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तनु कोलॉइडी निलंबनों के प्रक्रमण हेतु स्वदेशी स्पर्श-रेखीय प्रवाह निस्यंदन प्रणाली का विकास

मीनू प्राशर और कुलवंत सिंह
पदार्थ विज्ञान प्रभाग, भाभा परमाणु अनुसंधान केंद्र, ट्रांबे, मुंबई-400085, भारत

प्रयोगशाला-स्तर की स्पर्शरिखीय प्रवाह निस्यंदन (टीएफएफ) प्रणाली को भा.प.अ. केंद्र द्वारा स्व-विकसित किया गया

सारांश

कुशल अनुप्रवाह सांद्रण एवं तनु कोलॉइडी निलंबनों के शुद्धिकरण के लिए एक स्वदेशी, पूर्णत: स्वचालित स्पर्शरेखीय प्रवाह निस्यंदन (टीएफएफ) प्रणाली विकसित की गई । $0.1 m^2$ और पीएलसी/एचएमआई-आधारित स्वचालन के प्रभावी निस्यंदन क्षेत्र के साथ 10 kDa पॉलीइथरसल्फोन खोखला-तंतु झिल्ली मॉड्यूल से युक्त, प्लेटफॉर्म का उपयोग प्रयोगशाला संश्लेषित एल्युमिनोसिलिकेट नैनोपार्टिकल निलंबन के 4 L को संसाधित करने के लिए किया गया । 0.2 बार के ट्रांसमेम्ब्रेन दबाव पर, प्रणाली ने 500 mL तक आठ गुना आयतन की कमी हासिल की। 15 चक्रों में, बाद के डायाफिल्ट्रेशन ने अवशिष्ट Na^+ और Cl^- आयनों को हटा दिया, जैसा कि वास्तविक समय पारगम्य चालकता एवं एक्स-रे विवर्तन विश्लेषण में कमी से पुष्टि हुई है। ये परिणाम नैनो-पदार्थ प्रक्रम के लिए एक स्केलेबल और पर्यावरण के अनुकूल प्लेटफॉर्म को प्रदर्शित करते हैं।

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Development of an Indigenous Tangential Flow Filtration System for the Processing of Dilute Colloidal Suspensions

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Indigenously developed laboratory scale TFF system

ABSTRACT

An indigenous, fully automated tangential flow filtration (TFF) system was developed for the efficient downstream concentration and purification of dilute colloidal suspensions. Equipped with a 10 kDa polyethersulfone hollow-fibre membrane module with an effective filtration area of $0.1m^2$ and PLC/HMI-based automation, the platform was used to process 4L of a laboratory synthesized aluminosilicate nanoparticle suspension. At a transmembrane pressure of 0.2 bar, the system achieved an eight fold volume reduction to 500mL. Subsequent diafiltration over 15 cycles removed residual Na^+ and Cl^- ions, as confirmed by the decrease in real-time permeate conductivity and X-ray diffraction analysis. These results demonstrate a scalable and eco-friendly platform for nanomaterial processing.

KEYWORDS: *Tangential flow filtration, Colloidal suspension, Hollow-fibre*

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Introduction

Advanced inorganic nanomaterials play an increasingly important role in clean energy, environ mental remediation, catalysis, and biomedical technologies [1–3]. Many of these materials are synthesized as dilute colloidal suspensions, making efficient concentration and purification essential for obtaining high-quality products and enabling their large-scale utilization. To address this technological challenge, we designed an indigenous Tangential Flow Filtration (TFF) system for the fully automated post-synthesis processing of dilute colloidal suspensions. The integrated platform combines concentration and constant-volume diafiltration with automated process monitoring and control, providing an efficient and scalable processing.

Unlike conventional purification techniques such as sedimentation, dead-end filtration, and dialysis, TFF employs continuous tangential flow across the membrane surface, minimizing membrane fouling while enabling rapid removal of dissolved impurities and concentration of the product. TFF is well established in biopharmaceutical processing [4,5] and protein purification [6]. By extending it to advanced inorganic nanomaterials [7], the developed TFF setup provides a completely indigenous energy-efficient, eco-friendly, and resource-conserving separation strategy for dilute inorganic colloidal suspensions with high processing rates, reduced water consumption, and chemical-free operation, aligned with India's vision for sustainable materials development.

In this work, we demonstrate the performance of the indigenously designed, fully automated TFF system using a laboratory-synthesized colloidal suspension of aluminosilicate nanoparticles. The system achieved rapid volumetric concentration, effective removal of residual synthesis salts (Na^+ and Cl^-), and recovery of high-purity nanoparticles, demonstrating the its potential as a scalable downstream processing for advanced nanomaterials.

Experimental Set-Up

An indigenous integrated Tangential Flow Filtration system was designed and commissioned for the concentration and purification of dilute colloidal suspensions. The major components of the set-up are:

●**Feed/Retentate Reservoir:** A 5L stainless-steel reservoir serves as the unified feed and retentate vessel for continuous suspension recirculation. It is directly mounted on a precision digital load cell (±1 g resolution) to obtain real-time mass measurements and volumetric feedback during the concentration and diafiltration.

●**Recirculation & Buffer Pumps:** The suspension is circulated through the main feed line using a variable-speed peristaltic pump fitted with low-shear silicone tubing. A secondary peristaltic pump is also integrated into the fluid circuit to add buffer to the retentate vessel during diafiltration cycles.

●**Membrane Module:** The membrane-based separation is achieved using a polyethersulfone (PES) hollow-fibre membrane module with a molecular-weight cut-off (MWCO) of 10 kDa. The module has a total effective filtration area of 0.1m2.

●**Pressure Control & Temperature Monitoring:** The operating transmembrane pressure (TMP) is regulated using a stainless-steel needle backpressure valve installed downstream of the retentate outlet. The permeate line is kept completely open ($P_{permeate}$ = 0 bar) to allow atmospheric discharge during filtration. An inline temperature indicator is installed on the retentate return line to monitor fluid heating during continuous recirculation.

●**Pressure Transducers:** Digital piezoresistive pressure sensors are positioned at the feed inlet (P_{Feed}) and retentate outlet ($P_{Retentate}$) to continuously monitor hydraulic pressure and calculate TMP according to the equation below:

$$TMP = \frac{(P_{feed} + P_{Retentate})}{2} - P_{permeate} \quad \text{.............................(1)}$$

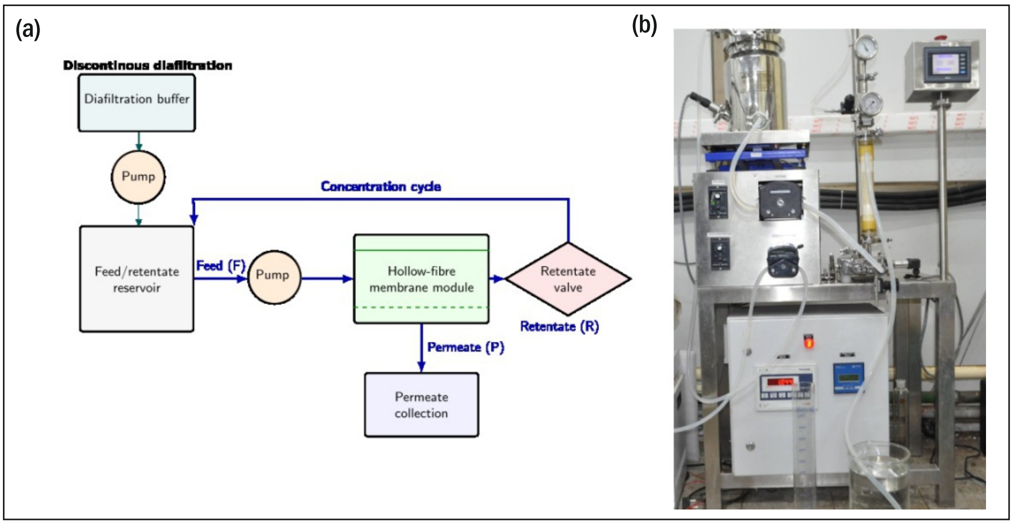

Fig.1: (a) Process-flow schematic of the TFF system showing the feed/retentate reservoir, recirculation pump, hollow-fibre membrane module, retentate cycle, permeate collection, and buffer addition for diafiltration, (b) Photograph of the indigenously developed laboratory scale TFF system with its feed/retentate reservoir, pumps, membrane module, pressure monitoring instruments, and HMI-based control panel.

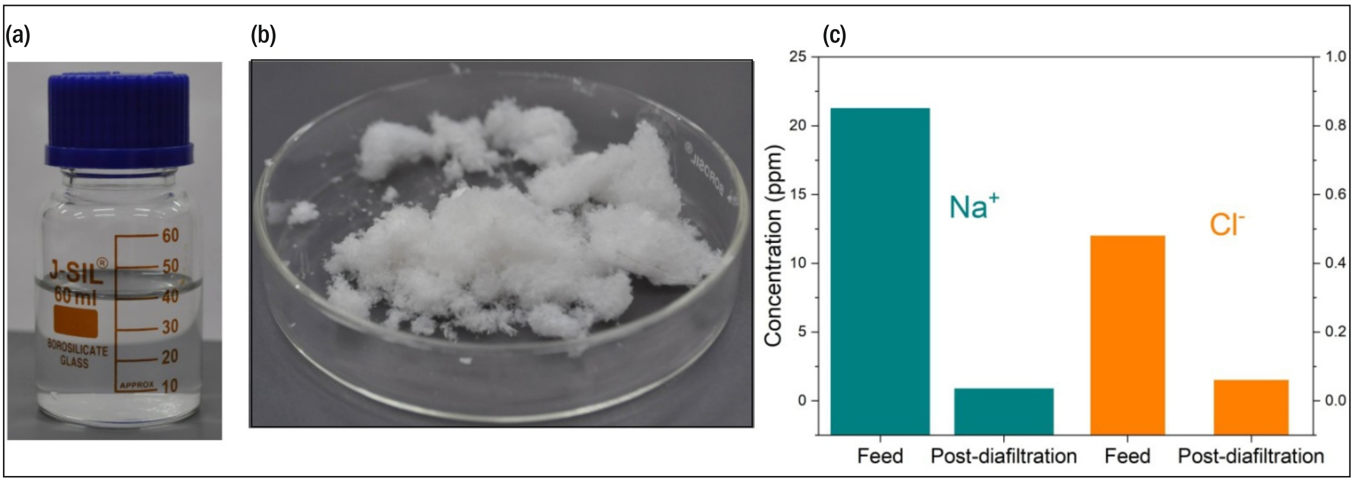

Fig.2: (a) As-synthesized aluminosilicate colloidal suspension (b) white, fluffy powder obtained after freeze-drying the diafiltered suspension. (c) Concentrations of residual Na^+ and Cl^- in the initial feed and the post-diafiltration retentate, demonstrating substantial removalof soluble ionic impurities.

●**Inline Analytical Probes:** A conductivity cell is inserted directly into the feed tank to continuously track ionic strength and impurity washout in real time.

●**HMI/PLC Control System:** A programmable logic controller (PLC) interfaced with a touch screen human–machine interface (HMI) automates recirculation pumpcontrol, discontinuous diafiltration buffer addition, conductivity monitoring, and high-pressure safety alarm.

Fig.1(a) depicts the schematic flow paths and operating modes of our TFF assembly, while Fig.1(b) shows the actual system equipped with a 10 kDa hollow-fibre membrane module. During concentration, the feed pump circulates colloidal suspension from the feed/retentate reservoir and delivers it through the feed line (F) to the membrane module. At a given transmembrane pressure (TMP), the the solvent and low-molecular-weight impurities are forced through the membrane pores into the permeate (P) stream, while the retained nanoparticles pass through the retentate (R) line and recirculated to the reservoir. In the set-up, diafiltration is operated in a discontinuous mode. During the diafiltration (purification) stage, buffer pump adds a volume of fresh buffer equal to the retentate volume (V), to make total volume 2V. The feed pump then concentrates the volume to remove permeate until the suspension returned to its baseline volume V, thereby completing one diafiltration cycle (1 DV). This sequence is repeated in an auto mode for N cycles, till the desired purification level is achieved.

Experimental Results

To demonstrate the purification efficiency of the developed TFF system, an inorganic aluminosilicate colloidal suspension synthesized in laboratory and used as the model feed. The as-synthesized opalescent suspension (pH = 3.0), hereafter referred to as the Feed, (shown in Fig.2(a)), is a stable colloidal suspension with zeta potential of +42 mV. During TFF process, the feed volume of 4 L is first concentrated to 500 mL (volume concentration factor = 8) and then 15 discontinuous diafiltration cycles were performed at TMP = 0.2 bar. This concentrated and diafiltred retentate upon freeze-drying yields

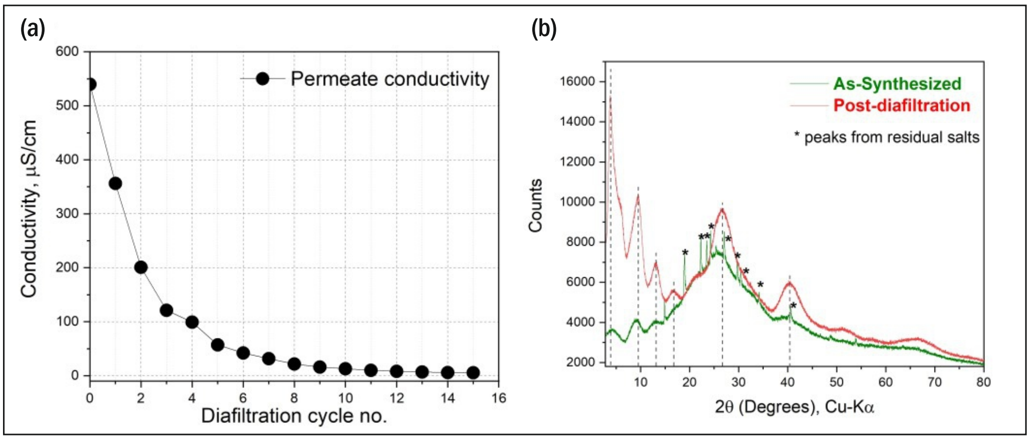

Fig.3: . (a) Variation in permeateconductivity with diafiltration cycle number, showing the progressive removal of dissolved ionic species, (b) Powder XRD patterns of the as-synthesized and post-diafiltration retentate (powder samples obtained after freeze-drying the suspensions). The disappearance of sharp reflections associated with residual salt phases (indicated by asterisk (*)) in the post-diafiltered sample indicates high purity final product.

final white, fluffy, cotton-like material (Fig.2(b)), with mass con centration of nearly 1 gL^{-1}. Fig.2(c) compares the residual Na^+ and Cl^- concentrations in the initial feed and final retentate, demonstrating near-complete removal of these ionic species. As shown in Fig.3(a), the rapid decrease in the permeate conductivity up to first 8 diafiltration cycles and subsequent saturation, confirming the rapid removal of soluble ionic species. Fig.3(b) shows the comparison between the powder X-ray diffraction (XRD) patterns of freeze-dried samples obtained from the concentrated feed before diafiltration and from the final retentate after 15 diafiltration cycles. Disappearance of the sharp reflections associated with residual salt phases after diafiltration and enhanced broad reflections from the purified product validates the successful purification step.

Conclusion

An indigenous, fully automated Tangential Flow Filtration system was successfully designed, developed and commissioned for the scalable concentration and purification of dilute colloidal sus pensions. During batch processing of an aluminosilicate nanoparticle suspension, the feed volume was successfully concentrated eightfold from 4 L to 500 mL, with near-complete removal of residual Na^+ and Cl^- ions over 15 diafiltration cycles. The PLC/HMI-based control system enabled precise, real-time monitoring of process hydraulics and impurity washout. Through this work, a robust and modular indigenous platform for the high-purity downstream processing of advanced functional nanomaterials at larger operational scales is established.

Acknowledgment

We gratefully acknowledge the constant support and guidance of the Head, Materials Science Division, and the Associate Director, Materials Group, BARC. We thank the Analytical Chemistry Division, BARC for carrying out the ICP-OES & ion-chromatography measurements and the Chemistry Division, BARC for zeta potential measurements. Meenu Prasher also acknowledges Mr. Samir Momin for his technical assistance with the maintenance and operation of the set-up.

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