

A Proposed Experimental-Design Framework for Evaluating Aluminium Oxide Nanoparticle Ultrafiltration: Concentration, Transmembrane Pressure and Membrane MWCO

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Abstract

Engineered aluminium oxide (Al_2O_3) nanoparticles provide a model particulate for examining membrane transport, aggregation and fouling in water-treatment systems. This article proposes a methodological framework for evaluating permeate flux during crossflow ultrafiltration with polyethersulfone membranes. The planned factors are Al_2O_3 concentration (20, 30 and 40 mg/L), transmembrane pressure (0.5, 1.0 and 1.5 bar), and membrane molecular weight cut-off (MWCO; 1, 5 and 10 kDa). Because the MWCO levels are unequally spaced and represent discrete membrane products, MWCO is treated as a categorical factor rather than as an equally spaced continuous variable. The proposed design comprises 27 unique factor combinations, with additional replication to estimate pure error. The primary response is prespecified as time-averaged permeate flux; aluminium rejection and pre-/post-filtration pure-water flux are recommended secondary measurements. No experimental results, fitted statistical model or optimum operating condition are reported; the article is limited to the design and analysis protocol. The specific methodological contribution is a transparent design and analysis protocol that defines response variables, identifies testable interaction hypotheses, and specifies the measurements required to evaluate flux, rejection and fouling without presenting theoretical expectations as empirical findings.

Keywords: aluminium oxide nanoparticles; crossflow ultrafiltration; permeate flux; transmembrane pressure; molecular weight cut-off; membrane fouling; critical flux; nanoparticle deposition; surface charge

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INTRODUCTION

The development and widespread application of engineered nanomaterials have increased interest in their environmental behaviour, transport and removal from aqueous systems. Nanomaterials are incorporated into industrial and consumer products because their high specific surface area and distinctive surface chemistry can provide useful functional properties. Their life-cycle release, transformation and persistence in environmental compartments remain important research questions, including for aluminium oxide (Al_2O_3) nanoparticles detected or investigated in aquatic systems [1], [2].

Water-treatment technologies must address contaminants that do not behave like conventional dissolved pollutants. Depending on pH, ionic strength, concentration and surface chemistry, nanoparticles may remain dispersed or form aggregates. Their separation can therefore involve size exclusion, aggregation, adsorption and particle-membrane interactions. Pressure-driven membrane processes, including ultrafiltration (UF), are relevant to particulate and colloidal separation, but their performance depends on membrane structure and feed chemistry rather than nominal size alone [3], [4].

Ultrafiltration (UF) is a pressure-driven membrane process positioned between microfiltration and nanofiltration in terms of nominal separation characteristics. UF membranes are commonly applied to the removal or concentration of macromolecules, colloids, microorganisms and other particulate materials [4], [5]. The performance of UF is normally assessed through parameters such as permeate flux, solute or particle rejection, permeability and fouling behaviour. Permeate flux represents the rate of permeate production per unit membrane area and is therefore a fundamental operational parameter for assessing membrane productivity [6].

The relationship between applied pressure and permeate flux is, however, more complex than a simple proportional relationship. Under conditions in which membrane resistance is dominant and concentration polarisation and fouling are limited, an increase in hydraulic pressure can increase the driving force for permeation. As the filtration process proceeds, however, material may accumulate near or on the membrane surface. Concentration polarisation results from the development of a concentration gradient near the membrane, whereas fouling involves the accumulation or deposition of material that contributes additional resistance to filtration. These phenomena can alter the effective driving force and resistance of the membrane system [6], [7].

Field and Wu emphasised the importance of distinguishing concentration polarisation from fouling when interpreting flux behaviour. Concentration polarisation can develop rapidly following the establishment of filtration, whereas longer-term changes in flux can involve the formation and development of a fouling layer. Consequently, flux decline over a relatively extended filtration period should not automatically be attributed to concentration polarisation alone [6]. This distinction is particularly relevant when nanoparticle suspensions are investigated because particle accumulation at the membrane surface can modify local hydrodynamics and filtration resistance.

Membrane fouling remains one of the principal limitations affecting the performance of pressure-driven membrane processes. Fouling can involve adsorption, pore blocking, deposition and cake-layer formation, with the relative

contribution of each mechanism depending on the membrane material, feed composition and operating conditions [7], [8], [9].

The resistance-in-series concept provides one useful framework for understanding these effects. In its simplest form, the total resistance to filtration can be represented as the sum of the intrinsic membrane resistance and additional resistances associated with deposited or retained material. Although the resistance-in-series approach has limitations and should not be interpreted as a complete description of all fouling mechanisms, it provides a useful conceptual basis for analysing changes in membrane permeability [8], [10].

Polyethersulfone (PES) is widely used as a polymeric membrane material because of its chemical stability, thermal stability, mechanical properties and suitability for membrane fabrication [11]. Nevertheless, PES has a relatively hydrophobic character, which may contribute to interactions with foulants and the development of membrane fouling [12], [13]. The surface properties and pore structure of PES membranes can therefore influence both permeability and separation performance. Previous research has examined various approaches to improving PES membrane hydrophilicity and reducing fouling, including blending, surface modification and incorporation of hydrophilic materials [12], [13], [14]. Even when commercially available membranes are used without modification, their nominal MWCO remains an important membrane characteristic in evaluating their expected separation behaviour.

MWCO is commonly used to describe the nominal separation capability of UF and related membranes. It is normally associated with the molecular weight of a reference solute at a specified retention level, although the exact definition and determination can vary according to the test method and membrane system [15]. MWCO should therefore not be interpreted as a universal pore-size measurement. The relationship between MWCO, pore structure, solute transport and permeate flux can be affected by the membrane material and by the characteristics of the feed. Recent work has further demonstrated that MWCO determination can become particularly complex when the membrane operates near the boundary between UF and nanofiltration because retention can involve factors beyond simple size exclusion [15].

The influence of membrane MWCO on filtration behaviour is therefore of particular interest in nanoparticle separation. A membrane with a lower nominal MWCO would generally be expected to offer greater resistance to the passage of larger species and potentially lower hydraulic permeability, whereas a membrane with a higher nominal MWCO may exhibit higher permeability. This does not mean, however, that MWCO alone determines nanoparticle retention. Nanoparticles are not molecular solutes, and their transport may depend on particle size distribution, aggregation, surface chemistry, hydrodynamic interactions and membrane-particle interactions. Consequently, the use of membranes with different MWCO values provides an opportunity to investigate how nominal membrane selectivity interacts with the hydraulic conditions of crossflow filtration.

The pressure-flux relationship depends on feed properties, membrane resistance and hydrodynamic conditions. An increase in TMP raises the driving force, but it may also increase particle delivery to the membrane; whether net flux increases or plateaus depends on the balance between permeation and added resistance [6], [16], [17].

Previous membrane studies have demonstrated the usefulness of statistical experimental design for examining several operating factors simultaneously. Response surface methodology provides a structured statistical approach for studying the relationships between independent variables and one or more responses. Rather than changing one factor at a time, RSM allows researchers to investigate main effects, quadratic effects and interactions within a defined experimental region [18], [19]. Central composite design (CCD) is one of the commonly used experimental designs within RSM and can be applied to develop second-order response models. In membrane research, RSM has been used to examine the effects of variables such as TMP, feed concentration, temperature, flow conditions and pH on permeate flux and separation performance [18], [19], [20], [21].

Published crossflow ultrafiltration research demonstrates that response-surface methodology (RSM) can examine interactions among operating factors [19], [20]. However, results from other feed and membrane systems cannot be transferred directly to Al_2O_3 /PES filtration. The present study is consequently framed as an experimental methodology for evaluating concentration, transmembrane pressure (TMP) and molecular weight cut-off (MWCO); no numerical optimum or statistical effect is claimed without the corresponding experimental observations.

The application of RSM to nanoparticle-containing feeds is especially relevant because the factors influencing flux are unlikely to operate independently. Increasing nanoparticle concentration can increase the amount of material available for accumulation at the membrane surface. Increasing TMP can increase the hydraulic driving force but may simultaneously influence particle deposition and the structure of a retained layer. Changing membrane MWCO can alter hydraulic permeability and the interaction between the membrane and suspended particles. These factors can therefore produce effects that cannot be adequately represented by examining each variable in isolation.

Although membrane processes for nanoparticle-containing water have received increasing attention, there remains value in establishing systematic experimental frameworks that simultaneously consider feed nanoparticle concentration, TMP and membrane MWCO. Such a framework can provide a more controlled basis for distinguishing membrane-dependent effects from operating-condition effects. It can also facilitate the use of statistical modelling where sufficient experimental observations are available.

This manuscript is presented as a methodological and experimental-design article, not as a report of completed experiments. Its objectives are to (1) define a reproducible protocol for evaluating permeate flux across Al_2O_3 concentration and TMP levels; (2) treat membrane MWCO appropriately as a three-level categorical factor because the 1, 5 and 10 kDa levels are not equally spaced; (3) specify testable hypotheses for concentration–pressure, concentration–membrane and pressure–membrane interactions; and (4) define the statistical and validation procedures required before any optimum condition can be claimed. No measured flux data or fitted model are reported in this manuscript.

The proposed design is a mixed-factor full-factorial experiment rather than a conventional three-factor central composite design. Concentration (20, 30 and 40 mg/L) and TMP (0.5, 1.0 and 1.5 bar) are quantitative factors; MWCO (1, 5 and 10 kDa) is treated as categorical. The $3 \times 3 \times 3$ arrangement gives 27 unique factor

combinations. Replication should be added at preselected conditions to estimate pure error, and the final run order should be randomised. This design avoids assigning an artificial numerical midpoint to the unequally spaced MWCO levels.

MATERIALS AND METHODS

Materials

Aluminium oxide (Al_2O_3) nanoparticles supplied as a fine powder with a nominal primary particle size of approximately 30 nm are specified as the model inorganic nanoparticle material. Distilled water is used as the dispersion medium and for pure-water membrane testing.

Polyethersulfone (PES) ultrafiltration membrane cassettes with nominal MWCO values of 1, 5 and 10 kDa are used as the membrane materials. The use of three nominal MWCO levels enables the influence of membrane characteristics to be considered within the selected experimental range. The membrane MWCO values are treated as nominal manufacturer or specification characteristics rather than direct measurements of membrane pore diameter, consistent with the recognised limitations of MWCO as a membrane descriptor [15].

Preparation of Aluminium Oxide Suspensions

The proposed feed concentrations are 20, 30 and 40 mg/L. These levels are deliberately elevated laboratory challenge concentrations, comparable to industrial or polishing-effluent loadings, chosen to examine membrane response; they are not intended to represent environmental exposure concentrations.

For each feed, the weighed Al_2O_3 is dispersed in distilled water using a documented sonication protocol, with the sonicator model, delivered power, duration, suspension temperature, stirring speed, pH and ionic strength recorded. Because Al_2O_3 aggregation depends strongly on pH, the working pH is measured and reported together with the hydrodynamic diameter, polydispersity index and zeta potential, as these measurements are required for reproducibility.

The prepared suspension is kept under continuous mixing during the filtration process to minimise uncontrolled settling and to maintain a more uniform feed concentration. This procedure is particularly relevant for nanoparticle systems because aggregation and sedimentation can influence the concentration actually delivered to the membrane surface.

Crossflow Ultrafiltration System

The protocol uses a crossflow ultrafiltration system equipped with PES membrane cassettes. To ensure reproducibility, the membrane manufacturer, cassette model, effective membrane area, channel dimensions, system hold-up volume, pump model, pressure-sensor positions and operating mode are reported. A total-recycle arrangement is recommended so that retentate and permeate are returned to the feed reservoir and the nominal feed concentration remains approximately constant; if concentration mode is used instead, feed concentration must be measured throughout each run.

TMP is proposed at 0.5, 1.0 and 1.5 bar, subject to confirmation that all values fall within the membrane manufacturer's operating limits. TMP should be controlled using the retentate valve and/or pump speed, with feed-side and retentate-side pressures recorded. Crossflow velocity or feed flow rate and feed temperature are measured and held constant across runs, and the apparatus (pump, reservoir,

membrane module, pressure gauges, flow meter and permeate collection point) is documented with a schematic.

Before each experimental series, the membrane should be compacted using a documented pressure and duration until a predefined flux-stability criterion is met. Pure-water flux should then be measured at all three TMP levels, allowing hydraulic permeability and membrane resistance to be estimated from the slope of flux against TMP rather than from a single point. The compaction pressure, duration, stability criterion and temperature are recorded for each membrane.

Transmembrane Pressure

TMP is calculated from the measured feed-side pressure, retentate-side pressure and permeate pressure according to

$$\text{TMP} = \frac{P_f + P_r}{2} - P_p \quad (1)$$

where P_f is the feed pressure, P_r is the retentate pressure and P_p is the permeate pressure.

The use of the average of the feed and retentate pressures is appropriate for a crossflow system in which pressure can change along the membrane channel. TMP therefore represents the effective pressure driving permeation across the membrane rather than simply the inlet feed pressure.

Pure-Water Flux

Pure-water flux is determined from the volume of permeate collected over a specified time interval and normalised by the effective membrane area:

$$J_w = \frac{V}{A t} \quad (2)$$

where J_w is the pure-water flux, V is the collected permeate volume, A is the effective membrane area and t is the filtration time.

The resulting value provides a reference for the hydraulic performance of the membrane under the selected operating condition. Pure-water permeability is commonly used in membrane studies to distinguish changes associated with the membrane itself from changes resulting from feed-membrane interactions [6], [10].

Membrane Hydraulic Resistance

The intrinsic hydraulic resistance of the membrane can be represented using Darcy's law:

$$J_w = \frac{\Delta P}{\mu R_m} \quad (3)$$

and therefore

$$R_m = \frac{\Delta P}{\mu J_w} \quad (4)$$

where R_m is the membrane hydraulic resistance, ΔP is the effective pressure difference across the membrane, μ is the permeate viscosity and J_w is the pure-water flux.

This relationship provides a conceptual basis for examining how membrane structure influences hydraulic permeability. A membrane with greater intrinsic resistance will generally require a greater driving force to produce the same flux, assuming equivalent fluid properties and filtration conditions.

The resistance calculation is not intended to imply that all filtration resistance during nanoparticle processing originates from the membrane. Once the nanoparticle suspension is introduced, additional resistance may arise from adsorption, pore blocking, particle deposition and cake-layer development [8], [10].

For nanoparticle filtration, the total effective filtration resistance can be represented by the Darcy-type relationship.

$$J = \frac{\Delta P}{\mu R_t} \quad (5)$$

where J is the permeate flux, ΔP is the effective transmembrane pressure, μ is the dynamic viscosity of the permeating fluid and R_t is the total filtration resistance.

The total resistance R_t represents the combined resistance of the clean membrane and additional resistance associated with concentration polarisation, particle deposition, pore blocking and fouling. Thus, R_t may increase during filtration as nanoparticles accumulate near or on the membrane surface.

Nanoparticle Filtration

After membrane preparation and pure-water testing, the Al_2O_3 suspension is introduced into the crossflow filtration system. The suspension is maintained under continuous stirring to minimise settling during the filtration experiment.

A provisional filtration period of 10 minutes, with permeate collected at two-minute intervals, is specified; this period should not be assumed to establish quasi-steady operation. The filtration duration should be justified by a preliminary flux-time test and extended until a predefined stability criterion is reached, or explicitly described as an early-stage filtration test. Permeate volume should be recorded at fixed intervals. The primary response for modelling should be defined in advance; the recommended response is the time-averaged permeate flux over a stated, identical interval for every run, calculated from permeate volume, effective membrane area and collection time. Instantaneous interval fluxes and the full flux-time profiles should also be reported.

$$J = \frac{V}{A t} \quad (6)$$

where J is the permeate flux, V is the collected permeate volume, A is the membrane area and t is the corresponding filtration time.

The use of short collection intervals provides a means of examining changes in permeate production during the filtration period. This is relevant because membrane flux may change with time as the system approaches a condition influenced by concentration polarisation, particle deposition or fouling.

Experimental Variables

Three independent variables are considered in the experimental design (Table 1).

Table 1. Experimental factors and levels in the proposed $3 \times 3 \times 3$ mixed-factor design (MWCO treated as a categorical factor)

Factor	Variable	Low level	Centre level	High level
A	Al ₂ O ₃ concentration	20 mg/L	30 mg/L	40 mg/L
B	TMP	0.5 bar	1.0 bar	1.5 bar
C	Membrane MWCO	1 kDa	5 kDa	10 kDa

These variables were selected because they represent three different classes of factors affecting membrane performance. Al₂O₃ concentration represents feed loading, TMP represents hydraulic driving force, and MWCO represents membrane characteristics.

Experimental Design and Response Surface Methodology

Because the three MWCO levels (1, 5 and 10 kDa) are not equally spaced and MWCO is a discrete membrane property, a conventional three-factor central composite design is not appropriate unless a defensible coding scheme and its statistical consequences are demonstrated. The proposed analysis therefore treats MWCO as a categorical factor and uses a $3 \times 3 \times 3$ full-factorial design for concentration, TMP and MWCO. This yields 27 unique combinations; replicate runs should be added at selected conditions to estimate pure error and support lack-of-fit assessment. Run order should be randomised, with the randomisation procedure recorded.

The general second-order model can be expressed as:

$$Y = \beta_0 + \sum_{i=1}^k \beta_i X_i + \sum_{i=1}^k \beta_{ii} X_i^2 + \sum_{i < j} \beta_{ij} X_i X_j + \epsilon \quad (7)$$

where Y represents the response, β_0 is the intercept, β_i represents the linear coefficient, β_{ii} represents the quadratic coefficient, β_{ij} represents the interaction coefficient, X_i and X_j are the coded independent variables and ϵ represents the experimental error.

The proposed design comprises 27 unique combinations (3 concentration levels $\times$ 3 TMP levels $\times$ 3 categorical MWCO levels), before replication. The final number of runs will equal 27 plus the number of replicate runs selected in the protocol. A complete run matrix should list both actual factor values and coded values for the two quantitative factors; MWCO should be represented by indicator variables rather than a falsely equally spaced numeric code. The exact run order must be generated and recorded before experimentation.

RSM has been widely applied in membrane research because it can provide a structured approach to the simultaneous investigation of multiple operating variables. Published studies have used CCD or related response-surface designs to evaluate membrane flux, rejection and fouling behaviour under different combinations of pressure, feed characteristics and hydrodynamic conditions [18], [19], [20], [21].

Statistical Analysis Framework

The primary response is the prespecified time-averaged permeate flux, reported in $\text{L m}^{-2} \text{h}^{-1}$ and calculated as permeate volume divided by effective membrane area and collection time. The temperature at which viscosity is evaluated

must be recorded; if flux is corrected to a reference temperature, the correction equation and reference temperature must be stated. A second response, nanoparticle rejection, should be added by measuring aluminium concentration in feed and permeate using ICP-OES or AAS, or by using a validated turbidity method if elemental analysis is unavailable. Flux alone cannot establish nanoparticle removal.

Analysis of variance (ANOVA) is normally used to assess the statistical significance of model terms. Model quality can also be evaluated using the coefficient of determination (R^2), adjusted R^2 , predicted R^2 , residual analysis and lack-of-fit assessment. These statistical measures should be interpreted together rather than independently because a high R^2 value alone does not guarantee that a model provides a scientifically adequate description of experimental behaviour.

Response-surface and contour plots can subsequently be used to visualise interactions between pairs of variables while holding the third variable within the experimental region. Such graphical analysis is particularly useful for identifying whether the effect of one factor depends on the level of another factor.

This article reports no measured flux values, ANOVA results, fitted coefficients, R^2 values, response surfaces or optimum conditions. Once data are collected, analysis should report the software and version, significance level, model hierarchy, residual diagnostics, lack-of-fit test, adjusted and predicted R^2 , adequate precision, and any transformation considered (including the rationale for a Box–Cox transformation). Confirmation runs at the predicted optimum should be compared with model predictions.

PROPOSED ANALYTICAL FRAMEWORK AND TESTABLE HYPOTHESES

Relationship between Operating Variables and Permeate Flux

Permeate flux is one of the most important performance indicators in ultrafiltration because it represents the rate at which liquid passes through a unit area of membrane. From a fundamental perspective, flux is determined by the balance between the effective driving force and the total resistance encountered by the permeating liquid [6], [16], [22].

For an idealised membrane system, the relationship is expressed by the Darcy-type equation given in Eq. (5),

where R_t represents the total effective resistance of the filtration system. In a clean membrane, this resistance is predominantly associated with the membrane itself. During filtration of a suspension, however, additional resistance may arise from concentration polarisation, adsorption, pore blocking and deposited material.

This distinction is important for interpreting the three factors selected in the present study. TMP primarily modifies the driving force, MWCO reflects membrane structure and expected permeability, and nanoparticle concentration affects the quantity of particulate material presented to the membrane. Their effects are therefore physically different but potentially interconnected.

An increase in TMP would ordinarily increase the pressure difference available for permeation. Nevertheless, the increase in flux does not necessarily remain proportional to the pressure increase throughout the operating range. When retained material accumulates near the membrane surface, the additional pressure may be accompanied by increased concentration polarisation or compaction of the deposited layer. Consequently, the effective resistance may increase simultaneously with the driving force [6], [16].

A transition from pressure-sensitive flux to a plateau or declining flux is consistent with increasing concentration polarisation or fouling. The critical-flux framework provides one way to interpret this transition, but its threshold must be measured for the specific suspension and membrane system [23], [24].

The Al_2O_3 concentration is similarly expected to influence the filtration environment. A higher nanoparticle concentration increases the quantity of suspended particulate matter entering the membrane module. Depending on the stability and transport of the suspension, this can increase the likelihood of particle accumulation at the membrane surface. The resulting layer can modify the local mass-transfer conditions and increase the resistance to permeation.

However, it would be inappropriate to assume that increasing nanoparticle concentration must always produce the same magnitude or direction of flux change under all operating conditions. Particle-particle interactions, aggregation, crossflow shear and membrane surface interactions can alter the behaviour of the retained phase. The relationship between concentration and flux is therefore better treated as an experimentally testable relationship rather than a predetermined linear response.

Concentration Polarisation and Fouling

Concentration polarisation and fouling are central considerations in the interpretation of ultrafiltration flux. Concentration polarisation refers to the accumulation of retained species within a concentration boundary layer adjacent to the membrane. Fouling involves the deposition or attachment of material to the membrane or within its pores and produces additional resistance to filtration [6], [7].

Concentration polarisation and fouling should not be treated as synonymous. Concentration polarisation describes the build-up of retained species near the membrane, whereas fouling involves deposition, adsorption or pore blocking that adds filtration resistance [6], [7]. Because the provisional filtration period is only 10 minutes, flux decline alone cannot establish whether a persistent fouling layer has formed.

The presence of Al_2O_3 nanoparticles introduces a particulate component to the feed that may contribute to surface deposition. Depending on particle dispersion and crossflow conditions, nanoparticles may remain suspended, form aggregates or accumulate near the membrane surface. The resulting filtration layer may influence the hydraulic resistance of the membrane system.

Critical flux and fouling-model interpretation. Field et al. introduced the critical-flux hypothesis for microfiltration, under which a flux below a system-specific threshold may avoid measurable time-dependent fouling; Bacchin et al. later clarified critical and sustainable flux concepts and their dependence on membrane, suspension and hydrodynamic conditions [23], [24]. The present design varies TMP rather than directly imposing constant flux, so it cannot identify a critical flux without additional flux-stepping experiments or suitable time-resolved resistance analysis. The observed flux-time records should instead be analysed cautiously as evidence consistent with, but not definitive proof of, particular deposition mechanisms.

Pore-blocking models provide a complementary interpretation of flux-time data. Complete blocking assumes that arriving particles block available pore entrances; standard blocking describes progressive constriction of pores;

intermediate blocking allows both pore blocking and deposition on previously retained material; and cake filtration represents growth of a surface layer. These models are idealisations, and model selection should be based on residuals and the adequacy of the time-series data rather than visual fit alone [25].

Electrostatic interactions are another plausible control on deposition. Alumina particles commonly have a pH-dependent surface charge, while PES surfaces are often negative around neutral pH. If the suspension pH is below the alumina isoelectric point, attractive particle-membrane interactions may enhance deposition despite the small primary particle size. Suspension pH, ionic strength and zeta potential are therefore specified as required measurements in the proposed protocol and must be obtained before electrostatic attraction is assigned as the dominant mechanism [26].

Crossflow operation can reduce the residence time of particles at the membrane surface by providing tangential shear. Nevertheless, crossflow does not eliminate fouling. Instead, it changes the balance between convective transport towards the membrane and shear-induced transport away from the surface. The resulting behaviour depends on particle concentration, particle size and aggregation, membrane characteristics and operating conditions.

The distinction between reversible and irreversible fouling is also relevant. A loosely deposited particle layer may be more readily removed by hydraulic or mechanical action, whereas adsorption or pore penetration can result in more persistent resistance. Resistance-in-series models provide one way of conceptualising these contributions, although the individual resistances should not automatically be regarded as completely independent [8], [10].

For the present study, pure-water flux establishes a baseline hydraulic condition. Comparing clean-water and nanoparticle-feed fluxes can help identify changes in apparent resistance, although post-filtration water-flux or cleaning-recovery measurements would be needed to distinguish reversible deposition from persistent fouling.

Influence of Membrane MWCO

The three PES membranes selected in the study represent different nominal MWCO levels. MWCO is a widely used descriptor in ultrafiltration because it provides a practical indication of membrane selectivity. Nevertheless, it is not equivalent to a single, precisely defined pore diameter [15].

Membrane pore structures normally exhibit distributions rather than a single uniform pore dimension. Consequently, two membranes with nominally similar MWCO values may exhibit different permeability and retention characteristics depending on their material, fabrication method and surface properties.

The general relationship between membrane MWCO and hydraulic permeability is nevertheless important. A membrane with a larger nominal MWCO generally possesses a more open structure and can allow greater water transport under otherwise comparable conditions. Published research concerning PES membranes has demonstrated that membrane structure and MWCO can influence flux and separation behaviour [12], [15], [27].

For nanoparticle filtration, however, membrane MWCO should be interpreted carefully. Al_2O_3 nanoparticles are particulate entities rather than molecular solutes used in conventional MWCO determination. The nominal MWCO therefore does not

directly indicate the percentage of nanoparticles that will pass through the membrane.

Nanoparticle retention can be influenced by particle diameter, aggregation state, surface charge, membrane surface characteristics and hydrodynamic interactions. A nanoparticle suspension can therefore behave differently from a solution of dissolved molecules with an equivalent nominal molecular weight.

This distinction reinforces the value of examining MWCO experimentally rather than using the nominal MWCO value as a direct prediction of nanoparticle removal. The selected 1, 5 and 10 kDa membranes provide a controlled range of membrane characteristics through which changes in hydraulic performance and particle-membrane interaction can be examined.

Influence of Transmembrane Pressure

TMP provides the principal hydraulic driving force for pressure-driven membrane filtration. At relatively low resistance, increasing TMP generally increases permeate flux [6], [16]. However, the critical-flux concept distinguishes a regime in which particle deposition is limited from one in which increasing the driving force promotes accumulation and flux decline. The critical flux is system-specific and cannot be inferred from TMP alone; it must be determined from flux or resistance measurements under controlled conditions [23], [24]. Above a sustainable operating flux, the rate of fouling may become operationally unacceptable even if an initial increase in pressure raises flux [24].

The use of 0.5, 1.0 and 1.5 bar in the present design creates a defined operating range within which pressure effects can be examined. The centre level of 1.0 bar also provides a reference point for evaluating the response surface around the middle of the experimental domain.

The pressure effect may also interact with nanoparticle concentration. At a higher nanoparticle loading, an increase in TMP may increase the rate at which liquid is forced towards the membrane surface. If the rate of particle transport towards the surface exceeds the rate at which particles can be removed by crossflow, the retained material may accumulate more rapidly. The resulting additional resistance could reduce the apparent benefit of increased pressure.

This interaction is one reason why response-surface analysis is appropriate for the experimental framework. A one-factor-at-a-time approach could demonstrate that pressure affects flux but would provide limited information about whether the magnitude of the pressure effect changes with nanoparticle concentration.

The pressure effect can also interact with membrane MWCO. A membrane with a lower hydraulic permeability may respond differently to an increase in TMP than a membrane with a more open structure. The increased pressure may improve water transport, but the degree to which this occurs will depend on the resistance of the membrane and the additional resistance created during filtration.

Influence of Aluminium Oxide Nanoparticle Concentration

Al₂O₃ is widely used in industrial and technological applications, and its occurrence in water-related research has generated interest in separation and environmental behaviour. Nanoparticles differ from dissolved molecular contaminants because their transport can involve aggregation, deposition and particle-surface interactions.

At lower nanoparticle concentrations, the amount of material available for deposition at the membrane surface is comparatively limited. At higher concentrations, the increased particle loading can increase the potential for formation of a concentration boundary layer or deposited particle layer.

Nevertheless, nanoparticle concentration should not be treated as a simple measure of fouling intensity. Particle stability is an important factor. A well-dispersed suspension can exhibit different transport characteristics from an aggregated suspension. The effective hydrodynamic size of aggregated particles can be substantially larger than the primary particle size.

The continuous stirring adopted in the present experimental framework is therefore important because it helps maintain suspension uniformity and reduce uncontrolled sedimentation. It does not, however, guarantee that all nanoparticles remain individually dispersed. Nanoparticle aggregation can still occur depending on the physicochemical properties of the suspension.

The concentration range of 20 to 40 mg/L provides a defined experimental domain for evaluating the effect of increasing Al_2O_3 loading. The centre concentration of 30 mg/L allows the response to be examined around an intermediate feed condition.

Role of Crossflow Hydrodynamics

Crossflow hydrodynamics influence membrane performance by altering mass transfer and the accumulation of retained material at the membrane surface. For the nominal 30 nm Al_2O_3 particles used here, Brownian diffusion is expected to contribute importantly to back-transport. Crossflow shear may reduce concentration polarisation, but it does not guarantee particle removal: particle-membrane attraction, aggregation and surface deposition can still dominate. Shear-induced diffusion and inertial lift are generally more relevant to micrometre-sized particles than to nanoparticles of this scale [17].

In nanoparticle filtration, the relationship between crossflow and particle transport can be particularly important. Stronger shear can reduce surface accumulation, whereas weak shear may permit greater deposition. The resulting balance can influence the transition between stable flux, concentration polarisation and fouling.

Although crossflow velocity is not treated as an independent factor in the present experimental design, the crossflow configuration is held as part of the experimental system. This approach allows the study to focus on the selected variables of concentration, TMP and MWCO.

Keeping hydrodynamic conditions consistent is important for experimental comparability. If crossflow velocity varied substantially between experimental runs, changes in flux could be attributed to hydraulic differences rather than the intended independent variables.

The literature on membrane filtration confirms that fluid dynamics and fluid-particle interactions can influence the structure and characteristics of fouling layers [7]. Therefore, the crossflow configuration should be regarded as part of the controlled experimental environment rather than an irrelevant operating detail.

Importance of Polyethersulfone Membrane Properties

PES is frequently selected for pressure-driven membrane applications because of its useful mechanical and chemical properties. Its surface charge and that of the

particles should also be considered: PES is commonly negatively charged near neutral pH, while alumina surface charge depends on pH and is often positive below its isoelectric point (typically around pH 8–9, depending on material and solution conditions). Under such conditions, electrostatic attraction may promote particle deposition; at other pH values or ionic strengths, the interaction may differ. Because pH and zeta-potential data are not yet available, this mechanism should be treated as a plausible hypothesis rather than a demonstrated result [26].

The membrane surface is not simply a passive barrier. Surface chemistry can influence adsorption and deposition, while pore structure controls hydraulic resistance and retention [28]. Consequently, membrane MWCO should be considered together with the underlying material characteristics.

Studies of PES modification have demonstrated that increasing membrane hydrophilicity can improve water permeability and reduce the tendency for fouling [12], [13]. Although membrane modification is outside the scope of the present framework, these findings demonstrate why membrane material must be considered when interpreting filtration performance.

Using PES membranes with different nominal MWCO values provides a controlled means of examining how membrane characteristics influence flux. It also creates an opportunity to separate, at least conceptually, the effects associated with membrane structure from those associated with the nanoparticle suspension.

Response Surface Methodology as an Analytical Framework

RSM provides a useful framework for membrane experiments because several variables can influence the response simultaneously. Traditional one-factor-at-a-time experimentation can identify individual effects but is inefficient when interactions between variables are important.

A second-order RSM model incorporates linear, quadratic and interaction terms. The linear terms represent the primary influence of each factor, quadratic terms allow curvature to be represented, and interaction terms determine whether the effect of one factor depends on the level of another factor.

The application of RSM to membrane processes has been demonstrated in studies involving different feed systems and membrane configurations. For example, crossflow UF research has applied statistical modelling to assess the influence of feed concentration, temperature and TMP on permeate flux and treatment performance [19]. Other studies have used RSM to optimise permeate flux and fouling-related responses by considering TMP, temperature and flow rate simultaneously [20].

These applications demonstrate the methodological value of RSM but should not be interpreted as evidence for specific numerical relationships in the Al_2O_3 /PES system considered here. Different membranes, feeds and hydrodynamic conditions can produce different responses.

The present framework therefore uses RSM as an analytical structure rather than assuming that relationships established in other membrane systems will apply directly to Al_2O_3 nanoparticle filtration.

Interactions among Concentration, TMP and MWCO

The central scientific value of combining the three independent variables is that membrane flux is a system response rather than a consequence of a single parameter.

Consider the interaction between Al_2O_3 concentration and TMP. At low concentration, an increase in pressure may produce a comparatively direct increase in flux because the particulate resistance is relatively limited. At higher concentration, however, greater pressure may increase the transport of particles towards the membrane and contribute to surface accumulation. The net flux response could therefore differ between concentration levels.

Similarly, the effect of TMP may depend on membrane MWCO. A membrane with lower hydraulic permeability may exhibit a different pressure response from a membrane with greater permeability. This TMP–MWCO (B×C) interaction is therefore stated as a testable hypothesis rather than an expected result.

The interaction between nanoparticle concentration and MWCO is also physically meaningful. A more open membrane may offer greater water permeability but may exhibit different particle-membrane interactions from a tighter membrane. Conversely, a tighter membrane may provide greater retention while producing greater intrinsic hydraulic resistance.

These relationships illustrate why a factorial or response-surface framework is preferable to interpreting each variable separately. The purpose of the experimental design is not merely to identify whether a factor matters but to establish how the response changes across the selected multidimensional operating region.

Interpretation of Flux Decline

Flux decline is commonly observed during filtration when the effective resistance of the membrane system increases. Candidate mechanisms include concentration polarisation, adsorption, complete or standard pore blocking, intermediate blocking and cake filtration. Hermia-type blocking laws can be fitted to suitable flux–time data to compare these mechanisms, but a short experiment and sparse measurements may not uniquely identify one dominant mechanism [25].

The interpretation of flux decline should therefore remain conservative. A reduction in flux during the 10-minute filtration period should not automatically be described as irreversible fouling. Establishing irreversible fouling would require additional evidence, such as post-filtration pure-water permeability or cleaning and recovery measurements.

The experimental design does, however, provide a basis for examining temporal changes in permeate production. Collecting permeate at two-minute intervals allows the flux behaviour to be considered over the filtration period rather than relying only on a single cumulative value.

This temporal approach is particularly useful because a stable flux and a progressively declining flux represent different membrane-process behaviours. A stable flux may indicate that the filtration system has approached a quasi-steady condition under the selected operating parameters, whereas a continuing decline may indicate increasing resistance.

The distinction is consistent with the broader membrane literature, which emphasises that flux-time behaviour contains information about the mechanisms governing membrane performance [6], [16].

Pure-Water Baseline and Resistance Measurements

Pure-water testing is an essential reference procedure because membrane flux is influenced by intrinsic membrane resistance. Without a baseline measurement, it

becomes difficult to distinguish between a naturally low-permeability membrane and a reduction in permeability caused by nanoparticle filtration.

Membrane resistance should be estimated from the slope of pure-water flux against TMP after compaction, using viscosity measured at the recorded test temperature. This avoids relying on a single operating point and provides a baseline for comparison with post-filtration flux.

As defined in Eqs. (3)–(4), pure-water flux depends on the effective pressure difference, water viscosity and intrinsic membrane hydraulic resistance.

The comparison between pure-water and nanoparticle-feed filtration should nevertheless be made carefully. Water viscosity, temperature, pressure and hydrodynamic conditions must be controlled or accounted for when comparing measurements.

The inclusion of pure-water testing in the present methodology therefore strengthens the experimental framework by establishing a reference state before exposure to the Al_2O_3 suspension.

METHODOLOGICAL IMPLICATIONS FOR NANOPARTICLE WATER TREATMENT

The separation of engineered nanoparticles from water presents challenges because nanoparticle behaviour depends on primary size, aggregation, surface chemistry and interactions with membrane materials. Membrane filtration can provide a practical separation route, but permeate flux describes hydraulic performance and does not by itself quantify nanoparticle rejection. Direct analysis of Al_2O_3 in feed, permeate and retentate would be required to establish removal efficiency.

Membrane filtration provides an attractive separation approach because it can be operated continuously and integrated with other water-treatment processes [29]. Nevertheless, the practical performance of membrane filtration depends on maintaining sufficient flux while controlling fouling and ensuring the required degree of retention.

The selected Al_2O_3 concentration range is therefore useful for examining feed-loading effects under controlled conditions. The three TMP values represent a moderate pressure range suitable for examining the relationship between hydraulic driving force and membrane resistance. The three PES MWCO values allow the role of nominal membrane selectivity to be considered.

The combined framework is consequently more informative than an investigation based on only one operating variable. It recognises that membrane productivity is determined by the interaction between the membrane, feed and hydraulic conditions.

LIMITATIONS OF THE PRESENT STUDY FRAMEWORK

The scientific interpretation of the study should recognise several methodological limitations.

First, the nominal particle size of approximately 30 nm represents the reported primary particle size rather than necessarily the hydrodynamic size of particles during filtration. Nanoparticle aggregation can alter the effective size distribution of the suspension. A complete physicochemical characterisation would therefore ideally include measurements such as dynamic light scattering, zeta potential and particle-size distribution under the actual feed conditions.

Second, nominal membrane MWCO should not be interpreted as a direct measurement of pore diameter. MWCO is influenced by the method used for its determination and by the reference solute. Recent literature has emphasised the complexity of relating MWCO to membrane pore structure and separation behaviour [15].

Third, a short filtration period provides information about initial and early-stage filtration behaviour but does not necessarily reproduce long-term membrane operation. Longer-term experiments would be required to distinguish transient concentration polarisation from sustained fouling development and to evaluate membrane cleaning requirements.

Fourth, permeate flux alone does not establish nanoparticle rejection. If the principal objective is to quantify nanoparticle removal, the concentration of Al_2O_3 in the permeate and retentate would need to be measured using an appropriate analytical technique. Flux describes hydraulic performance, whereas rejection describes separation performance. The two responses should therefore not be treated as interchangeable.

Fifth, a full fouling analysis would benefit from post-filtration pure-water flux measurements. Such measurements could help distinguish reversible changes in hydraulic resistance from more persistent fouling. Resistance-in-series analysis can provide additional information but should be interpreted in light of its underlying assumptions [8], [10].

These limitations do not invalidate the experimental framework. Rather, they define the scope of the conclusions that can reasonably be drawn from permeate-flux measurements.

PRACTICAL AND SCIENTIFIC RELEVANCE

The systematic investigation of nanoparticle concentration, TMP and membrane MWCO has relevance to membrane-based water treatment because each factor represents a controllable aspect of process design.

Feed concentration is important because membrane systems treating contaminated water may encounter variable particle loading. Understanding the relationship between loading and flux can assist in determining appropriate operating ranges and pretreatment requirements.

TMP is important because increasing pressure can increase productivity but may also influence concentration polarisation and fouling. An appropriate operating pressure should therefore be considered in relation to membrane resistance and feed characteristics rather than selected solely on the basis of maximum hydraulic driving force.

MWCO is important because membrane selection represents a fundamental design decision. A membrane that is excessively tight may provide greater retention but lower permeability, whereas a more open membrane may provide greater water productivity but different separation behaviour.

The combined consideration of these variables supports a more systematic approach to membrane process design. RSM can subsequently be used, when complete experimental observations are available, to quantify the relationships between the independent variables and the response.

Published research demonstrates that statistical optimisation can be valuable in membrane systems where several operating parameters interact [18], [19], [20],

[21]. Nevertheless, the resulting statistical model must always be derived from actual experimental observations. A response-surface model cannot legitimately be generated from literature values belonging to different membrane systems.

CONCLUSION

This methodological article defines a proposed experimental and analytical protocol for examining Al_2O_3 concentration, TMP and PES membrane MWCO in crossflow ultrafiltration. It does not report completed experiments or empirical findings. The contribution is the explicit treatment of unequally spaced MWCO levels as categorical, the prespecification of flux and rejection responses, and a set of testable interaction hypotheses. The protocol should be completed with verified equipment and material specifications before implementation.

Established membrane-filtration theory relates permeate flux to the effective pressure difference and total hydraulic resistance. In the absence of experimental observations, this manuscript does not demonstrate a factor effect in the Al_2O_3 /PES system. It proposes that any observed response will depend on the joint effects of TMP, feed loading and membrane properties, which must be tested using the protocol described above.

The use of pure-water flux before nanoparticle filtration provides an important reference for evaluating membrane hydraulic behaviour. The calculation of membrane resistance through Darcy's law provides a complementary theoretical basis for interpreting changes in permeability. Permeate collection at two-minute intervals over the filtration period further provides a means of examining changes in flux with time.

Published membrane-filtration literature identifies concentration polarisation, fouling, membrane structure and hydrodynamics as mechanisms that can influence flux. Their relative contributions in the proposed Al_2O_3 /PES system remain to be measured and should not be inferred from theory alone.

The methodological contribution is a protocol that distinguishes quantitative operating variables from the categorical MWCO factor, defines flux and rejection responses before analysis, and specifies the measurements needed to assess hydraulic performance and fouling. The proposed concentration-TMP ($A \times B$), concentration-MWCO ($A \times C$) and TMP-MWCO ($B \times C$) interactions are hypotheses, not findings, until supported by experimental data.

The proposed protocol may support laboratory assessment of membrane performance under controlled, elevated nanoparticle loadings relevant to selected industrial or polishing-treatment scenarios. Its relevance to natural waters cannot be claimed without evidence that the tested concentrations and suspension chemistry represent those waters.

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