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# Graft Polymerization of Allylamine for the Modification of PET Track-Etched Membrane

Track-etched membranes (TMs), characterized by their precisely controlled pore size, geometry, and distribution, offer a promising platform for the development of advanced membrane systems and serve as model membranes for testing and optimizing surface modification techniques. This study presents a perspective modification of poly(ethylene terephthalate) track-etched membranes (PET TM) based on photo-induced graft polymerization of allylamine (AlAm) to introduce primary amine groups on the membrane surface. The polymerization process was optimized by evaluating key parameters, including reaction time, monomer concentration, solvent, and distance from UV-lamp. Optimal conditions for photoinduced graft polymerization were found: grafting time 60 minutes, AlAm monomer concentration 50 %, 2-propanol as a solvent and distance to UV lamp 10 cm. These parameters allowed effective modification of the polymer while maintaining the integrity of the membrane porous structure. The modified membranes were characterized using SEM-EDX, ATR FTIR, and UV-spectroscopy. The results demonstrate the successful fabrication of membranes with a high amino group content (up to 10.6±0.3 μmol/g) while preserving their porous structure. This functionalization enhances the practical potential for the environment and biomedical fields.

*Keywords*: track-etched membrane, poly(ethylene terephthalate), allylamine, photoinduced graft polymerization, modification, polymeric membranes, porous structure, surface functionalization, UV irradiation

## *Introduction*

Track-etched membranes (TM) are a unique class of polymeric membranes obtained through heavy-ion irradiation of polymer films, followed by chemical etching of the ion tracks to create well-defined pores [1, 2]. This fabrication process allows precise control over pore size, shape, and density, enabling the development of membranes with tailored properties for specific applications [3]. Several polymers, such as polyethylene terephthalate (PET), polycarbonate (PC), polyimide (PI), polypropylene (PP) and poly(vinylidene fluoride) (PVDF) are widely used for ion-tracking technology. Poly(ethylene terephthalate) has been found to have a number of applications in the form of functionalized TM owing to its chemical stability, mechanical strength, and thermal stability [4]. PET TM are widely used in medical and analytical applications, including drug purification, virus filtration, and plasma separation [5–8]. TM are also used for quality control of food and water, air and liquid filtration for environments, drinking water purification systems, and analytical monitoring of various substances [9–14].

Most polymeric materials have a pristine surface with low surface energies. Thus, the control of chemistry at polymer surfaces has become increasingly important for at least the major way of applications [15, 16]. The use of chemical and physical modification is one of the most effective approaches to improve membrane properties. Various surface modification techniques have been applied to enhance the functionality of TM, including radiation, chemical, photochemical, and plasma — induced initiation techniques and physical or chemical adsorption [17–20]. These methods are therefore usually employed to obtain TM with specific properties, such as adhesiveness, wettability, biocompatibility, and antifouling, to carry out their intended tasks. The UV-induced surface graft polymerization has several advantages over other surface modification techniques. These advantages include a rapid reaction rate, low processing cost, simple experimental setup, and high potential for industrial scalability. One significant benefit is that the grafted polymer chains are confined to a thin surface layer, which minimizes alteration to the bulk properties of the material. This makes photo-induced grafting a powerful tool for precisely tuning surface characteristics without compromising the structural integrity of the substrate [21].

In our study, we presented the modification of PET TM with allylamines (AlAm) using photo-induced graft polymerization. The AlAm is an attractive category of amines with its molecular structure consisting of highly dense primary amines at the side chain [22]. Additionally, AlAm is a suitable binding material in supported amine systems due to cohesive forces between the chains and adhesive forces with the supports [23, 24]. The allyl group is used for the modification of the molecule onto various templates, including polymers and inorganic supports [25]. In addition, the allyl group allows for efficient participation in radical polymerization, while the introduced amine groups can serve as reactive sites for further chemical functionalization or biomolecule immobilization. Functionalized AlAm systems have been developed to address, among other things, issues related to oxidative and thermal stability in $CO_2$ capture processes [26].

## *Experimental*

### *Reagents*

Allylamine (98 %), benzophenone (BP) (97 %), N, N-dimethylformamide (99,9 %), sodium hydroxide (98 %), acetic acid (99.5 %), ethanol (98 %), 2-propanol (99.8 %) were produced from Sigma-Aldrich. To eliminate stabilizers, AlAm underwent purification via aluminum oxide packed chromatographic columns. The deionized water utilized in experimental procedures was purified through an “Aquilon-D301” purification system, ensuring a resistivity level of 18.2 MΩ.

### *Preparation and Modification of Track-Etched Membrane*

For all measurements, PET TM with a thickness of 12 μm, pore density of $1\cdot10^6$ pore/cm$^2$, and an effective pore diameter of ~250 nm was used. The membranes were fabricated by irradiating a PET film with $^{84}Kr^{15+}$ ions accelerated to an average energy of ~1.75 MeV/nucleon using a DC-60 ion accelerator (Institute of Nuclear Physics of Kazakhstan), followed by chemical treatment in 2.2M NaOH solution at 85 °C. The samples were rinsed with 5 % acetic acid and DI water and stored in air at room temperature.

The 5×5 cm TM was immersed in a 5 % solution of BP in DMF for 24 hours. Following this treatment, the membranes were rinsed with ethanol and subsequently air-dried. The quantitative determination of BP adsorbed on the surface of PET TM was performed using a spectrophotometric assay. The samples with adsorbed BP were briefly washed with ethanol at room temperature, dried, and then immersed in pure ethanol at 40 °C to desorb loosely bound BP from the membrane surface. The amount of released BP was subsequently measured by UV–vis spectroscopy at 253 nm. The PET TM was placed in a solution containing AlAm. Chloroform, 2-propanol, acetonitrile, and deionized water were used as solvents. To eliminate dissolved oxygen, the reaction mixture was purged with Ar. The photo-induced graft polymerization was conducted under irradiation using an OSRAM Ultra Vitalux E27 lamp (UVA: 315–400 nm, 13.6 W; UVB: 280–315 nm, 3.0 W) for different durations.

### *Characterization of the Morphological and Chemical Properties*

The chemical structure of the membrane was investigated through the Fourier Transform Infrared Spectroscopy (FTIR) technique using InfraLUM FT-08. The FTIR spectra were recorded in the range of 400–4000 cm$^{-1}$, with a resolution of 1 cm$^{-1}$ and an average number of 20 scans, by using an attenuated total reflectance (ATR) module. The ATR-FTIR analyses were performed on both sides of the membranes.

The change of morphology and pore size after modification was evaluated by scanning electron microscopy (SEM). Before the analyses, a 10 nm thick layer of gold was applied on the surface. The pore size determination was performed using the Phenom Image Viewer program. The elemental composition of the sample was studied by using the energy-dispersive X-ray spectroscopy (EDX) system Bruker Xflash MIN SVE at an accelerating voltage of 15 kV. The results of the analysis are presented as averages based on three data points.

The number of available amino end-groups was quantified using 500 μmol/L acid orange (AO) solution (in HCl, pH=3). Samples of modified PET TM of 1 cm$^2$ in size were immersed in AO. The adsorption of AO on the membrane surface is carried out for 12 hours. After the samples were removed, it was washed twice in a HCl (pH=3) solution, and dried. The AO was desorbed in 5 ml of NaOH solution (pH=12) for 15 min on a shaker. The concentration of the amino group was determined on UV-vis — spectrophotometer SPECORD-250 at a wavelength of 495 nm according to the calibration curve.

## *Results and Discussion*

Photo-induced graft polymerization is typically carried out by the pre-immobilization of a photoinitiator onto the surface of the membrane to be modified. In this approach, BP is one of the most commonly used photoinitiators [27]. The grafting process was carried out in two stages: in the first stage, the covalent immobilization of the sensitizer BP onto the surface of the PET TM was performed. In principle, upon UV irradiation, BP molecules are first excited to a singlet state, followed by a transition to a triplet state via intersystem crossing [28]. Studies have shown that triplet-state BP and its derivatives can abstract hydrogen atoms from nearby polymer substrates, leading to the formation of surface-bound radicals ($R^{\cdot}$) that are capable of initiating graft polymerization. The resulting benzopinacol radicals ($BP–OH^{\cdot}$) are comparatively less reactive and do not readily participate in free radical polymerization. Instead, they tend to terminate the reaction through radical coupling, rather than propagating the polymer chains [21]. In the second stage, the graft polymerization was conducted in the presence of an AlAm monomer solution. The PET TM surface was modified by scheme presented in Figure 1.

The interaction of BP with the polymer surface occurs through electrostatic and van der Waals forces, and is influenced by the hydrophilic–hydrophobic balance and the crystalline–amorphous characteristics of the polymer. A hydrophilic polymer such as PET can form more ordered and stronger bonds with BP. When BP was dissolved in alcohol, the maximum adsorbed concentration on the membrane surface did not exceed 19 µmol/g. The low concentration is associated with the leaching of adsorbed BP [29]. The dissolution of BP in DMF resulted in an adsorbed BP concentration of 580 µmol/g on the membrane surface [14]. The increase in adsorbed initiator concentration results from BP molecules in DMF permeating the template and forming additional active centers [30]. An increase in the concentration of the photoinitiator of PET TM leads to more efficient AlAm grafting.

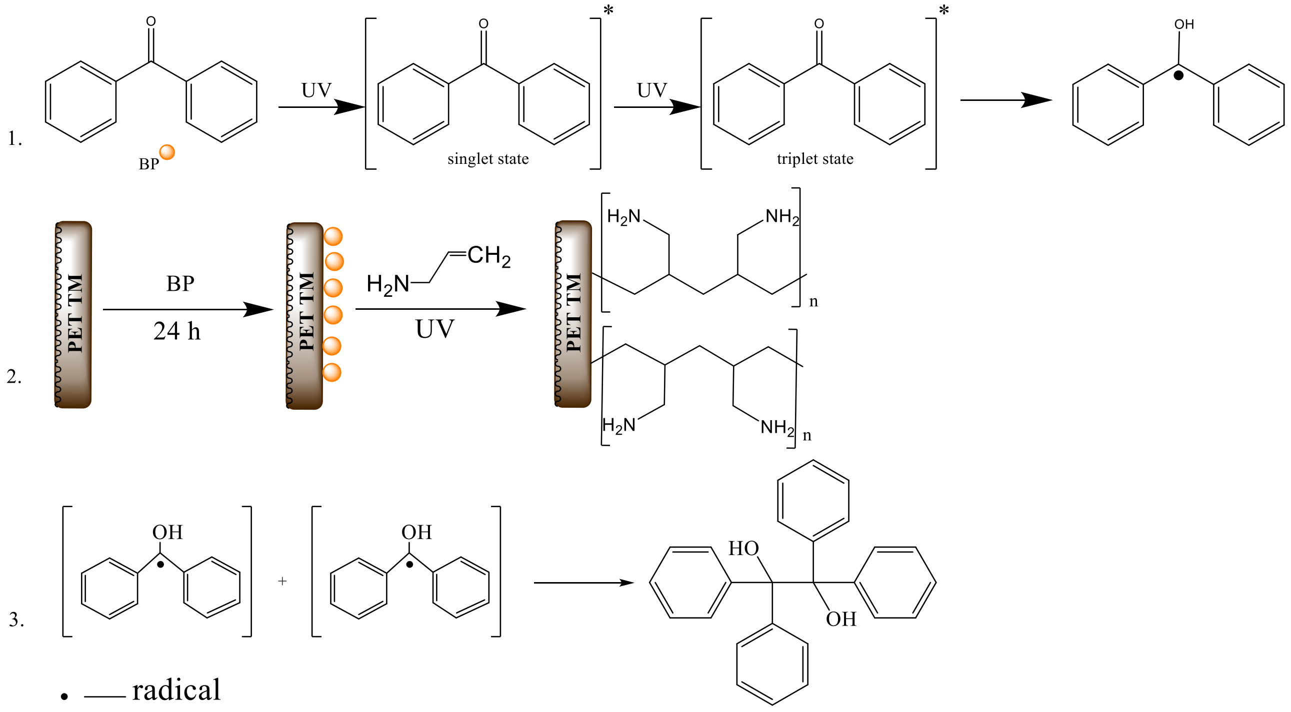

Figure 1. Scheme of modification of PET TM

The polymerization process was impacted by monomer concentration, solvent, irradiation dose, and polymerization time. Firstly, a series of experiments was conducted to determine the optimal solvent for graft polymerization. Allylamine is highly soluble in water, alcohols, and nonpolar solvents. When the reaction was conducted in deionized water, chloroform, and acetonitrile, it led to the decomposition of the sample. The degradation was attributed to the pH level (10-11) of the reaction mixture, where a polymer PET degradation occurs. Additionally, chloroform has a low boiling point, and it easily evaporates from the reaction mixture. The 2-propanol was found to provide a more stable environment, minimizing polymer degradation of the sample.

The distance from UV-irradiation affected the polymerization process, an increase in this distance leads to a reduction in the irradiation dose. The optimal distance from UV-lamp was determined at 10 cm. A reduction to 7 cm adversely affected the membrane quality. The PET TM became brittle and prone to breakage. On the other hand, an increase in the distance to 15 cm resulted in a significantly lower polymerization of AlAm.

The photo-induced graft polymerization was carried out at a range of monomer concentration from 10 % to 50 %. After each experiment, the concentration of amino groups in the PET TM was analyzed. Table 1 shows the variation of the content of amino-groups at different parameters.

According to the data presented in Table 1, an increasing trend in AlAm concentration from 10 % to 50 % leads to an increase in the amino group concentration (10.6 ± 0.31 μmol/g). This suggests that higher monomer content enhances amine group concentration. However, reducing the reaction time from 60 to 45- or 30 min results in a decrease in amino group concentration, indicating that a longer reaction time is necessary for effective polymerization. Further increase in monomer concentration and polymerization time can lead to sample degradation due to increased pH, making membrane removal difficult. This suggests that in order to achieve a high level of amino group functionalization without compromising membrane integrity, an optimal balance between monomer concentration and reaction time is critical.

Table 1

**Results of elemental analysis, pore size, and concentration of amino group for PET TM under various parameters of graft polymerization**

| № | Concentration of AlAm, % | *t*, min | Concentration of amino group, μmol/g | Pore size (from SEM analysis), nm | Concentration of N (from EDX analysis), % |
|---|---|---|---|---|---|
| 1 | – | – | – | 322±21 | – |
| 2 | 10 | 60 | 5.7±0.6 | 305±18 | 2.7±0.1 |
| 3 | 30 | 60 | 6.6±0.1 | 269±25 | 8.9±2.2 |
| 4 | 50 | 60 | 10.6±0.3 | 267±22 | 9.2±0.7 |
| 5 | 50 | 45 | 9.6±0.3 | 303±18 | 8.5±0.1 |
| 6 | 50 | 30 | 5.8±0.3 | 304±19 | 2.2±0.1 |

The ATR FTIR spectra of the pristine and modified PET TM are presented in Figure 2. The adsorption bands at 3100–2800 cm$^{-1}$ have been attributed to aromatic and aliphatic –C–H bonds stretching, 1719 cm$^{-1}$ to the ester carbonyl bond, 1240 cm$^{-1}$ to the ester group stretching [17]. After polymerization FTIR spectra shows the appearance of a small peak at 3365 cm$^{-1}$ corresponding to the N–$H_2$ stretching vibration [18]. The intensity of this peak increases slightly with increasing monomer concentration.

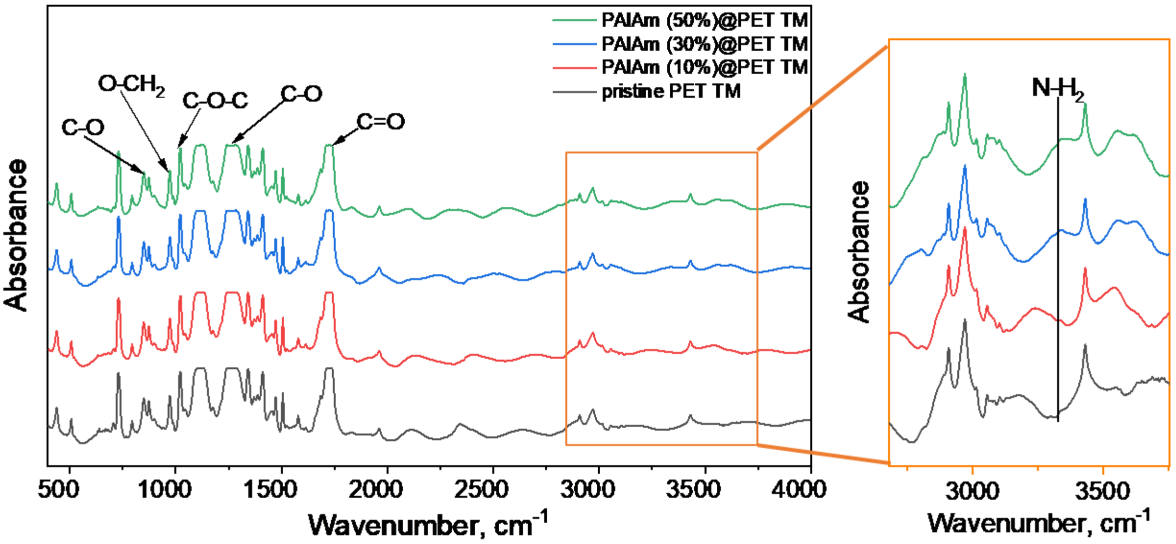

Figure 2. ATR FTIR spectra of the PET TM without and with layer modification

The morphological differences of the functionalized membranes were examined using SEM, with the corresponding results presented in Figure 3. The microphotographs illustrate that the pores of pristine PET

TM are distinctly visible with well-defined edges. After modification with increasing monomer concentration from 10 % to 50 % the pore size slightly decreased from 305±5 nm to 267±3 nm. Although, according to the obtained EDX analysis results (Table 1), an increase in AlAm concentration leads to a gradual growth of the N content from 2.7±0.1 % to 9.2±0.7 %.

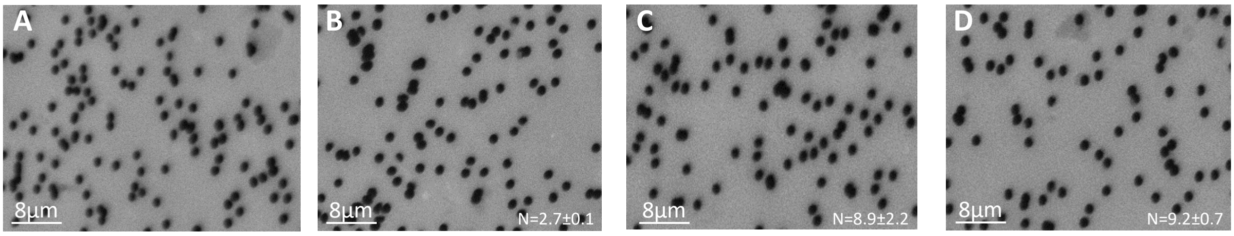

Figure 3. SEM images of pristine PET TM (*A*) and AlAm functionalized membranes at different concentrations: 10 % (*B*), 30 % (*C*), and 50 % (*D*)

The grafting of AlAm in different polymerization times in the PET TM surface gave rise to the appearance of a new peak in the FTIR spectra related to the AlAm structure, such as at 3365 cm$^{-1}$, which has been attributed to amino group stretching. Figure 4 shows ATR FTIR spectra of pristine and modified membranes in different polymerization times.

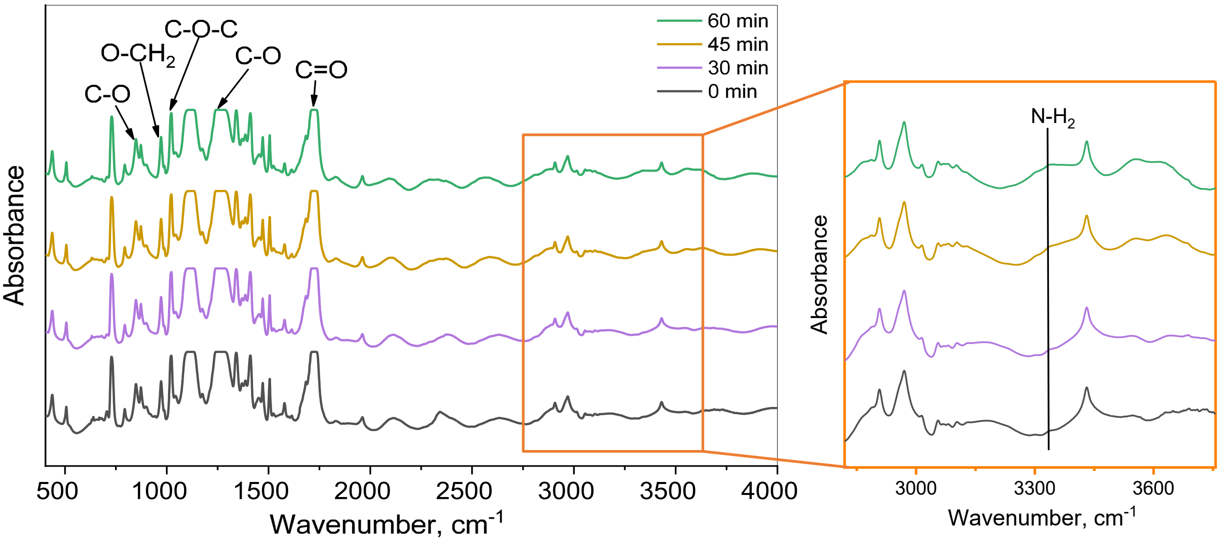

Figure 4. ATR FTIR spectra of pristine and AlAm functionalized membranes at various graft polymerization times

The impact of grafting time on the morphology of TM is illustrated in Figure 5, showing differences between the unmodified and AlAm functionalized surface. The SEM images reveal a smooth surface and a small reduction in the pore size of the TM, which indicates the formation of an AlAm polymer layer. As shown in Table 1, an increase in nitrogen concentration and amino groups is observed with longer polymerization times. At a grafting time of 30 minutes, the nitrogen concentration reached 2.21±0.05 %, while the amino group content, as determined by UV-spectroscopy, was 5.80±0.27 µmol/g.

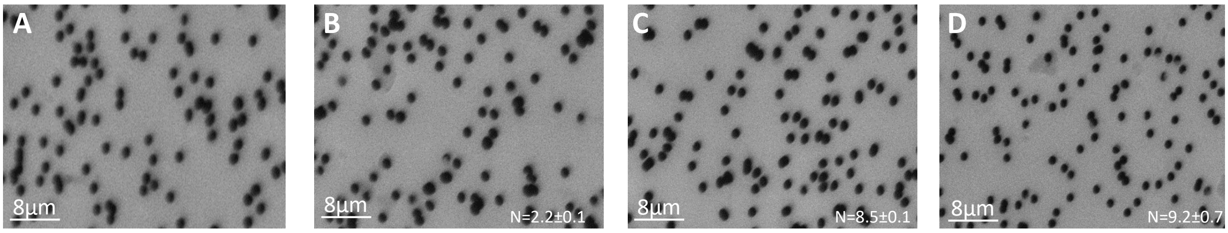

Figure 5. Microphotographs of pristine PET TM (*A*) and PAlAm modified PET TM at various grafting times: 30 min (*B*), 45 min (*C*) and 60 min (*D*)

Experimental findings indicate that the most efficient photo-induced graft polymerization of AlAm occurs under a monomer concentration of 50 %, a distance from the UV-lamp of 10 cm, and a reaction time of 60 min. The formation of a polymer layer on the PET TM surface is supported by evidence from SEM micrographs, EDX, and FTIR spectroscopy. The photo-induced graft polymerization of AlAm in TM offers an approach for introducing primary amine groups on the membrane surface and significantly enhancing its functionality. The amine groups increase surface hydrophilicity, allowing for reversible interactions with gases like $CO_2$ through carbamate formation, and providing reactive sites for further chemical modification or biomolecule immobilization. The next stage of this research, the transport properties, such as gas and water permeability, will be investigated to evaluate the performance of the modified TM in practical applications. Additionally, due to the high content of amino groups on the surface of PET TM, this modification can be utilized as a template for the synthesis of MOFs and for the development of adsorbents for carbon dioxide capture and removal of different water pollutants.

## *Conclusions*

In conclusion, the results confirm that PET TM can be effectively modified by photo-induced AlAm graft polymerization, leading to a market increase in the concentration of surface amino functionalities. The optimal parameters for the photo-induced graft polymerization of AlAm were determined as a UV lamp distance of 10 cm, an AlAm concentration of 50 %, and a grafting time of 60 min, ensuring the preservation of the porous membrane structure, as confirmed by SEM-EDX, ATR FTIR, and UV spectroscopy. The modification enhances the chemical functionality of PET TM, offering opportunities for a variety of applications. These modified membranes can be utilized for $CO_2$ separation and capture, water purification and electrochemical and biosensing technology.

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## *Author Contributions*

All authors contributed to the preparation of the manuscript. All authors approved the final version of the manuscript. CRediT: **Aigerim Khairatovna Shakayeva** investigation, writing — original draft, data curation, visualization; **Dias Dumanuli Omertasov** — investigation; **Zhanna Kalanbekovna Zhatkanbayeva** — supervision; **Ainash Turlybekovna Zhumazhanova** investigation, data curation; **Maxim Vladimirovich Zdorovets** — supervision, data curation; **Ilya Vladimirovich Korolkov** conceptualization, supervision, writing review and editing, project administration, funding acquisition.

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### *Conflicts of Interest*

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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