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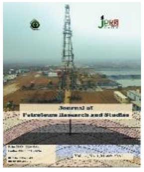

Comparison Study Performance Between the Photocatalytic Process and Membrane Filtration for Oily Wastewater Treatment

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Abstract

In this work, a comparison of performance between the photocatalytic process and ultrafiltration membrane was investigated for removing oil from oily wastewater. The eco-friendly calcium oxide (CaO) was prepared from tomato plant waste as a photocatalyst, and the membrane was prepared from polyacrylonitrile (PAN). The DLS and EDX analyses of the prepared photocatalyst were investigated, and it was noted that the photocatalyst is nanosized and belongs to the type CaO. The contact angle of the PAN membrane was measured, and it was about 68.53°. The impact of the amount of CaO (0–0.35 g/L) and irradiation time (15–150 min) on oil photocatalytic activity was tested, as well as transmembrane pressure (1–3 bar) and operation time (15–60 min) on the removal of oil and flux of water and oily wastewater by the PAN ultrafiltration membrane. The findings demonstrated that when the amount of CaO and irradiation time increase, the oil removal increases, but it slightly decreases as the transmembrane pressure increases, while the permeability of the PAN membrane rises when transmembrane pressure rises and declines as operation time rises. The findings illustrated that the maximum oil removal efficiency (92.19%) was obtained by ultrafiltration membrane compared to the photocatalytic process (80.93%) at optimum conditions (CaO = 0.25 g, irradiation time = 2 hr, transmembrane pressure = 1 bar).

Keywords: Eco-friendly, Oily wastewater, PAN, Photocatalytic, Ultrafiltration membrane.

دراسة مقارنة بين أداء عملية التحفيز الضوئي والترشيح الغشائي لمعالجة مياه الصرف الصحي الزيتية

الخلاصة

في هذا العمل، تم إجراء مقارنة بين أداء عملية التحفيز الضوئي وغشاء الترشيح الفائق لإزالة الزيت من مياه الصرف الصحي الزيتية. تم تحضير أكسيد الكالسيوم الصديق للبيئة (CaO) من نفايات نبات الطماطم كمحفز ضوئي، وتم تحضير الغشاء من بولي أكريلونيتريل(PAN). تم التحقيق في تحليلات DLS و EDX للمحفز الضوئي المحضر، ولوحظ أن المحفز الضوئي بحجم النانو وينتمي إلى نوع CaO تم قياس زاوية تلامس غشاء PAN، وكانت حوالي 68.53 درجة. تم اختبار تأثير كمية CaO (0-0.35) غرام/ لتر ووقت التشعيع (15-150 دقيقة) على النشاط الضوئي للزيت، وكذلك ضغط عبر الغشاء (1-3 بار) ووقت التشغيل (15-60 دقيقة) على إزالة

الزيت وتدفق الماء ومياه الصرف الصحي الزيتية بواسطة غشاء الترشيح الفائق PAN . وأظهرت النتائج أنه عند زيادة كمية CaO وزمن الإشعاع، تزداد إزالة الزيت، لكنها تقل قليلاً مع زيادة الضغط عبر الغشاء، بينما ترتفع نفاذية غشاء PAN مع ارتفاع الضغط عبر الغشاء وتنخفض مع ارتفاع زمن التشغيل. وأوضحت النتائج أن أقصى كفاءة لإزالة الزيت (92.19٪) تم الحصول عليها بواسطة غشاء الترشيح الفائق مقارنة بعملية التحفيز الضوئي (80.93٪) في الظروف المثلى (CaO = 0.25 غرام، زمن الإشعاع = 2 ساعة، ضغط عبر الغشاء = 1 بار).

1. Introduction

The amount of oil consumed increases with industrial progress, while other technical and management advancements lag for different reasons that are not ideal, causing a lot of oil to enter the water and cause pollution [1]. Oily wastewater holds oil at different concentrations and is generated from different sources, like oil and gas exploration, petrochemicals, textiles, food processing, transportation, mining, oil refining, metal finishing, etc. [2]. Oily materials, including petroleum hydrocarbons, polyaromatic hydrocarbons, and phenols, are considered poisonous substances and prevent the growth of animals and plants [3]. They cause carcinogenic and mutagenic hazards, which menace the health of humans; thus, the regulations of the government prohibit oily wastewater from being directly discharged [4]. The only practical way to deal with these dire circumstances is to recycle contaminated water or lower the concentration of contaminants to a manageable level [5]. Numerous approaches, including electrochemical treatment, adsorption, flotation, gravitational settling, hydrocyclones, biological processes, and coagulation/flocculation, are physical, chemical, and biological approaches applied for the elimination of pollutants from oily wastewater, which must satisfy the discharge standards demand before they are being discharged into the environment [6][7]. Some of these techniques have drawbacks in their applications, like low elimination efficiency, secondary contaminated products, and rising costs [8]. As a result, the development of effective, economical, and ecologically sustainable methods for the treatment of oily wastewater has emerged as a critical challenge for scientists and environmental professionals worldwide [9]. Specifically, photocatalysis seems to be a successful method for purifying water because of its nontoxicity, low cost, and high efficacy [10]. It is an effective technology for degrading organic pollutants (grease and oil) in oily wastewater by photocatalysts under a UV light source [11]. Many metal oxides, such as TiO_2, ZnO, Fe_2O_4, CuO, SnO_2, CaO, CeO_2, etc., are employed as efficient photocatalysts due to their low toxicity and ability to be easily oxidized into hydroxides or oxides [12]. The synthesis of photocatalysts from the waste of plants is more stable and less expensive, enabling the creation of NPs in different sizes and forms [13]. Thus, due to their relative abundance and inexpensive cost, green photocatalysts are a possible substitute [14]. Because agricultural wastes are plentiful and naturally occurring, and because they have a large number of surface functional groups, they can serve as cost-effective alternatives to expensive semiconductor photocatalysts [15].

One of the common promising techniques for treating oily wastewater is membrane filtration [16]. Compared with conventional methods, membranes offer many benefits for oily wastewater separation, like excellent elimination efficiency of oil, no additive need for a chemical, and a more compact design [17]. Usually, ceramic and polymeric membranes are employed to eliminate the oil. It is believed that polymeric membranes are easy to scale up, inexpensive, and provide good organic removal, minimal energy usage, and complete automation [18]. Polyacrylonitrile (PAN), polypropylene (PP), polyvinylidene fluoride (PVDF), cellulose acetate (CA), polyethersulfone (PES), and polysulfone (PSF) are among the polymers that researchers have commonly employed in oily wastewater purification [19]. PAN is considered a commonly employed polymer for the treatment of oily wastewater due to its good physical and chemical stability, low cost, simplicity of functionalization, and ease of electrospinning [20].

This work aims to compare the performance of the photocatalytic process using green CaO prepared from the waste of tomato plants for the first time and membrane filtration using a PAN ultrafiltration membrane for the removal of oil from oily wastewater to determine the best method for treating. Different parameters, such as the amount of CaO, contact time, oil concentration, and membrane pressure, were investigated to determine their effect on the efficiency of oil removal.

2. Materials and methods

2.1. Materials

The tomato plant waste was supplied from groves. High-purity polyacrylonitrile (PAN) $[-CH_2-CH(CN)-]_n$ (solubility parameter = 26 $MPa^{0.5}$, purity = 99.5%) was obtained from Biosynth, USA. Dimethylformamide (DMF) $[H_3C-N(=O)-CH_3]$ (purity = 99.95%) was purchased from Shanghai Baoqu Chemical Co., Ltd., China. Oily wastewater was provided from the oil field in Iraq.

2.2. Calcium Oxide (CaO) Preparation

CaO is synthesized by the thermal method, which involves the calcination of $CaCO_3$ existence in the sepal's tomato plant to obtain CaO, as appeared in Figure (1). For this purpose, firstly, the outer parts of the flower of the tomato plant (sepals) were collected and cleaned to remove the waste, soil, and dust; after that, they dried for 2 h at 60 °C for removal of any moisture and were ground by a grinding device (MM8-300, NIMA, Japan) to determine the smaller particles of this powder (75 µm). Finally, the prepared powder was burned for 2 h at 900 °C to ensure the completely converted $CaCO_3$ to CaO to obtain CaO [21].

Fig. (1): Thermal method for synthesis of CaO

2.3. PAN Membrane Preparation

The phase inversion method was applied for the preparation of the PAN membrane as appeared in Figure (2). First, the PAN is heated for 2 h at 60 °C to remove humidity, and then 80 wt.% of DMF and 20 wt.% of PAN are blended at 200 rpm, 60 °C, and 5 h by a magnetic stirrer. Then, this mixture is teeming onto a glass plate. Finally, the flat sheet film obtained is submerged in distilled water to harden the flat sheet PAN membrane prepared and remove the DMF [22].

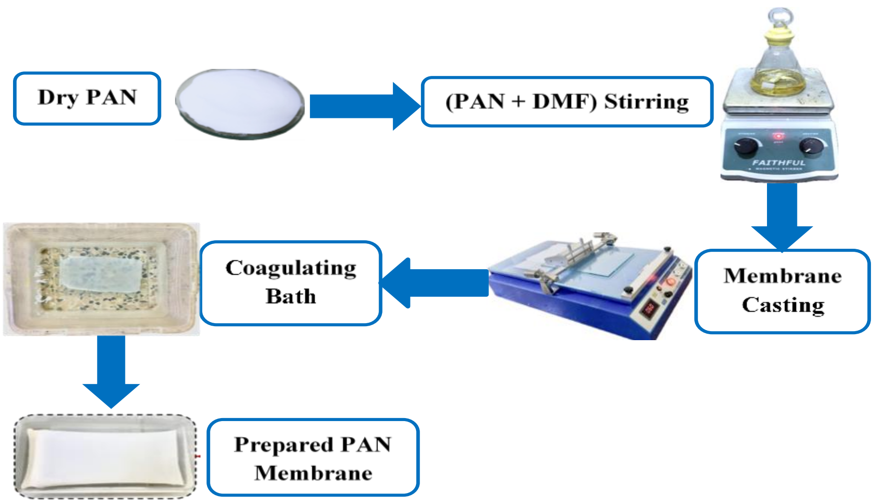

Fig. (2): Phase inversion method for synthesis of PAN membrane

2.4. Oily wastewater

The oily wastewater samples were obtained from the oil field in Iraq. The specification of oily wastewater used in this study is illustrated in Table (1).

Table (1): Specifications of the oily wastewater

Test	Unit	Result
Chloride	mg/L	140200
pH	-	5.8
Density at 15 °C	g/cm^3	1.15
Oil in water	mg/L	160
Particle size	µm	Max = 9.7 Min = 3.1

2.5. The experimental setup

Figure (3) illustrates the photocatalytic reactor that is applied to the degradation of oily wastewater. The photocatalytic process experiments were conducted at an oil concentration of 160 ppm, with a CaO dosage between 0 and 1 g/L, an irradiation time between 15 and 150 min, and an aeration rate of 1 L/min. CaO doses with 500 ml of oily wastewater were mixed in the dark for 0.5 h at 300 rpm speed to reach the case of adsorption-desorption equilibrium. Then, irradiate the mixture under four UVC light lamps (power = 8 W, intensity = 0.7 mW/m^2, wavelength = 254 nm) to remove the oil from oily wastewater. The sample of oily wastewater after treatment was taken to evaluate the oil concentration after a certain time.

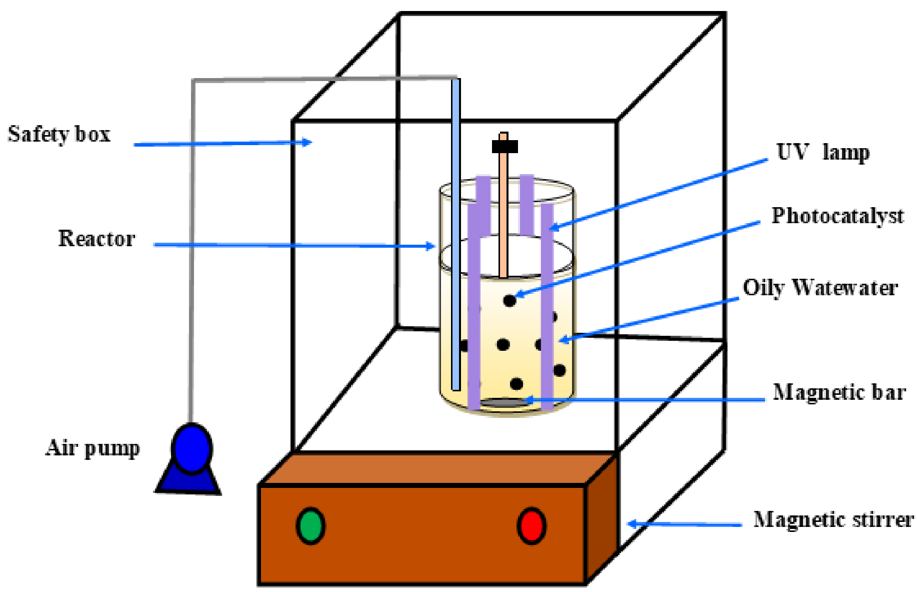

Fig. (3): Photocatalytic reactor system experimental setup

The ultrafiltration membrane experiment was conducted in the cross-flow mode system via a flat sheet PAN membrane with an area of 15.2 cm² at different pressure values (1, 2, and 5 bar) as appeared in Figure (4). In the beginning, the PAN membrane is run at 4 bar for 15 min employing distilled water, and then pressure is reduced to 2 bar (0.7 L/min) to reach the stable state; after 15 min, the flux of water across the PAN membrane is evaluated. After the distilled water test, the feed tank is discharged from distilled water and filled with oily wastewater.

The removal of oil efficiency was calculated by equation (1).

$$\text{Oil removal } \% = \left(1 - \frac{C}{C_o}\right) \times 100 \qquad (1)$$

where C_o and C refer to the initial concentration of oil and over time concentration of oil [23].

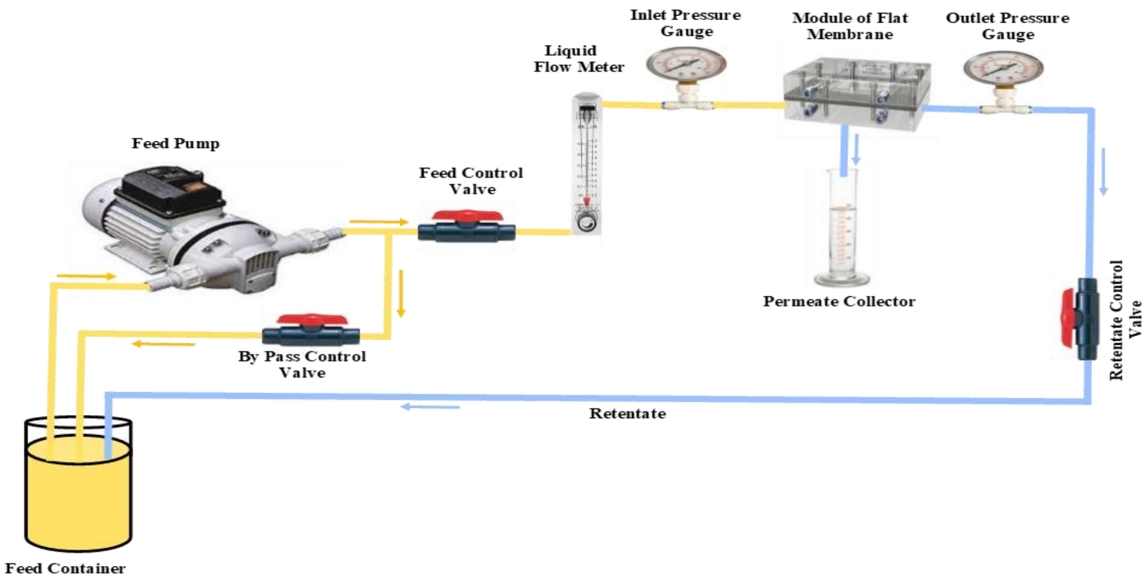

Fig. (4): Ultrafiltration cross-flow membrane system experimental setup

3. Results and Discussion

3.1. CaO Characterization

CaO effective particle size was evaluated using the DLS analysis (90 plus, Brookhaven Instruments Corp., USA) at the University of Technology, Iraq, as appeared in Figure (5). The mean CaO particle size reached 88.7 nm, and this indicates that the CaO synthesized in nanoscale particle size, which enhances the effectiveness of degradation of oil in the photocatalytic process due to increasing the surface area and active sites for adsorption of oil [24].

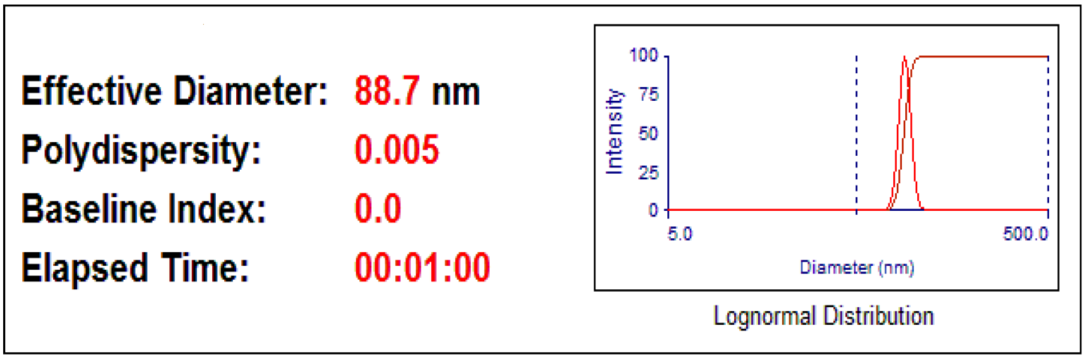

Fig. (5): CaO particle size distribution

Figure (6) represents the EDX analysis by EDX spectrometer (TM4000, Hitachi, Germany), at center BPC analysis, at Iraq of the photocatalyst. From Figure (6), it appears that the basic compositions of the photocatalyst are O and Ca with the highest values of 22.7 wt.% and 20.0 wt.%, respectively, compared with other elements Cl, K, C, Al, Na, S, Mg, Si, P, and Fe. This result proves that the type of prepared photocatalyst is the CaO class.

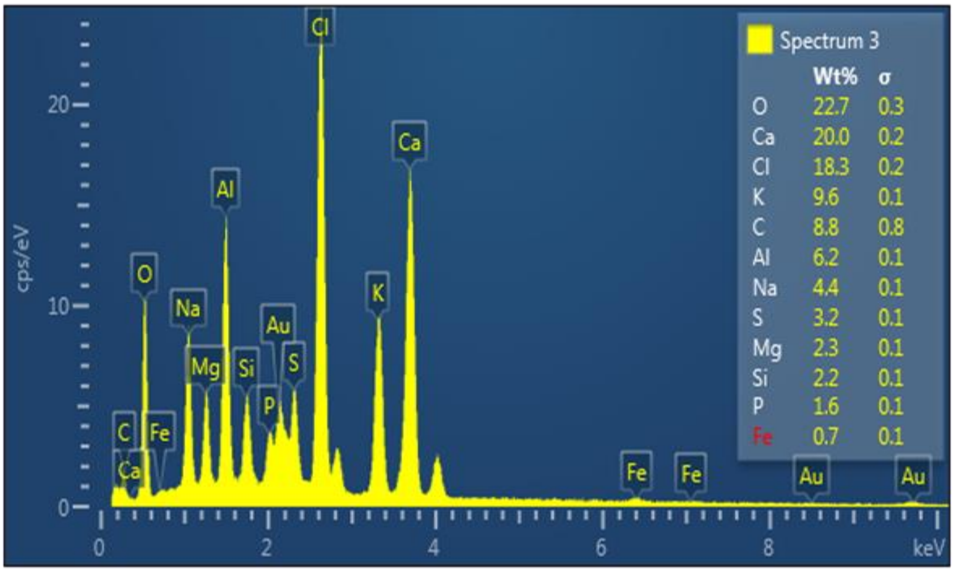

Fig. (6): CaO EDX analysis

3.2. PAN Characterization

Hydrophilicity represents one of the most important membrane properties that influence the permeability and antifouling characterization of the membrane [25]. By contact angle measurement using a contact angle meter (CAM 110-O4W, Tainan, Taiwan), at the University of Technology, Iraq, the hydrophilicity of the PAN membrane arrived at 68.53° (see Figure 7).

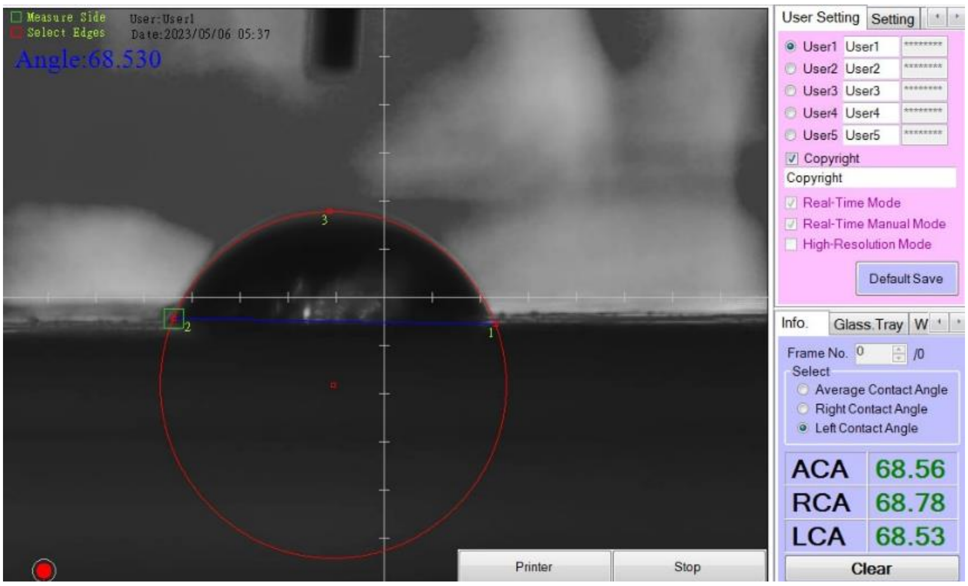

Fig. (7): PAN contact angle

Other significant properties of the membrane are average pore size and porosity, which impact the performance and separation efficiency of the membrane [26]. The PAN membrane's average pore size and porosity were 42.40 nm and 69%, respectively. This value of PAN pore size is smaller than the particle size of oil in oily wastewater, which indicates that oily droplets can't be permeated across the PAN membrane, thereby reaching excellent separation efficiency of oil from oily wastewater.

3.3. The impact of some variables on the photodegradation of oil

3.3.1. Impact of CaO dose

Figure (8) clarified the impact of CaO doses (0-0.35 g/L) on oil removal from oily wastewater at 160 ppm oil concentration under UV for 1.5 hr and a 1 L/min aeration rate. From Fig. 8 it can be noted that the oil removal rises with the increased amount of CaO until it reaches its optimum value, and later the oil removal decreases with increased CaO dose. The oil removal rises from 21.88% (125 ppm) to 73.13% (43 ppm) as the CaO dose rises from 0 to 0.25 g/L. This is because the increase in the amount of CaO can increase the number of active sites available for light absorption and the formation of hydroxyl and superoxide radicals, thereby increasing the oil removal efficiency [27,28]. The oil removal declines from 71.25% (46 ppm) to 70% (48 ppm) with CaO rising from 0.3 to 0.35 g/L. This is due to the high suspension of CaO, which contributed to prohibiting photon flux penetration due to agglomeration, thus reducing the catalyst surface area available for absorption of light and, consequently, oil removal efficiency decreases [29,30].

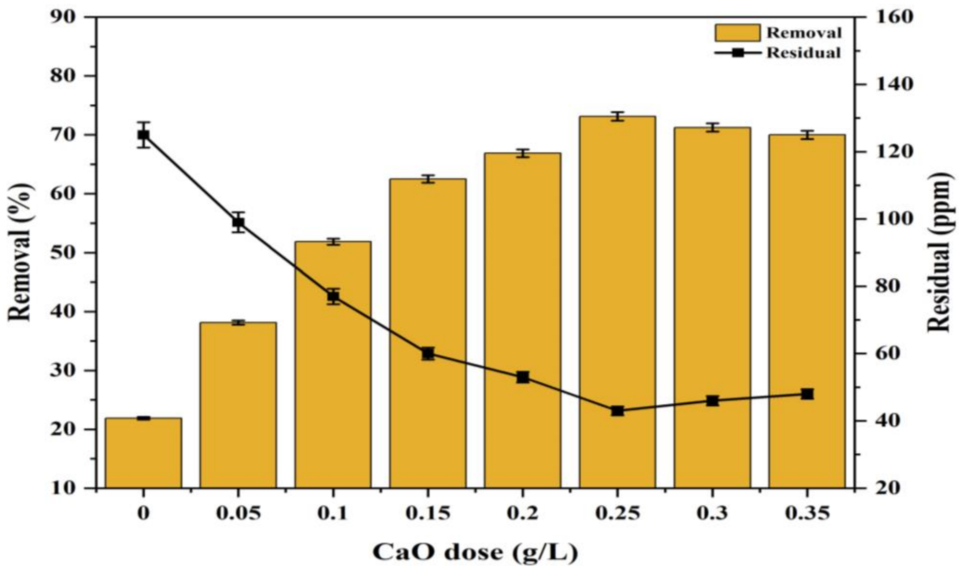

Fig. (8): Impact of CaO doses on oil removal efficiency

3.3.2. Impact of irradiation time

Figure (9) illustrates the impact of UV exposure time (15-150 min) on oil removal efficiency using 0.25 g/L of CaO. From Fig. 9, it displays that the removal of oil rises from 46.25% to 80.94% with an increase in the exposure time for UV from 15 to 120 min. This can be explained by the contact between CaO and oil increasing; the CaO surface becomes more activated with an increase in UV exposure time, thereby increasing the surface area for photocatalytic reactions. With increased irradiation time, more photons are absorbed, leading to the generation of reactive species, which enhances the oil removal [31,32]. Furthermore, it is noted that after 120 min of UV exposure, the oil removal remained constant due to the reaction reaching equilibrium as a result of saturating the active sites on the CaO surface; thereby, the oil removal reaches equilibrium [33,34]. The same findings are gained by Al-Ansari et al. [35].

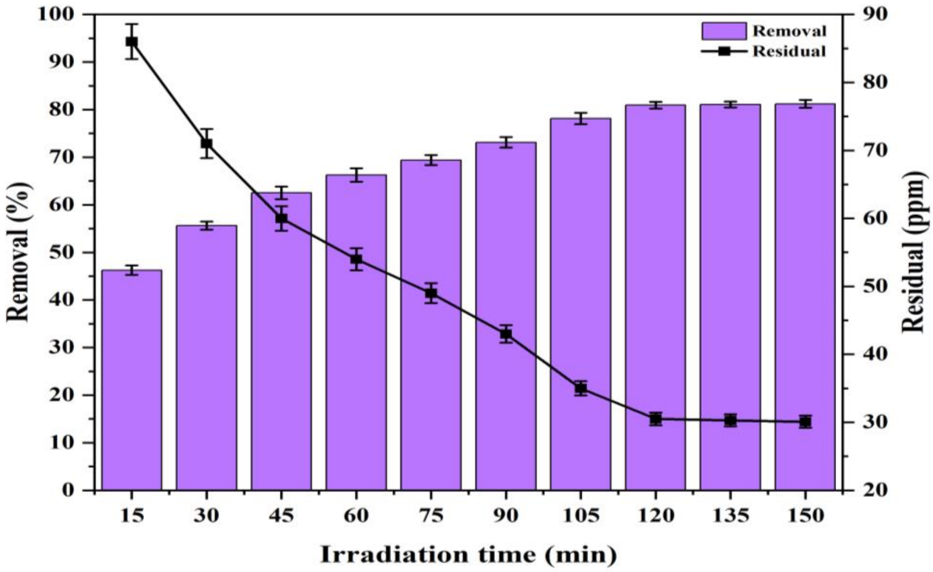

Fig. (9): Impact of irradiation time on oil removal efficiency

3.4. The impact of some variables on the PAN membrane performance

3.4.1. Impact of transmembrane pressure

Figures (10) and (11) display the impact of transmembrane pressure on oil removal and on the flux of oily wastewater and distilled water by applying the PAN membrane under operation pressure between 1 and 3 bar. Figure (10) proves that oil removal declined from 92.19% to 87.50% as the pressure increased from 1 to 3 bar. This result is attributed to exacerbating fouling that causes blocking pores and cake layer formation, which creates additional resistance to filtration with the rising transmembrane pressure, which reduces the efficiency of oil removal [36,37].

From Figure (11), it is observed that the flux of distilled water rises from 78.78 to 220.01 L/m^2. hr while the flux of oily wastewater rises from 29.50 to 98.87 L/m^2. hr when the pressure rose from 1 to 3 bar. This is due to a higher pressure difference with high pressure; according to Darcy's law, the flux is directly proportional to the pressure difference, which increases the shear stress force, which causes the decreased skin layer thickness of the membrane, and this contributes to passing high water or oil flux across the PAN membrane [38]. Therefore, the flux increases with rising pressure. In addition, it is observed that the flux of oily wastewater is less than that of distilled water because of the agglomeration of oil drops on the surface of the PAN membrane, which causes it to block its pores, thereby lowering the flux of oily wastewater compared to the flux of distilled water [39].

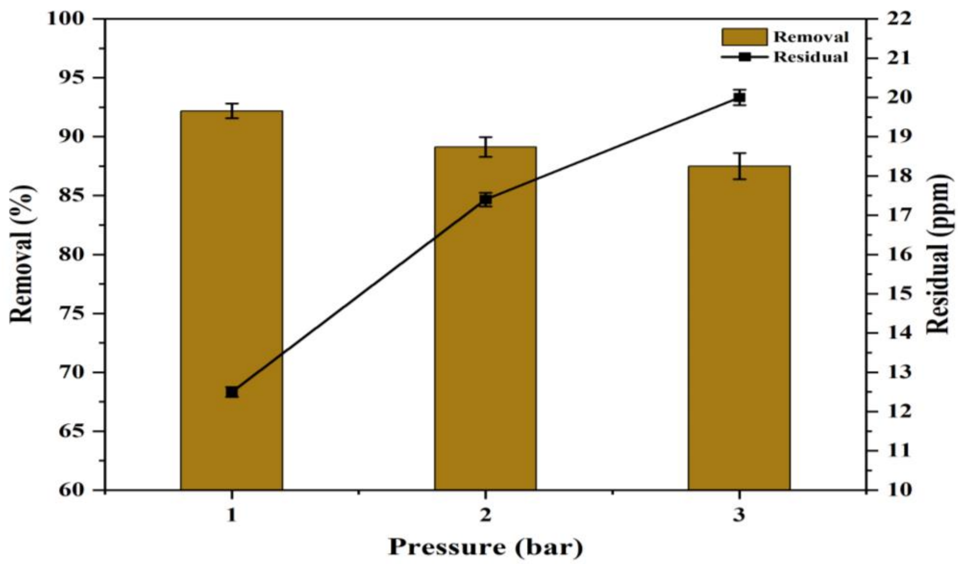

Fig. (10): Impact of transmembrane pressure on oil removal

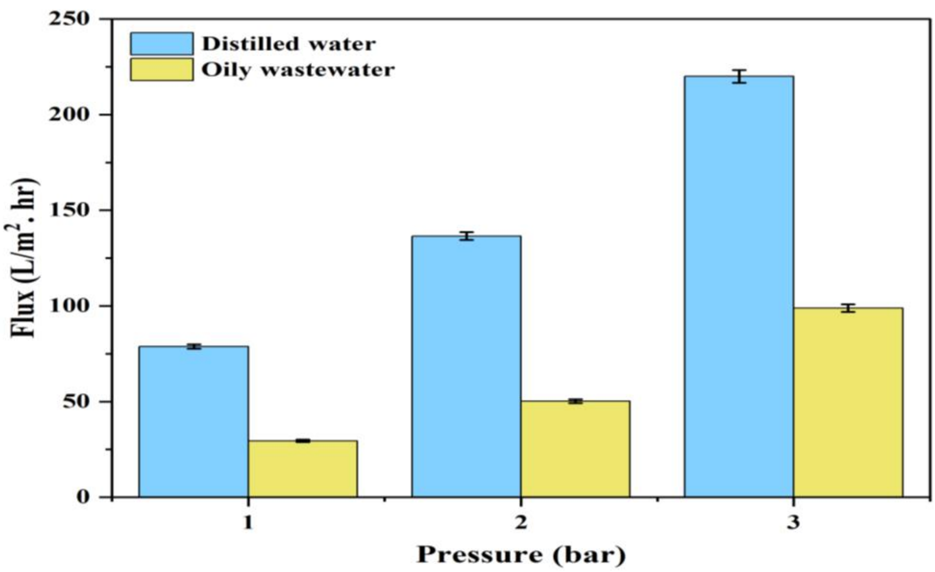

Fig. (11): Impact of transmembrane pressure on the flux of water and oil

3.4.2. Impact of time

Figure (12) offers the impact of operation time (15–60 min) on the permeate flux, which was evaluated by the PAN membrane. According to the findings, it is observed that the permeate flux declined from 60.7 to 39.56 L/m^2. hr with rising in time from 15 to 60 min. This is due to the accumulation of fouling and the formation of the cake layer on the PAN membrane surface and its growth as time progresses [40]. Therefore, this cake layer resistance causes the decline of permeation flux with increased operation time [41].

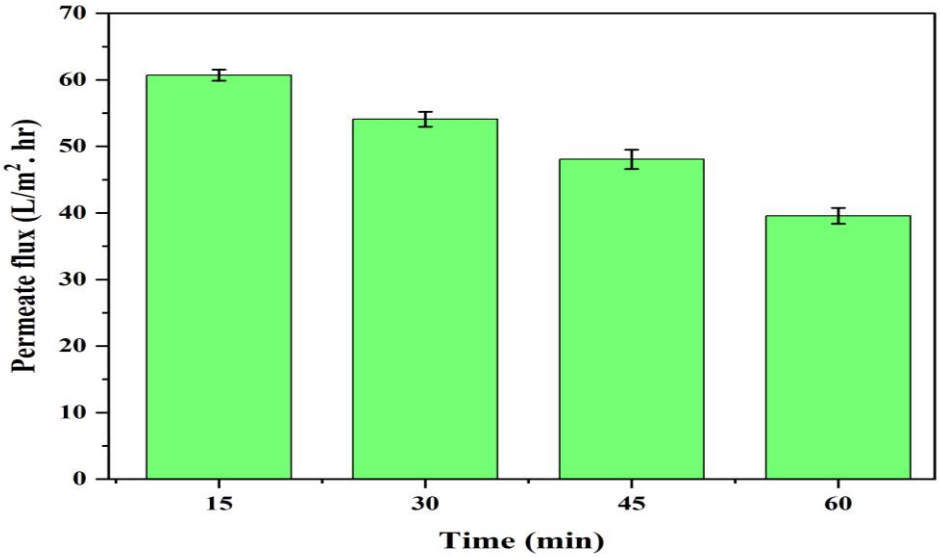

Fig. (12): Impact of time on oil permeate flux

3.5. Comparison between photocatalytic process and membrane filtration

The comparison of the performance of photocatalytic utilizing green CaO and the performance of an ultrafiltration membrane by PAN membrane appears in Table (2), under optimum conditions for each treatment process. It is noted from Table (2) that the oil removal from oily wastewater by ultrafiltration PAN membrane is higher than by the photocatalytic process; it reached about 92.19% with an oil residual of about 12.5 ppm by using ultrafiltration PAN membrane while reaching about 80.93% with an oil residual of about 30.5 ppm by employing photocatalytic. These results are due to the presence of a high amount of chloride in oily wastewater, which affects the performance of photocatalytic processes. The chloride affects the photodegradation of oil by adsorbing inorganic anions onto the photocatalyst surface. This adsorption decreases the number of holes, ·OH radicals, and reaction rate, where chloride anions block the surface sites that are normally available at the CaO for adsorption and electron transfer, thereby oil removal effectiveness utilizing photocatalytic is lower compared to UF membrane.

Table (2): Comparative between photocatalytic process and membrane filtration

Process treatment	Oil removal (%)	Oil residual (ppm)	Optimum parameters
Photocatalytic	80.93	30.5	CaO = 0.25 g, time = 2 hr
Membrane filtration	92.19	12.5	Preesure = 1 bar

4. Conclusions

This work prepares a green CaO as a photocatalyst from the sepals of the tomato plant for applied CaO in the photocatalytic process and prepares an ultrafiltration membrane from PAN for applied PAN in membrane filtration for removal of oil from oily wastewater and compares performance between these processes. The DLS and EDX analyses of the synthesis photocatalyst confirm it to be nanosized and of the CaO class. The contact angle of PAN reaches 68.53°. This work demonstrated that the oil removal increased with an increased amount of CaO and irradiation time and slightly decreased with increased transmembrane pressure, while the permeability of the PAN membrane increased with increased transmembrane pressure and decreased with increased operation time. The findings proved the highest oil removal arrived at 92.19% (residual = 12.5 ppm) by using an ultrafiltration PAN membrane at an oil concentration of 100 ppm and a transmembrane pressure of 1 bar, while it reached 80.93% (residual = 30.5 ppm) by using the photocatalytic process at CaO of 0.25 g/L under 120 min of irradiation by UV. Therefore, the ultrafiltration PAN membrane is better than the photocatalytic for oil removal from the oily wastewater.

Abbreviations

CaO Calcium oxide
CA Cellulose acetate
CeO_2 Cerium oxide
CuO Copper oxide
DMF Dimethylformamide
DLS Dynamic light scattering
EDX Energy dispersive x- ray spectroscopy
Fe_2O_3 Iron oxide
PAN Polyacrylonitrile
PES Polyethersulfone
PDF Polyvinylidene fluoride
PP Polypropylene
PSF Polysulfone
SnO_2 Tin oxide
TiO_2 Titanium dioxide
UV Ultraviolet
ZnO Zinc oxide

Author Contributions Statement: Eman H. Khader contributed to the Conception; Methodology; Investigation/ Experiments; Data Analysis and Interpretation. Thamer J. Mohammed contributed to the Conception; Methodology; Writing – Original Draft; Writing – Review & Editing. Talib M. Albayati contributed to the Conception; Methodology; Writing –

Original Draft; Writing – Review & Editing. Mahdi S. Mahdi contributed to the Conception; Methodology; Investigation/ Experiments; Data Analysis. All authors have read and approved the final version of the manuscript.

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