

Article

Graphene Oxide-Doped Polycarbonate Track-Etched Membranes Grafted with Poly(acrylic acid) as Gel Polymer Electrolytes for All-Solid-State Supercapacitors

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Abstract

Track-etched polycarbonate membranes (PC-TeMs) offer a structurally supported and well-defined nanoporous architecture, but their intrinsically low ionic conductivity limits their direct application as solid-state electrolytes in supercapacitors. Here, we grafted poly(acrylic acid) (PAA) into the cylindrical nanopores of PC-TeMs via reversible addition–fragmentation chain-transfer (RAFT) polymerization, incorporating graphene oxide (GO) at 0–1 wt% to enhance ion transport and structural stability. Structural, spectroscopic, and morphological analyses indicated effective PAA grafting along the nanochannels and homogeneous GO dispersion within the membrane matrix. Electrochemical tests revealed a non-linear dependence of performance on GO content: an optimal GO content of 0.5 wt% with PAA grafting delivered the highest ionic conductivity ($0.57 \text{ mS}\cdot\text{cm}^{-1}$) and specific capacitance ($1.14 \text{ F}\cdot\text{g}^{-1}$ at $5 \text{ mV}\cdot\text{s}^{-1}$). This corresponds to improvements of approximately $\sim 6\times$ and $\sim 800\times$ compared to the ungrafted GO-free membrane ($0.09 \text{ mS}\cdot\text{cm}^{-1}$ and $1.42 \text{ mF}\cdot\text{g}^{-1}$), and $\sim 3\times$ and $\sim 42\times$ compared to the ungrafted GO-containing membrane ($0.18 \text{ mS}\cdot\text{cm}^{-1}$ and $26.96 \text{ mF}\cdot\text{g}^{-1}$), respectively. In contrast, at a GO content of 1 wt%, pore blockage and a decline in performance were observed. These results demonstrate that finely tuned GO incorporation into RAFT-grafted PAA/PC-TeMs enables dimensionally stable solid-state electrolytes for high-performance solid-state supercapacitors.

Keywords: track-etched membranes; RAFT grafting; poly(acrylic acid); graphene oxide; gel polymer electrolyte; all-solid-state supercapacitor; ionic conductivity



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1. Introduction

The increasing demand for sustainable, safe, and high-performance energy storage systems has intensified research into advanced electrochemical energy storage technologies. Among these, supercapacitors have attracted considerable attention due to their high power density, rapid charge–discharge capability, and long cycle life, positioning them as key components in next-generation energy storage devices [1–3]. In recent years, particular emphasis has been placed on the development of all-solid-state supercapacitors, motivated

by their superior safety, structural integrity, and operational stability compared to liquid-electrolyte-based systems [4–6]. By eliminating volatile liquid electrolytes, solid-state architectures mitigate leakage, evaporation, and thermal runaway risks, thereby enabling safer operation across wider temperature ranges [7–12].

A central challenge in realizing high-performance solid-state supercapacitors lies in the development of efficient gel polymer electrolytes (GPEs) that combine high ionic conductivity with adequate structural integrity [3,13–16]. GPEs play a critical role in facilitating ion transport between electrodes while maintaining dimensional stability, making them indispensable for both conventional energy storage devices and emerging applications such as wearable and flexible electronics [17–25]. Consequently, designing GPE architectures that provide continuous ion-transport pathways without compromising dimensional stability remains a key research focus.

In this context, polycarbonate (PC) track-etched membranes (TeMs) offer a unique platform for GPE design. PC is widely valued for its excellent structural properties, chemical resistance, and processability, making it a robust and cost-effective substrate for electrochemical applications [26–28]. Track-etching technology enables the fabrication of membranes with highly uniform, cylindrical nanochannels of well-defined geometry and controllable dimensions [29,30]. These nanochannels provide a large internal surface area that can be selectively functionalized, offering an effective route to introduce ion-conductive polymers throughout the membrane thickness [31–33]. As a result, PC TeMs can support continuous ion-transport networks while maintaining dimensional stability, an essential requirement for solid-state supercapacitors [34–36].

Functionalization of nanochannels via grafting polymerization is a particularly powerful strategy to impart ion conductivity to otherwise electrically inert polymer membranes. However, conventional free-radical grafting methods often suffer from limited control over polymer growth, leading to pore blockage, surface-localized grafting, and restricted accessibility of the internal nanochannels [37,38]. Controlled polymerization techniques, such as reversible addition–fragmentation chain-transfer (RAFT) polymerization, provide a distinct advantage by enabling precise regulation of molecular weight and polymer architecture [39]. This level of control is especially critical in nanoconfined systems, where preservation of open nanochannels is essential for diffusion-controlled processes such as ion transport [40–42]. Accordingly, RAFT polymerization represents a suitable and robust approach for achieving uniform grafting along the entire length of TeM nanochannels.

Beyond polymer grafting, the incorporation of ion-conductive fillers has emerged as an effective strategy to further enhance electrolyte performance. In addition to its high surface area, graphene oxide (GO) contains abundant oxygen-containing functional groups (e.g., hydroxyl, epoxy, and carboxyl groups), which can facilitate ion transport by providing additional coordination sites and improving interfacial interactions with the membrane matrix [43,44]. Furthermore, GO incorporation can influence the wettability and structural properties of the membrane, contributing to the formation of more efficient ion-transport pathways while also improving its structural stability. In the present work, GO was incorporated into the PC matrix prior to ion irradiation and track-etching, enabling the fabrication of track-etched membranes from composite precursor films rather than conventional single-component polymers. The resulting GO-containing nanochannel architecture was subsequently functionalized with PAA by RAFT-mediated graft polymerization, providing an ion-conductive polymer network within the confined membrane structure. Importantly, RAFT-mediated PAA grafting was performed on the already prepared GO-containing PC track-etched membranes, allowing for the controlled functionalization of a pre-existing GO-modified nanochannel architecture [45–47]. Thus, the novelty of the present approach lies in the integration of GO modification of the polycarbonate matrix, controlled track-etched

nanochannel formation, and PAA grafting within the nanoconfined structure into a single self-supported membrane platform for gel polymer electrolyte applications. To the best of our knowledge, this combination of a GO-doped PC TeM with controlled PAA grafting for solid-state supercapacitor applications has not been previously reported.

However, the effect of GO strongly depends on its concentration, as excessive loading may lead to aggregation and partial obstruction of nanochannels.

In this study, GO-doped composite precursor films were converted into track-etched membranes and subsequently grafted with ion-conductive polymers to establish continuous ion-transport pathways within their nanochannels. Initial investigations focused on poly(vinyl alcohol)-based systems, which are widely reported for their high ionic conductivity [48–51]. However, achieving sufficient functionalization of internal pore surfaces within nanoconfined geometries proved challenging. Poly(acrylic acid) (PAA), which also contains a high density of polar functional groups and strong electrolyte affinity, was therefore selected as an alternative grafting polymer and introduced via RAFT-mediated polymerization to ensure controlled and uniform modification of the nanochannels. The resulting membranes provided a self-supported and ion-conductive gel polymer electrolyte platform for solid-state supercapacitor applications, as illustrated in Figure 1.

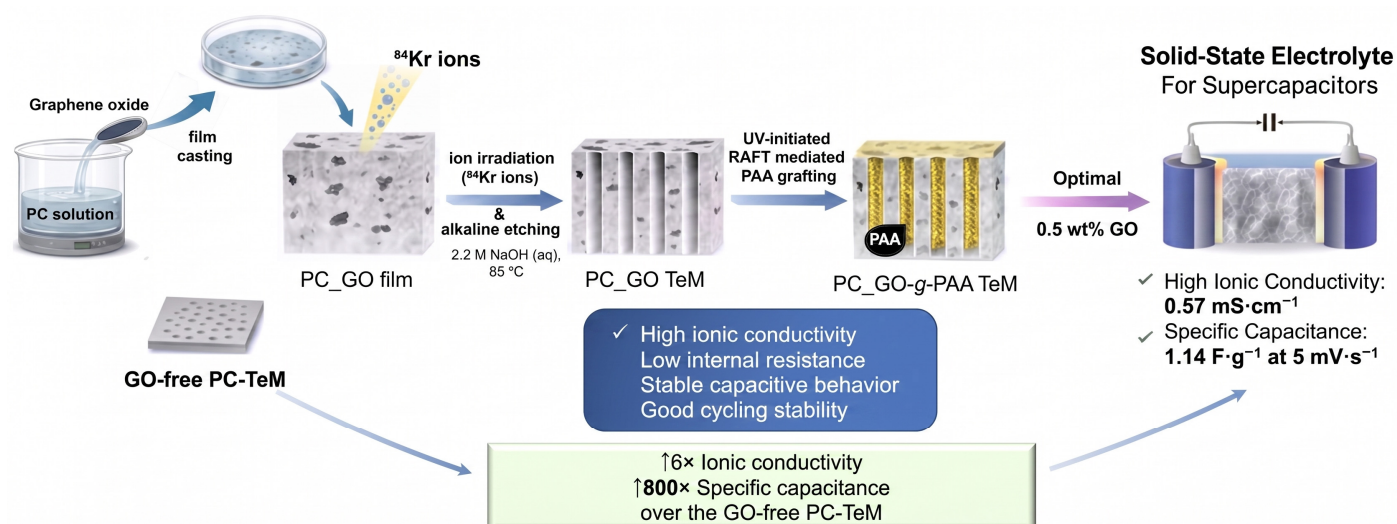


Figure 1. Schematic illustration of the fabrication and application of GO-doped, PAA-grafted polycarbonate track-etched membranes (PC_GO-g-PAA TeMs) as solid-state electrolytes for supercapacitors.

2. Materials and Methods

2.1. Materials

Benzophenone (BP), ethanol, methanol, sodium hydroxide (NaOH), hydrogen peroxide (H_2O_2), vinyl acetate (VAc), acrylic acid (AA), hydrochloric acid (HCl), and the RAFT agent 4-cyano-4-(phenylcarbonothioylthio)pentanoic acid (CPADB) were purchased from Sigma-Aldrich (Steinheim, Germany). Prior to use, VAc and AA were purified by passing them through a basic alumina column to remove polymerization inhibitors. Graphene oxide (GO) and conductive carbon black (TIMCAL Super P) were purchased from Nanografi (Ankara, Türkiye).

2.2. Creation of PC TeM Doped with GO

The fabrication of GO-doped PC track-etched membranes (PC_GO TeMs) was carried out in two successive steps: preparation of GO-doped PC precursor films, followed by ion irradiation and chemical etching. In the first step, PC sheet waste was thoroughly washed with deionized water, dried, and mechanically cut into small pieces. Subsequently, 3 g

of PC was dissolved in 20 mL of dichloromethane under continuous stirring to obtain a homogeneous polymer solution. GO was then added to the PC solution at concentrations ranging from 0.1 to 1.0 wt% with respect to the PC mass. To ensure homogeneous dispersion, GO was subjected to ultrasonic treatment in the polymer solution prior to casting. Ultrasonication promotes exfoliation of GO sheets and prevents their aggregation, enabling stable distribution within the polycarbonate matrix. This approach ensured uniform incorporation of GO, as later confirmed by morphological and compositional analyses.

The resulting PC_GO solutions were cast onto clean glass substrates using a solution-casting technique and dried in a vacuum oven at room temperature for 24 h to remove residual solvent, yielding free-standing GO-doped PC films. These films served as precursor materials for subsequent track-etching.

Ion irradiation of the PC_GO films was carried out using ^{84}Kr ions, with irradiation parameters selected based on the SRIM (Stopping and Range of Ions in Matter) program to ensure continuous latent track formation across the membrane thickness (Table S1, Supporting Information). After irradiation, the samples were stored under ambient conditions prior to chemical etching. Track-etching was performed using a standard alkaline etching procedure in a 2.2 M NaOH aqueous solution at $85 \pm 1^\circ\text{C}$, resulting in the formation of cylindrical nanochannels throughout the membrane. Under these conditions, a final pore density of $9.2 \times 10^7 \text{ pores}\cdot\text{cm}^{-2}$ was obtained for the GO-doped PC track-etched membranes.

The effective pore diameter of the studied TeMs was determined by porometry using gas permeance measurements. The pore diameter was calculated from the measured gas flow using the gas-permeance relationship derived for Knudsen diffusion through cylindrical pores, following the approach established for track-etched polycarbonate membranes (1) [52,53]:

$$Q = \frac{8\pi}{3MRT} \sqrt{\frac{n r^3 \Delta P}{l}} \quad (1)$$

where ΔP —pressure difference, dyn cm^{-2} ; M —molar mass of the gas, g mol^{-1} ; R —universal gas constant, $\text{erg mol}^{-1} \text{ K}^{-1}$; n —number of pores in the membrane sample; r —pore radius, cm; l —membrane thickness, cm; T —temperature, K.

All measurements were performed at least 5 times for every sample of PC_GO TeMs.

2.3. Grafting of AA Within the Nanochannels and Surface of PC TeMs

Benzophenone (BP) is widely used as a photoactive tethering reagent for UV-initiated surface grafting. The amount of BP immobilized on the substrate surface plays a key role in determining the grafting efficiency. To enhance BP immobilization, the surface of both pristine PC TeMs and GO-modified PC TeMs was oxidized to increase the concentration of surface carboxyl groups, following a previously reported procedure [54]. BP immobilization was achieved by immersing the oxidized TeMs in a 5 wt% BP solution prepared in tert-butanol under continuous stirring (150 rpm) at room temperature for 24 h. Subsequently, the membranes were thoroughly washed with deionized water and ethanol to remove unbound BP, dried, and immediately used for grafting experiments.

Grafting polymerization from BP-immobilized TeMs was carried out via UV-initiated RAFT polymerization using 4-cyano-4-(phenylcarbonothioylthio)pentanoic acid (CPADB) as the RAFT agent. The molar ratios of monomer to RAFT agent were fixed at $[\text{AA}]/[\text{CPADB}] = 1000$ for acrylic acid and $[\text{VAc}]/[\text{CPADB}] = 1000$ for vinyl acetate. Monomer concentrations of 10, 20, 30, 40, and 50 vol% were investigated in a total reaction volume of 10.0 mL. Ethanol, propanol, and butanol were employed as reaction solvents.

The polymerization solutions were transferred into sealed flasks containing BP-immobilized TeMs (approximately $1 \times 2 \text{ cm}^2$) and degassed with argon for 10 min prior to UV irradiation. UV-assisted grafting was performed using a 300 W Ultra-Vitalux UV

lamp (Osram, Augsburg, Germany) positioned at a distance of 7 cm from the samples, with irradiation times ranging from 3 to 7.5 h.

After completion of the grafting reaction, the membranes (PAA-g-TeMs and PVAc-g-TeMs) were removed from the reaction medium and thoroughly washed with deionized water using periodic solvent replacement to eliminate ungrafted polymer. The degree of grafting (DG) was determined gravimetrically by measuring the membrane mass before and after grafting using an analytical balance (Mettler Toledo, Columbus, OH, USA) with an accuracy of ± 0.05 mg, according to Equation (2):

$$DG = \frac{W_f - W_i}{W_i} \times 100\% \quad (2)$$

where W_f is the mass of the grafted membrane and W_i is the mass of the BP-immobilized PC TeM.

2.4. Material Characterization

Fourier-transform infrared (FTIR) spectra were recorded using an InfraLUM FT-08 spectrometer (Lumex Instruments, Mission, BC, Canada) equipped with an ATR accessory (GladiATR, PIKE Technologies, Madison, WI, USA). Spectra were collected in the range of $400\text{--}4000\text{ cm}^{-1}$ with a resolution of 2 cm^{-1} over 20 scans to identify chemical functional groups before and after grafting.

The morphology and nanochannel structure of pristine and grafted track-etched membranes were examined using scanning electron microscopy (SEM). Surface and cross-sectional images were acquired using a Phenom ProX Desktop SEM (Thermo Scientific, Waltham, MA, USA) operated at an accelerating voltage of 20 kV, with magnifications ranging from $10,000\times$ to $50,000\times$. Elemental composition and distribution were analyzed using a Hitachi TM3030 SEM (Hitachi Ltd., Chiyoda, Tokyo, Japan) equipped with a Bruker XFlash MIN SVE energy-dispersive X-ray spectroscopy (EDS) system, operated at 15 kV.

Thermal analysis (TGA/DTA) was performed using a Simultaneous Thermal Analyzer (Themys One Plus, Setaram Instrumentation, Caluire, France) under a nitrogen atmosphere with a flow rate of $20\text{ mL}\cdot\text{min}^{-1}$. The measurements were conducted from 25 to $600\text{ }^{\circ}\text{C}$ at a heating rate of $10\text{ }^{\circ}\text{C}\cdot\text{min}^{-1}$ to evaluate the thermal stability and grafted polymer content.

Nitrogen adsorption–desorption isotherms were recorded using a V-Sorb 2800P analyzer (Gold APP Instrument, Xi'an, China) to estimate the specific surface area and pore volume of the membranes. The specific surface area was calculated using the Brunauer–Emmett–Teller (BET) method. These measurements were employed as complementary characterization, while the nanochannel dimensions were primarily evaluated by SEM and porometry due to the macroporous nature of track-etched membranes.

X-ray photoelectron spectroscopy (XPS) analysis was carried out using a Thermo Scientific K-Alpha spectrometer (Waltham, MA, USA) equipped with a monochromatized Al K α X-ray source ($h\nu = 1486.6\text{ eV}$). Survey spectra were recorded with a pass energy of 200 eV and a step size of 1.0 eV, while high-resolution core-level spectra were collected at a pass energy of 30 eV with a step size of 0.1 eV. All measurements were performed at a take-off angle of 90° , and the chamber pressure was maintained below 2×10^{-9} Torr.

A Krüss DSA 100 model CA goniometer (KRÜSS GmbH, Hamburg, Germany) was used to characterize the wettability of pristine PC GO-modified PC TeM samples at ambient temperature by placing a drop of water ($10\text{ }\mu\text{L}$) on the dry sample surface.

The surface morphology of the composite membranes was studied using a scanning probe microscope (SmartSPM-1000, NT-MDT, Novato, CA, USA) in semi-contact mode. An NSG10 rectangular-shaped silicon cantilever (length $95 \pm 5\text{ }\mu\text{m}$, width $30 \pm 5\text{ }\mu\text{m}$, thickness $1.5\text{--}2.5\text{ }\mu\text{m}$, probe tip radius = 10 nm, resonance frequency = 200 kHz) TipsNano (Tallinn,

Estonia) was employed for this purpose. An initial scan of a $10 \times 10 \mu\text{m}^2$ sample area was performed at a speed of $5.0 \mu\text{m/s}$. The data obtained were processed and analyzed using IAPro software (version 3.2.2., 2012, NT-MDT, Novato, CA, USA).

2.5. Electrochemical Measurements

All-solid-state supercapacitors were assembled using carbon-based electrodes separated by a gel polymer electrolyte membrane (PC_GO-g-PAA TeM). Prior to cell assembly, the membranes were soaked in 1-ethyl-3-methylimidazolium tetrafluoroborate (EMIMBF₄, 6.54 M) for 24 h to ensure sufficient electrolyte uptake. After impregnation, the membranes were vacuum-treated at 40°C for 2 h to remove excess surface liquid and residual moisture, while the nonvolatile EMIMBF₄ retained within the porous/granted membrane structure served as the ionic conducting phase. After this treatment, the membranes remained self-supporting and no macroscopically free-flowing liquid phase was observed. The term “all-solid-state” is therefore used here at the device-configuration level to denote a self-supporting, non-flowing gel polymer electrolyte without a separate free-flowing liquid-electrolyte phase, rather than a completely liquid-free solid polymer electrolyte.

The commercial carbon-coated aluminum foil (Nanografi) was used as the electrode material. The aluminum substrate thickness was $20 \mu\text{m}$, and the total measured mass of the electrode (2 cm^2) was 16.0 mg. To determine the exact mass of the active material, the theoretical mass of the aluminum substrate (10.8 mg) was subtracted from the total mass, yielding a total carbon coating mass of 5.2 mg for both sides. Since only one side of the electrode is in direct contact with the solid electrolyte, the actual active mass (m) per single electrode used for specific capacitance calculations was 2.6 mg (0.0026 g), which corresponds to an active material mass loading of $1.3 \text{ mg}\cdot\text{cm}^{-2}$.

Supercapacitor cells were assembled in an argon-filled glove box using coin cell components (case, spring, spacer, and gasket) made of 304 stainless steel (Xiamen Tob New Energy Technology Co., Ltd., Xiamen, China). Electrochemical measurements, including cyclic voltammetry (CV), galvanostatic charge–discharge (GCD), and electrochemical impedance spectroscopy (EIS), were performed at room temperature using a VersaSTAT3 potentiostat (AMETEK Scientific Instruments, Oak Ridge, TN, USA).

The CV tests were conducted with the initial potential (E_i) set to 0.000 V depending on the open circuit potential (E_{oc}) and a scan rate ranging from 5 to 1000 mV/s .

The specific capacitance (C_{sc} , in $\text{F}\cdot\text{g}^{-1}$) was calculated from CV data using the equation

$$C_{sc} = \frac{2x \int IdV}{ms\Delta V} \quad (3)$$

where m is the mass of the active carbon coating (g), s is the scan rate ($\text{V}\cdot\text{s}^{-1}$), and ΔV is the potential window in V, while $\int IdV$ represents the integral or area under the CV curve. The multiplier 2 is introduced because the symmetric supercapacitor cell consists of two equivalent electrodes connected in series, meaning the measured capacitance of the full cell is half the capacitance of a single electrode.

The ionic conductivity of the films (16 mm diameter, round punched) was measured by electrochemical impedance spectroscopy (EIS). The EIS measurements were performed over a frequency range from 100 kHz to 10 mHz with an AC voltage amplitude of 10 mV at open-circuit potential. The conductivity (σ , S/cm) was determined using

$$\sigma = \frac{L}{A \times R} \quad (4)$$

where L is the thickness (cm), A is the contact area (cm^2), and $R = R_b$ (Ω) is the bulk resistance. The bulk resistance was determined directly from the high-frequency intercept

on the real axis (Z') of the Nyquist plot, representing the intrinsic resistance of the ion-conducting gel without the need for complex equivalent circuit fitting.

3. Results

This section examines the structural, physicochemical, and electrochemical properties of poly(acrylic acid)-grafted polycarbonate track-etched membranes prepared from pristine and GO-modified substrates. Emphasis is placed on how GO incorporation and RAFT-mediated grafting affect membrane structure, nanochannel accessibility, and electrochemical performance, beginning with the fabrication and structural characterization of the pristine and GO-modified PC track-etched membranes used as host matrices.

3.1. Fabrication of Pristine and GO-Modified PC Track-Etched Membranes

Pristine PC and GO-modified PC films were first prepared by solution casting prior to ion irradiation in order to obtain their nanoporous track-etched counterparts. For this purpose, pristine PC was dissolved in dichloromethane, followed by the addition of different amounts of GO. Uniform dispersion was achieved using ultrasonic treatment rather than simple mechanical stirring. Ultrasonication facilitates the exfoliation of GO sheets and prevents their aggregation, enabling stable dispersion within the polycarbonate solution. Furthermore, interactions between GO functional groups and polymer chains contribute to additional stabilization of the dispersion. The resulting homogeneous distribution of GO in the matrix is supported by the uniform coloration of the films (Figure 2b) compared to the pristine PC film in Figure 2a.

These cast films served as precursor materials for the formation of track-etched membranes. Subsequently, the films were irradiated with ^{84}Kr ions and chemically etched to generate nanoporous track-etched membranes (TeMs) and detailed information about the irradiation step is provided in the Supporting Information file. Surface SEM images of pristine PC TeMs (Figure 2c,d) and GO-modified PC TeMs (Figure 2e,f) show uniformly distributed, well-defined pores with comparable pore sizes and morphologies. The similarity between pristine and GO-doped membranes indicates that the presence of GO does not hinder ion-track formation or subsequent pore development during chemical etching.

Cross-sectional SEM analysis (Figure 2g) further confirms the formation of straight, cylindrical nanochannels extending continuously through the entire membrane thickness. Importantly, the GO-modified PC TeMs exhibit empty and well-aligned through-pores, demonstrating that GO incorporation does not obstruct the conversion of ion tracks into continuous nanochannels. These observations verify the effectiveness of the track-etching process for GO-doped PC membranes.

The effect of etching time on pore development is summarized in Figure 2h. No through-pore formation was detected for etching times shorter than 120 s, whereas prolonged etching beyond 200 s resulted in partial mechanical degradation of the membranes. For all samples, the pore growth behavior followed a similar trend: increasing etching time led to a proportional increase in pore diameter. Under optimized conditions, pore diameters reached approximately 290–330 nm after 180 s of etching. These conditions were therefore selected for subsequent grafting experiments. Overall, these results demonstrate the successful irradiation and controlled chemical etching of both pristine and GO-modified PC films, yielding mechanically intact track-etched membranes with uniform, cylindrical nanochannels suitable for subsequent grafting and electrochemical studies.

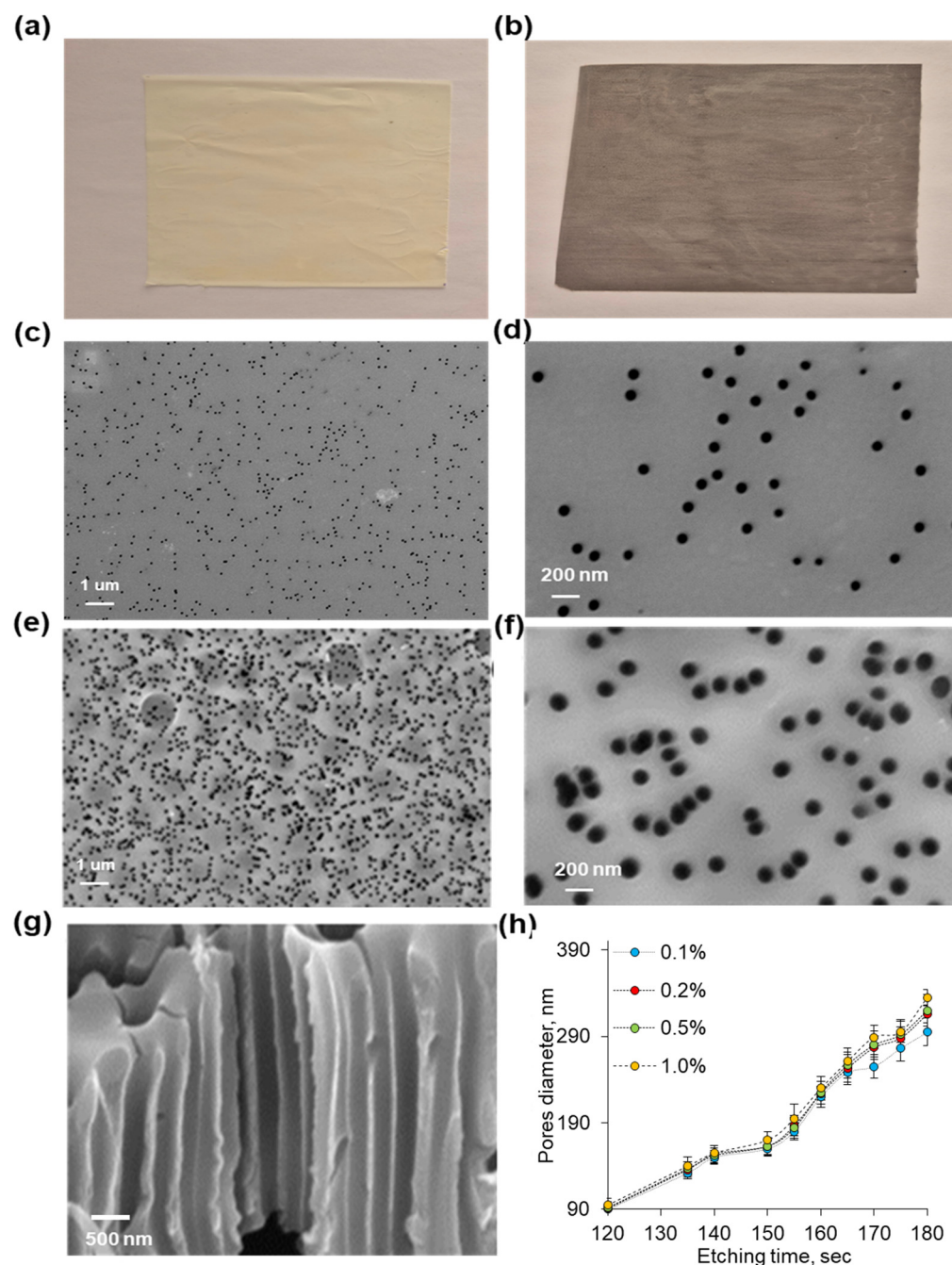


Figure 2. (a) Pristine PC film prior to ion irradiation. (b) GO-modified PC film prepared by solution casting, showing homogeneous brown coloration indicative of uniform GO dispersion within the polymer matrix. (c,d) Surface SEM images of the pristine polycarbonate track-etched membrane (PC TeM) and (e,f) GO-modified PC track-etched membrane (PC_GO TeM) recorded at different magnifications after ion irradiation and chemical etching. (g) Cross-sectional SEM image of the PC_GO TeM showing cylindrical nanochannels through the membrane thickness. (h) Evolution of pore diameter of the PC_GO track-etched membrane as a function of chemical etching time.

Figure 3 presents the XPS analysis of pristine PC and PC_GO hybrid TeMs, revealing their surface elemental composition and chemical states. The survey spectra (Figure 3a) of both samples are dominated by the characteristic C 1s and O 1s signals located at approximately 285 and 532 eV, respectively, in agreement with the chemical structure of polycarbonate [55]. Quantitative analysis, normalized considering the C and O contributions, gave 82.53 at.% C and 17.47 at.% O for pristine PC TeM and 83.84 at.% C and 16.16 at.% O for PC_GO TeM, corresponding to C/O atomic ratios of 4.72 and 5.19, respectively.

No significant additional peaks related to foreign elements were detected, except for a minor Si 2p signal (1.81 at.%) observed only for the pristine PC sample at approximately 103.03 eV, which was attributed to a minor extrinsic surface contribution rather than an intrinsic component of the membrane. The close similarity of the wide-scan spectra further shows that the incorporation of 0.5 wt% GO does not introduce any new detectable elemental species and that its effect is more clearly reflected in the high-resolution spectra.

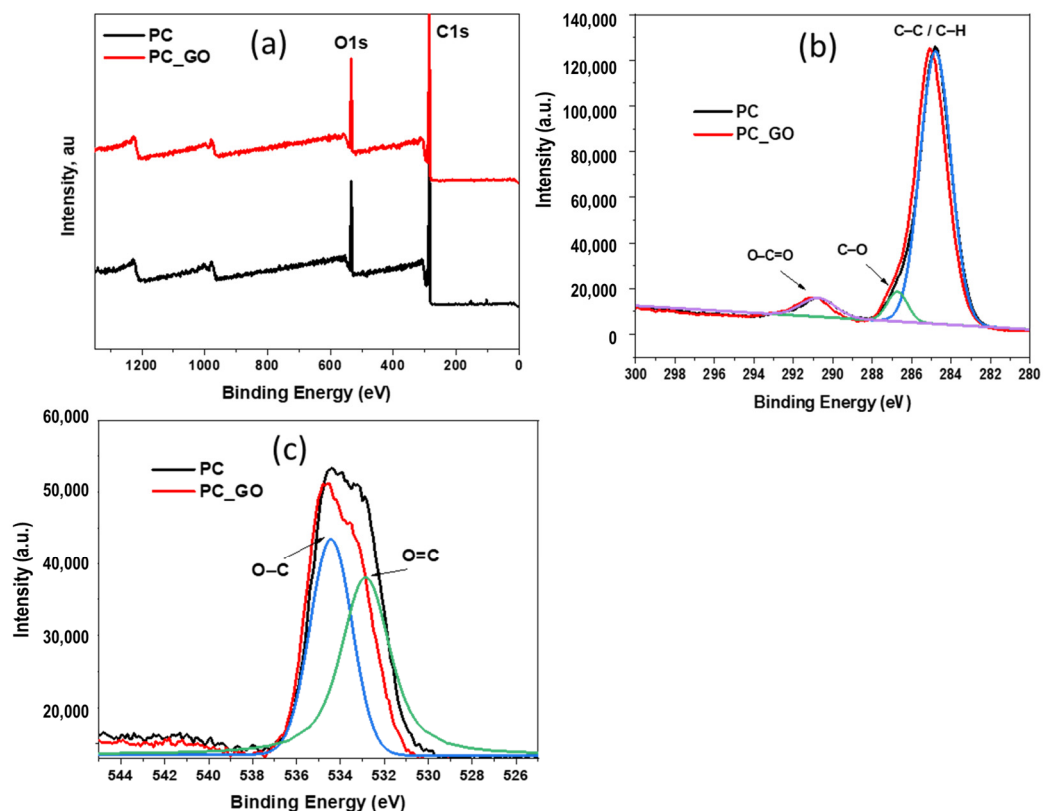


Figure 3. X-ray photoelectron spectra of PC and PC_GO TeMs: (a) survey spectrum, (b) high-resolution C 1s spectrum, (c) high-resolution O 1s spectrum.

The high-resolution C 1s spectra (Figure 3b) are dominated by the main hydrocarbon/aromatic carbon peak centered at about 284.8 eV. For pristine PC (black line), deconvolution resolves three main contributions: the C-C/C-H component at ~284.8 eV (blue), the C-O component at ~286.7 eV (green), and the high-binding-energy feature near 291 eV assigned to the O-C=O carbon of the carbonate groups (purple) [56,57]. After GO incorporation, the C 1s spectrum of PC_GO (red line) remains largely unchanged in terms of peak position and overall line shape, indicating that the carbon chemical environment is still predominantly governed by the PC matrix. Only slight variations are observed in the relative intensity of the oxygen-containing carbon region and the carbonate-related feature, suggesting that the contribution of GO to the overall C 1s response is limited at this loading level.

The high-resolution O 1s spectra (Figure 3c) exhibit an asymmetric profile, indicating the presence of more than one oxygen-containing chemical state. For pristine PC (black line), the O 1s peak can be deconvoluted into two components centered at approximately 532 and 534.1 eV, assigned to the carbonyl oxygen (O=C, green) of the carbonate group and the oxygen in C-O-C linkages (blue), respectively, consistent with the intrinsic oxygen environments of polycarbonate [56,58]. In the PC_GO spectrum (red line), the overall peak position remains nearly unchanged; however, the relative intensity distribution is altered. In particular, the higher-binding-energy side around 534.1 eV becomes more pronounced

compared with pristine PC, indicating an increase in the relative contribution of O-C-type oxygen species [59]. This behavior is consistent with the oxygen-containing functional groups introduced by GO, particularly epoxy and hydroxyl environments overlapping with the O-C region [59,60].

3.2. Grafting Strategy and Optimization on Pristine PC TeMs

Background and need for ion-conductive grafting in nanochannels

Although polycarbonate track-etched membranes provide a dimensionally stable and well-defined nanoporous architecture, their intrinsic ionic conductivity remains insufficient for electrochemical applications such as gel polymer electrolytes. The incorporation of GO into the polycarbonate matrix introduces additional polar functional groups and can partially facilitate ion transport; however, GO modification alone is not sufficient to establish continuous and efficient ionic pathways across the full membrane thickness. In particular, ion transport in track-etched membranes is governed by the accessibility and chemical functionality of the nanochannel walls, and therefore requires the presence of ion-conductive polymer chains distributed not only on the membrane surface but also along the inner walls of the nanochannels.

Grafting ion-conductive polymers onto track-etched membranes therefore represents a critical strategy for transforming these mechanically stable yet electrically limited substrates into functional gel polymer electrolytes, while preserving their ordered nanochannel architecture [40]. By covalently anchoring ion-conductive polymers within the nanochannels and on the membrane surface, grafting enables the formation of a continuous ion-transport network that cannot be achieved through membrane modification or filler incorporation alone. Such covalent immobilization is important for maintaining long-term stability and homogeneous ion-conductive pathways across the full membrane thickness. In this context, the relatively low porosity of track-etched membranes does not necessarily limit their performance in electrochemical applications. Instead, the presence of uniform, straight nanochannels enables efficient and predictable ion transport when the internal pore surfaces are properly functionalized. The grafting of ion-conductive polymers within these channels helps establish continuous ion-transport pathways, effectively compensating for the low volumetric porosity of the membrane. This approach highlights the importance of controlled nanochannel architecture over simply maximizing porosity.

UV-initiated grafting is a versatile method for introducing functional groups or polymers onto polymer substrates. By using UV light to initiate surface reactions, it enables the attachment of desired materials and thereby modifies surface properties and functionality [61]. An increase in the amount of grafted polymer is generally associated with a higher density of functional groups on the substrate surface [62]. However, an optimal degree of grafting (DG) is a pivotal factor in enhancing polymer substrate performance, because it requires a careful balance to ensure that the nanochannel entrances remain unblocked [63]. This balance preserves both a sufficiently functionalized surface and open nanochannels, allowing for efficient grafting along the interior pore walls without compromising accessibility.

To fine-tune the DG, we systematically examined the influential factors of reaction time, monomer concentration, and solvent selection [64]. Given that GO is a costly and specialized material, preliminary optimization studies were first conducted using pristine PC TeMs without GO incorporation. This enabled us to investigate the main grafting parameters, such as solvent type, reaction time, and monomer concentration, in the absence of GO, and subsequently apply these optimized conditions to GO-containing membranes.

Preliminary grafting trials: PVAc/PVA grafting, conventional grafting method and limitations

In graft copolymerization strategies, two main approaches are generally distinguished: conventional free-radical grafting and controlled grafting techniques that offer improved control over chain growth and spatial distribution [65]. While conventional grafting is widely used due to its simplicity, controlled methods are often required to achieve uniform functionalization within confined nanostructures and to preserve nanochannel accessibility, which is critical for both pristine and GO-modified PC track-etched membranes [66,67].

In the initial stage of this study, conventional UV-initiated free-radical grafting polymerization was considered due to its simplicity and widespread use in polymer surface modification, where the involvement of RAFT agents is not required [68]. Poly(vinyl alcohol) (PVA) was first considered as a grafting polymer because of its established ionic conductivity and widespread use in gel polymer electrolytes [69]. Owing to the thermodynamic instability of vinyl alcohol, poly(vinyl acetate) (PVAc) was grafted as a precursor, followed by hydrolysis to generate PVA within the membrane structure. Despite systematic variation in solvent, monomer concentration, and irradiation time, the resulting degree of grafting remained below 2%, which is insufficient to form a continuous ion-conductive network across the membrane thickness.

This limited grafting performance can be attributed primarily to the strong competition between homopolymerization in solution and surface-initiated grafting, a well-documented limitation of conventional free-radical systems [70]. In addition, unfavorable interfacial interactions between the PC substrate and the vinyl acetate monomer likely hindered effective monomer diffusion into the nanochannels, thereby preventing uniform functionalization of the internal pore surfaces. As a result, the formation of a continuous ion-conductive network across the PC TeM could not be achieved. Consequently, PVAc/PVA was deemed unsuitable for the targeted electrolyte design.

PAA was subsequently selected owing to its high electrolyte uptake capacity and excellent ionic conductivity, which make it particularly attractive for supercapacitor applications [71]. PAA-based GPEs exhibit high alkaline electrolyte uptake and ionic conductivity, making them promising for supercapacitor applications [72]. However, due to their tendency to absorb significant quantities of water, PAA-based GPEs often suffer from reduced mechanical strength, resulting in inadequate physical support between the battery electrodes and a significant increase in ohmic polarization [73]. To address this limitation, PAA was grafted onto PC TeMs in order to exploit the rigid membrane framework as a mechanical support, while simultaneously enabling ion-conductive functionality throughout the membrane cross-section [74,75].

Initial PAA grafting experiments were carried out using conventional UV-initiated free-radical polymerization. As illustrated in Figure 4, this approach resulted in uncontrolled polymer chain growth, leading to pronounced clustering of PAA on the membrane surface and likely restricting access to the nanochannel entrances [45]. Although higher overall grafting levels were achieved compared to PVAc, a significant reduction in membrane porosity was observed at early stages of polymerization [76]. This behavior suggests that the formation of high-molecular-weight polymer chains and the absence of growth control hindered monomer diffusion into the nanopores, restricting grafting predominantly to the membrane surface [77]. These observations clearly indicate that conventional grafting fails to provide the level of control required to preserve nanochannel accessibility in PC TeMs.

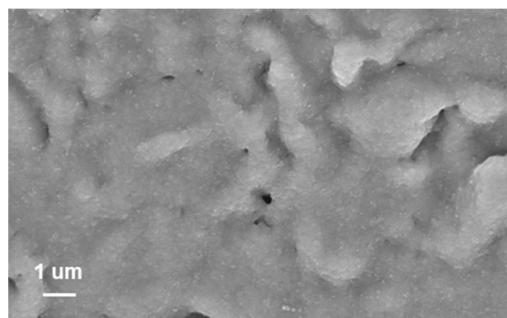


Figure 4. Grafting of PAA on the surface of PC TeM by conventional free radical polymerization (solvent: ethanol, duration: 6 h, monomer concentration: AA, 20% (*v/v*)).

RAFT-mediated PAA grafting and optimization

The severe loss of open nanochannels observed in Figure 4 highlights a drawback of conventional grafting for diffusion-controlled applications such as ion transport. In addition, high degrees of surface grafting were accompanied by reduced membrane transparency and flexibility, serving as macroscopic indicators of excessive surface-localized polymer accumulation rather than uniform internal functionalization. These macroscopic changes are consistent with the microscopic observations of pore blockage and confirm that polymer growth predominantly occurs at the membrane surface, as discussed above.

To overcome these limitations, reversible addition–fragmentation chain transfer (RAFT) polymerization was adopted as a controlled grafting strategy. RAFT polymerization is a well-established controlled radical polymerization technique that enables linear chain growth as a function of monomer conversion, offering precise control over molecular weight and polymer architecture [78,79]. While conventional grafting techniques can introduce substantial chemical modification, their lack of control over chain growth and architecture often limits their applicability in nanostructured systems [80]. In the present work, maintaining open and accessible nanochannels was identified as a primary design requirement, particularly for ion-conduction-dependent applications [81]. RAFT polymerization addresses this requirement by suppressing uncontrolled chain propagation and promoting uniform grafting along the nanochannel walls, thereby enabling comprehensive functionalization without pore blockage [82].

Following the selection of RAFT polymerization, systematic optimization of the grafting conditions was undertaken. The influence of solvent type was first investigated, as the solvent must not dissolve the PC substrate while still providing sufficient interaction to facilitate monomer diffusion into the nanochannels. Additionally, the solvent must effectively solubilize the acrylic acid monomer, the growing PAA chains, and the RAFT agent. Based on these criteria, ethanol, propanol, and butanol were examined. Among them, ethanol resulted in significantly higher grafting efficiencies after 6 h of polymerization in a 20% (*v/v*) monomer solution, as shown in Figure 5a. Notably, membranes grafted in ethanol retained their transparency, providing qualitative indication of less severe surface-localized polymer accumulation.

Subsequent optimization focused on polymerization time and monomer concentration. As depicted in Figure 5b, increasing monomer concentration resulted in a higher degree of grafting, particularly at lower concentrations; however, concentrations exceeding approximately 30% (*v/v*) led to increased membrane fragility, which is consistent with excessive polymer accumulation at or near the membrane surface. Figure 5c shows that the degree of grafting increased with polymerization time, reflecting ongoing grafting reactions, before reaching a plateau at longer irradiation durations. Prolonged irradiation times (≥ 7.5 h) led to a deterioration of mechanical properties, highlighting the importance

of balancing grafting efficiency with structural stability. Accordingly, a monomer concentration of 30% and a polymerization time of 6 h were identified as optimal conditions for subsequent experiments.

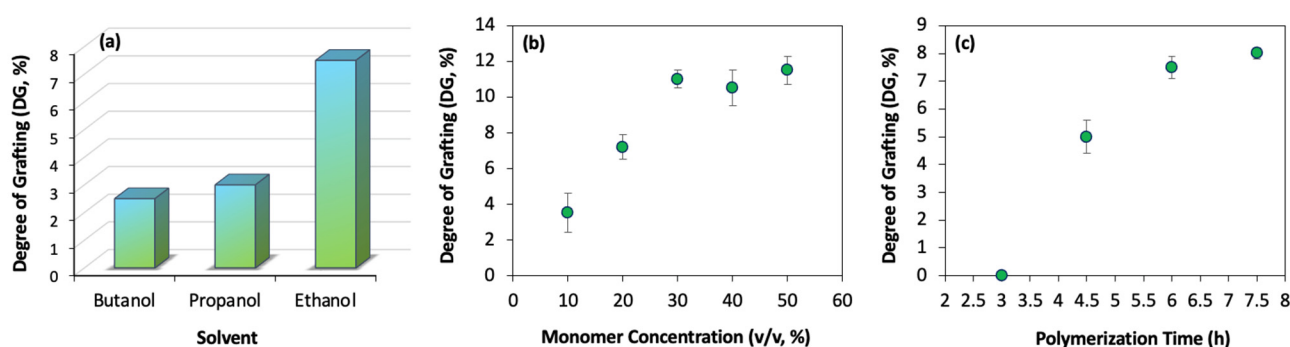


Figure 5. (a) The impact of solvent selection on grafting (AA: 20%, *v/v*, time: 6 h), (b) the effect of monomer concentration on the degree of grafting (solvent: ethanol, time: 6 h), and (c) the influence of polymerization time on the degree of grafting (solvent: ethanol, AA: 20%, *v/v*), [AA]/[CPADB] = 1000). Error bars represent the standard deviation (SD) of DG values from three independent syntheses (*n* = 3).

Our optimization endeavors, focusing on the parameters of solvent, time, and monomer concentration, found further validation through FTIR results, as depicted in Figure 6. The chemical structure of the PC track-etched membrane forming the carrier matrix was first verified to assess the integrity of the polymer backbone after irradiation and grafting. The FTIR spectrum of the pristine PC_GO membrane displays the characteristic peaks of the PC membrane, corresponding to the carbonate group C=O stretching vibration at approximately 1773 cm^{-1} , aromatic C=C stretching vibrations at 1501 cm^{-1} , C–O–C stretching vibrations near 1224 cm^{-1} , C–O stretching at 1015 cm^{-1} and aromatic C–H bending vibrations around 883 cm^{-1} [83].

The FTIR results of PAA-grafted PC samples show that, after irradiation and subsequent grafting, these characteristic bands remain present without significant shifts or intensity loss, and no additional peaks associated with polymer degradation are observed. This confirms that the polycarbonate backbone is preserved under the applied modification conditions. PAA-grafted PC samples were examined by FTIR spectroscopy. The appearance of a new carbonyl band at 1698 cm^{-1} after grafting is assigned to the C=O stretching vibration of carboxylic acid groups in PAA [84]. Its position at lower wavenumbers compared to the carbonate C=O band of PC indicates the successful introduction of carboxyl functionalities onto the membrane surface. Minor shifts in this band are attributed to hydrogen bonding, carboxyl group dimerization, and variations in the local chemical environment [85]. In addition, the broad band observed in the $3000\text{--}3600\text{ cm}^{-1}$ region after grafting is consistent with O–H stretching vibrations of carboxylic acid groups and hydrogen-bonded species. The increase in intensity and breadth of this band with increasing monomer concentration and reaction time reflects the growing contribution of PAA to the surface chemistry. To minimize ambiguity arising from adsorbed moisture, changes in this region were evaluated alongside the evolution of the carbonyl bands and gravimetrically determined grafting yields [86]. Consistent with these observations, the intensity of the PAA-related carbonyl band increases systematically with monomer concentration (Figure 6a) and polymerization time (Figure 6b), in good agreement with the gravimetrically determined grafting yields.

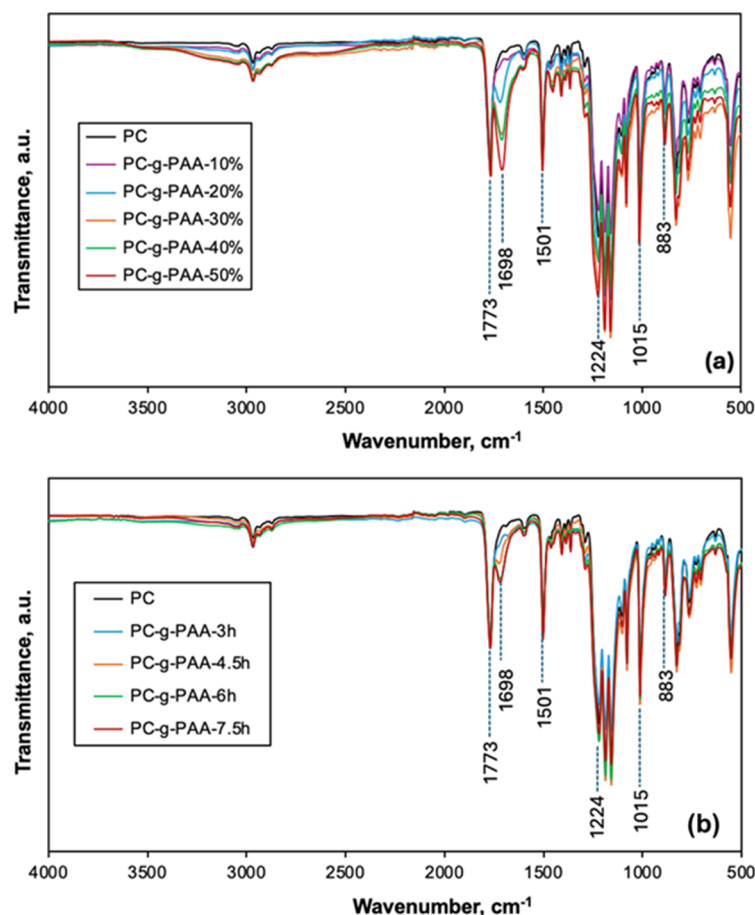


Figure 6. FTIR spectra of PAA-grafted PC TeMs (PC-g-PAA TeM) obtained as a function of (a) monomer concentration (solvent: ethanol, time: 6 h, $[AA]/[CPADB] = 1000$), (b) reaction time (solvent: ethanol, AA: 20%, v/v , $[AA]/[CPADB] = 1000$).

SEM images of PAA-grafted PC TeMs prepared by RAFT polymerization at different monomer concentrations and reaction times are presented in Figure 7. When comparing Figure 7a,b, the nanochannel diameters gradually decrease with increasing polymerization time at a constant monomer concentration (20% v/v). This progressive reduction in pore diameter provides direct morphological evidence of grafting within the nanochannels.

In Figure 7b–d, obtained after a reaction time of 6 h, grafting is observed to increase both on the membrane surface and within the nanochannels as the monomer concentration increases, as indicated by the systematic narrowing of the channels. This trend is consistent with the grafting behavior shown in Figure 5b. The correlation between decreasing nanochannel diameter and increasing grafting ratio confirms the level of control achieved by RAFT polymerization.

At a monomer concentration of 50%, the nanochannel diameters are significantly reduced, indicating intensive grafting along the channel walls. At these high grafting levels, morphological features associated with surface-localized polymer accumulation become more pronounced. Due to the higher density of radiation-induced functional groups, the nanochannel walls represent the most chemically active regions of the membrane. As a result, initiator adsorption and subsequent grafting reactions are expected to occur preferentially in these regions. In wide-area SEM images, grafting appears to concentrate around the nanochannels and progressively spread across the membrane surface as the degree of grafting increases.

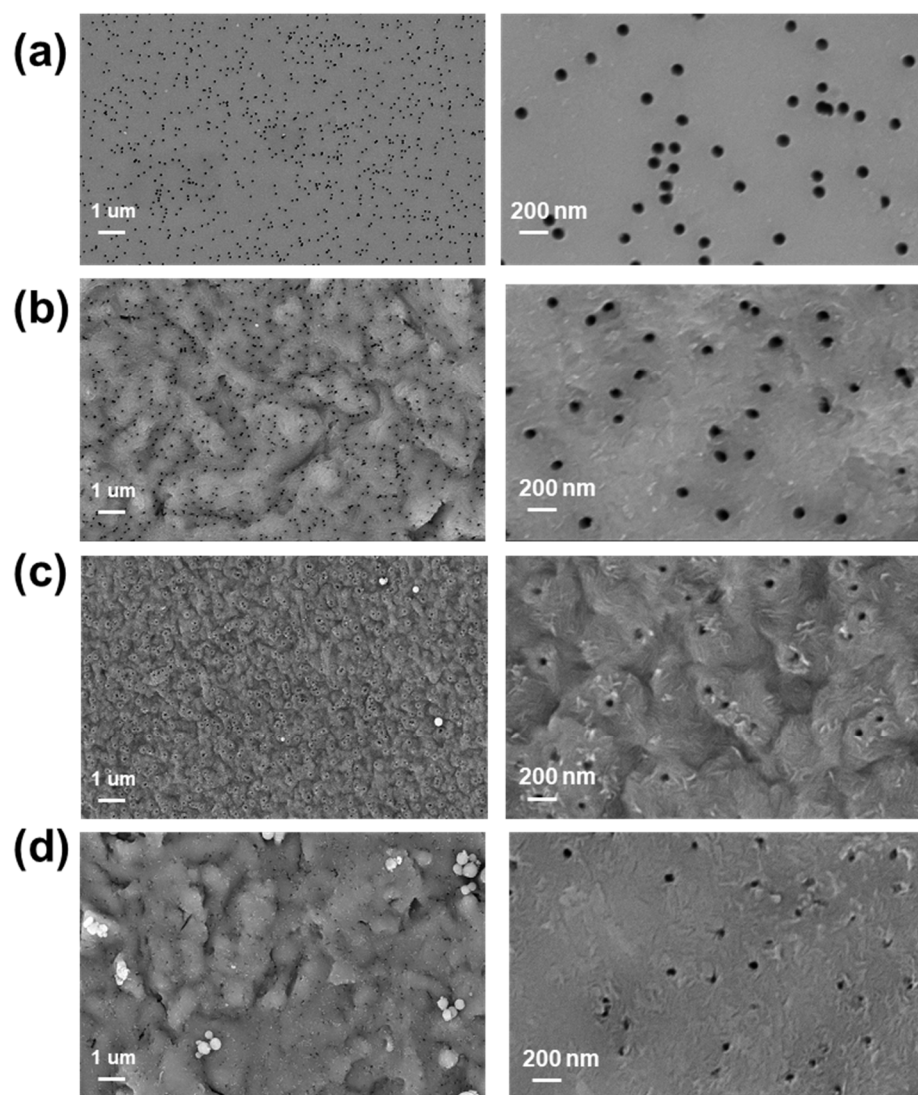


Figure 7. SEM images of PAA-grafted polycarbonate track-etched membranes (PC-g-PAA TeMs) prepared under different grafting conditions: (a) 20% AA, 4.5 h; (b) 20% AA, 6 h; (c) 30% AA, 6 h; (d) 50% AA, 6 h.

Extension of grafting strategy to RAFT polymerization on GO-modified PC TeMs

Following the optimization of RAFT-mediated PAA grafting on pristine PC TeMs, the applicability of the optimized conditions was extended to GO-modified PC TeMs to assess the influence of GO incorporation on grafting behavior. Gravimetric analysis was utilized to monitor the grafting efficiency of PAA onto the GO-modified PC membranes over time. The grafting of PAA was conducted in an ethanol solution containing 30% (*v/v*) monomer for a duration of 6 h, a condition previously identified as optimal for pristine PC membranes. These conditions were directly transferred to GO-modified PC TeMs in order to evaluate the influence of GO incorporation on grafting behavior while minimizing unnecessary consumption of GO. The results, depicted in Figure 8a, demonstrate that the amount of grafted PAA increased over time, stabilizing at approximately 8.5% after 6 h. In contrast, the grafting ratio for the pristine PC membranes without GO was approximately 12%.

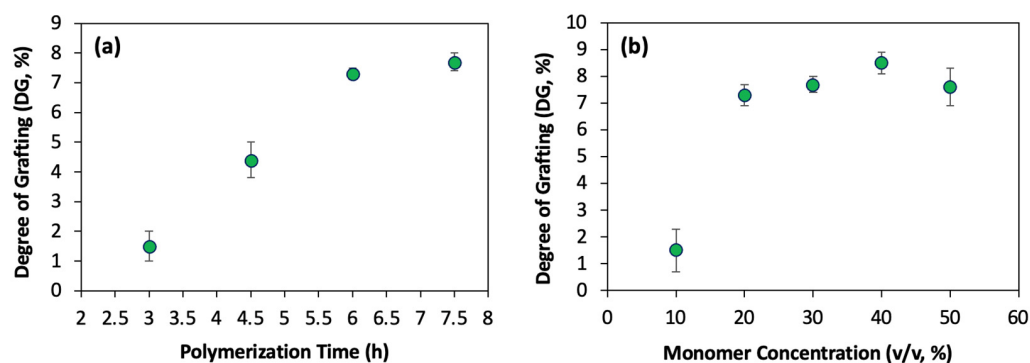


Figure 8. The effect of (a) polymerization time (solvent: ethanol, AA: 30%, *v/v*) and (b) monomer concentration (*v/v*) on the degree of PAA grafting onto PC_GO (solvent: ethanol, time: 6 h, [AA]/[CPADB] = 1000). Error bars represent the standard deviation (SD) of DG values from three independent syntheses (*n* = 3).

To explore the potential for increasing the grafting yield, the monomer (AA) concentration was also varied. However, as shown in Figure 8b, increasing the monomer concentration did not result in a further increase in the grafting yield beyond approximately 9%.

The lower grafting efficiency observed for GO-modified PC TeMs compared to pristine PC TeMs indicates that the presence of GO influences the grafting process. Water contact angle measurements were performed to further examine this effect. The contact angle decreased from 85° for pristine PC TeMs to 71° for GO-modified membranes, indicating an increase in surface hydrophilicity due to the presence of oxygen-containing functional groups of GO (Figure 9).

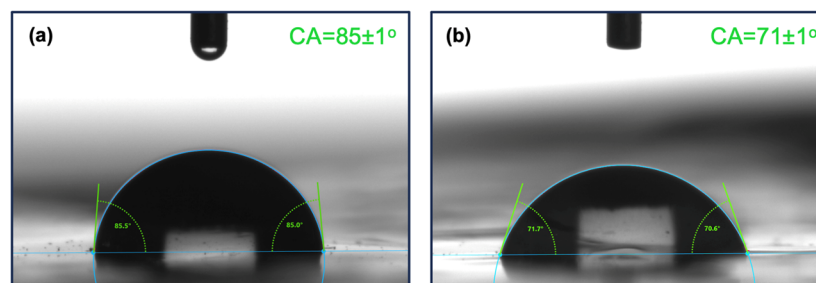


Figure 9. Contact angle measurements of (a) pristine PC TeM and (b) PC_GO TeM (0.5 wt%). The solid green lines indicate the tangents at the three-phase contact points, while the green dashed arcs indicate the measured contact angles. Contact angle values are reported as mean ± standard deviation (SD) from measurements at five different locations on each sample (*n* = 5).

In BP-initiated UV grafting, UV-excited BP abstracts hydrogen from accessible aliphatic C–H groups in PC, generating surface radicals that initiate graft polymerization [87,88]. The lower grafting efficiency observed for GO-modified membranes may therefore arise from changes in interfacial properties rather than from bulk chemical modifications. In particular, the decrease in contact angle indicates increased surface hydrophilicity after GO incorporation, which may alter the local photochemical environment of BP and reduce its hydrogen abstraction efficiency. This interpretation is consistent with literature reports showing that BP reactivity is influenced by the polarity of its surrounding environment [89]. ATR-FTIR results (Figure 10) further support this view, as no significant chemical changes were observed in the polymer matrix. Despite the reduced grafting yield, RAFT-mediated polymerization remained effective for GO-modified membranes.

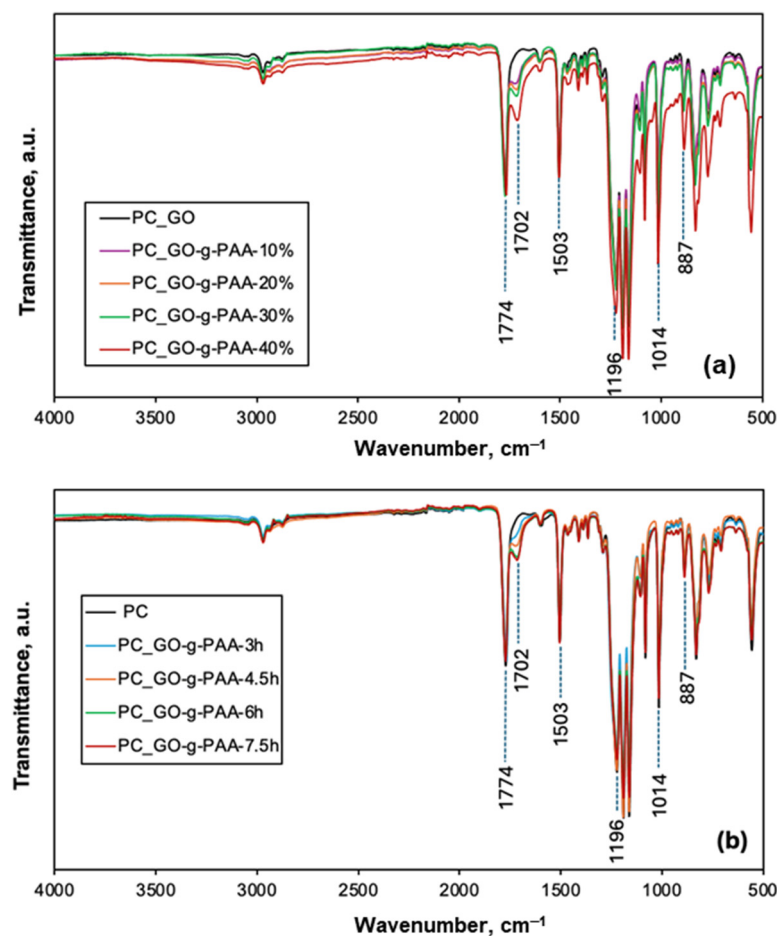


Figure 10. FTIR spectra of grafted PC_GO-g-PAA TeMs synthesized at (a) different monomer ratios (solvent: ethanol, time: 6 h, [AA]/[CPADB] = 1000) and (b) different grafting times (solvent: ethanol, AA: 20%, *v/v*, [AA]/[CPADB] = 1000).

3.3. Structural and Surface Characterization of PC_GO-g-PAA TeMs

FTIR spectroscopy was employed to characterize the PAA-grafted PC_GO membranes (Figure 10). The FTIR spectrum of the pristine PC_GO membrane displays the characteristic peaks of the PC membrane [83], with no pronounced changes due to the low GO content. In the FTIR spectra of the PAA-grafted PC_GO membranes, the carbonyl (C=O) stretching band of PAA appears at approximately 1702 cm⁻¹, observed at lower wavenumbers relative to the carbonate C=O band of the pristine PC_GO membrane. As the grafting time increased, the intensity of the PAA carbonyl band increased up to 6 h, indicating progressive incorporation of PAA onto the membrane. This trend is consistent with the gravimetric grafting results discussed above, confirming the time-dependent nature of the grafting process.

SEM analysis provides detailed insight into the morphological evolution of PC_GO TeMs upon grafting (Figure 11). As shown in Figure 11a, the surface of the pristine PC_GO TeM is characterized by uniformly distributed, circular nanochannels with an average diameter of approximately 270.8 ± 12.9 nm, determined by gas permeability measurements based on Equation (1) as an average of five independent measurements, while the corresponding elemental mapping (Figure 11b) confirms a homogeneous spatial distribution of the constituent elements across the membrane surface. Following PAA grafting, a pronounced and systematic reduction in nanochannel diameter is observed (Figure 11c), indicating that polymer growth occurs not only on the membrane surface but also along the internal walls of the nanochannels. This effect is further highlighted

by the high-magnification images (Figure 11d,e), which visually indicate a decrease in nanochannel diameter from approximately 270 nm to about 150 nm after grafting. A comparable nanochannel constriction was also observed earlier in this work for pristine PC track-etched membranes (Figure 7). In addition, comparable pore-constriction behavior resulting from surface-initiated grafting under confined geometries has been reported for other track-etched membrane systems in the literature [75]. The cross-sectional SEM image (Figure 11f) further confirms the presence of continuous nanochannels throughout the membrane thickness and reveals a layered morphology consistent with the incorporation of GO within the membrane cross-section.

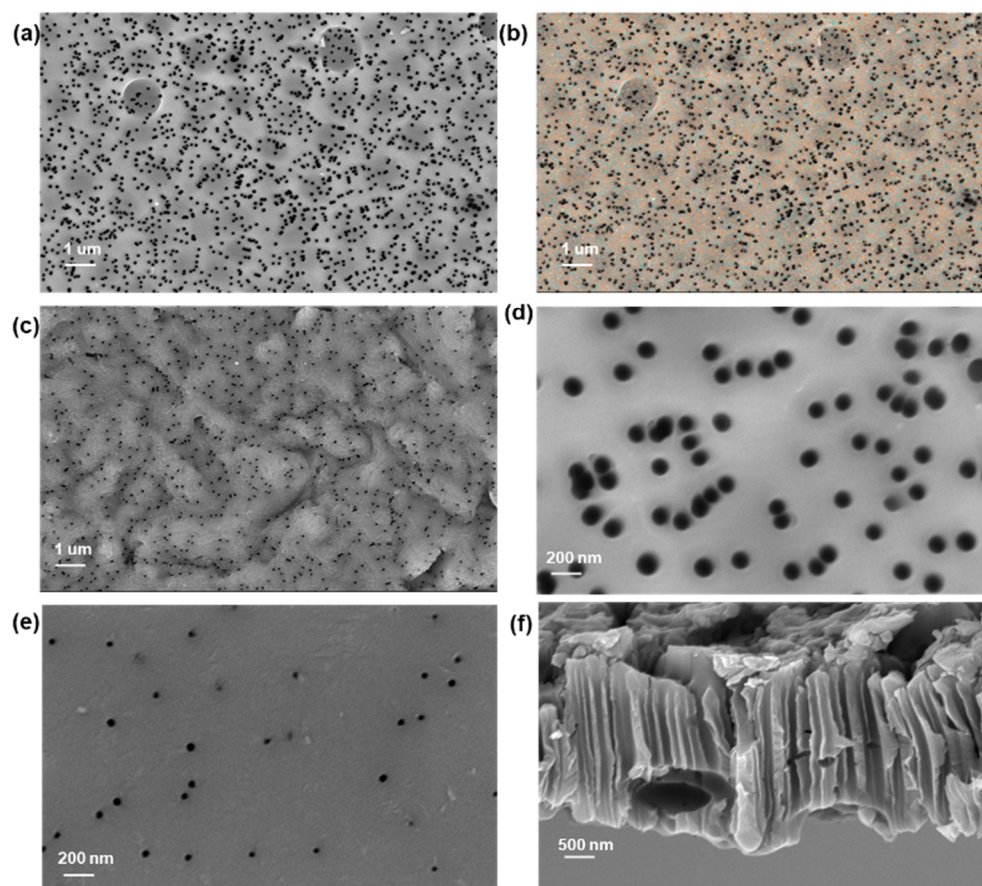


Figure 11. SEM images of GO-modified PC track-etched membranes (PC_GO TeMs) before and after PAA grafting: (a) pristine PC_GO TeM surface and (b) corresponding elemental distribution; (c) surface SEM image of PAA-grafted PC_GO TeM; high-magnification SEM images of nanochannels in (d) pristine and (e) PAA-grafted PC_GO TeMs; (f) cross-sectional SEM image of PC_GO TeM. The average nanochannel diameter of the pristine PC_GO TeM was 270.8 ± 12.9 nm ($n = 5$), determined from gas permeability measurements according to Equation (1).

Further evidence of morphological changes before and after grafting is provided by the AFM images in Figure 12a,b. The pristine and grafted PC_GO membranes show a clear reduction in nanochannel diameter after grafting, consistent with the SEM observations, together with slight changes in surface roughness. In addition, localized height increases are observed around the nanochannel regions in the grafted samples. Although AFM does not directly resolve the full membrane thickness, these topographical changes support the conclusion that grafting alters the surface morphology in and around the nanochannels.

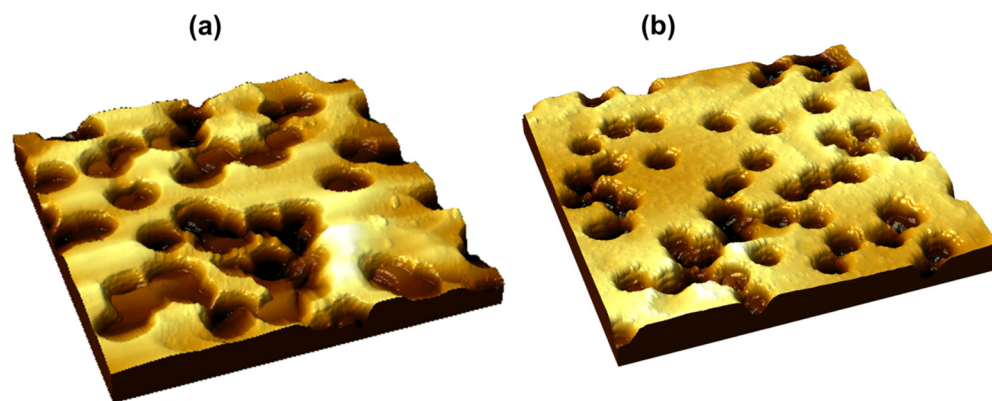


Figure 12. AFM images of (a) pristine PC_GO TeM and (b) PAA-grafted PC_GO TeM recorded over a $10 \times 10 \mu\text{m}^2$ scan area.

To complement the qualitative AFM observations, quantitative surface roughness parameters were determined from the AFM height maps. For the $10 \times 10 \mu\text{m}^2$ scan area, the pristine PC_GO TeM exhibited an average roughness (R_a) of 165.76 nm and an R_{MS} roughness of 206.49 nm. After PAA grafting, the corresponding values were 161.74 and 203.52 nm, respectively. The comparable R_a and R_{MS} values indicate that PAA grafting does not substantially alter the overall surface roughness at the investigated scan scale, although localized topographical changes are evident around the nanochannels.

Thermal behavior and textural properties

The thermal behaviour of the PC_GO-g-PAA samples depends on GO loading. In the TGA curves (Figure 13a), all samples remain nearly unchanged at low and intermediate temperatures, while the main mass loss occurs at higher temperatures. However, the degradation behaviour is not monotonic with increasing GO content. Pristine PC shows the earliest sharp weight loss, whereas the addition of GO shifts the main degradation region to higher temperatures. This shift is most pronounced for PC_GO (0.2 wt%), while the 0.5 and 1.0 wt% GO samples degrade earlier than the 0.2 wt% sample. In addition, the GO-containing samples retain a larger residue at 500–550 °C than pristine PC, indicating increased char yield at the final stage of decomposition [90,91]. The derivative thermogravimetric curves (Figure 13b) show the same trend. The temperature of maximum mass-loss rate is approximately 380 °C for PC, 471.6 °C for PC_GO (0.2 wt%), 430 °C for PC_GO (0.5 wt%), and 390 °C for PC_GO (1.0 wt%). Thus, the highest decomposition temperature is reached at 0.2 wt% GO rather than at the highest GO loading. The 0.2 wt% sample also shows a sharper DTG peak, whereas the 0.5 and 1.0 wt% GO samples exhibit broader peaks. Overall, these results indicate that the thermal stability does not increase continuously with GO content, but reaches an optimum near 0.2 wt% [92,93], while higher GO loadings mainly increase char retention rather than further delaying the main decomposition step [94,95]. The improved thermal resistance observed at low GO loading may be associated with the barrier effect of well-dispersed GO sheets and restricted polymer-chain mobility, as commonly reported for GO-containing polymer systems [96,97]. At higher GO contents, this stabilizing effect becomes less pronounced, which is consistent with the broader derivative thermogravimetric features and previous works [40]. To further examine the thermal behaviour of the GO-immobilized polycarbonate track-etched membrane (0.5%, wt), DTA analysis was also performed (Figure S5). The curve shows no distinct melting or crystallization peaks, consistent with the predominantly amorphous nature of the material. A baseline transition is observed at around ~140–150 °C, which can be assigned to the glass transition temperature (T_g) and is consistent with literature-reported values for polycarbonate [98,99].

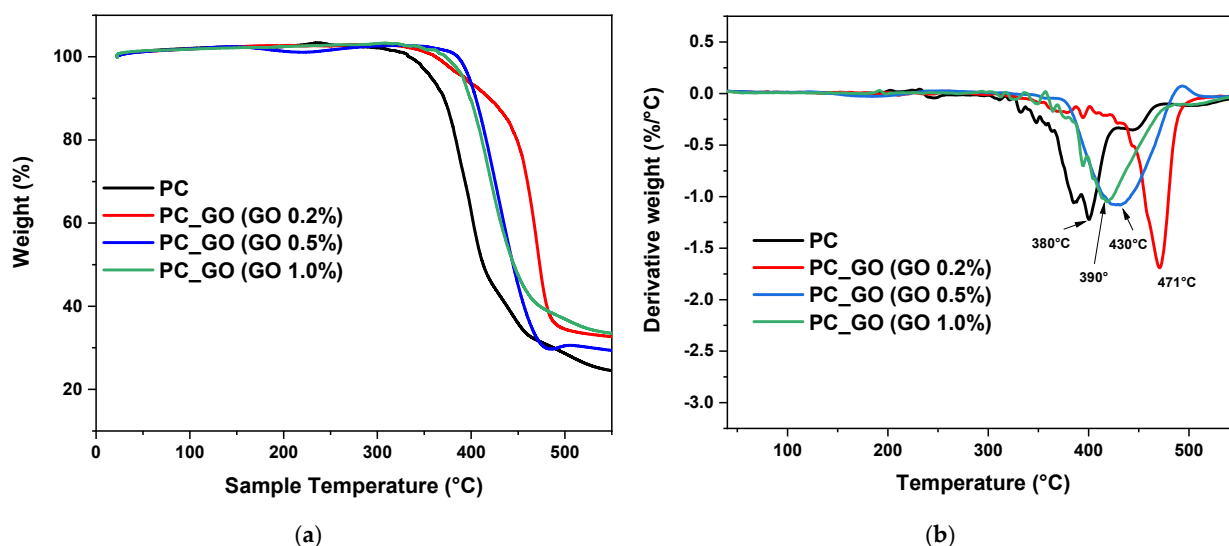


Figure 13. (a) Thermogravimetric and (b) derivative thermogravimetric curves of PAA-grafted PC_GO TeM containing 0, 0.2, 0.5 and 1 wt% GO (N_2 , $10\text{ }^\circ\text{C min}^{-1}$).

To complement the thermal analysis, the textural properties of the membranes were examined by nitrogen adsorption–desorption measurements. The isotherms recorded at 77 K (Figure 14a) show type-IV behaviour with an H3 hysteresis loop for all membranes. According to the IUPAC classification, this hysteresis type is associated with structural or textural heterogeneity rather than a specific pore geometry. In the present system, this behaviour is attributed to heterogeneity introduced by GO incorporation and PAA grafting, and does not imply a transformation of the original cylindrical track-etched pores, which remain the dominant structural feature according to SEM observations. The pristine PC TeM exhibited a low BET surface area ($S_{\text{BET}} = 5.3\text{ m}^2\text{ g}^{-1}$) and a total pore volume of $0.046\text{ cm}^3\text{ g}^{-1}$, consistent with the smooth internal surface and well-defined cylindrical pore structure of track-etched polycarbonate membranes (Figure 14b). The BJH pore-size distribution showed a broad maximum centered at $\sim 35\text{ nm}$, indicating that the native membrane is dominated by wide meso- to macropores with limited internal surface area available for adsorption. The substantial difference between the pore sizes determined by SEM/porometry (270–330 nm) and the BJH analysis ($\sim 35\text{ nm}$) stems from the fundamental limitations of the nitrogen physisorption technique. The BJH method is highly sensitive to mesopores (2–50 nm) but is physically incapable of accurately characterizing macropores larger than 50 nm, as capillary condensation does not occur efficiently in wide, straight cylindrical microchannels. Therefore, the 9–35 nm values obtained from BJH do not represent the primary track-etched channels; rather, they likely arise from secondary nanoscale textural heterogeneity, such as surface roughness and interstitial regions associated with PAA grafting and GO incorporation.

The introduction of 0.2 wt% GO (Figure 14c) resulted in minimal changes in both the isotherm shape and S_{BET} ($5.2\text{ m}^2\text{ g}^{-1}$), along with a slight reduction in average pore diameter and pore volume. This suggests that at low GO loading, the primary effect is limited to surface modification without significant alteration of the overall pore structure, in agreement with reported behavior of graphene-based polymer systems at low filler content [100].

At 0.5 wt% GO, a pronounced increase in S_{BET} ($22.3\text{ m}^2\text{ g}^{-1}$) and pore volume ($0.140\text{ cm}^3\text{ g}^{-1}$) was observed (Figure 14d), accompanied by a broadening of the pore-size distribution. This result indicates increased structural heterogeneity and a larger adsorption-accessible surface. This remarkable increase is attributed to the synergistic

effect of both components: the optimally dispersed GO nanosheets alter the local surface energy of the nanochannels, promoting the formation of a highly textured, rough PAA-grafted layer rather than a smooth polymer film. Similar increases in apparent surface area have been reported for graphene-based polymer composites [101].

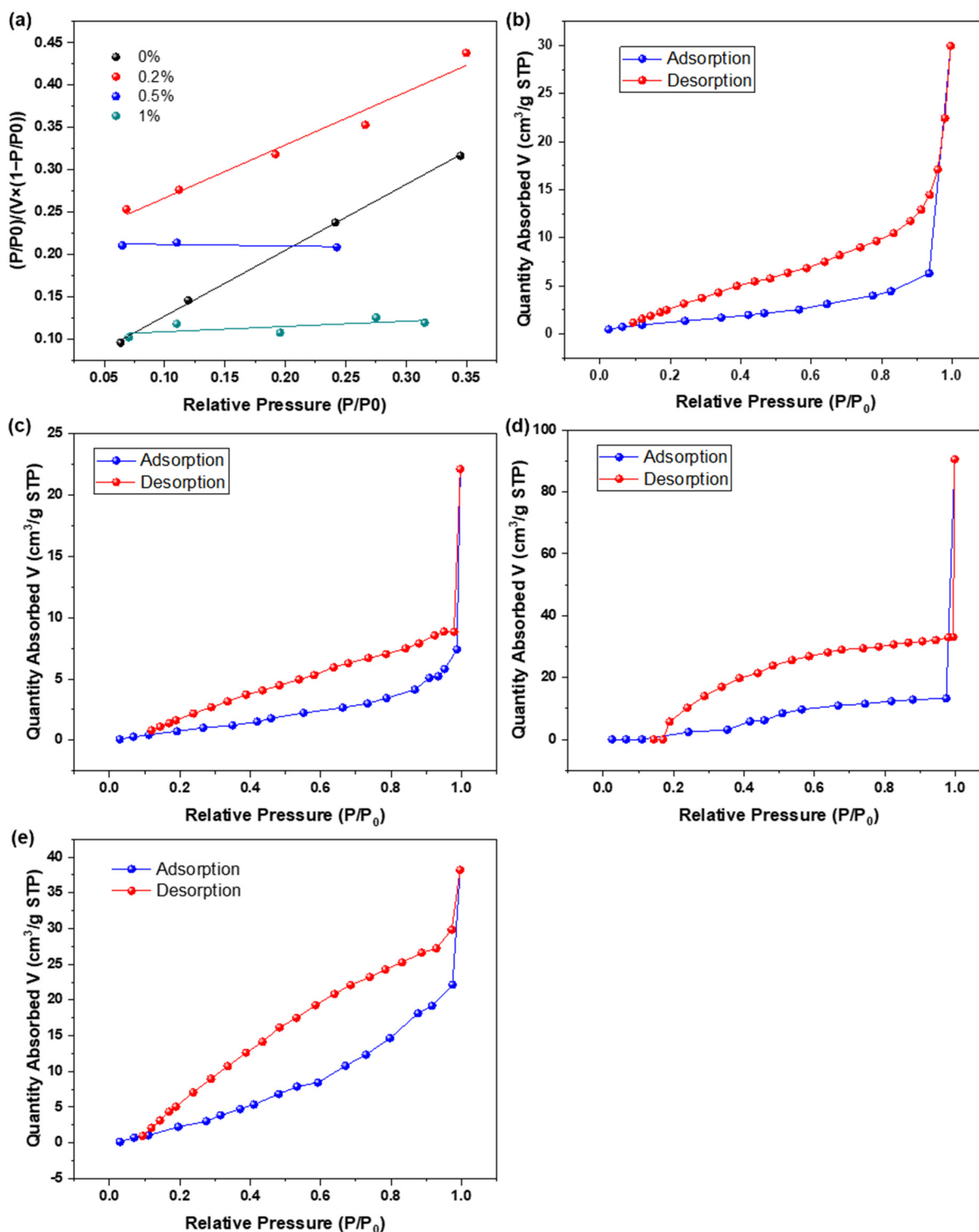


Figure 14. (a) N_2 adsorption–desorption isotherms of PC_GO-g-PAA membranes; corresponding BET/BJH results for membranes containing (b) 0, (c) 0.2, (d) 0.5, and (e) 1.0 wt% GO.

When the GO content was increased to 1 wt%, the BET surface area (Figure 14e) further increased to $26.4 \text{ m}^2 \text{ g}^{-1}$, while the total pore volume decreased to $0.059 \text{ cm}^3 \text{ g}^{-1}$ and the

characteristic pore size shifted toward smaller values (~ 9 nm). This combination of higher surface area and lower pore volume indicates a redistribution of adsorption-accessible space in the dry membrane. These changes reflect modifications in textural organization rather than a fundamental change in the primary cylindrical pore architecture [102,103].

Overall, the BET results (Table 1) indicate a non-monotonic evolution of membrane texture: negligible change at 0.2 wt% GO, maximum development of adsorption-accessible heterogeneity at 0.5 wt% GO (driven by the interplay between well-dispersed GO sheets and the highly textured PAA-grafted chains), and partial densification at 1 wt% GO. This trend is consistent with the electrochemical results, which also show an optimum GO content at 0.5 wt%. It should be emphasized that nitrogen adsorption–desorption characterizes the membranes in their final dried state after modification. Accordingly, the BET-derived surface area and pore-volume parameters should be regarded as descriptors of the geometric and textural characteristics of the porous PAA-grafted architecture, rather than as direct quantitative measures of the ion-transport pathways operative under electrolyte-swollen conditions.

Table 1. BET surface area, total pore volume, and average pore width of PC_GO-g-PAA membranes with different GO contents.

GO Content	0%	0.2%	0.5%	1%
BET surface area, m^2/g	5.26	5.23	22.27	26.40
Total pore volume, cm^3/g	0.04	0.034	0.14	0.05
Average pore width, nm	35.22	26.13	25.14	8.94

3.4. Electrochemical Performance

The electrochemical evaluation of the developed materials begins with the determination of electrolyte ionic conductivity, which is a key parameter for their application in solid-state supercapacitors. Figure 15a,b show the Nyquist plots of PC_GO and PC_GO-g-PAA electrolytes with different GO contents. The nearly vertical shape of the Nyquist plots indicates predominantly capacitive blocking behavior typical of ion-conducting electrolytes. The high-frequency intercept with the real axis was assigned to the bulk resistance of the gel polymer electrolyte. The introduction of GO significantly facilitates ion transport and thus reduces the internal resistance of the gel polymer electrolyte. With increasing GO content, the conductivity of PC_GO-g-PAA gradually increases due to ion transport facilitated by oxygen-containing groups. The maximum conductivity is achieved at a GO content of 0.5%, as shown in Figure 15c. With a further increase in the GO fraction, the conductivity gradually decreases, presumably due to GO aggregation, which hinders ion transport.

Cyclic voltammograms (CVs) of the symmetric cell with the PC_GO-g-PAA (0.5% GO) gel polymer electrolyte were recorded within a potential window of 0–1.0 V at scan rates of 5–1000 mV s^{-1} (Figure 15d). The choice of this composition is justified by electrochemical impedance spectroscopy results, which show that it exhibits the highest ionic conductivity and the lowest resistance (in the Nyquist plot), indicating optimized ion transport both within the electrolyte bulk and at the electrolyte/electrode interface [104]. Across the entire range of scan rates, a systematic increase in current response with increasing scan rate is observed, which is consistent with a predominantly capacitive charge storage mechanism. At low scan rates (5–20 mV s^{-1}), the CV curves approach a quasi-rectangular shape, indicating effective utilization of the accessible surface area and sufficient time for ion migration within the gel. Deviations from the ideal rectangular shape (curve tilting and branch asymmetry) suggest the presence of ohmic contributions and partial diffusion limitations, which are typical of gel-polymer-electrolyte-based solid-state systems [105]. At higher scan rates of 500–1000 mV s^{-1} , the CV loops become broader and the difference

between the anodic and cathodic branches increases. This behavior is associated with reduced time for ion transport and incomplete utilization of the internal regions of the porous structure and/or the ion-conducting channels of the electrolyte. Meanwhile, the absence of pronounced redox peaks over the entire potential window indicates good electrolyte stability and the dominance of capacitive behavior without noticeable parasitic Faradaic processes under the selected conditions [106].

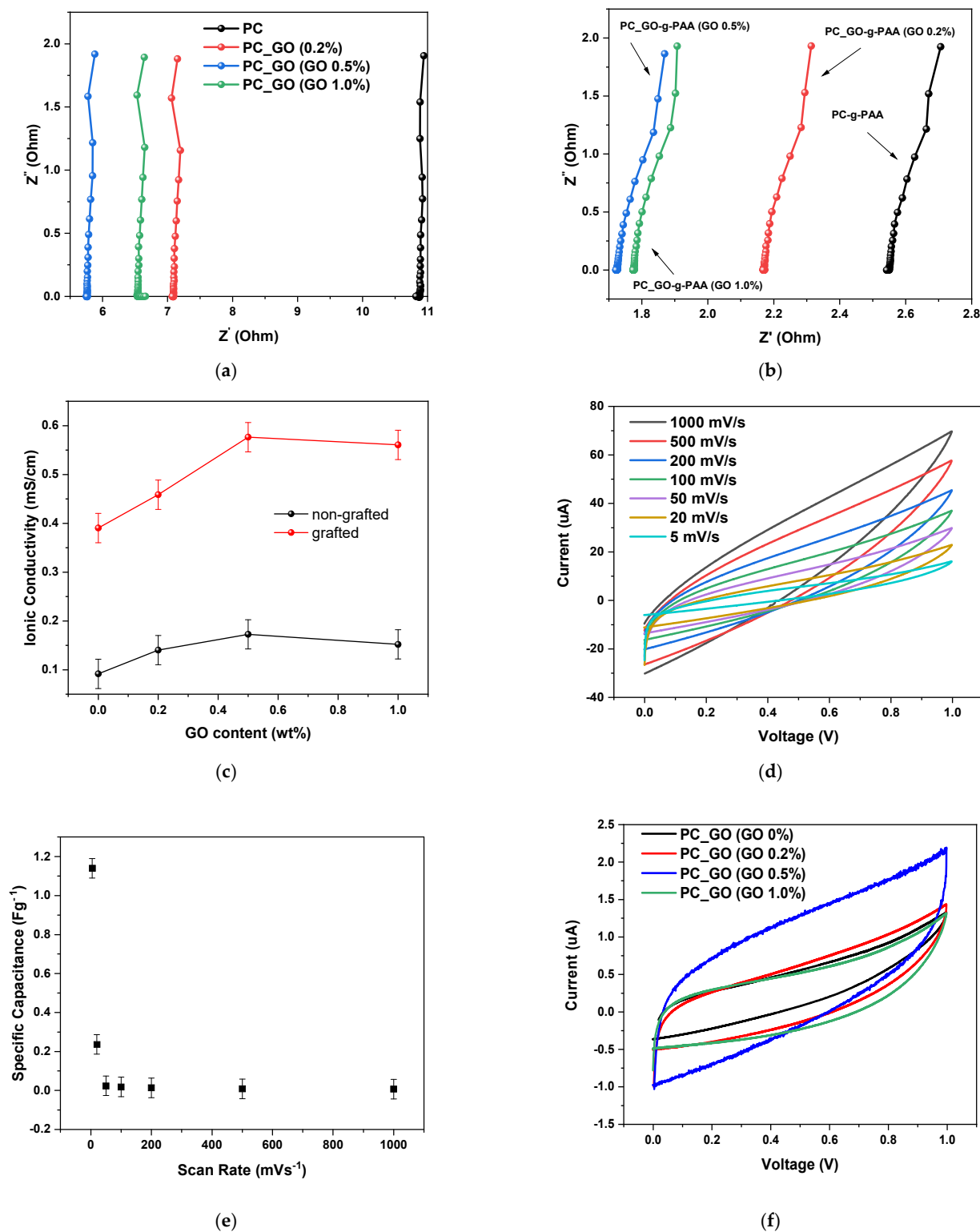


Figure 15. Cont.

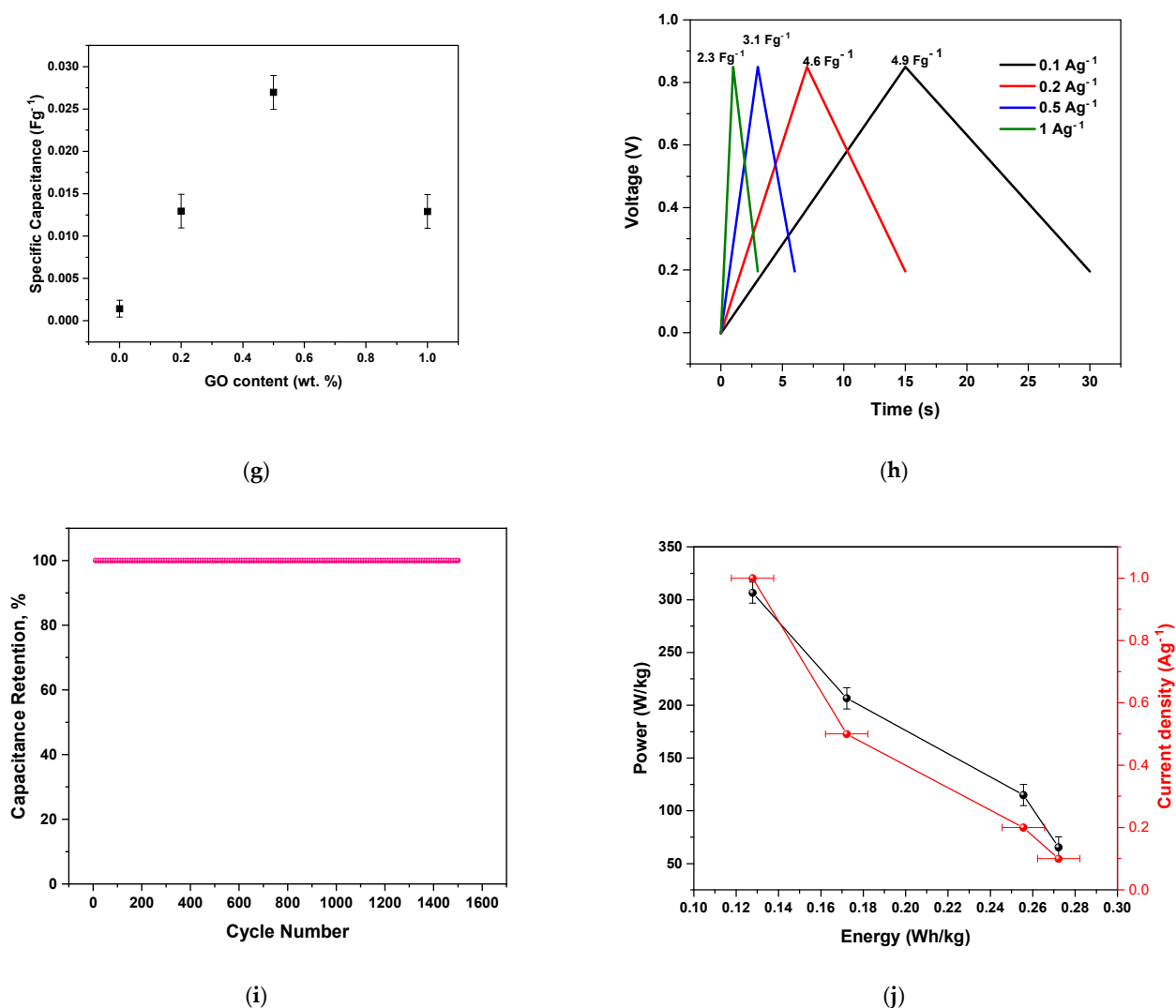


Figure 15. Nyquist plots of PC_GO (a) and PC_GO-g-PAA (b) electrolytes with different GO contents. (c) Ionic conductivity varies according to GO content. (d) CV curves of the PC_GO-g-PAA (0.5% GO) solid-state supercapacitor. (e) Specific capacitance of the optimized PC_GO-g-PAA (0.5 wt% GO) solid-state supercapacitor as a function of scan rate. (f) CV curves of the PC and PC_GO solid-state supercapacitors. (g) Dependence of specific capacitance on the graphene oxide (GO) content at a constant scan rate of 5 mV s^{-1} . (h) Galvanostatic charge–discharge curves at different current densities. (i) The cycling stability of PC_GO-g-PAA (0.5% GO) at 0.1 A g^{-1} for 1500 cycles. (j) Ragone plot of the PC_GO-g-PAA (0.5% GO) solid-state supercapacitor at different current densities. Error bars represent the standard deviation of three independent measurements ($n = 3$). Error bars smaller than symbols are omitted for clarity.

The specific capacitance calculated from the CV data (Figure 15e) exhibits a typical dependence on scan rate: the highest values are achieved at 5 mV s^{-1} (approximately $\sim 1.14 \text{ F g}^{-1}$), followed by a gradual decrease with increasing scan rate. This behavior reflects a transition from a regime in which ions can access deeper regions of the structure to a regime in which, at high scan rates, charge storage is dominated by the outer or most easily accessible surface. Such scan-rate-dependent behavior is widely reported for gel-polymer-electrolyte-based solid-state supercapacitors. Importantly, the 0.5% GO composition provides the most favorable compromise between the polymer matrix structure and the conductive/ion-transport enhancement introduced by GO. While the addition of GO can improve ion transport efficiency (e.g., through the formation of additional transport pathways and enhanced interfacial interactions), the optimum is achieved at moderate

concentrations, whereas excessive filler content may lead to increased heterogeneity and impaired transport. The obtained CV characteristics for PC_GO-g-PAA (0.5% GO) are consistent with literature report [40] indicating that GO modification of gel electrolytes can effectively enhance ionic conductivity and overall performance of solid-state supercapacitors. The obtained results demonstrate a significant contribution of PAA grafting to the increase in specific capacitance compared to ungrafted PC and PC_GO (Figure 15f,g).

Galvanostatic charge–discharge (GCD) measurements of the symmetric cell with the PC_GO-g-PAA (0.5% GO) gel polymer electrolyte were carried out within a potential window of 0–0.9 V at current densities of 0.1, 0.2, 0.5, and 1 A g^{−1} (Figure 15h). All curves exhibit an almost linear, triangular shape without a pronounced IR drop at the beginning of the discharge, which is characteristic of electric double-layer capacitors (EDLCs) and indicates a low internal resistance of the system. This behavior is consistent with the minimal resistance values observed in the impedance spectra (Nyquist plots), confirming efficient ion transport within the gel polymer electrolyte and good electrolyte/electrode interfacial contact. Similar linear GCD behavior has been widely reported in the literature for gel polymer supercapacitors with optimized electrolytes and minimal interfacial resistance losses [72].

With increasing current density, the charge–discharge time decreases as expected, while the curve shapes remain symmetric and linear, indicating a stable capacitive nature of the process without a noticeable contribution from pseudocapacitive or Faradaic reactions. The preservation of the triangular shape of the GCD curves at higher current densities suggests fast ion transport both in the electrolyte bulk and at the electrode surface, reflecting good ionic conductivity and effective accessibility of the porous electrode network [107].

The cycling stability of the cell was evaluated by repeated GCD cycling up to 1500 cycles (Figure 15i). A comparison of the discharge profiles for cycles 1–10 and 1490–1500 shows nearly identical shapes and discharge times. Specifically, the device demonstrated a high capacitance retention of 99.6% after 1500 cycles, with the discharge time decreasing marginally from 45.2 s to 45.0 s. The absence of significant performance degradation indicates the electrochemical stability of the GO electrolyte as well as the robustness of the electrolyte/electrode interface during repeated cycling, without signs of degradation or accumulation of side products. This high reversibility of the charge–discharge process and good capacitance retention are key requirements for practical supercapacitor applications [14].

The observed stability and retention of capacitive performance correlate well with effects reported in the literature for optimized gel polymer matrices containing moderate concentrations of GO. In particular, the moderate incorporation of GO promotes the formation of additional ion-conducting pathways and improves the structural integrity of the polymer without significantly increasing resistance, which is supported by both the enhanced CV characteristics and the high-quality GCD curves [108].

The Ragone plot of the symmetric supercapacitor based on the PC_GO-g-PAA (0.5% GO) gel polymer electrolyte illustrates the relationship between energy density and power density at different current densities (0.1–1 A g^{−1}) (Figure 15j). The device exhibits a typical trade-off behavior, where higher energy density is achieved at lower current densities, while higher power density is obtained at increased current densities. This trend is consistent with the galvanostatic charge–discharge results, reflecting efficient ion transport within the gel polymer electrolyte and good accessibility of the porous electrode structure. The relatively small decrease in energy density with increasing power indicates favorable electrochemical performance and low internal resistance of the system.

Table 2 presents the values of the key electrochemical parameters of GO-based gel polymer electrolytes (GPEs). It can be concluded that the PC_GO-g-PAA (0.5% GO) membranes

developed in this work exhibit electrochemical performance that is competitive within the class of dimensionally stable gel polymer electrolytes. Although the ionic conductivity is lower than that of some free-standing hydrogel systems, the PC_GO-g-PAA membranes provide a distinct practical advantage. As self-supported membranes, they can be handled directly, integrated onto surfaces, and adapted to solid-state configurations more easily than highly swollen gel systems. Their self-supported nature and dimensional integrity make them particularly suitable for solid-state supercapacitor architectures.

Table 2. Comparison of ionic conductivity values of selected GO-based gel polymer electrolytes.

GO-Based GPE	Ionic Conductivity (mS cm ^{−1})	Ref.
PAA/GO hydrogel	168.1	[93]
B-PVA/KCl/GO	47.5	[109]
Agarose/GO gel polymer electrolyte	73.8	[110]
PVDF-HFP/EMIMBF ₄ /GO ion-gel	25.0	[43]
GO/PVA hydrogel	0.466	[111]
PVDF-HFP_GO@PET-g-PAA/EMIMBF ₄ (0.5% GO)	0.039	[40]
PC_GO-g-PAA/EMIMBF ₄ (0.5% GO)	0.577	This study

4. Conclusions

In this study, a new class of gel polymer electrolytes based on poly(acrylic acid) (PAA)-grafted polycarbonate track-etched membranes (PC TeMs) modified with GO was successfully developed and systematically investigated. The membranes were prepared first by incorporating GO into polycarbonate precursor films, followed by ion irradiation and chemical etching to generate well-defined nanoporous architectures, and subsequent RAFT-mediated grafting of PAA throughout the nanochannels and membrane surface.

Structural and spectroscopic analyses confirmed that the track-etching process yields structurally intact membranes with uniform, cylindrical nanochannels even in the presence of GO within the polymer matrix. RAFT polymerization enabled controlled grafting of PAA without blocking the nanochannels, ensuring continuous ion-conductive pathways across the full membrane thickness. The combination of nanoconfinement and controlled grafting proved essential for balancing high functionalization levels with preserved pore accessibility.

Electrochemical analysis revealed a strong dependence of ionic transport properties on GO content. A moderate GO loading of 0.5 wt% resulted in the lowest bulk resistance and the highest ionic conductivity, indicating the formation of an optimized ion-transport network within the grafted membrane. Further increases in GO content led to reduced performance, which can be attributed to partial pore obstruction and increased structural heterogeneity. These trends were consistent with the textural evolution observed by nitrogen adsorption–desorption analysis and with the electrochemical impedance results.

Symmetric supercapacitor cells assembled with the optimized PC_GO-g-PAA (0.5 wt% GO) electrolyte exhibited predominantly capacitive behavior, low internal resistance, and stable charge–discharge characteristics over a wide range of scan rates and current densities. Testing over 1500 cycles demonstrated good cycling stability, with a capacitance retention of 99.6% and negligible changes in the discharge profiles.

Beyond the electrochemical performance, this work introduces a previously unexplored hybrid membrane platform by adapting track-etching technology to GO-doped polycarbonate films and subsequently functionalizing the resulting nanochannels via controlled grafting. To the best of our knowledge, such hybrid precursor films have not previously been integrated with ion-irradiation track-etching technology to generate composite TeM architectures. The strategy applied in this study expands the design space

of track-etched membranes beyond conventional single-polymer systems to composite films and demonstrates their potential as multifunctional, dimensionally stable, and ion-conductive platforms.

Overall, the PC_GO-g-PAA membrane with 0.5 wt% GO offers an optimal combination of high ionic conductivity, low internal resistance, stable capacitive behavior, and good cycling stability. These results highlight the promise of GO-modified, grafted track-etched membranes as gel polymer electrolytes for all-solid-state supercapacitors and suggest broader applicability of this hybrid membrane concept in membrane science and energy storage technologies.

5. Patents

A patent application resulting from the work reported in this manuscript has been filed with the Patent Office of the Republic of Kazakhstan: Application Registration No. 2026/1228.2, filed on 29 May 2026, entitled “Method for Preparing a Gel Polymer Electrolyte.”

Supplementary Materials: The following supporting information can be downloaded at <https://www.mdpi.com/article/10.3390/batteries12090376/s1>, Table S1. Energy losses and total mileage of krypton ion ^{84}Kr PC TeM with different GO contents; Table S2. Calculation of the parameters of the operating mode of the DC-60; Table S3. Transmission coefficient of the krypton ion beam; Figure S1. The spectrum of krypton ions by charge; Figure S2. Axial injection channel setup (a), cyclotron magnetic system setup (b) and cyclotron RF system setup (c); Figure S3. Setting up the ion-optical elements of the transport channel (a), the profile of the krypton ion beam $^{84}\text{Kr}^{15+}$ on the profilometer in diagnostic unit No. 2 with the beam scanning system turned on (b) and ion currents $^{84}\text{Kr}^{15+}$ on FPM2, FPM3 and FPM4 units (c); Figure S4. Flat film irradiation module; Figure S5. Determination of the glass transition temperature (T_g) for the PC_GO TeMs (0.5%, wt).

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Abbreviations

The following abbreviations are used in this manuscript:

BET	Brunauer–Emmett–Teller
BP	Benzophenone
BPO	Benzoyl peroxide
CA	Contact angle
CPADB	4-Cyano-4-(phenylcarbonothioylthio)pentanoic acid
CV	Cyclic voltammetry
DCM	Dichloromethane
DG	Degree of grafting
EDX	Energy-dispersive X-ray analysis
EIS	Electrochemical impedance spectroscopy
FTIR	Fourier transform infrared spectroscopy
GCD	Galvanostatic charge/discharge
GO	Graphene oxide
GPE	Gel polymer electrolyte
PAA	Polyacrylic acid
PC	Polycarbonate
PET	Polyethylene terephthalate
PVA	Polyvinyl alcohol
PVAc	Polyvinyl acetate
RAFT	Reversible Addition–Fragmentation chain Transfer
SEM	Scanning electron microscope
TeM	Track-etched membrane
PC TeM	Pristine polycarbonate track-etched membrane
PC_GO TeM	GO-doped polycarbonate track-etched membrane
PC-g-PAA TeM	PAA-grafted pristine PC TeM
PC_GO-g-PAA TeM	PAA-grafted GO-containing PC TeM

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