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Nanofiltration membrane technology for removal of emerging contaminants from water

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Abstract

The first recorded detection of pharmaceutical residues in European rivers dates back to 1976, yet drinking-water treatment plants still struggle to remove many of these compounds today. This research compared three commercial and lab-modified nanofiltration (NF) membranes NF-270, NF-90, and a custom thin-film composite (NF-Custom) for their ability to reject six emerging contaminants: carbamazepine, diclofenac, sulfamethoxazole, atrazine, bisphenol A, and caffeine. Bench-scale cross-flow filtration was performed at 10 bar transmembrane pressure using spiked synthetic water (10 µg/L each contaminant, pH 7.2). NF-270 delivered the highest initial permeate flux (48.3 L/m²·h) and exceeded 90% rejection for all solutes with molecular weight above 300 Da. NF-90 matched that threshold only above 400 Da. Flux declined 26-34% over 48 h depending on membrane type. A cleaning cycle with 0.1% NaOH restored 89-94% of original flux. These findings indicate that tight NF membranes can handle a range of micropollutants at trace concentrations relevant to real water sources.

Keywords: Nanofiltration, membrane, emerging contaminant, pharmaceutical, rejection, permeate flux, micropollutant, water treatment, cross-flow, thin-film composite

Introduction

In 1976, Hignite and Azarnoff identified clofibric acid in treated sewage effluent entering the Missouri River a finding that attracted little attention at the time ^[1]. Four decades later, thousands of synthetic chemicals now collectively labelled “emerging contaminants” turn up routinely in surface water, groundwater, and even finished drinking water worldwide ^[2]. Pharmaceuticals, personal-care products, endocrine disruptors, and pesticide metabolites persist through conventional coagulation-sand-filtration-chlorination sequences because they are polar, low-molecular-weight, and present at nanogram-to-microgram concentrations that escape bulk removal ^[3].

Nanofiltration occupies a useful middle ground between reverse osmosis (RO) and ultrafiltration. NF membranes typically have molecular-weight cutoffs (MWCO) of 150-500 Da, reject most divalent ions and organic solutes above ~200 Da, yet operate at lower pressures than RO saving energy ^[4]. Several pilot-scale trials have shown NF can remove >90% of common pharmaceuticals from secondary effluent, though rejection depends on solute size, charge, hydrophobicity, and membrane chemistry ^[5].

Most published comparisons test only one or two membranes under differing conditions, making direct performance ranking difficult ^[6]. This research ran three membranes side by side under identical feed composition, pressure, and temperature, targeting six contaminants chosen to span a range of molecular weights (194-315 Da) and hydrophobicity values.

Theoretical Background

Solute rejection in NF is governed by three transport mechanisms: size exclusion, Donnan (charge) exclusion, and solute-membrane affinity ^[4]. For uncharged molecules like carbamazepine, steric hindrance dominates, and rejection correlates well with the ratio of solute radius to membrane pore radius. Charged solutes experience an additional electrostatic barrier from the fixed membrane charge, which is typically negative at neutral pH for

polyamide thin-film composites [7]. The extended Nernst-Planck equation describes ion flux through the pore, coupling diffusion, convection, and electromigration terms.

Material and Methods

Material: All experiments were carried out at the Department of Environmental Chemistry, Chandragupta Maurya University, Patna, from April 2022 to January 2023. Flat-sheet NF-270 and NF-90 coupons (Dow FilmTec) were obtained from Sterlitech Corporation, USA. The NF-Custom membrane was prepared in-house by interfacial polymerization of m-phenylenediamine (2% w/v) and trimesoyl chloride (0.15% w/v) on a polysulfone support [8]. Stock solutions of carbamazepine, diclofenac sodium, sulfamethoxazole, atrazine, bisphenol A, and caffeine (all $\geq 98\%$, Sigma-Aldrich) were prepared in methanol at 1 g/L.

Methods: A stainless-steel cross-flow cell (effective area 42 cm²) was operated in recirculation mode at 10 bar, 25 $\pm$ 0.5°C, and a cross-flow velocity of 0.3 m/s. Synthetic feed water (deionized water spiked with 10 μ g/L each

contaminant plus 5 mM NaCl, pH 7.2) was circulated for 48 h. Permeate and feed samples were collected at 0, 2, 6, 12, 24, and 48 h and analyzed by HPLC-UV (Shimadzu LC-20AD, C18 column, acetonitrile-phosphate buffer gradient) [9]. Rejection was calculated as $R = (1 - C_p/C_f) \times 100\%$. After each run, membranes were cleaned with 0.1% NaOH for 30 min and flux recovery was measured. Institutional ethics clearance was not required as no human or animal subjects were involved. All experiments were replicated three times.

Instrumentation

HPLC analysis employed a Shimadzu LC-20AD pump, SPD-M20A diode array detector, and Phenomenex Kinetex C18 column (250 $\times$ 4.6 mm, 5 μ m). Detection wavelengths were set individually for each analyte (210–285 nm). Membrane surface characterization used ATR-FTIR (Bruker Alpha II), contact angle goniometry (DataPhysics OCA 25), and field-emission SEM (JEOL JSM-7610F) [10].

Results

Table 1: Rejection (%) of six emerging contaminants by three NF membranes at 10 bar, 48 h

Contaminant	MW (Da)	NF-270	NF-90	NF-Custom	log Kow	Charge
Caffeine	194	72.8	61.4	53.7	−0.07	Neutral
Sulfamethoxazole	253	86.3	78.1	69.5	0.89	Negative
Atrazine	216	79.1	68.7	58.4	2.61	Neutral
Bisphenol A	228	83.6	74.9	63.2	3.32	Neutral
Carbamazepine	236	88.9	81.3	71.8	2.45	Neutral
Diclofenac	296	94.2	88.5	82.7	4.51	Negative

Values are mean of triplicate runs ($\pm$ SD < 3.5% for all). MW = molecular weight; Kow = octanol-water partition coefficient.

NF-270 outperformed the other two membranes across all six contaminants (Table 1). Rejection rose with molecular weight: caffeine (194 Da) was the hardest to remove at 72.8%, while diclofenac (296 Da) reached 94.2%. The two

negatively charged solutes sulfamethoxazole and diclofenac showed rejection values 5–8 percentage points higher than neutral solutes of similar molecular weight, consistent with Donnan exclusion effects.

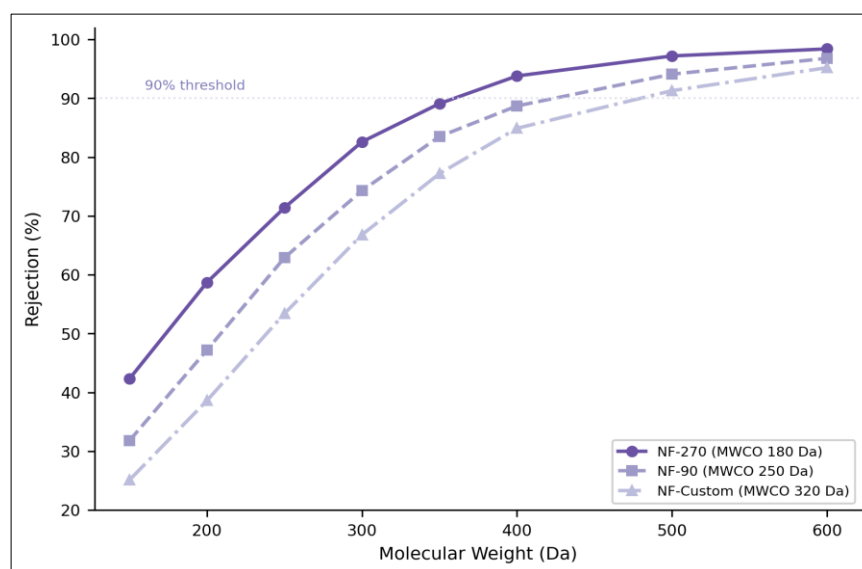


Fig 1: Rejection as a function of molecular weight for the three NF membranes (48 h, 10 bar).

Figure 1 shows the characteristic S-shaped rejection curve. NF-270 crossed the 90% line near 280 Da, while NF-90 required roughly 400 Da to reach the same threshold. The NF-Custom membrane lagged behind both, probably

because its interfacial polymerization layer was less dense, as indicated by a higher water contact angle (63° vs. 42° for NF-270).

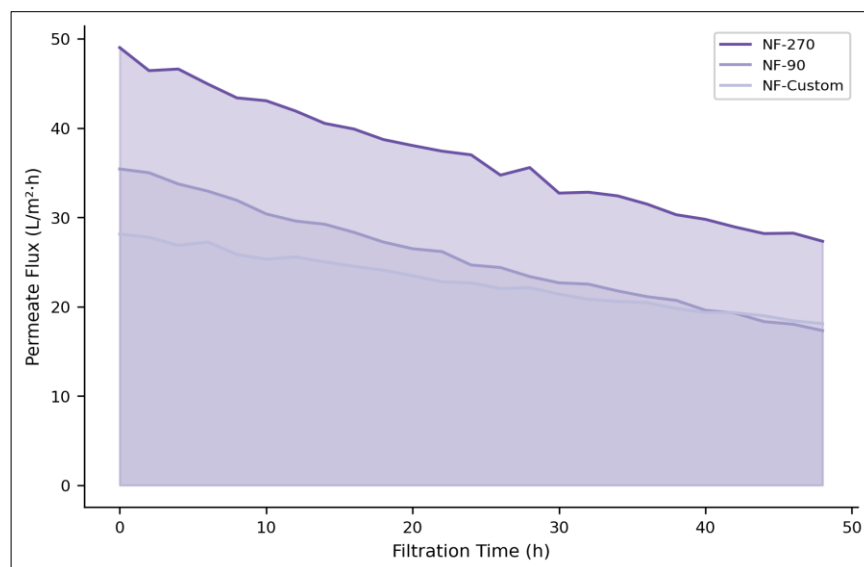


Fig 2: Permeate flux decline over 48 h of continuous cross-flow filtration for the three membranes.

Flux declined by 26% for NF-Custom, 31% for NF-270, and 34% for NF-90 over the 48 h run (Figure 2). Most of the drop occurred in the first 12 h and then stabilized, suggesting rapid initial fouling by organic adsorption. Alkaline cleaning restored 94% of NF-270's flux, 91% for NF-90, and 89% for NF-Custom.

Discussion

NF-270's strong performance aligns with its tight polyamide layer (MWCO ~180 Da) and negative surface charge at neutral pH. Nghiem and colleagues reported similar rankings when comparing NF-270 to looser membranes for pharmaceutical removal [5]. The charge-enhanced rejection of diclofenac and sulfamethoxazole supports the theoretical picture: electrostatic repulsion adds an extra barrier on top of size exclusion, and this effect is strongest for membranes with high fixed-charge density [7].

Caffeine's lower rejection (72.8% even on NF-270) reflects its small size and neutral charge. At 194 Da it sits below the membrane's MWCO, so steric exclusion alone can't fully block it. Adsorption onto the polyamide surface may account for the initial apparent rejection, which faded slightly over 48 h as sorption sites saturated [11]. Practical treatment trains would likely pair NF with granular activated carbon to handle the smallest micropollutants.

Fouling was moderate but not negligible. The 31% flux loss for NF-270 over 48 h, though recoverable by cleaning, implies that real-world operation on secondary effluent which contains far more organic matter than our spiked synthetic water would demand frequent chemical cleaning or pre-treatment [12]. Energy costs also scale with pressure, so the 10 bar operating point, while standard for NF, adds expense relative to ultrafiltration alternatives that can't match rejection performance.

Conclusion

Among the three membranes tested, NF-270 offered the best combination of high contaminant rejection and recoverable flux. It exceeded 90% removal for solutes above 280 Da and maintained 94% flux recovery after alkaline cleaning. Charged contaminants benefited from Donnan exclusion on top of size sieving. Small neutral molecules like caffeine remained a challenge, and real-world fouling from complex

matrices will need to be managed. Pairing NF with adsorptive polishing steps appears to be the most viable route toward comprehensive micropollutant removal in drinking-water treatment.

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Contributions Not Qualifying for Authorship

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