

## CONSUMER PROTECTION AND THE LONGEVITY OF NANOMATERIALS BLENDED ULTRAFILTRATION MEMBRANES FOR CIRCULAR WATER SYSTEMS

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### **Abstract**

*The development of closed loop water system technology, which is treated water is continuously recycled within a defined system boundary, requires advanced treatment methods guaranteeing high quality treated water. Polymeric ultrafiltration membranes are widely used for water recovery. Nevertheless, their lifespan is often restricted by fouling. In this paper, nanomaterials blended ultrafiltration membranes incorporating nano sized zinc oxide (ZnO NP) and multi walled carbon nanotubes (MWCNT) were comparatively examined regarding their suitability for closed loop water systems from performance and consumer protection points of view.*

*The incorporation of 1 wt% ZnO NP increased flux from 54 to 58 L m<sup>-2</sup> h<sup>-1</sup>, and solute rejection from 81% to 86%. However, it did not significantly affect the porosity and hydraulic resistance. On the other hand, the addition of 1 wt% MWCNT resulted in an increase in flux from 29 to 107 L m<sup>-2</sup> h<sup>-1</sup>, and improved rejection from 45% to 80%. The hydraulic resistance decreased from  $13 \times 10^{12}$  to  $11 \times 10^{12}$  m<sup>-1</sup>. These results suggest performance enhancement by improving pore structure with ZnO NP, and by pore connectivity with MWCNT. From the perspective of consumer protection, both nanomaterial systems show improved properties which can lead to better efficiency and reliability in terms of wastewater reuse process. Especially reduced hydraulic resistance of the MWCNT modified membrane may offer additional operational advantages.*

**Key words:** MWCNT, ZnO NP, ultrafiltration, consumer protection, nanomaterials, water treatment

### **Introduction**

Membranes are extensively used in water and wastewater treatment as well as industries such as food, petrochemical, and energy (Daufin et al., 2001, Ravanchi et al., 2009, Wang et al., 2011, Li et al., 2011, Lee et al., 2014). Membrane technologies are low cost and more energy efficient than other water treatment techniques (Nasrollahi et al., 2018, Aziz et al., 2024).

Ultrafiltration can be considered as a porous membrane with a pore size of typically ranging from 0.001 to 0.1 µm (Mulder, 1997). Ultrafiltration membranes often have an anisotropic structure, having a dense layer on top of a microporous layer. The separation process is carried out in the dense layer, and mechanical strength is provided by the

microporous layer (Baker, 2004). Small, suspended particles and dissolved macromolecules pass through the ultrafiltration membrane, while larger molecules are mainly rejected (Idris et al. 2006).

Polymeric membranes are flexible, tough, economical, and have good separation properties, but their mechanical, thermal, and chemical resistance are low (Peng et al., 2007, Mollahosseini et al., 2025). In addition, fouling problem is one of the most significant disadvantages of membrane technology (Nguyen et al., 2012, Balta et al., 2012). The reduction in the separation performance of a membrane is referred to as membrane fouling. When a membrane becomes fouled, the amount of permeate obtained decreases (Koyuncu, 2018). Colloids, particles, macromolecules, salts, and similar substances accumulate within the membrane pores and/or on the membrane surface, causing fouling (Geise et al., 2010). Membrane fouling is divided into reversible and irreversible fouling. Reversible fouling can be removed by hydraulic cleaning methods. However, irreversible fouling is eliminated by cleaning the membrane with chemical solutions. The use of chemicals shortens membrane lifetime (Meng et al., 2009).

Studies have shown that increasing membrane hydrophilicity reduces membrane fouling by decreasing hydrophobic interactions between foulants and the membrane surface (Gohari et al., 2014, Damodar et al., 2009, Leo et al., 2012). Therefore, in recent years, the production of low cost polymeric membranes with high fouling resistance have attracted significant attention. There are three main methods to increase membrane hydrophilicity. These methods are blending the polymer with another hydrophilic material, grafting the polymer with hydrophilic monomers or functional groups, and coating the membrane with a hydrophilic material (Celik Madenli et al., 2017).

Nanoparticles such as aluminium oxides ( $\gamma$ -Al<sub>2</sub>O<sub>3</sub>,  $\alpha$ -Al<sub>2</sub>O<sub>3</sub>), zirconium oxide (ZrO<sub>2</sub>), titanium oxide (TiO<sub>2</sub>), cerium oxide (CeO<sub>2</sub>), silicon dioxide (glass) (SiO<sub>2</sub>), and zinc oxide (ZnO), are widely used as additives for membrane synthesis (Aksu, 2019, Koyuncu, 2018). It was shown in literature that TiO<sub>2</sub> nanoparticles are very effective for fouling control (Rahimpour et al., 2011, Bae et al., 2006). However, ZnO NP is an excellent alternative to TiO<sub>2</sub> nanoparticles for fouling control due their high surface to volume ratio and low cost. It was shown that ZnO NP addition increases membrane hydrophilicity and permeate flux (Ahmad et al., 2018, Celik Madenli et al., 2023). Multi walled carbon nanotubes (MWCNT) are also attractive nanomaterial with exceptionally high aspect ratio, strength and stiffness (Gojny et al., 2004). It was shown that CNT blended membranes are more hydrophilic, with a higher flux (Celik et al., 2011a, Celik et al., 2011b).

The use of nanomaterials for membrane preparation has significant implications for consumer protection and environmental sustainability. The challenges include nanoparticle release into treated water, stability of nanoparticle blended membranes. The treated water is reused for domestic and industrial purposes in circular systems. Hence ensuring consumer protection and regulatory compliance is as important as achieving high technical performance. Therefore, this study aims to assess the performance, longevity, and consumer protection effects for the MWCNT, and ZnO NP blended membranes.

## Membrane Preparation and Characterisation

Membranes were synthesized based on the nanoparticle doped membrane synthesis method through phase inversion method as described in the literature (Celik et al 2011a, Celik et al. 2011b, Celik and Choi 2011, Celik Madenli and Ciftci 2022, Celik Madenli et al. 2023). In membrane synthesis, ZnO NP or MWCNT were used as additives, and N-methyl-2-

pyrrolidone (NMP) or N,N-Dimethylformamide (DMF) were used as solvents. Before use MWCNT were functionalized in an acid mixture, as described elsewhere (Celik et al. 2011a, Celik et al. 2011b, Celik Madenli et al. 2017, Celik Madenli et al. 2021, Celik Madenli and Cakmakci 2017, Celik Madenli and Ciftci 2022, Celik Madenli and Kesiktas, 2023). During the synthesis, nanoparticles were first added to the solvent. Then, polyethersulfone was added to obtain the membrane solution. The obtained membrane solution was poured onto a glass plate using an automatic film applicator. Membranes were formed by placing the glass plate into a coagulation bath. The prepared membranes were washed with and stored in distilled water until use. Compositions of the membranes prepared are presented in Table 1.

**Table 1. Composition of membranes**

| <i>Membranes</i>    | <i>Polyethersulfone<br/>Ratio</i> | <i>nZnO<br/>Ratio</i> | <i>CNT<br/>Ratio</i> | <i>NMP<br/>Ratio</i> | <i>DMF<br/>Ratio</i> |
|---------------------|-----------------------------------|-----------------------|----------------------|----------------------|----------------------|
| <i>0 wt% ZnO NP</i> | 15                                | 0                     | 0                    | 85                   | 0                    |
| <i>1 wt% ZnO NP</i> | 15                                | 1                     | 0                    | 85                   | 0                    |
| <i>0 wt% MWCNT</i>  | 15                                | 0                     | 0                    | 8.5                  | 76.5                 |
| <i>1 wt% MWCNT</i>  | 15                                | 0                     | 1                    | 8.5                  | 76.5                 |

The porosity ( $\varepsilon$ ) of the membranes was determined by measuring the dry ( $w_d$ , g) and wet weights ( $w_w$ , g) of the membranes by using the following equation.

$$\varepsilon = (w_w - w_d) / (\rho \times A \times h).$$

where  $A$  represents the membrane area ( $\text{cm}^2$ ),  $h$  represents the membrane thickness (cm), and  $\rho$  represents the density of pure water. For each membrane, the mean and standard deviation were determined based on at least two independent measurements

Contact angle measurement was performed using the sessile drop technique by using a contact angle goniometer. A drop of pure water (1  $\mu\text{L}$ ) was placed on the membrane surface, and a measurement was taken. To minimise experimental error, at least five replicate measurements were conducted for each sample, and the results are reported as the mean and standard deviation.

Membrane filtration tests were conducted by using a crossflow membrane filtration unit for ZnO NP blended membranes and dead-end membrane filtration unit for MWCNT blended membranes. 0.5 bar of constant transmembrane pressure was applied during permeability and selectivity tests. The water flux ( $J_w$ ,  $\text{L}/\text{m}^2\text{h}$ ) was determined using the following equation.

$$J_w = V / (A \times \Delta t)$$

where  $V$  represents the volume of the permeate (L), and  $\Delta t$  represents time (h).

One of the typical pollutants found in water treatment is protein-like substances that cause serious fouling during membrane filtration (Liu et al. 2013, Ding et al. 2019, Huner et al 2017, Vanangarnudi et al. 2017). In this study, bovine serum albumin was used as a model protein. Bovine serum albumin filtration for 1 hour was performed to examine the selectivity and fouling resistance. Bovine serum albumin rejection ratio ( $R$ , %) and hydraulic resistance of the membranes ( $R_m$ ,  $\text{m}^{-1}$ ) were determined by using the following equations.

$$R = (C_f - C_p) / C_f$$

$$R_m = \Delta P / (\mu \times J_p)$$

where  $\mu$  represents water viscosity,  $\Delta P$  represents transmembrane pressure (Pa), and  $J_p$  represents the protein filtration flux ( $\text{L}/\text{m}^2\text{h}$ ).

## Results and Discussion

### *Nanomaterial Incorporation Effects on Membrane Structure*

The addition of nanomaterials in ultrafiltration membranes had significant effects on the structure and properties of the membranes, reflected by changes in porosity, contact angle, bovine serum albumin rejection, and hydraulic resistance, as shown in Table 2. These effects are strongly dependent on the type of nanomaterial used. Hence, additive selection is very critical when designing membranes for circular water systems.

**Table 2. Properties of the membranes**

| <i>Membranes</i>    | $\varepsilon$ | <i>Contact Angle (°)</i> | $R_m (m^{-1})$      |
|---------------------|---------------|--------------------------|---------------------|
| <i>0 wt% ZnO NP</i> | 0.43          | 66                       | $17 \times 10^8$    |
| <i>1 wt% ZnO NP</i> | 0.43          | 64                       | $17 \times 10^8$    |
| <i>0 wt% MWCNT</i>  | 0.25          | 67                       | $13 \times 10^{12}$ |
| <i>1 wt% MWCNT</i>  | 0.29          | 62                       | $11 \times 10^{12}$ |

For the ZnO NP blended membranes, addition of 1 wt% ZnO NP led to a decrease in contact angle from 66° to 64°. Reduced contact angle shows increased hydrophilicity. This increase in hydrophilicity is likely due to the hydrophilic nature of ZnO NP, which promotes faster solvent and nonsolvent exchange during phase inversion and may contribute to formation of a more interconnected pore structure. An increase in hydraulic resistance indicates an increase in fouling. The unchanged hydraulic resistance suggests ZnO NP addition in blended membranes did not significantly alter the fouling behaviour.

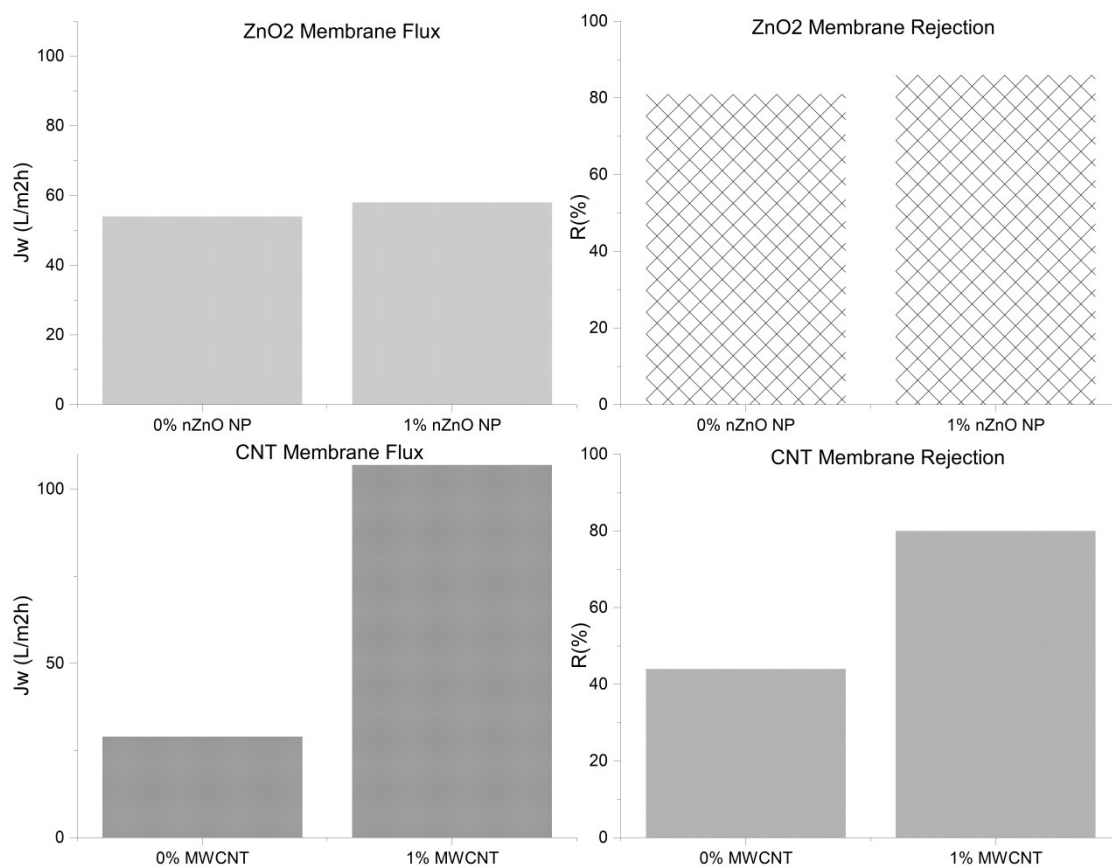
For the MWCNT blended membranes, addition of 1 wt% MWCNT resulted in an increase in membrane porosity from 0.27 to 0.32. The reduction in hydraulic resistance from  $13 \times 10^{12}$  to  $11 \times 10^{12} \text{ m}^{-1}$  suggests increasing fouling resistance of the MWCNT blended membranes. In addition, contact angle reduced from 66° to 64° shows increased hydrophilicity.

### *Permeability and Rejection Performance*

While permeability enhancements are desired to reduce energy consumption in circular water systems, sufficient solute rejection is crucial in terms of consumer protection. In ZnO NP blended membranes, 1 wt% ZnO NP addition increased the rejection from 81% to 86%, together with an increase in flux from 54 to 58 L/m<sup>2</sup>h (Figure 1). Such simultaneous improvement suggests that ZnO NP may have enhanced pore size distribution uniformity and/or surface interactions with the rejected solute, thus offsetting the effect of increased porosity.

A different trend was observed for the MWCNT blended membranes. The flux increased, from 29 to 107 L/m<sup>2</sup>h. This considerable improvement in permeability was accompanied by a significant improvement in rejection, which increased from 45% to 80% with 1 wt% MWCNT addition. This implies that MWCNT incorporation not only enhanced hydraulic performance but also contributed to a more controlled pore structure, probably because of reduction in macrovoids or reinforcement of the polymer matrix. Improvement in rejection with increased permeability points out the ability of MWCNT to favorably overcome the commonly confronted permeability-selectivity trade-off in polymeric membranes.

When high flux and high removal are achieved simultaneously during membrane filtration, concentration polarization occurs due to the accumulation of rejected solutes near the membrane surface. Therefore, controlling concentration polarization is important in ultrafiltration processes (Celik and Choi 2011, Vankelecom et al., 2005).



**Figure 1. Flux and rejection of the nanomaterials blended membranes**

#### ***Membrane Longevity and Operational Stability***

Membrane longevity is closely related to hydraulic resistance, structural integrity, and fouling tendency. A decrease in the hydraulic resistance upon the addition of MWCNT indicates improved water transport characteristics during pressure driven operation. Lower hydraulic resistance also points toward a reduction of energy consumption and lessened mechanical stress during prolonged filtration cycles. On the other hand, the hydraulic resistance values were in the order of  $10^{12} \text{ m}^{-1}$  for the MWCNT blended membranes, which is significantly higher than that of the ZnO NP blended membranes, which were of the order of  $10^8 \text{ m}^{-1}$ . This represents a distinct difference between the pore structures of the membranes.

#### ***Consumer Protection***

From the perspective of consumer protection, these two nanomaterials have different advantages and challenges. ZnO NP possesses higher rejection, which is favourable for water reuse. While the case for ZnO NP is based on strong material immobilization, that of MWCNT relies on strong material embedding within the polymer matrix. The strong enhancement of permeability achieved with low additive load, alongside high rejection,

indicates that MWCNT have strong potential for delivering strong performance with minimal additive content.

From a consumer protection and regulatory law perspective, the use of nanomaterials in ultrafiltration membranes for water treatment engages a clear framework of rights and obligations applicable to all actors involved in the water supply chain. Consumers have a fundamental right to have access to safe, clean, and legally compliant water, as this right is firmly included in EU Drinking Water Directive (EU) 2020/2184. In turn, the manufacturers of filtration membranes, water utilities and system operators face legal obligations to ensure that innovative materials, including nanomaterials, do not impair water quality over their intended lifetime. This encompasses activities such as carrying out appropriate risk assessments, avoiding accidental release of nanoparticles, and ensuring long-term material performance in line with public health protection requirements. Membrane longevity, in this regard, is not only a technical benefit but also a legal safeguard toward continued regulatory compliance and customer confidence.

In parallel, the product safety and chemical regulation framework also lays down obligations for diligence, transparency, and accountability for economic operators in the market with regard to the placing of nanomaterial-based products. The European General Product Safety Framework and the REACH Regulation hold the manufacturer liable for the safety of the materials used by the manufacturer, including the traceability and performance of the water supplier. Failure to comply with such obligations may give rise to regulatory enforcement or liability claims, particularly where damage to consumer health or the environment is claimed. Legal design considerations for ultrafiltration membranes with durability, stability, and safety-by-design principles, therefore, represent a direct legal underpinning for the fulfilment of such obligations and reinforce consumers' rights to informed, safe, and sustainable water services in circular water systems.

### ***Relevance to Circular Water Systems***

In the case of circular water systems, membranes must be able to perform for longer durations, and in the process, they also need to ensure the quality of the water that is produced. The results show that the role of ZnO NP and MWCNT in the membranes brings about an improvement in the performance of the membranes compared to their pristine form, but in different ways. While the role of ZnO NP brings about an ideal balance between the permeability and rejection properties of the membrane, the role of MWCNT achieves greater permeability efficiency with higher selectivity. Thus, the role of nanomaterials in membranes needs to be assessed in an integral way, taking into consideration performance parameters, as well as longevity and consumer protection.

### ***Limitations***

MWCNT leaching during membrane filtration was evaluated using UV–Vis spectroscopy based on Beer–Lambert’s law at 810 nm in deionised water. No MWCNTs were detected in either the permeate or retentate streams. Hence, MWCNT release was either below the detection limit or negligible under the experimental conditions. In contrast, although ZnO NP is widely reported in the literature as a low-toxicity nanomaterial, its leaching behaviour was not measured in this study. The systematic assessments of ZnO NP leaching in membrane systems remain limited.

In this study all membranes were prepared and tested at the laboratory scale. Hence, scaling up to pilot or industrial scale may introduce challenges such as homogeneous distribution of nanomaterials, reproducibility, and stability under actual process conditions.

To evaluate nanomaterial blended membranes for regulatory purposes, especially for water treatment, it is important to conduct standard tests for ecotoxicity and safety, which are not within the scope of this research and should be addressed in future research.

## Conclusions

This study comparatively analysed nanomaterials blended ultrafiltration membranes using ZnO NP and MWCNT, regarding the performance, durability, and consumer protection within the scope of the circular water management. The findings indicate a significant improvement in membrane properties with the addition of nanomaterials. However, the nature of the impact is different depending on the nanomaterials used.

ZnO NP blending caused a moderate increase in the porosity and permeability of the membranes but at the same time provided enhanced rejection of solutes. Such desirable properties of ZnO NP, confer advantages to ZnO NP blended membranes in applications where water quality assurance are of high priority. However, there is a need to evaluate the stability of the ZnO NP blended membranes and the potential release of ZnO NP to ensure consumer safety in water reuse.

The MWCNT incorporation led to a significant increase in the permeability of the membranes as well as an improved solute rejection and lower hydraulic resistance. The significant level of permeability enhancement obtained at a relatively low MWCNT content confirms effective water transport channels and high interconnectivity without the sacrifices in selectivity. These factors suggest that MWCNT blended membranes can be used in energy efficient environments without sacrificing long-term performance in circular water management. In conclusion, the results demonstrate that nanomaterials blended ultrafiltration membranes are useful in overcoming the limitations of traditional membranes.

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