.

A maximum of 0.6 L was withdrawn as permeate during the fouling phase (run in feed and bleed mode, with retentate being recirculated back to the feed tank) of the experiments. In addition, up to 0.25 L was withdrawn before the lignin was added. This combined volume of permeate corresponded to at most 5.6 vol% of the

Please cite this article in press as: T. Mattsson et al., The use of fluid dynamic gauging in investigating the thickness and cohesive strength of cake fouling layers formed during cross-flow microfiltration, Separ. Purif. Technol. (2017), http://dx.doi.org/10.1016/j.seppur.2017.01.040