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# Compression and expansion properties of filter cake accompanied with step change in applied pressure in membrane filtration

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#### ABSTRACT

In order to well elucidate the true nature of filter cake formed in membrane filtration of aqueous colloids, the transient flux decline behaviors accompanied with step-up and step-down changes in the applied filtration pressure during the course of constant pressure filtration were investigated by carrying out both microfiltration experiments of kaolin slurry and ultrafiltration experiments of nanosilica sol under various experimental conditions. When the applied pressure was increased stepwise, for each colloid the plot of the reciprocal filtration rate vs. the filtrate volume per unit membrane area referred to as the Ruth plot tends to approach a linear relationship obtained in constant pressure filtration conducted under the increased pressure condition from the beginning of filtration, after a transient period when the preformed cake under the initial pressure was compressed. In contrast, in the case of the step-down change in the applied pressure, when the volume of the preformed cake is adequate, the plot after a transient period tends to approach a higher straight line parallel to a linear relationship for constant pressure filtration conducted under the decreased pressure condition from the beginning. Such flux decay behaviors were well described by the expression having two constants: one is the constant representing the final filterability for the preformed filter cake, and the other is the rate constant describing the change rate in the structure of preformed cake resulting from a pressure jump. Moreover, the influence of experimental conditions on the cake reversibility and the rate constant was revealed both for kaolin slurry and for nanosilica sol.

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#### 1. Introduction

Membrane filtration of aqueous colloids has a broad variety of applications in widely diversified fields ranging from industry to environmental protection. One of the critical issues obstructing more widely spread use of membrane filtration may be a significant flux decline occurring with the progress of filtration, arising from the membrane fouling. Especially, when a highly compressible filter cake forms on the membrane surface during filtration, the increase in the applied filtration pressure does not necessarily lead to a noticeable increase in the overall performance of membrane filtration [1]. Therefore, an understanding of properties of filter cake such as the cake compressibility is of paramount significance to controlling filtration behaviors. Until now, the properties of compressible filter cakes have been evaluated by a number of researchers through pressure filtration tests [2-9], compression-permeability (C-P) cell tests [10-14], and centrifugation tests [15–19], etc.

The expansion properties of filter cake are of significance in the practical applications such as the rebound of water content of cake at the time of pressure release seen in the cake discharge [20] and variable pressure filtration in which the applied pressure varies rapidly with time, in addition to the basic understanding of cake properties. However, the expansion properties of filter cake have been less examined compared to the work on the compression behaviors [21,22], with the exception of the studies on the expansion behaviors of compressed cakes produced by expression operation [23,24]. Okamura and Shirato [25] investigated the temporal variation of the filtration rate in stepwise pressure filtration of slurry for ignition plug and Gairome clay when the applied filtration pressure stepwise increased or decreased during the course of cake filtration operation conducted under a constant pressure condition. As a result, they found that the constant pressure filtration coefficient defined by Ruth [26,27] after the pressure increase was closely accorded with that in constant pressure filtration conducted at the increased pressure from the beginning of filtration, but that the coefficient after pressure decrease was in disagreement with that in constant pressure filtration conducted at the decreased pressure from the beginning. Subsequently, Shirato

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#### Nomenclature

ds	mean specific surface area size of particles (m)	C
Κ	Ruth coefficient of constant pressure filtration $(m^2/s)$	0
Ke	value of <i>K</i> corresponding to cake structure finally	0
	exhibiting under later pressure for filer cake preformed	6
	at initial pressure $(m^2/s)$	7
т	ratio of mass of wet to mass of dry cake $(-)$	3
n	empirical constant in Eq. $(1)$ (–)	3
р	applied filtration pressure (Pa)	ŋ
$p_a$	empirical constant in Eqs. (1) and (2) (Pa)	θ
R <sub>c</sub>	resistance of filter cake $(m^{-1})$	θ
R <sub>t</sub>	total filtration resistance to filtrate flow (m <sup>-1</sup> )	$\mu$
S	mass fraction of particles in colloid $(-)$	f
t	elapsed filtration time after pressure change (s)	f
v	cumulative filtrate volume per unit effective membrane	
	area $(m^3/m^2)$	S
$v_{\rm t}$	cumulative filtrate volume per unit effective membrane	1
-	area when filtration pressure changes $(m^3/m^2)$	2
		-

Greek letters average specific cake resistance (m/kg)  $l_{av}$ empirical constant in Eq. (1) (m/kg) l1 empirical constant in Eq. (2) (-)pressure drop across filter cake (Pa)  $\Delta p_{c}$ average cake porosity (–) av empirical constant in Eq. (2) (-)rate constant in Eq. (7) (s<sup>-1</sup>) filtration time (s) filtration time when filtration pressure changes (s) viscosity of filtrate (Pas) density of filtrate  $(kg/m^3)$ true density of particles (kg/m<sup>3</sup>) )<sub>s</sub> Subscripts initial stage later stage

et al. [28] obtained a similar result for dead-end ultrafiltration of nanosilica sol. Reihanian et al. [29] examined the flux decline behaviors when the filtrate flow was interrupted due to filtration cell depressurization during the course of filtration in ultrafiltration of bovine serum albumin (BSA) solutions.

The focus of the present study is to delineate the transient flux decline behaviors in dead-end membrane filtration under step-up pressure and step-down pressure conditions, in order to obtain an in-depth understanding of the nature of filter cake formed on the membrane surface in membrane filtration of colloids. Membrane filtration experiments are performed in the dead-end mode using kaolin slurry and nanosilica sol forming compressible filter cakes. The empirical filtration rate equation is proposed in this paper, and two adjustable fitting parameters in the equation are examined by carrying out the membrane filtration experiments under various experimental conditions.

#### 2. Materials and methods

#### 2.1. Materials

The materials used in the experiments were kaolin (Sigma-Aldrich) and nanosilica sol (ST-XS in Snowtex (ST)-series, Nissan Chemical Industries). Test colloids were prepared by dispersing preweighed quantities of the powder or solution in ultrapure, deionized water (resistivity of at least 18 M $\Omega$  cm) produced by purifying tap water through ultrapure water systems equipped both with Elix-UV20 and with Milli-Q Advantage (Bio-POD) for laboratory use (Millipore). The particle concentration by weight fraction was prepared at 0.15 and 0.30 for kaolin slurry and at 0.03 for nanosilica sol. The concentration of kaolin slurry was set to much higher values than that of nanosilica sol in order to avoid precipitation of kaolin particles. Table 1 lists the mean specific surface area size  $d_s$  and true density  $\rho_s$  of colloidal particles, and the properties of filter cake. The mean specific surface area sizes of kaolin

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Properties of test colloids.

and nanosilica particles shown in the table were calculated from the particle size distributions measured by a laser diffraction particle size analyzer (SALD-2200, Shimadzu Corp.) and a dynamic light scattering (DLS) photometer (DLS-8000, Otsuka Electronics), respectively. The true density of particles was measured by a pycnometer. The average specific cake resistance  $\alpha_{av}$  and the average cake porosity  $\varepsilon_{av}$  are, respectively, related to the pressure drop  $\Delta p_c$  across the filter cake as [9,30]

$$\alpha_{\rm av} = \alpha_1 \left( 1 + \frac{\Delta p_{\rm c}}{p_{\rm a}} \right)^n \tag{1}$$

$$1 - \varepsilon_{av} = (1 - \varepsilon_1) \left( 1 + \frac{\Delta p_c}{p_a} \right)^{\beta}$$
<sup>(2)</sup>

where  $\alpha_1$ ,  $p_a$ , n,  $\varepsilon_1$ , and  $\beta$  are the empirical constants. The values of these constants were determined with reference to dead-end membrane filtration methods outlined elsewhere [9,31] and listed in the table. It is found that the filter cakes of both kaolin and nanosilica sol exhibit compressible behaviors.

The membranes employed are mixed cellulose ester microfiltration membranes (Advantec Toyo Corp.) with a nominal pore diameter of 0.1  $\mu$ m for microfiltration of kaolin slurry and regenerated cellulose ultrafiltration membranes (Millipore) with a nominal molecular weight cutoff of 10 kDa for ultrafiltration of nanosilica sol. The complete rejection of colloidal particles was assured throughout the course of membrane filtration with the use of these membranes.

#### 2.2. Experimental apparatus and technique

Membrane filtration experiments were conducted in the deadend mode by using unstirred batch filtration cells with effective membrane areas of 19.40 and 24.61 cm<sup>2</sup> for microfiltration and ultrafiltration, respectively. The filtration cells were fabricated in our laboratory. In the filtration cell for microfiltration experiments,

Material	<i>d</i> <sub>s</sub> (μm)	$\rho_{s}\left(g/cm^{3}\right)$	$\alpha_1 (m/kg)$	p <sub>a</sub> (kPa)	n (-)	ε <sub>1</sub> (-)	β(-)
Kaolin	3.09	2.60	$\begin{array}{c} 7.57 \times 10^{11} \\ 2.07 \times 10^{14} \end{array}$	28.4	0.753	0.772	0.231
Nanosilica sol	0.0048	2.27		15.9	0.629	0.977	0.524

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