



Hydraulic impedance spectroscopy tracks colloidal matter accumulation during ultrafiltration



M.C. Martí-Calatayud^a, M. Wessling^{a,b,*}

^a RWTH Aachen University, Chemical Process Engineering, Forckenbeckstr. 51, 52074 Aachen, Germany

^b DWI Interactive Materials Research, Forckenbeckstr. 50, 52074 Aachen, Germany

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ABSTRACT

Membrane fouling is one of the most pressing obstacles during the operation of filtration membranes. Its different physical stages of concentration polarization, fouling layer deposition and reorganisation are difficult to access experimentally. Normally, the phenomenon is observed during membrane filtration when the flux turns time variant or the flux increase does not follow the applied pressure difference linearly. Flux or pressure stepping methods are state of the art to detect the onset of fouling, however they give only limited information on the properties of the deposition layer in the fluid phase adjacent to the membrane surface.

Here we present a new hydraulic spectroscopy method allowing to fingerprint the properties of the deposition layer. At different flux levels, we vary the flux around an average value in a sinusoidal fashion. From the measured pressure drop values and the time delay as compared to the imposed flux, we can calculate the so-called hydraulic impedance. Varying the frequency of the flux at a set amplitude, we can explore the frequency dependent hydraulic impedance and the different single contributions to its overall magnitude. We have applied this method to a variety of different solutions and filtration configurations showcasing its sensitivity. The method can be easily established in any laboratory and will pave the way to more systematic fouling studies.

1. Introduction

Frequency response analysis (FRA) is a dynamic method to investigate technical systems in which different physical phenomena occur superimposed on each other at different time-scales [1]. These FRA techniques apply an input signal with a sinusoidal pattern to the system under study and analyze the response of an output variable of the system in the frequency domain. In materials science, techniques such as infrared spectroscopy are capable of isolating the vibrational modes of different molecules, thus being useful to characterize the chemical composition of numerous substances. Quartz crystal microbalance is applied to monitor thin film deposition processes [2]. In electrochemical engineering and electro-membrane transport studies, electrochemical impedance spectroscopy (EIS) is used to determine properties of electrodes, as well as to differentiate charging and faradaic processes [3–5]. EIS has gained more relevance in the last years as it is useful to determine the electrical conductivity of ion-exchange membranes. Thus, it has been used to systematically compare polymer electrolytes which find their application in diverse systems, like fuel cells, batteries or deionization devices [6–8].

Pressure-driven membrane processes have important analogies with

electro-membrane processes. In both cases, the difference in a driving force promotes the transfer of a component present in an aqueous solution through a permselective membrane and generates concentrated and diluted streams. Moreover, concentration polarization limits mass transfer through the membranes in both pressure-driven and electric field-driven separations [9,10]. Such analogies have recently broadened the application of EIS to investigate porous membranes [11–14]. Bannwarth et al. developed a four-point impedance measurement device to characterize hollow fiber membranes [15]. Scale or biofouling formation in reverse osmosis membranes has been monitored also by means of EIS [16–18]. Usually, the application of EIS implies the need for a supporting electrolyte in order to obtain reliable measurements of the electric impedance.

Curiously, the investigation of transmembrane pressure (*TMP*) responses in the frequency domain is unexplored in the field of membrane science. Yet, analysis of the so-called hydraulic impedance has been object of study in other disciplines, such as hydrology, microfluidics or even cardiology [19–21]. These techniques can be especially helpful for the development and understanding of microfluidic multiplexors and micropumps [22,23].

In the present work, we introduce the concept and experimental

* Corresponding author at: RWTH Aachen University, Chemical Process Engineering, Forckenbeckstr. 51, 52074 Aachen, Germany.
E-mail address: manuscripts.cvt@avt.rwth-aachen.de (M. Wessling).

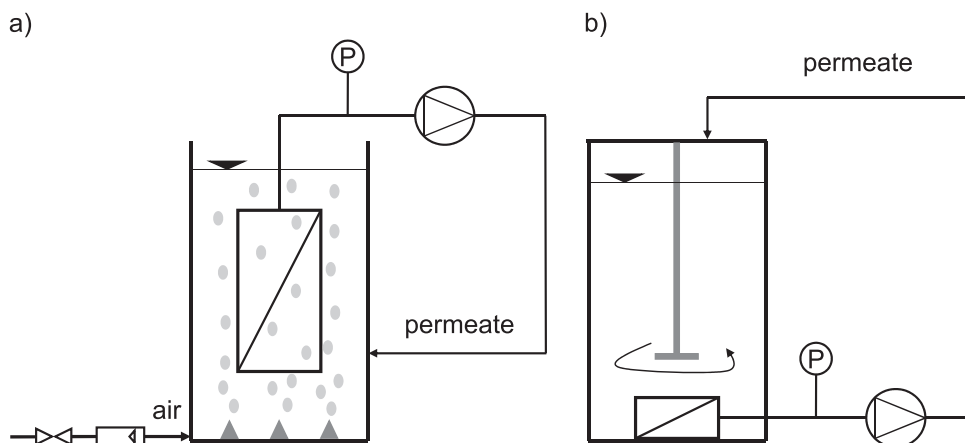


Fig. 1. Schematic representation of the experimental setups used to obtain the fluidic impedance of the different investigated membrane systems: (a) immersed membrane reactor, (b) dead-end stirred cell.

details of hydraulic impedance spectroscopy as a novel approach to investigate multiple phenomena occurring in pressure-driven membrane processes. In contrast with EIS, this method does not require from additional supporting electrolytes, as it does not rely on the conductivity of the tested solutions. Hydraulic impedance spectroscopy measures variations occurring in a membrane system directly caused by the actual driving force of the process. This enables the characterization of different types of membrane filtration systems without the necessity of changing the ionic environment of the tested solutions, which has been demonstrated to significantly alter fouling phenomena in ultrafiltration [24]. Evaluation of the alternating pressure transients allowed us to identify the accumulation of fluid energy in membrane systems filtering different colloidal solutions, which is manifested in the form of a retarded increase of transmembrane pressures upon sinusoidal flux variation. We interpret this phenomenon to be inherent to colloid retention and is concomitant to the creation of reversible concentration polarization layers and potential fouling deposits when the pressure response is not reversible.

2. Theory

In analogy with electrical systems where current can have a phase shift with regards to potential, we hypothesize that the processes taking place in membrane filtration can also induce a phase shift between the permeate flux and the pressure drop across the membrane. We also hypothesize that the electric-fluidic analogy can be extrapolated to different basic elements. For example, a tube transporting a low-viscous fluid can be considered as equivalent to conducting wires in electrical circuits. Electrons are transported along the wire as the fluid flows through the pipe both without experiencing any significant resistances. However, a membrane acts as an energy-dissipating barrier which exerts resistance to the fluid flow. This hydraulic resistance associated with the membrane results in an increase of TMP when a fluid is permeating through. The accumulation of rejected matter in front of the membrane surface or inside the pores contributes to increase the resistance of the system. If the TMP buildup takes place simultaneous to the flow of fluid, no phase shift is created between both variables and membrane filtration units can be seen as analogues to electric resistors. Interestingly, we will demonstrate below that the time-scales of colloid accumulation and flow resistance increase occur on different time-scales and can be therefore clear separated from each other. This represents an experimental finesse not available at classical flux-pressure stepping methods [25]. We describe the theoretical and experimental details below.

In electro-membrane transport systems, apart from pure resistances, a dielectric charge difference is formed between the membrane fixed charges and the solution ions having opposite charge sign; i.e. electrical

charge double layers. Energy is momentarily accumulated in these thin layers, thus resembling the properties of electric capacitors. Double layers contribute or even govern ion selectivity at electro dialysis and nanofiltration processes. Double layer effects in alternating electrical fields induce a phase shift where current leads voltage, which is usually visible at frequencies in the order of kHz. It is difficult to conceptualize an elementary hydraulic analogue to double layers in pressure-driven membrane processes. Nevertheless, the fluidic capacitance in hydraulic systems, which is more commonly known as compliance, can be manifested when fluid energy is stored by virtue of pressure in a specific component of the system [26]. Compressible fluids and flexible encapsulating materials are the most typical sources of compliance in fluidic systems [27,28].

In analogy with EIS, where the electrochemical impedance is defined as the opposition that a system poses to the passage of current when a certain voltage is applied; here we define the hydraulic (or fluidic) impedance (Z_H) as the opposition that a membrane system poses to permeation when a transmembrane pressure (TMP , measured in bar) is applied:

$$Z_H = \frac{TMP}{j_p} \quad (1)$$

Where j_p ($L \cdot m^{-2} \cdot h^{-1}$, or LMH) represents the permeate flux. Let us consider a system composed of a membrane unit, such as one of those shown in Fig. 1, which filtrates an aqueous solution. Water is transported through the membrane as a consequence of the negative pressure exerted by the pump at the permeate side. The flux can be controlled by setting the revolutions per minute of the pump head. Now, a sinusoidal function is applied to the pump, which will result in the following expression for the permeate flux j_p :

$$j_p(\omega t) = j_{p,0} \cdot \sin(\omega t) \quad (2)$$

Where $j_{p,0}$ is the amplitude of the permeate flux and ω is the frequency of the sine function. The output variable, namely TMP , has an analogous form to that of the input variable. However, it may be displaced in time and exhibit differing amplitude:

$$TMP(\omega t) = TMP_0 \cdot \sin(\omega t + \varphi) \quad (3)$$

In the previous equation TMP_0 and φ represent the amplitude of the output signal and the phase shift between TMP and j_p signals, respectively. If the frequency-dependent expressions for both variables are then substituted in Eq. (1) and we apply the Euler's formula, the hydraulic impedance can be expressed only as a function of frequency:

$$Z_H(\omega) = \frac{TMP_0 \cdot e^{j(\omega t + \varphi)}}{j_{p,0} \cdot e^{j\omega t}} = \frac{TMP_0}{j_{p,0}} \cdot e^{j\varphi} = \frac{TMP_0}{j_{p,0}} \cdot (\cos(\varphi) + j \cdot \sin(\varphi)) \quad (4)$$

In consequence, the complex impedance can be given as the sum of

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