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Centrifugal ultrafiltration for determination of filter cake properties of colloids

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ABSTRACT

The article is devoted to the application of analytical centrifugation for the measurement and analysis of centrifugal ultrafiltration kinetics. The model of centrifugal ultrafiltration of colloids accounting for the cake formation, compression and possible decompression is proposed. The method for the determination of filter cake properties (pressure dependency of specific cake resistance) from the results of batch centrifugal ultrafiltration experiment is presented.

The method was tested for the determination of filterability of model samples: aqueous solutions of protein (bovine serum albumin) and nanoparticle suspension (Laponite RD). The pressure dependency of specific cake resistance was determined in the range of 10 − 500 kPa from the results of centrifugal ultrafiltration experiments at different centrifugal rotation speeds. The obtained values of specific resistance were in good correspondence with those measured in a series of reference constant pressure dead-end filtration tests.

1. Introduction

At the early stage of membrane filtration-related research (optimization of filtration parameters, sample (i.e., solution, suspension) pretreatment or membrane choice) there can be a necessity of rapid characterization of filtration kinetics at different experimental conditions (e.g., transmembrane pressure), for different sample properties (pH, concentration, solvent composition) and membrane type (material, molecular weight cut-off). Besides the simple comparison of filtrate flux values obtained at different conditions, the research can be aimed at the analysis of membrane-solute interaction (membrane fouling) and characterization of behavior and properties (formation, permeability and compressibility) of the polarized layer (or filter cake).

Currently, such a multiparametric research demands a series of separate filtration tests, which is explained by technical limitations of dead-end filtration method. Several approaches were proposed in order to simplify/shorten filtration experiments or replace them by the measurement of a certain pertinent and accessible parameter (simple sample fluid/membrane characterization):

(i) ones are based on empirical correlations obtained in case studies: e.g., Dahdouh et al. [1,2] demonstrated a correlation between different characteristics (total soluble solids content, pH, particle diameter) and filtrate flux measured in a cross-flow microfiltration of various orange juice samples and suggested possible empirical prediction of filtration kinetics from the determined key properties of a tested fluid; Manalo et al. [3] suggested the measurement of the fluorescence intensity of biofilms formed on the surface of membrane samples after their contact with studied liquid in order to predict the fouling intensity during the reverse osmosis experiments;
(ii) others propose the characterization of filtration kinetics for a studied fluid from the results of a single filtration experiment: e.g., Gesan-Guiziou et al. [4] and Espinasse et al. [5] proposed different methodologies for the characterization of cross-flow filtration kinetics (critical flux, fouling reversibility) by a successive variation of operational parameters (transmembrane pressure, permeation flux and wall shear stress);
(iii) Iritani and co-authors systematically developed original methods for the complete characterization of filterability (pressure depen-

Abbreviation: CFUF, centrifugal ultrafiltration; DEF, dead-end filtration

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Nomenclature

a	parameter of Eq. (14)
a_c	centrifugal acceleration (m s^{-2})
b	parameter of Eq. (14).
c_0	solid concentration in the initial sample (kg m^{-3})
h	sample column height in centrifugal filtration cell (m)
h_0	initial sample column height in centrifugal filtration cell (m)
J	filtrate flux (m s^{-1})
n	parameter of Eq. (11) (dimensionless)
P	pressure (Pa)
P_c	liquid pressure drop across filter cake (Pa)
P_m	liquid pressure drop across membrane (Pa)
P_T	transmembrane pressure (Pa)
p_s	local solid pressure (Pa)
R	distance to the membrane from the axis of rotation (m)
r_c	filter cake resistance (m^{-1})
r_f	fouled membrane resistance (m^{-1})
r_m	membrane resistance (m^{-1})
r_T	total hydraulic resistance (m^{-1})
S_m	cross-sectional area of filtration membrane (m^2)
s	sedimentation coefficient (s)
t	time (s)
V	filtrate volume (m^3)
w_c	solid in filter cake per unit of membrane cross sectional area (kg m^{-2})

Greek letters

α	local specific cake resistance (m kg^{-1})
$\alpha_{c,av}$	average specific cake resistance of filter cake (m kg^{-1})
α_o	parameter of Eq. (11) (m kg^{-1} Pa^{-n})
μ	liquid viscosity (Pa s)
ρ_l	fluid sample (solution, suspension) specific weight (kg m^{-3})
ρ_s	specific weight of solid (kg m^{-3})
Ω	centrifugal rotation speed (rad s^{-1})
ω	material coordinate (m)
ω_o	total solid height in filter cake (m)

dency of specific cake resistance and cake porosity in a wide pressure range) from the results of a single dead-end filtration experiment [6–14];

(iv) application of various high throughput filtration systems for the simultaneous filtration of different samples at various operational conditions [15–20].

In this regard, analytical centrifugation attracts attention due to the possibility of automated analysis of different samples behavior under centrifugal force [21]. Initially developed for the characterization of dispersion stability and particle size analysis [22–24], analytical centrifugation was later applied for the indirect evaluation of filterability of different samples (suspensions, emulsions, protein solutions) from kinetics of centrifugal settling and consolidation (papers of Iritani and co-authors [25–28] should be added to those briefly reviewed in [29,30]). Recently, it was demonstrated that analytical centrifugation equipment can be used for direct characterization of filterability of concentrated suspensions (pressure dependency of cake dryness and specific cake resistance) via centrifugal filtration-consolidation experiments [29]. This gives incentive to further development of centrifugal filtration methods and theory for data analysis.

Different theories of batch centrifugal filtration, accounting for material properties and various filtration and centrifugation phenomena (particle sedimentation, pressure distribution in a filter cake, pressure dependency of particle volume fraction and filtration cake resistance, filter cake compression and dewatering), were developed since 1950 [31–49] and can be potentially applied for the analysis of the results of centrifugal filtration experiment and characterization of filterability. In addition, different theories of gravity filtration can be adapted for this purpose [50–57]. However, the cited methods and theories were developed for the cake filtration of particulate suspensions and do not concern with the membrane fouling during the (centrifugal) micro- and ultrafiltration of solutions and colloidal suspensions.

At the same time, centrifugal ultrafiltration is successfully applied in pharmaceutical, food engineering, water treatment, nano-, bio- and chemical technology laboratories for concentration, purification, clarification and washing of various colloids (proteins, DNA, nanoparticles, extracts) [58–65]. The centrifugal filtration is usually performed with simple equipment that complicates the centrifugal filtration kinetics measurement and limits the data analysis (it is necessary to stop the centrifugation in order to measure the filtrate volume). To the best of the author's knowledge, very limited number of papers devoted to centrifugal ultrafiltration analysis is available in the literature. In this regard, systematic reports of Hideo Nakakura and co-authors on centrifugal ultrafiltration must be referenced herein (e.g. [66]).

Recently, applications of analytical centrifugation equipment for direct characterization of ultrafiltration kinetics of different samples (protein, pectin and tannin solutions) were reported [67,68]. The feasibility of semi-quantitative characterization of fluid samples filterability from results of centrifugal ultrafiltration experiments was demonstrated; however, the possibility of more rigorous centrifugal ultrafiltration data analysis was not discussed.

The present study reports the application of centrifugal ultrafiltration for characterization of filterability of colloidal samples (polymer solutions, nanoparticle suspensions). The theoretical part of this study was focused on the development of centrifugal ultrafiltration model accounting for the peculiarities of pressure variation during the batch centrifugal filtration with cake formation compression and decompression. The experimental part was devoted to the analysis of centrifugal ultrafiltration curves for the quantitative characterization of membrane fouling and filter cake properties: fouled membrane resistance, pressure dependency of specific cake resistance and reversibility/irreversibility of cake compression.

2. Model for the centrifugal ultrafiltration with cake formation

2.1. General assumptions

The proposed model is based on following general assumptions.

1) The model is developed for one-dimensional batch centrifugal filtration (Fig. 1).

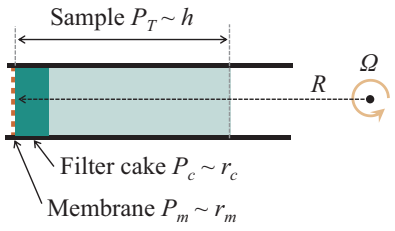

Fig. 1. Explanation for the centrifugal ultrafiltration model (P_T – transmembrane pressure, h – fluid sample height, P_c and P_m – pressure drop across the cake and the membrane, respectively, r_c and r_m – hydraulic resistance of the cake and the membrane, respectively).