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Effective removal of anionic dyes from aqueous solution using polyethersulfone based membrane reinforced by montmorillonite

Farnaz Mortezapour¹, Nasrin Shadjou^{1,2*} and Mehdi Mahmoudian^{1,2*}

Abstract

The study highlights the development and characterization of a novel polymeric membrane composed of montmorillonite (MMT) and polyethersulfone (PES) using the phase inversion process. The membrane incorporates polyethylene glycol (PEG) as a pore-forming agent and N-methyl pyrrolidone (NMP) as a solvent. The addition of MMT significantly enhances the membrane's properties, including hydrophilicity, porosity, antifouling capacity, hydraulic resistance, water uptake, and dye removal efficiency. Characterization techniques such as FT-IR spectroscopy, FE-SEM, EDX spectroscopy, water flux measurements, water uptake analysis, contact angle studies, and fouling assessments confirm the improved performance of the PES/MMT composite membrane. The presence of MMT increases the negative surface charge of the membrane, making it particularly effective in removing anionic dyes like Congo red (CR), Quinoline yellow (QY), and Methyl orange (MO). The study demonstrates that membranes with up to 5 wt% MMT exhibit high porosity (68.2%) and enhanced water flux (35 L/m²·h), achieving dye rejection rates of 99% for CR, 92% for MO, and 81% for QY. The integration of MMT into PES membranes presents a significant advancement in sustainable water purification technologies. These modified membranes demonstrate enhanced mechanical strength, improved structural stability, and an increased surface area, making them highly effective for dye adsorption. Compared to traditional materials, PES/MMT membranes exhibit superior performance in wastewater treatment and dye removal, offering a promising and eco-friendly alternative for addressing environmental challenges.

Keywords Water purification, Polymeric membrane, Environmental technology, Montmorillonite-polyethersulfone, Water remediation, Anionic dye removal

Introduction

The global textile dye industry significantly contributes to water pollution, releasing hazardous synthetic dyes into aquatic ecosystems [1, 2]. Annually, around 700,000 tons of dyes are produced, with 10-25% wasted during the

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dyeing process and 2–20% directly discharged into water bodies. These synthetic dyes are particularly concerning due to their toxicity and resistance to degradation, with some persisting in the environment for up to 46 years [3]. To address this environmental challenge, advanced methods such as ultrasonication combined with ultrafiltration, activated sludge systems, and adsorption-based techniques have been developed [4, 5]. Membrane technologies, in particular, play a vital role in capturing dye molecules and ensuring wastewater meets environmental standards before being released. Hence, researchers continue to focus on designing more efficient membranes to combat this pressing issue [6, 7].



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Modified polyethersulfone (PES) membranes exhibit exceptional performance in dye removal from polluted water, surpassing their unmodified versions. The incorporation of various additives, including iron nanoparticles [8], graphene oxide [9], metformin [10], ZnO [11], Cu-doped GO nanoparticles [12], and carboxylated-MWCNT [13], substantially improves their attributes. These enhancements markedly increase hydrophilicity, raise pure water flux, and strengthen resistance to fouling. Additionally, they improve membrane roughness, increase porosity, and enhance mechanical durability, all while maintaining the selectivity essential for effective dye separation. As a result, these modifications successfully address the inherent shortcomings of unmodified PES membranes, such as their hydrophobic nature and susceptibility to fouling, leading to a notable improvement in filtration efficiency [14].

Montmorillonite (MMT), a clay mineral with a layered structure, is widely recognized for its effectiveness in adsorbing various dyes from wastewater. This makes it both cost-effective and efficient for environmental applications [15]. It has been proven to successfully remove dyes such as Reactive Yellow 15, Reactive Yellow 42 [16], malachite green [17], and Basic Red 13 [18]. The adsorption efficiency of MMT depends on factors such as pH levels, contact time, dye concentration, and the amount of adsorbent used. Modifications, such as the addition of sodium dodecyl sulfate, can further enhance its adsorption capabilities. Additionally, MMT-based adsorbents are sustainable, as they can be regenerated and reused in wastewater treatment processes [19]. When modified montmorillonite is incorporated into polyethersulfone (PES) membrane matrices, it significantly improves the removal of anionic dyes from water. This combination results in a highly effective adsorbent for addressing dye pollution in aqueous environments. Research indicates that montmorillonite's high surface area and ionexchange properties substantially enhance membrane performance, leading to increased dye removal efficiency [20]. Furthermore, the integration of modified montmorillonite into PES membranes enhances their porosity, water flux, and anti-fouling characteristics. These improvements make the membranes highly efficient in removing various dyes from water sources, achieving high removal percentages and contributing to more effective wastewater treatment solutions [21-23].

This study explores the development and application of PES/MMT nanocomposite membranes for the efficient removal of anionic dyes such as Methyl Orange (MO), Congo Red (CR), and Quinoline Yellow (QY). These membranes, created through a phase inversion process, incorporate varying amounts of montmorillonite (MMT) to enhance their performance in dye separation.

Characterization techniques were employed to evaluate the membranes' effectiveness, focusing on the influence of montmorillonite's natural negative charge and the molecular size of the dyes. The dual functionality of MMT, serving as both an adsorbent and antifouling agent, plays a critical role in improving the hydrophilicity and antifouling properties of the membranes. These modified membranes demonstrate enhanced mechanical strength, improved structural stability, and an increased surface area, making them highly effective for dye adsorption. These advancements contribute to better performance in water treatment applications (Fig. 1).

Experimental

Materials

Polyethersulfone (PES, MW = 58,000 g/mol)), Polyethylene glycol (PEG, Mw:10,000 g/mol), and N-methyl pyrrolidone (NMP), were purchased from Merck. Montmorillonite K10 (MMT), methylene blue, methyl orange, congo red, and quinoline yellow were supplied from Sigma-Aldrich. All the mentioned materials were utilized in their original state without undergoing additional purifications.

Instruments

FTIR spectroscopy was utilized to analyze the chemical composition of nanostructures and fabricated membranes over a scanned range of 400 to 4000 cm⁻¹ using a WQF-510A spectrophotometer. The shape and morphology of the membranes were examined using FESEM (HITACHI S-4160), while EDAX was applied to exhibit the alteration and distribution of particles within the membrane matrix.

Preparation of membranes

All membranes were synthesized using phase inversion process and characterized according to our previous published article [23]. In brief, polyethersulfone (PES) as polymer and polyethylene glycol (PEG) as pore forming agent, and MMT (as additive) were dissolved in *N*-methyl pyrrolidone (NMP) as the solvent and stirred for 24 h at 40 °C (Table S1 (see supporting information)). Then, the obtained mixture was drawn on a smooth and clean glass plate using a suitable blade and then immersed in a coagulation bath (water, anti-solvent, room temperature) to form the nanocomposite film. After separation from the glass surface, the membranes were immersed in water for 24 h and then dried in a vacuum oven at 50 °C. In the synthesis process, four types of membrane with different amount of MMT were prepared.



Fig. 1 Schematic structure of polyethersulfone based membrane reinforced by montmorillonite towards efficient removal of anionic dyes from aqueous solution

Water uptake analysis

To analyze water uptake, a membrane piece with a surface area of 1 cm² was trimmed and subjected to 24 h of drying at 50 °C in a vacuum oven. Following this, the sample was meticulously weighed (W_d) and submerged in deionized water. After 24 h, the membrane specimen was taken out from the water and reweighed after eliminating surface water (W_w) . The percentage of water uptake by the membrane was then calculated using Eq. 1.

Water uptake (%) =
$$\frac{W_w - W_d}{W_w} \times 100$$
 (1)

The measurements were conducted three times, and the average value was recorded as the amount of water absorbed.

Measurement of porosity and pore radius in membranes

The porosity of the membrane was assessed using a gravimetric method based on the degree of expansion in deionized water (Eq. 2).

$$\varepsilon (\%) = \frac{(W_w - W_d)}{A \times l \times d_w} \times 100$$
⁽²⁾

This equation represents the porosity percentage denoted as ε . W_d and W_w represent the weight of dry and wet membranes in grams, respectively. The density of water, marked as d_w , is 1 g/cm³, and A stands for the membrane's surface area in cm², while 1 denotes the membrane thickness in cm.

Furthermore, the membranes' average pore radius was determined utilizing the velocity filtration method based on the Guerout-Elford-Ferry theory (Eq. 3).

$$R_m = \sqrt{\frac{(2.9 - 1.75\varepsilon) \times 8\eta lQ}{\varepsilon \times A \times \Delta P}}$$
(3)

 R_m , η, l, Q, A, and ΔP represent the average pore radius (m), viscosity of water (Pa.s), thickness of the membrane (m), flow rate through the membrane (m³/s), surface area (m²), and applied pressure (Pa), respectively.

Contact angle measurement

During the experiment, tiny deionized water droplets were positioned at various locations on the membrane surface, and their images were captured by high-speed camera. The contact angle formed by the water droplets on the surface was quantified using Image J software. This procedure was implemented to assess the water contact angle across different regions of the membrane surface, and the average of these readings was documented. Furthermore, the standard deviation for the collected data was computed.

Membrane performance evaluation *Pure water flux (PWF)*

A hand-made nanofiltration cell was utilized to measure the pure water flux. The testing membrane employed in this experiment exhibited a 3.73 cm² circular surface area and was situated at the membrane loading location within the cell system, which was sealed and filled with deionized water as the feed. Nitrogen gas served as the driving force at pressures ranging from 3 to 10 bar. The pure water flux was determined using the equation denoted as Eq. 4.

$$PWF = \frac{Q}{A.t} \tag{4}$$

In this regard, the PWF is assessed as kg/m^2 .h. Additionally, Q represents the collected water quantity in kg, while A stands for the membrane surface area in square meters. The duration of measurement is denoted as t(h).

Dye removal

The filtration experiments were carried out using a homemade dead-end setup with a membrane surface area of 3.73 cm^2 and a processing volume of 300 mL. In order to assess the efficiency of MMT-containing membranes in dyes removal, a solutions containing MB, CR, QY, and MO dyes at concentrations of 1.25, 2.5, 5.0, 10 and 20 ppm were prepared as a feed. In every test, the pressure was set to 5 bar, and the temperature was controlled at 25 °C. Additionally, the effect of applied pressure and dye concentration was investigated in dye removal efficiency. The permeate solution that passed through the membrane was collected and analyzed to determine the percentage of dye removal (Eq. 5). The concentration of dyes was measured using a UV spectrophotometer (PG, China) equipped with a 1 cm quartz cell.

$$\text{Rejection}(\%) = 1 - \left(\frac{C_p}{C_f}\right) \times 100 \tag{5}$$

 C_p and C_f represent the concentrations of dyes in feed and permeate solutions, respectively. Experiments were carried out to investigate membrane fouling and its longterm efficiency by utilizing dye solutions and measuring flux. Also, the absorption peaks of Methylene Blue, Congo Red, Quinone Yellow, and Methyl Orange in UV–visible spectroscopy are observed at approximately 665 nm, 499 nm, 400–500 nm, and 464 nm, respectively.

Determination of fouling in membranes

The fouling behavior of the fabricated membranes was evaluated through a static test, which determined the quantity of dye adsorption on the membrane surface after exposure to a 100 ppm methylene blue solution.

Results and discussion

Characterization of engineered membranes FT-IR analysis of montmorillonite and montmorillonite modified membranes

FT-IR spectra of MMT, unmodified and modified membranes were recorded (Figure S2, see supporting information). In the MMT spectrum the peak in 3622 cm^{-1}

is associated with the stretching vibration of hydroxyl groups within the layers and the peak at 3438 cm⁻¹ indicates the presence of water absorption on the sample. Observing a peak of 1634 cm⁻¹ suggests the vibration of water molecules between the layers. Moreover, the 1054 cm⁻¹ peak is related to the stretching vibration of O-Si bond. Similarly, the 527 cm⁻¹ peak is attributed to the stretching vibration of the O-Al bond, while the 467 cm^{-1} peak indicates the bending vibration of the O– Si bond. In the pure polysulfone membrane, the peaks at 1104 cm⁻¹ and 1301 cm⁻¹ are related to the symmetric and asymmetric stretch in vibrations of sulfone groups, and the peaks at 1221 cm⁻¹, 1475 and 1560 cm⁻¹ are related to C-O-C bond, and aromatic ring vibrations, and peaks at 2949 and 3051 cm⁻¹ appear due to vibrations of aliphatic and aromatic C-H bonds, respectively. The spectrum of the nanocomposite membrane is similar to the neat PES membrane. However, the presence of a peak at 3500 cm^{-1} is related to the absorbed water in the MMT, inserted into the membrane structure.

FESEM of membranes

The FESEM analysis (Fig. S3, see supporting information) revealed notable morphological changes in the PES membrane upon the incorporation of MMT. Distinct differences were identified in the size and distribution of channels, as well as in cavity characteristics, when comparing pristine membranes to their nanocomposite variants. All examined membranes consisted of a thin, compact top layer supported by a porous substrate underneath. Surface imaging showed that the pristine membrane had a smooth appearance, while the nanocomposite membranes exhibited increasing surface roughness with rising MMT content, a result of particle inclusion. Cross-sectional imaging indicated that the pristine membrane featured prominent finger-like cavities, whereas adding MMT transformed the pore structure into a sponge-like configuration. Moreover, the incorporation of MMT particles affected both the membrane porosity and the microstructural thickness.

EDAX analysis of prepared membranes

EDAX analysis facilitated the assessment of MMT extent in polymer matrix. Hence, a membrane containing 5% MMT was chosen and the elemental analysis of the samples was carried out. The resulted graphs are shown in Figure S3 (see supporting Information). A comparison between the elemental analysis of MMT and PES/ MMT membrane provides the presence of MMT in the polymer matrix and confirmed successful modification of membrane by this additive.

The mean pore diameter (MPD), and porosity of PES based membranes

The MPD and porosity analysis of membranes was performed to investigate the effect of pore size and porosity on the removal efficiency. Table S2 (see supporting information) summarizes the membrane characteristics, indicating that the integration of more MMT into the polymer matrix leads to a decrease in average pore size and an increase in porosity, facilitating the capture of pollutants with lower molecular weights [24]. Specifically, a membrane with 10wt% of MMT shows a removal efficiency of over 90%. This highlights the effectiveness of mixed matrix membranes in enhancing pollutant removal capabilities by tailoring pore size and porosity through the incorporation of MMT into the membrane structure.

Pure water flux and water uptake tests

In order to evaluate the hydrophilic property of prepared membranes, pure water flux experiment was carried out using circular cut membranes with surface area of 3.73 cm² for filtration, in external pressures of 5 and 10 bar.

The pure water flax data are presented in Fig. 2A. The findings show that with increasing pressure as the driving force, the pure water flux has increased. Also, all the membranes containing MMT demonstrated a higher pure water compared to the neat PES membrane because of the MMT's hydrophilic nature and larger cavities in the modified membranes. Also, the hydroxyl groups on the surface of MMT facilitate hydrogen bonding with water molecules, which increases the membrane's wettability. This increase in hydrophilicity promotes more effective water transport through the membrane's structure. As

a result, membranes containing MMT, have increased pure water flux and improved dye removal due to their enhanced hydrophilicity and porosity [25, 26].

Water uptake demonstrates the amount of absorbed water by the membrane and shows the hydrophilicity of the membrane matrix. As shown in Fig. 2B, the water uptake in the neat membrane was 33% and, in the nanocomposite, membranes increased with addition of MMT content and in the membrane with 10 wt% of montmorillonites, it was more than 60%. The hydrophilic nature of montmorillonite and increased porosity have contributed to enhanced water uptake.

The effect of time on pure water flux

The effect of time on the pure water flux of pure PES nanocomposite membranes was investigated. This approach enables the assessment of membrane performance, which is essential for comprehending the effectiveness and longevity of filtration membranes in water treatment applications. The pure water flux of the fabricated membranes was measured during the initial and subsequent two hours under a 5-bar pressure. Figure S4 (see supporting information) shows the measured pure water flux. It indicated that the pure water flux of all membranes decreased over time. However, the drop in pure water flux was less notable in membranes with high MMT content compared to those with lower MMT. This difference can be primarily attributed to the uniform distribution of additive particles within the membrane, resulting in greater structural integrity during the water permeation process in membranes with higher MMT. The improved dispersion of MMT particles within the



Fig. 2 A, B Pure water flux and water uptake of the prepared membranes at 5 and 10 bar, respectively

polymer matrix serves to reinforce the membrane structure effectively.

Contact angle test

Water contact angle test is a reliable method for assessing membrane surface hydrophilicity by measuring the angle of a water droplet on the membrane. Membrane modification with hydrophilic additives leads to decreased contact angles, indicating increased hydrophilicity, which aligns with enhanced water flux and uptake data. Therefore, adjusting the surface properties of membranes through additives can significantly influence their hydrophilicity and overall functionality in various applications. The incorporation of MMT into PES membranes significantly improves their hydrophilicity, as evidenced by the reduction in water contact angle from 63° for pure PES to 38° for membranes with 10 wt% MMT. This increased hydrophilicity enhances the membrane's ability to attract water molecules, leading to improved operational performance, such as better water flow and enhanced dye rejection. The addition of MMT modifies the surface energy of the membranes due to its layered silicate structure, which increases surface area and promotes stronger interactions between the polymer matrix and water molecules. These changes contribute to higher surface energy and improved wettability, making the membranes more efficient for applications requiring high water affinity (Fig. 3).

Hydraulic resistance studies

The hydraulic resistance (R_m) of a membrane refers to its ability to resist the flow of a fluid through it. This property is influenced by various factors, including the membrane's structure, porosity, and surface chemistry. Therefore, hydraulic resistance indicates the tolerance of filtration of membranes relative to the hydraulic pressure.



Fig. 3 Contact angle test of bare PES and modified membranes (PES/ MMT)

Obtained results (Figure S5 (see supporting information)) show that a lower amount of hydraulic resistance means better performance of the membrane under high pressure. In addition, the hydraulic resistance of all modified membranes (PES/MMT) was lower than the neat PES membrane. Also, R_m decreased with increasing of MMT amount, because a higher percentage of the additive improved the mechanical strength of membrane.

Membrane fouling study

The study examined membrane fouling using methylene blue (MB), a cationic dye, as the filtration feed. Since the membrane surface carries a negative charge, the positively charged MB was utilized to assess the fouling rate. The investigation employed static methods to analyze the dye absorption rate on the membrane surface. Results indicated that the dye absorption rate increased with the integration of an additive and its higher concentration in the membrane. This outcome aligns with the formation of negatively charged functional groups on the membrane, which enhance electrostatic attraction with the positively charged dye (Figure S6 (see supporting information)).

Investigation of anionic dyes removal by the prepared membranes from water

This research explores the separation of three anionic dyes (CR, QY, MO) with different molecular weights by the prepared membranes (Fig. 4). At first, a concentration of 100 ppm for these dyes was prepared and stored away from sunlight. Then, the efficiency of neat and nanocomposite membranes in dye removal was evaluated using a nanofiltration cell. The findings revealed that increasing the concentration of MMT in the membranes, up to 5 wt%, improved the separation of dyes, indicating the positive impact of MMT on membrane functionality. However, a higher additional amount (10wt%) led to a slight reduction in membrane efficiency, probability due to additive accumulation, and a decrease in effective surface area. The cavities size could affect the dye removal efficiency. The presence of large cavities in the membrane wit 10 wt% of MMT has reduced its efficiency in separating dyes. Moreover, the improvement of the efficiency of nanocomposite membranes compared to neat polyethersulfone membrane can be justified due to the negative charges in the structure of montmorillonite. Polyethersulfone is inherently negatively charged. The presence of montmorillonite additive increases the density of negative charges on the surface of the membrane, and the accumulation of negative charges can help to remove the negative charge in the pigments. Also, the dye removal by the prepared membranes is directly dependent on the MW of dyes. As a result, mentioned dyes with larger MWs, were more effectively separated by the membrane.



Fig. 4 Anionic dye (MO, QY and CR) removal of the prepared membranes

For instance, the CR, which has a MW of 696 g/mol, exhibited greater removal compared to other dyes (MO and QY). The QY, with a MW of 477 g/mol, was treated more effectively than the MO dye with a MW of 327 g/mol.

The effect of time on dye removal

The evaluation of the optimized membrane's performance over a two-hour filtration period, as shown in Figure S7 (see supporting information), revealed a higher efficacy of dye removal during the initial hour compared to the subsequent hour. This difference can be attributed to the membrane's initially clean and efficient surface, facilitating enhanced dye capture. However, as filtration progresses, the membrane may become blocked or saturated with dyes, leading to a decrease in efficiency over time. This decline in effectiveness over the twohour period underscores the importance of monitoring membrane performance and considering factors such as fouling and saturation to optimize the efficiency of dye removal processes in wastewater treatment applications.

The effect of dyes' initial concentration on dye rejection

The performance of the optimized membrane (5% MMT) was evaluated by assessing the initial concentration of dye solutions (1.25, 2.5, 5.0, 10 and 20 ppm). As the initial dye concentration increases, the membrane's saturation may occur more rapidly, leading to a decrease in dye removal effectiveness over time (Fig. 5). Moreover, heightened dye concentrations can lead to increased

membrane fouling, diminishing its capacity to trap additional dye particles. Generally, an escalation in the initial dye concentration in wastewater is anticipated to lessen the overall efficiency of the filtration process utilizing the PES/MMT as a membrane. This underscores the crucial necessity to optimize operational parameters and membrane design to tackle higher dye concentrations in wastewater for effective treatment.

The study for the first time highlights the effectiveness of PES membranes reinforced with MMT in removing anionic dyes from water. Compared to earlier findings [27–32], the developed membrane exhibits enhanced dye removal efficiency. The addition of MMT not only boosts the membrane's dye elimination capabilities but also leverages its porous structure to achieve efficient separation based on molecular size. This innovation offers a significant step forward in advanced water purification technologies (Table 1).

Conclusion

In summary, the PES/MMT nanocomposite membrane fabricated via the phase inversion method exhibits excellent adsorption efficiency while being cost-effective and environmentally friendly. These nanocomposite membranes are employed because polymers and MMT are readily available and cost-effective. Enhancing the membrane's performance for removing anionic dyes has been significantly influenced by the presence of MMT, which has improved surface hydrophilicity and hydraulic resistance. Also, the presence of montmorillonite additive



Fig. 5 The effect of initial concentration (1.25, 2.5, 5.0, 10 and 20 ppm) of pigments (MO, QY and CR) solutions in dye rejection

Entry	Membrane	Dye removal (%)	Contact angle	Flux (Kg. m ² h)	Ref.
1	PES/TiO ₂ membrane	MG=80%		_	27
2	PES/GO	Direct red 16=99%	53.2	20.4 (4 bar)	28
3	PES/TiO ₂ membrane	RB21 = 73.01%	54.2	32.5 (3 bar)	29
4	PES/GO	Sunset yellow = 84.9% acridine orange = 48.4%	52.4	13 (1 bar)	30
5	amino-functionalized mesoporous/ PES	DR-16=97.5% MO=95.5%	40.78	55.56 (4 bar)	31
6	PES/ MMT-Fe ₃ O ₄	MO=87.5% MB=92.8%	64.5	_	32
7	PES/MMT	CR=99% MO=81% QY=92%	50	421 (5 bar)	This study

 Table 1
 Dye rejection performance comparison with previous studies

increases the density of negative charges on the surface of the membrane, and the accumulation of negative charges can help to remove the negative charge in the pigments. The results show that increasing the amount of MMT (up to 5 wt%) in the membrane structure, caused high porosity (68.2%) and improved water flux (35 L/m².h). Also, water contact angle of pure PES membrane was 63° and reduced gradually as the MMT content in the nanocomposite membranes increased, and reach to 38° in the membrane with 10wt% of MMT (M4). The dye removal for CR, QY, MO is 99%, 81%, and 92% respectively, which demonstrating the good performance of MMT on membrane functionality. Also, the dye removal by modified membranes is directly dependent on the molecular weight of dyes. As a result, dyes with larger molecular weights, owing to their bigger size, are more effectively separated by the membrane. In the present study, factors affecting adsorption including, temperature, initial dye concentration, and contact time was investigated. The potential future perspective lies in sustainable membrane design using PES reinforced by MMT, addressing the removal of anionic dyes for water purification while considering eco-friendly. These materials offer promising potential for treating industrial dyeing wastewater, particularly in alkaline and high-temperature conditions.

Supplementary Information

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Supplementary Material 1.

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Author contributions

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Availability of data and materials

The datasets used and/or analysed during the current study are available from the corresponding author on reasonable request.

Declarations

Ethics approval and consent to participate Not applicable.

Consent for publication

Not applicable.

Competing interests The authors declare no competing interests.

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