



CuO nanoparticles as surface additives for membranes in anaerobic digestate treatment applications

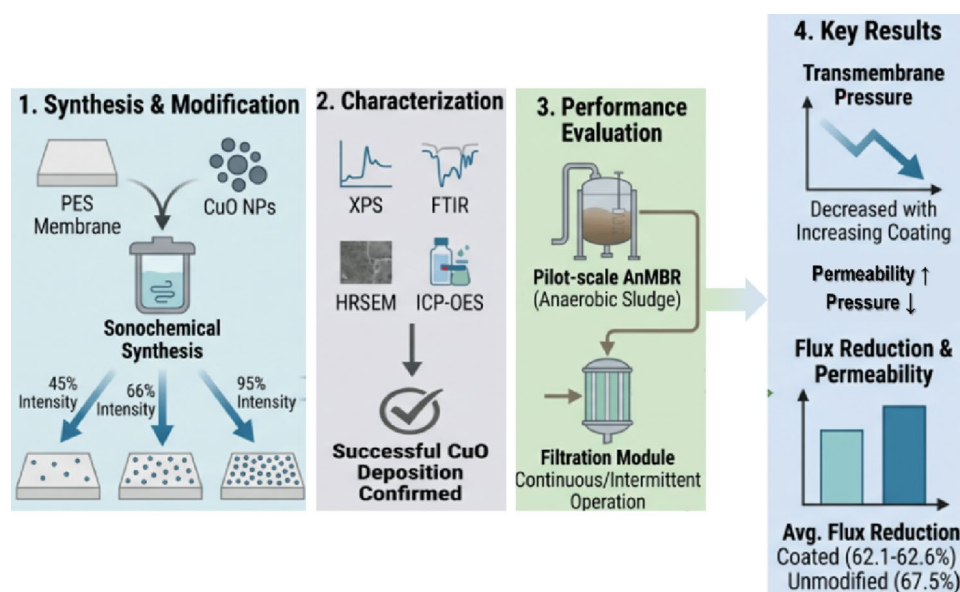
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Abstract

Application of CuO nanoparticles (NPs) deposited via sonochemical synthesis onto polyethersulfone (PES) ultrafiltration membranes for fouling mitigation in anaerobic digestate treatment was investigated. Modified membranes were prepared at three sonication intensities (45%, 66% and 95%) and evaluated alongside with unmodified samples using anaerobic digestate from a pilot-scale AnMBR under continuous and intermittent operation with reverse flow cleaning. Surface characterization was performed through XPS, FTIR, HRSEM and ICP-OES. The CuO NPs coating on the PES membrane was 0.11%, 0.65% and 0.90% w/w at sonication intensities of 45%, 66% and 95%. Under constant input flux conditions, transmembrane pressure decreased with increasing coating intensity, indicating progressive reduction in hydraulic resistance. Coated membranes exhibited moderately lower average flux reduction (62.1–62.6%) compared to the unmodified membrane (67.5%), though differences were within experimental uncertainty. When membrane performance was evaluated based on pressure-normalized permeability, the 66% and 95% coated membranes demonstrated 18.5% and 20.8% increased permeability compared to the unmodified membrane. These findings support CuO surface modification at moderate sonication intensity as an efficient antifouling strategy for anaerobic membrane digestion systems.

Graphical abstract



Keywords Ultrafiltration membranes · Anaerobic digestion · Anaerobic membrane digestion · CuO nanoparticles

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Abbreviations

NPs	Nanoparticles
PES	Polyethersulfone
COD	Chemical oxygen demand
PVDF	Poly(vinylidene fluoride)
TMP	Transmembrane pressure

Introduction

Global acceleration of organic waste generation including food residues, livestock manure and waste-activated sludge, has become a critical concern for environmental and public health practices. Conventional disposal treatments, including landfilling and incineration are major contributors to greenhouse gas emissions and resource depletion. In order to achieve sustainable carbon neutrality, waste management strategies are oriented towards resource recovery and circular economy (Awasthi et al. 2023; Zeng et al. 2025).

Anaerobic digestion (AD) is a promising technology, converting complex organic matter under oxygen-free conditions into valuable biochemical products (Edifor et al. 2020; Liu et al. 2019). Effective treatment or utilization of digestate plays a key role in the overall sustainability of the AD process (Skaggs et al. 2018). However, traditional separation technologies such as adsorption, distillation and solvent extraction often display limited selectivity and high energy consumption (Park et al. 2017; Reungoat et al. 2023). Membrane-based anaerobic digestion technologies have emerged as promising alternatives due to their scalability, versatility and reduced environmental footprint (Rongwong et al. 2018).

Membrane separation is increasingly employed for digestate treatment, however performance of filtration systems is often affected by fouling and flux decline. Polyether sulfone (PES) membranes are widely used for wastewater treatment due to their mechanical stability, chemical resistance and enhanced filtration abilities (Zheng et al. 2024). However, the hydrophobic nature of PES membranes often leads to membrane fouling by organic contaminants, which reduces the separation efficiency and stability. Surface modification with metal oxides into PES membranes addresses issues of hydrophobicity and fouling by organic pollutants, significantly enhancing the operational efficiency, as well as the longterm performance for anaerobic digestion applications (Maximous et al. 2010).

Recent studies have shown that incorporation of metal oxide nanoparticles (NPs) such as Al_2O_3 , ZrO_2 , and TiO_2 into PES membranes enhances membrane hydrophilicity, leading to improved water flux, while in several cases it also contributes to improved mechanical stability, depending on filler dispersion and loading conditions (Gao et al. 2025;

Li et al. 2025; Nhlengethwa et al. 2024; Zhu et al. 2019). Incorporation of CuO NPs can prove a promising antifouling approach as well, attributed to improvements in both surface hydrophilicity and antimicrobial properties. In one such application, a ceramic membrane anaerobic bioreactor equipped with CuO NPs-modified membranes demonstrated 93% COD removal efficiency alongside with decreased transmembrane pressure buildup relatively to the unmodified membrane (Qiu et al. 2023). Another research involving PVDF ultrafiltration membranes incorporating CuO in combination with graphene oxide has demonstrated improved flux recovery ratios, diminished irreversible fouling and effective antibacterial performance (Zhao et al. 2017). In addition, integration of morphologically engineered CuO nanomaterials in PVDF/PVP membranes displayed flux recovery exceeding 90% during application, alongside with enhanced hydrophilicity and miniscule release of Cu^{2+} into the permeate (Pakan et al. 2023).

The deposition method selected for introducing metal particle coating onto a membrane surface determines both the particle distribution and their adhesion to the substrate. Physical blending method disperses nanoparticles through the bulk of the polymer matrix, which can promote particle aggregation and the formation of non-selective interfacial voids that compromise mechanical properties (Arundhati et al. 2024), while dip-coating relies on physical adsorption and tends to produce coatings that are patchy and weakly attached to the membrane surface (Khorshidi et al. 2018). Sonochemical deposition instead generates the CuO nanoparticles in situ, at the membrane surface under ultrasonic cavitation, promoting closer contact between the nanoparticles and the membrane surface without modifying the bulk polymer (Deokar et al. 2021).

Building on the aforementioned reports, the present work explores the application of CuO NPs coated PES membranes as a method for fouling control and improved separation in anaerobic membrane bioreactor systems. Through membrane performance evaluation with digestate samples from a pilot plant reactor, the findings of this work aim to facilitate the adoption of surface-modified membranes in full scale anaerobic wastewater treatment infrastructures.

Materials and methods

NADIR[®] UP150 T Polyethersulfone (PES) ultrafiltration membranes (MANN+HUMMEL GmbH, Germany), with a nominal pore size of 40 nm and a molecular weight cutoff (MWCO) of 150 kDa, were used in this study. The CuO NPs coating process required Copper (II) acetate monohydrate (ACS reagent, $\geq 98\%$) and Ammonium hydroxide

Table 1 Main sludge characteristics of interest for the filtration process

Total Suspended Solids (g/L)	Volatile Suspended Solids (g/L)	pH
3.0	1.6	7.2

solution (ACS reagent, 28.0–30.0%), both obtained from Sigma-Aldrich.

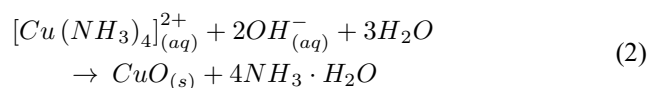
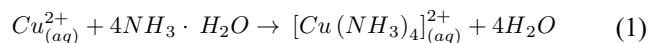
Greek EcoSite - Anaerobic digester

This research was conducted within the framework of the Horizon Europe SYMSITES project, which aims to advance Industrial-Urban (I-U) symbiosis by transforming waste streams and by-products into value-added materials with reduced environmental impact. Throughout its implementation the project has explored and validated a range of technologies and approaches with stakeholder involvement at four designated EcoSites spread across Europe. These sites span across a broad geographic and socioeconomic spectrum, from Scandinavia (Denmark) and central Europe (Austria) to the Mediterranean (Spain and Greece). At the EcoSite establishment in Greece, a 5.5m³ anaerobic membrane bioreactor (AnMBR) served as the primary treatment unit, enabling the recovery of energy (methane), irrigation water and compost. The anaerobic sludge utilized to assess the performance of the modified membrane was collected from the AnMBR operating at the Greek EcoSite. Its main characteristics are displayed in Table 1.

CuO nanoparticle deposition

Membrane functionalization with NPs was achieved via a sonochemical deposition process. Initially, copper(II) acetate was introduced into the sonochemical reactor (Fig. 1), followed by ultrasonic treatment for 1 h while maintaining the solution temperature at 60 °C. Afterward, ammonium hydroxide was added to adjust the pH to 8.5, promoting the

in situ formation of CuO nanoparticles through the reactions presented in Eqs. (1) and (2).



During synthesis, the membrane was continuously passed through the suspension to facilitate nanoparticle immobilization onto its surface under ultrasonic irradiation. The influence of sonotrode intensity was examined at 45.0, 66.0, and 95.0%. A schematic representation of the overall modification procedure is provided in Fig. 2.

Membrane characterization

The elemental analysis was carried out by X-ray photoelectron spectroscopy with the XPS, Nexsasprometer (England). The functional groups of CuO coated PES membranes were examined by FTIR spectrometer (Tensor 27 spectrometer (Bruker, Germany)) in the range of 4000–400 cm^{−1}. To analyse the morphologies of CuO/PES membrane, HRSEM (Quanta FEG 250, Thermo Fisher Scientific) was employed with various magnifications. To identify the concentration of CuO particles on the PES membranes, Inductively Coupled Plasma (ICP) was employed by SPECTRO ARCOS ICP- OES Multi view FHX22. For sample preparation, 1.5 × 1.5 cm² CuO-coated PES membranes were submerged in diluted nitric acid (HNO₃) and heated at 50 °C for 30 min. The resulting copper-containing solutions were then diluted to a final volume of 50 mL and analyzed using ICP-OES.

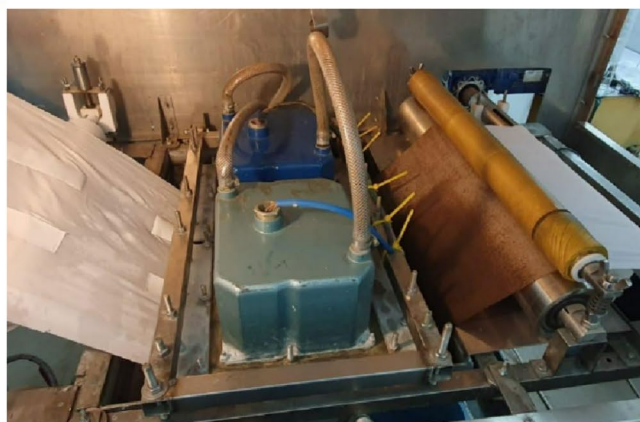
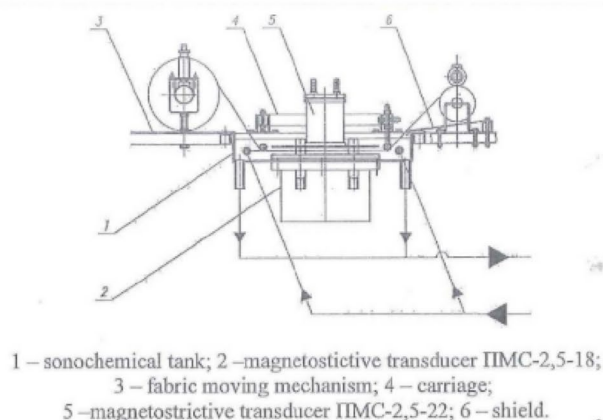


Fig. 1 Sonochemical tank, reagent tank and rolling mechanism used for membrane modification

Fig. 2 Schematic representation of membrane modification process with CuO nanoparticles

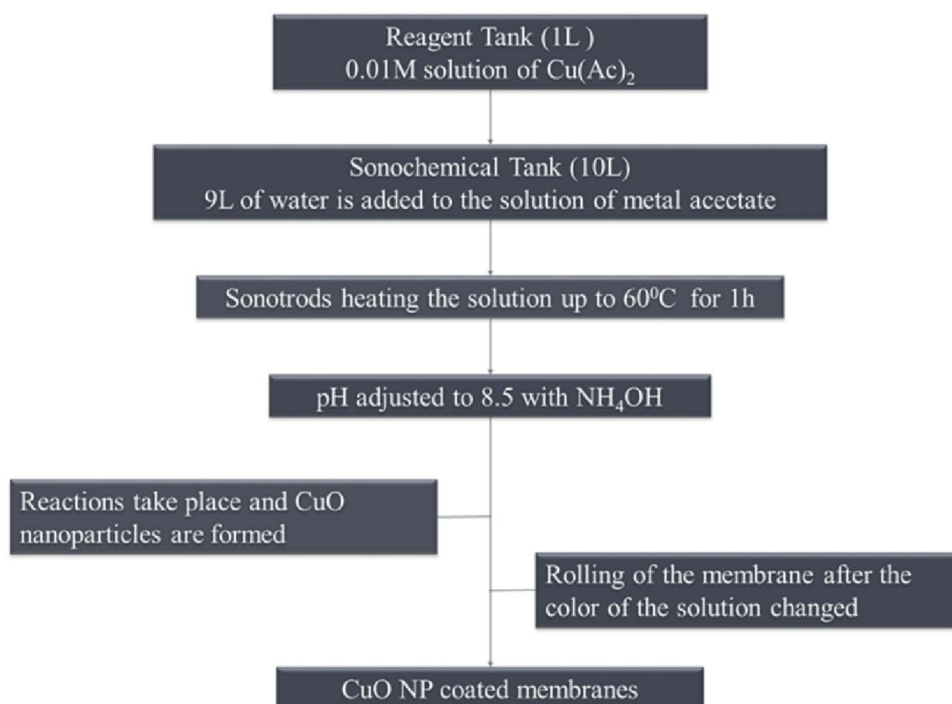


Fig. 3 Experimental setup, 1: Digestate, 2: Magnetic Stirrer, 3: Inlet & Outlet pumps, 4: Lab35 Membrane Module, 5: Manometer, 6: Permeate

Performance of modified membranes with AD sludge

The performance of both non-modified and modified membranes was investigated under continuous and intermittent operating conditions in order to assess permeate flux behavior and the effectiveness of reverse-flow cleaning. Each experiment consisted of 4 h of uninterrupted operation followed by a 4 h cycle incorporating reverse-flow cleaning at 60 min intervals. For consistency, identical permeate volumes were applied during each cleaning step. Throughout all tests, the anaerobic digestate feed flux and inlet/outlet

pump rotational speeds remained constant. Permeate flow and transmembrane pressure (TMP) evolution were continuously recorded. Experimental trials were conducted using a Lab35 membrane module supplied by SANI Membranes. and the experimental setup is presented in Fig. 3.

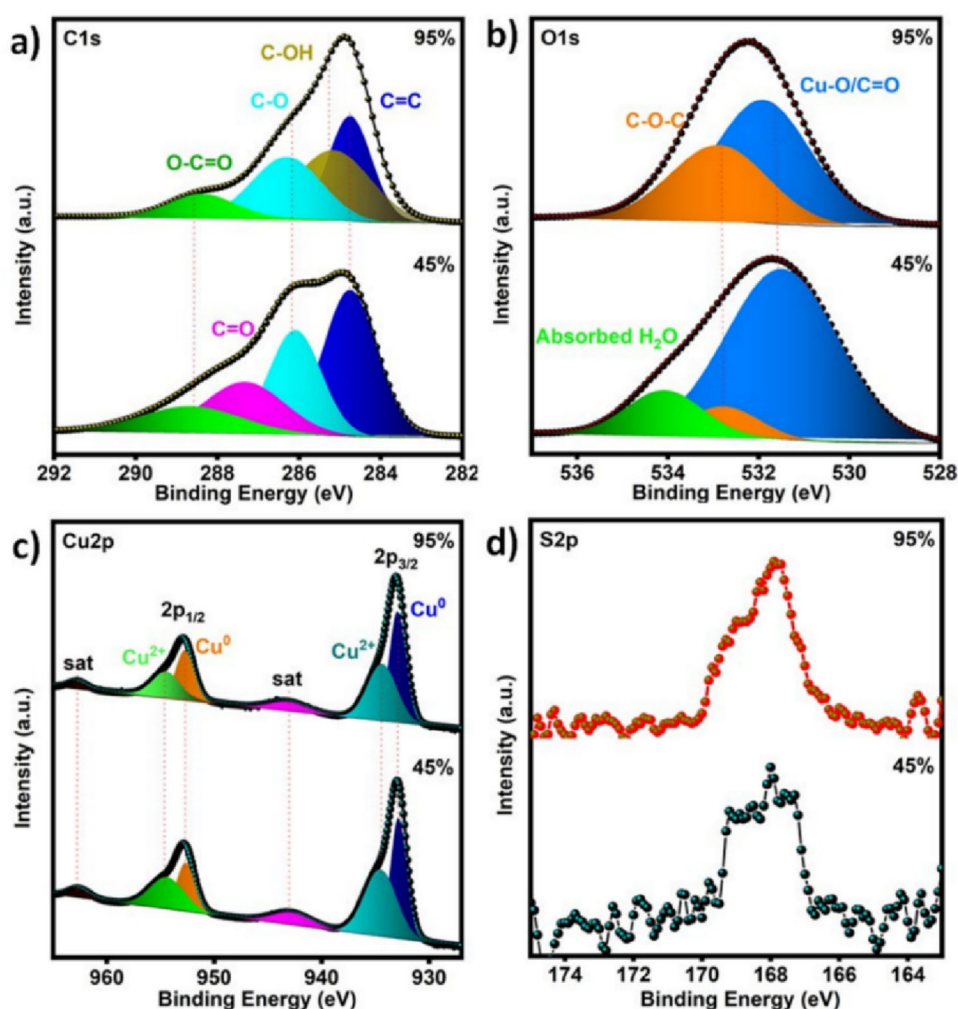
Results and discussion

Characterization of modified membranes

The surface modification of the CuO nanoparticles on the PES membrane was supported by ICP-OES analysis. Inductively Coupled Plasma Optical Emission Spectrometry (ICP-OES) was used to quantify the concentration of copper ions loading from CuO-coated PES membranes. The amount of copper deposited on the PES membrane was quantified as 0.11%, 0.65%, and 0.90% (w/w) at sonication intensities of 45%, 66%, and 95%, respectively. The copper loading on the PES membrane increased with increasing sonication intensity.

XPS analysis of CuO/PES membrane determines the surface properties, elemental composition and presence of functional groups including carbon, oxygen, copper and sulphur and the results are shown in Fig. 4. The CuO/PES-45 and CuO/PES-95 samples contain the prominent peaks C1s, O1s, Cu2p and S2p around 284 eV, 532 eV, 935 eV and 168 eV respectively. High resolution C1s spectra of CuO/PES-45 and CuO/PES-95 depicted in Fig. 4a. The C1s deconvoluted spectra of CuO/PES-45 exhibit the four C1s

Fig. 4 XPS of CuO/PES-45 and CuO/PES-95 shows the presence of C, O, Cu and S. (a) C 1s, (b) O 1s, (c) Cu2p and (d) S2p XPS spectra with respective deconvoluted profiles



peaks at 284.7 eV, 286.1 eV, 287.3 eV, and 288.7 eV corresponds to the C=C, C-O, C=O, O-C=O functional groups. The C1s deconvoluted spectra of CuO/PES-95 exhibit the four C1s peaks at 284.7 eV, 285.2 eV, 286.3 eV, and 288.4 eV corresponds to the C=C, C-OH, C-O, O-C=O functional groups. Due to the accumulation of CuO NPs and the intensity of ultrasonication, the reduction in C1s observed in CuO/PES-95 (Al-Ghafri et al. 2022). In Fig. 4b, the O1s high resolution spectra of CuO/PES-45 is divided into three peaks of Cu-O/C=O, C-O-C and adsorbed H₂O at 531.5 eV, 532.7 eV and 534.1 eV respectively. In the case of CuO/PES-95 membrane showed two O1s peaks at 531.9 eV (C=O), and 532.9 eV (C-O-C). Deconvoluted Cu2p spectra of CuO/PES-45 and CuO/PES-95 is depicted in Figure 4c. Cu2p spectra shown two distinct peaks for Cu 2p_{3/2} and Cu 2p_{1/2} at 935 eV and 952.2 eV accompanied with two satellite peaks at 943, and 965 eV. The Cu 2p_{3/2} XPS region deconvoluted into two peaks at 932.8 eV and 934.6 assigned to the Cu⁰ and Cu²⁺ oxidation states. The Cu 2p_{1/2} XPS spectrum deconvoluted into two peaks at 952.6 eV and 954.5 assigned to the Cu⁰ and Cu²⁺ oxidation states (Xu et

al. 2023). High resolution S2p spectrum in Fig. 4d reveals that peak at 168 eV indicating the presence of S-O functional group in the CuO/PES membrane.

The HRSEM images of the CuO-coated membranes and uncoated membranes are shown in Fig. 5. The original (uncoated) PES membrane exhibits a smooth and uniform surface at various magnifications (Fig. 5a, b). In the CuO/PES-45 sample (Fig. 5c, d), CuO particles are deposited on the PES membrane with a predominantly spherical morphology, exhibiting good homogeneity and a uniform distribution. The particle diameters range from approximately 400 to 600 nm, consistent with secondary aggregates formed from smaller, sonochemically nucleated CuO nanocrystals rather than discrete nanoparticles. Figure 5e, and f present the HRSEM images of the In CuO/PES-95 membrane, where the CuO particles maintain a spherical and isolated nature but appear closely bound on the membrane surface. Additionally, copper oxide flakes with sizes less than 100 nm - within the conventional nanoparticle size range - were observed on the PES membrane, likely formed due to the influence of high-intensity ultrasonic treatment

Fig. 5 HRSEM images of (a, b) Pristine PES, (c, d) PES/CuO-45 and (e, f) PES/CuO-95 at different magnifications

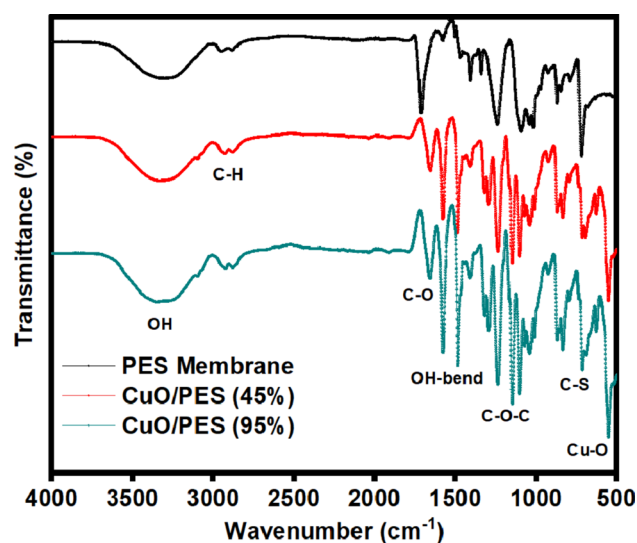
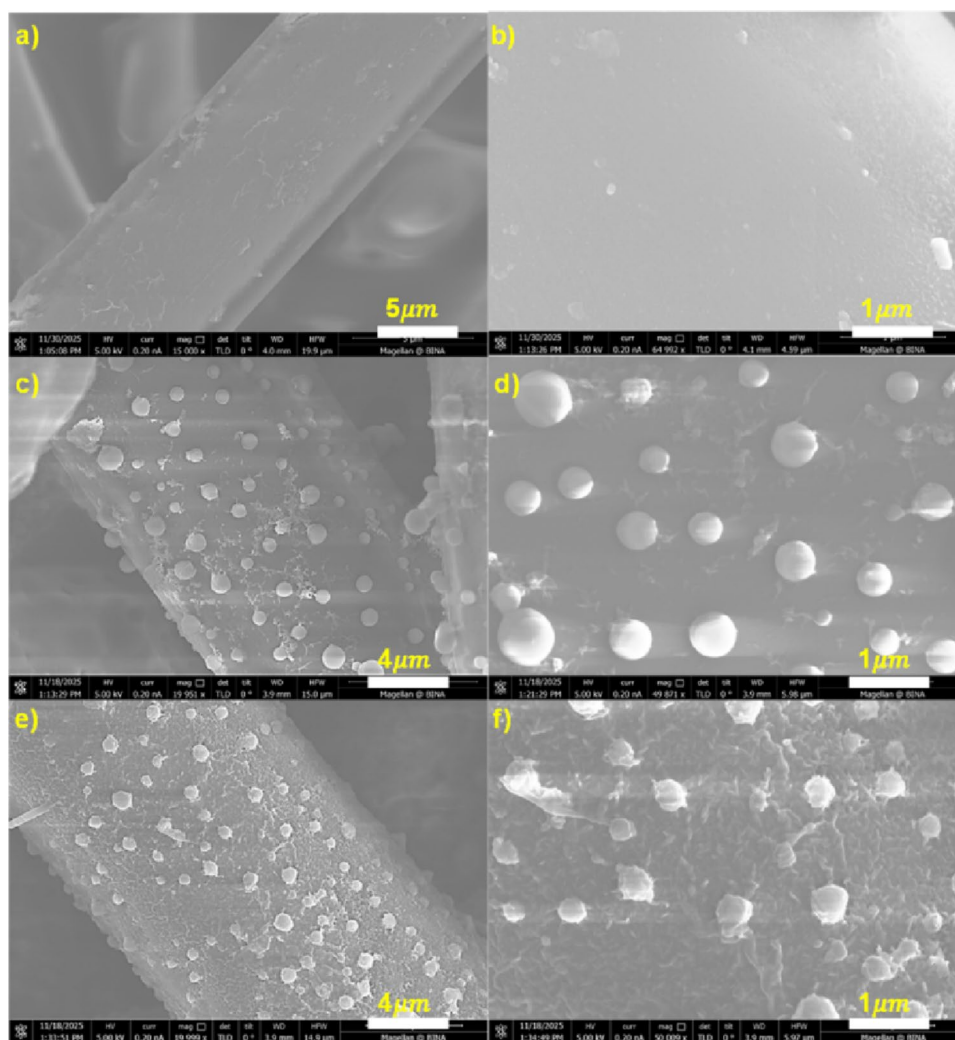


Fig. 6 FTIR images of the pristine and CuO coated PES membrane

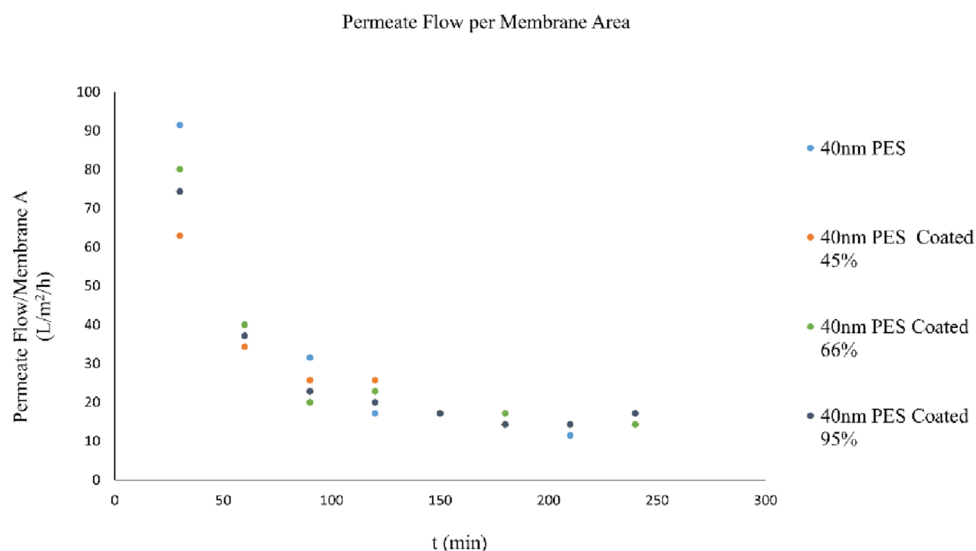
during deposition (Kajau et al. 2021). The presence of both spherical particles and sub-100 nm nanoparticle flakes indicates diverse CuO morphologies on the membrane surface, spanning nanoscale features to larger micron-range aggregates. The particle size range for the spherical aggregates in both sample types remains between 400 and 600 nm.

The FTIR spectra of CuO/PES exhibited typical Cu-O and PES functional groups depicted in Fig. 6. The peaks at 3317 cm^{-1} and 1577 cm^{-1} corresponds to the stretching OH and plane bending OH functional group. The peaks at 1660 cm^{-1} and 1147 cm^{-1} assigned to the stretching vibrations of C=O and C-O-C groups respectively. The peaks at 1320 cm^{-1} and 711 cm^{-1} corresponds to the presence of O=S=O and C-S functional groups respectively. Compared to PES membrane, a new peak is occurring in CuO/PES-45 and CuO/PES-95 spectra at 533 cm^{-1} which is due to the presence of CuO nanoparticles on the PES membrane. These observations are consistent with an interaction between CuO nanoparticles and PES functional groups; confirming the precise nature of this interaction (e.g. coordination

Fig. 7 NADIR® UP150 T membrane samples **a**: non-modified, **b**: coated 45% intensity, **c**: coated 95% intensity, **d**: coated 66% intensity (default)



Fig. 8 Evolution of permeate flux ($\text{L}/\text{m}^2 \cdot \text{h}$) for non-modified and modified membrane samples under continuous filtration



bonding) would require complementary techniques such as XPS binding-energy shifts or DFT-supported vibrational analysis (Al-Ghafri et al. 2022; Mobarak et al. 2022).

The ultrasonically-driven formation of CuO from the copper-ammine complex (Eqs. 1–2) nucleates nanoparticles that are immobilized onto the PES surface under cavitation, producing both physical surface coverage (seen by HRSEM) and a measurable surface interaction (the new 533 cm^{-1} FTIR band). The increasing Cu loading with sonication intensity (ICP-OES) is consistent with more extensive nanoparticle immobilization at higher cavitation energy.

Performance of modified membranes with AD sludge

Pictures of: the original NADIR® UP150 T (a) and the 3 modified samples at 45 (b), 95 (c) and 66% (d) sonotrod intensity are presented in Fig. 7. Samples from each membrane sheet were cut appropriately to fit the Lab35 Membrane Module and activated with DI water / isopropanol rinse before use.

Continuous operation

During continuous operation, the non-modified PES membrane exhibited the highest initial permeate flux of $91.4 \text{ L}/\text{m}^2/\text{h}$, while the CuO-coated membranes showed lower initial values of approximately 62.9 , 80.0 and $74.3 \text{ L}/\text{m}^2/\text{h}$ for 45%, 66% and 95% sonication intensities respectively (Fig. 8). The reduction in initial flux for coated membranes is consistent with the increased hydraulic resistance resulting from CuO nanoparticle deposition on the membrane surface, which partially occludes the pore structure, an effect also reported for other metal oxide-modified PES membranes (Maximous et al. 2010; Pakan et al. 2023). Despite its higher starting flux, the non-modified membrane exhibited a steep decline within the first 60 min of operation, losing 56.3% of its initial flux, while operating at higher Transmembrane Pressure (TMP, 1 bar) in comparison to coated membranes (0.6–0.8 bar). These observations are indicative of rapid pore blocking and cake layer formation by the matter present in the anaerobic sludge. In contrast, the coated membranes displayed a more gradual and stable flux decline over the same period. After approximately

Fig. 9 Cumulative permeate flow per membrane area (L/m^2) for non-modified and modified membrane samples under intermittent filtration with hourly cleaning cycles (vertical, dotted)

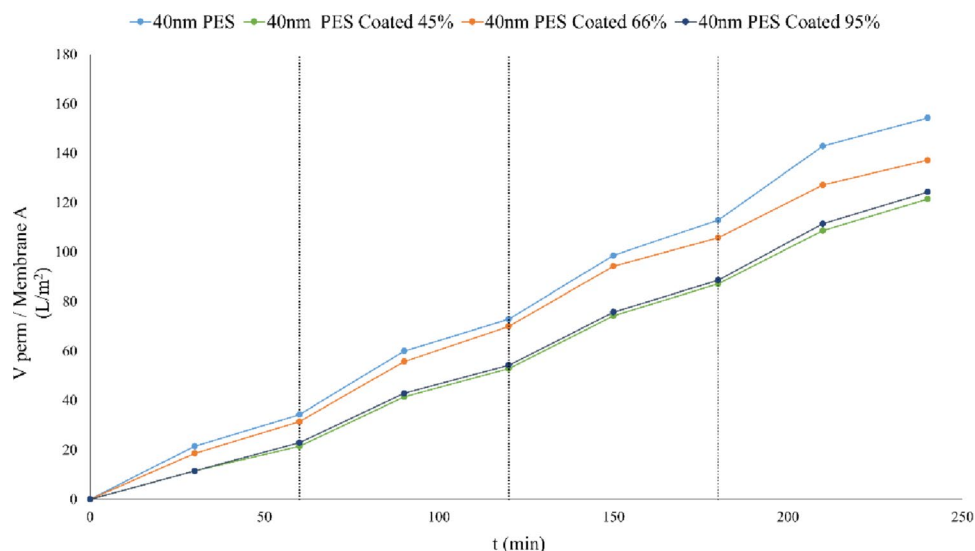


Table 2 Average flux reduction and recovery after cleaning, cumulative permeate volume/area, Transmembrane Pressure (TMP), and Permeability for each membrane tested during intermittent operation

	AVG Flux Recovery after cleaning (%)	AVG Flux Reduction (%)	Cumulative Permeate Volume/Area (L/m^2)	TMP (bar)	Permeability ($\text{L}/\text{m}^2/\text{h}/\text{bar}$)
40 nm PES non-coated	68.7 ± 5.1	67.5 ± 2.6	154.3	1.2	32.1
40 nm PES coated 45%	66.5 ± 2.1	62.6 ± 3.8	121.4	1.1	27.6
40 nm PES coated 66%	71.8 ± 4.1	62.6 ± 4.9	137.1	0.9	38.1
40 nm PES coated 95%	67.0 ± 3.6	62.1 ± 2.2	124.3	0.8	38.8

150 min of operation however, all membrane samples converged toward a similar steady-state flux range of 14–17 $\text{L}/\text{m}^2/\text{h}$, suggesting that at this stage fouling layer development dominates the membrane resistance, regardless of surface modification. This behavior is consistent with the transition from pore blocking to cake filtration mechanisms, observed in long-term UF membrane operation with anaerobic sludge (Le-Clech et al. 2006; Meng et al. 2009).

Intermittent operation

The cumulative permeate volume per unit membrane area during intermittent operation with reverse flow cleaning every 60 min is presented in Fig. 9. The dotted vertical lines indicate the cleaning events at 60, 120 and 180 min. During the first operational interval (0–60 min), the non-modified PES membrane accumulated the highest cumulative volume, reaching 34.3 L/m^2 , while the coated membranes produced lower cumulative volumes of 21.4, 31.4 and 21.4 L/m^2 for 45%, 66% and 95% intensities respectively. By the end of the 240-minute experiment, the non-modified membrane achieved the highest total cumulative volume of 154.3 L/m^2 , followed by the 66% coated membrane at 137.1 L/m^2 , while the 45% and 95% coated membranes reached similar final values of 121.4 and 124.3 L/m^2 respectively.

Operation under a stable input flow, resulted in varying transmembrane pressure (TMP) between the different membrane samples tested. Table 2 presents Average flux reduction and recovery after cleaning (%), cumulative permeate volume/area (L/m^2), Transmembrane Pressure (TMP - bar), and Permeability ($\text{L}/\text{m}^2/\text{h}/\text{bar}$) for each membrane tested during intermittent operation. All CuO-coated membranes exhibited moderately lower average flux reduction compared to the non-coated PES membrane ($62.1\text{--}62.6\%$ versus $67.5 \pm 2.6\%$), indicating a consistent improvement in fouling resistance upon surface modification. However, given the overlapping standard deviations across all samples, these differences cannot be considered statistically conclusive. Similarly, flux recovery after cleaning ranged from $66.5 \pm 2.1\%$ to $71.8 \pm 4.1\%$ across all membranes, with the 66% coated sample appearing to perform best. However, the substantial overlap between confidence intervals - particularly between the non-coated ($68.7 \pm 5.1\%$) and coated 66% ($71.8 \pm 4.1\%$) samples - prevents a definitive conclusion regarding the superiority of any specific coating intensity on the basis of flux recovery alone.

A substantial differentiation in membrane sample performance emerges when permeability - normalised for transmembrane pressure - is taken into consideration. The non-coated membrane, while achieving the highest cumulative permeate volume (154.3 L/m^2), operated at the

highest TMP of 1.2 bar, yielding a permeability of 32.1 L/m²/h/bar. In contrast, the 66% and 95% coated membranes achieved permeability values of 38.1 and 38.8 L/m²/h/bar respectively, an increase of 18.5 and 20.8% over the non-coated membrane, while operating at a reduced TMP of 0.9 and 0.8 bar. This decrease in TMP with increasing coating intensity can be an indicator of the progressive reduction in hydraulic resistance associated with the increased CuO surface coverage.

The progressive increase in Cu loading with sonication intensity (Sect. 3.1) parallels the progressive decrease in TMP and increase in permeability observed for the 66% and 95% coated membranes (Sect. 3.2), supporting a coating-density-dependent reduction in hydraulic resistance. It is also noted that the 45% coated membrane, despite having a measurable CuO loading, does not show a proportional performance improvement. This is discussed as a threshold effect below which the coating is too sparse to meaningfully reduce fouling.

When the cumulative permeate volume is contextualized against operating pressure, the apparent advantage of the non-coated membrane is diminished, as its higher throughput is achieved at the cost of higher applied pressure compared to the 66% and 95% coated membranes, which can be further translated into higher energy demand. The 66% coated membrane produces 137.1 L/m² at 0.9 bar versus 154.3 L/m² at 1.2 bar for the non-coated, offering a more energy-efficient operating point with comparable output. Overall, permeability provides a clearer basis for comparison, on which CuO-modified membranes at 66% and 95% intensity demonstrate superior efficiency, supporting CuO surface modification as an energy-efficient antifouling strategy for anaerobic digestate treatment.

Conclusions

CuO nanoparticles were successfully deposited onto PES ultrafiltration membranes via sonochemical synthesis, with ICP-OES analysis showing that the Copper coating density increased from 0.11%w/w at 45% sonication intensity to 0.90%w/w at 95% intensity. XPS, FTIR and HRSEM characterization confirmed the presence and distribution of CuO on the membrane surface. CuO-coated membranes exhibited moderately reduced average flux decline compared to the unmodified membrane (62.1–62.6% versus 67.5%). However, given the overlapping standard deviations, these differences were not statistically conclusive. Under constant input flux operation, transmembrane pressure decreased with increasing sonication intensity, suggesting that CuO NPs presence progressively reduced membrane hydraulic

resistance. Permeability provided a more solid comparison basis, as the 66% and 95% coated membranes achieved 38.1 and 38.8 L/m²/h/bar respectively, which correspond to 18.5% and 20.8% increase compared to the unmodified membrane (32.1 L/m²/h/bar), while the latter's higher cumulative permeate volume (154.3 L/m²) was attributed to its elevated TMP (1.2 bar). Further studies under longer operational duration and Cu loading quantification are recommended to fully characterize the long-term performance and environmental safety of CuO-modified membranes in anaerobic membrane digestion.

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Author contributions K.P.: Investigation, Conceptualization, Methodology, Writing – original draft, Writing – review & editing, Project administration, Supervision. C.P.: Methodology, Validation, Data Curation, Writing – review & editing. D.R.: Investigation, Formal analysis, Writing – original draft. I.P.: Methodology, Conceptualization, Writing – review & editing, Supervision. P.M.: Investigation, Validation. D.A.: Methodology, Conceptualization, Writing – review & editing and G.L.: Supervision, Methodology, Conceptualization, Writing – review & editing.

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Data availability The data presented in this study are available on request from the corresponding author.

Declarations

Conflict of interest On behalf of all authors, the corresponding author states that there is no conflict of interest.

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