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# Fouling behavior during microfiltration of silica nanoparticles and polymeric stabilizers



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## ABSTRACT

Nanotechnology applications give rise to new forms of water pollution, resulting in a need for reliable technologies that can remove nanoparticles from water. Membrane filtration is an obvious candidate. The tendency of nanoparticles to become instable in suspension and form aggregates strongly influences their filtration behavior. This experimental study investigated fouling and rejection during dead-end microfiltration of sterically stabilized nanoparticles. Polyvinylpyrrolidone (PVP) with different molecular weights at different concentrations was used as model steric stabilizer. The large difference between membrane pore size ( $\sim 200$  nm) and the size of the silica nanoparticles (25 nm) allowed a detailed investigation of the filtration process and fouling development. We characterized the feed solution with optical reflectometry, dynamic light scattering, zeta potential measurements and asymmetric flow field flow fractionation (AF4) combined with static light scattering. Subsequently, we looked at the influence of the steric stabilizer (PVP) on nanoparticle fouling development during pore blocking and cake filtration stages.

Our work demonstrates that molecular mass, concentration of the steric stabilizer (PVP) and filtration pressure significantly influence pore blockage and cake filtration. Using a stabilizer with a lower molecular mass generally led to better stabilization of the nanoparticles and the stabilizer contributed less to the fouling. While higher concentrations of the stabilizer enhanced the stability of the nanoparticles, they also caused faster fouling development due to the higher total solute load. Stabilizer with a higher molecular mass was found to contribute more to pore blockage and lead to faster fouling development. Use of a higher transmembrane pressure resulted in compression of the filtration cake, resulting in improved nanoparticle rejection at the expense of permeability.

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## 1. Introduction

The exponential growth of nanotechnology creates new sources of water pollution because engineered nanoparticles occur in many common products, such as pigments, coatings and cosmetics, nowadays and are eventually released into the aqueous environment [1–3]. Water pollution with nanoparticles is therefore expected to become an increasingly serious problem [4]. Although the number of studies into the effect of nanoparticles on living organisms is still low relative to their wide range of application, most researchers suggest that nanoparticles are toxic [5,6] and that their small size allows easy penetration into organs and cells [7]. Therefore, reliable technologies for nanoparticle removal from water sources are needed.

Membrane technology is effective in the removal of much larger colloidal particles, but not much is known about the filtration of engineered nanoparticles yet [8,9]. Up-to-date the application of membrane technology is limited by fouling [10] causing pore constriction and internal fouling; this usually necessitates the use

of sophisticated cleaning procedures [11–13]. Therefore, it is important to determine the mechanisms, factors and parameters that influence nanoparticle fouling in the initial fouling stage to enable the development of filtration strategies that minimize fouling.

Key factor in the development of fouling is the tendency of the nanoparticles to aggregate [14–16]. The stability of aqueous suspensions of nanoparticles depends on the nanoparticles' surface chemistry, the aquatic environment (pH, ionic strength), and on interactions with other compounds present in the solution [17]. Often, steric or electrostatic stabilizers are added to nanoparticle suspensions to keep the nanoparticles dispersed [18]. Steric stabilization is one of the most commonly used and studied forms of stabilization. Steric repulsion occurs after adsorption of a neutral polymer such as polyvinylpyrrolidone or polyethylene oxide onto the nanoparticle surface and prevents aggregation of nanoparticles. Other organic macromolecules, like humic substances, proteins or saccharides, which are present in many water sources, can also act as steric stabilizers [19–21]. As adsorption of such natural macromolecules leads to transformation of the

nanoparticles' surface characteristics and the resulting change in aggregation kinetics, it can facilitate their transport through porous media [22].

The interactions in such feed solutions containing macromolecules and nanoparticles are diverse and simple superposition of the individual fouling contributions cannot be used to describe membrane fouling development in such cases [22]. On the one hand, the higher stability due to steric stabilizers reduces near-membrane surface aggregation as a result of concentration polarization of the rejected nanoparticles [23]. This reduced aggregation of nanoparticles facilitates their transport through the porous membrane structure as individual particles and fouling decreases as the result of the formation of a porous cake [24,25]. On the other hand, the stabilizers themselves can be foulants; they can be retained by the membrane, increasing the thickness of the fouling layer, meanwhile also reducing the porosity of the formed filtration cake by filling the voids between the nanoparticles in the cake [26–28]. Furthermore, polymeric stabilizers may lead to more severe pore clogging due to flocculation bridging, which increases the size of the nanoparticles. Moreover, steric stabilization may not prevent nanoparticles from depositing on the membrane surface either [29]. Therefore, the presence of stabilizers may well result in a higher filtration resistance.

To date, however, there have been no systematic studies of the role of polymeric steric stabilizers during membrane filtration of engineered nanoparticles as far as we know. Therefore, the aim of the research we report here was to determine the effect of steric stabilizers (in this case, polyvinylpyrrolidone (PVP)) on fouling and rejection during microfiltration of nanoparticles. The emphasis of the study was on concentration and chain length (molecular weight) of the PVP and on applied filtration pressure. Whether the stability of the suspension becomes increased or reduced is a function of stabilizer type, concentration and molecular mass (in addition to pH and ionic strength). These properties can therefore strongly affect filtration behavior. The idea behind our work was that combining rejection and permeability data with mixture properties would allow us to identify mechanisms involved in fouling and rejection during microfiltration of sterically stabilized nanoparticle suspensions.

## 2. Theory

### 2.1. Steric stabilization and flocculation

Steric stabilization of nanoparticle suspensions is a matter of finding and maintaining a delicate balance. Adsorption of the stabilizer onto the surface of nanoparticles changes their surface properties. At high enough polymer concentrations, this leads to

the nanoparticles being covered with a dense polymer layer. Steric repulsion between polymer chains on the particle surfaces (Fig. 1a) prevents particle aggregation. On the other hand, at low polymer concentrations or with very long polymer chains (high molecular weight), a single polymer can adsorb onto more than one single nanoparticle. This so-called flocculation bridging (Fig. 1b) leads to the formation of clusters of polymer chains and nanoparticles, effectively promoting the aggregation of the nanoparticles [30,31].

In solutions, there is always an equilibrium between the amount of adsorbed stabilizer and the bulk concentration of the stabilizer [32–34]. At increased concentrations of polymer in the bulk, more polymer chains adsorb onto the nanoparticles. As a result, the conformation of the adsorbed polymer chains changes, as schematically shown in Fig. 2. The trains conformation that occurs at lower polymer concentrations changes to a loops and tails conformation at higher polymer concentrations. The adsorbed polymer chains become more elongated and the nanoparticle size increment as a result of stabilizer adsorption is greater.

### 2.2. Dead-end microfiltration of nanoparticles

In previous work [10], we found that constant pressure dead-end microfiltration of electrostatically stabilized suspensions of nanoparticles much smaller than the membrane pores takes place in the following stages: (1) adsorption, (2) free transport of the nanoparticles through pores, (3) pore blockage, (4) cake filtration, and (5) maturation of the cake. Since these filtration stages can only occur in a specific order and permeability decays at a different rate during each of these stages, they can be easily recognized in the filtration curve (Fig. 3a). Pore blockage and formation of the filtration cake, which acts as a secondary membrane, lead to a final nanoparticle rejection to about 90%, as visible in Fig. 3b.

## 3. Materials and methods

### 3.1. Materials

Colloidal silica nanoparticles Ludox TM-50 with a diameter of about 25 nm [10] were used as model nanoparticles. We purchased three types of polyvinylpyrrolidone (PVP), each with a different molecular mass; their average molecular masses were 10 kDa, 40 kDa and 360 kDa (Sigma Aldrich supplier data). Aqueous solutions of ACS grade HCl or NaOH, and NaCl, (Sigma Aldrich) were used to adjust the pH and ionic strength of the solutions, respectively. All solutions were prepared with ultrapure water (Milli-Q, resistivity > 18.2 MΩ cm); all chemicals were used without further purification.

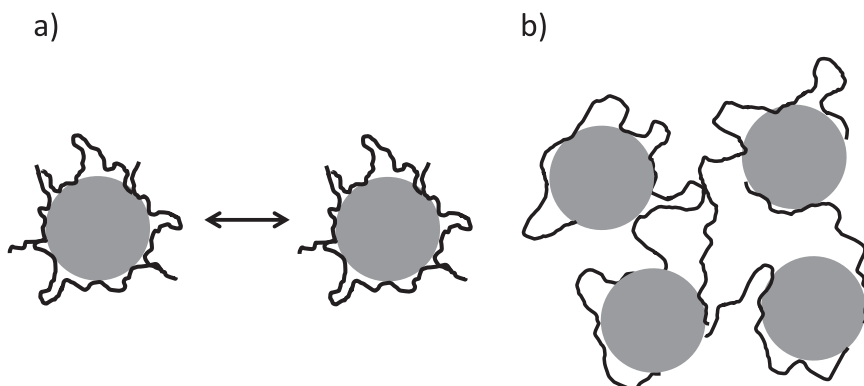


Fig. 1. Schematic representation of (a) steric stabilization and (b) flocculation bridging.

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