

SURFACE MORPHOLOGY AND STRUCTURAL CHARACTERIZATION OF ION-IRRADIATED PET FILMS AFTER ALKALINE ETCHING

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ABSTRACT

This study reports the fabrication and structural characterization of track-etched membranes based on a 12 μm -thick, locally produced polyethylene terephthalate (PET) film. PET films were irradiated with krypton (Kr) ions (2.0 MeV/nucleon, fluence 10^9 cm^{-2}) and subsequently etched in a 2.2 M NaOH solution at 82–85°C to convert the latent ion tracks into open, cylindrical micropores. X-ray diffraction (XRD) revealed a shift and broadening of the principal PET reflection after etching, indicating a reorganization of the crystalline domains rather than a simple increase or decrease in crystallinity. Fourier-transform infrared (FTIR) spectroscopy confirmed that all characteristic PET absorption bands were retained after etching, showing that the polymer's chemical backbone remained intact. Scanning electron microscopy (SEM) revealed a clear, etching-time-dependent evolution of the surface and pore morphology, with roughness and pore density increasing progressively between 150 and 180 s of etching. Considered jointly, the three techniques converge on a single mechanistic picture in which etching selectively removes radiation-damaged, amorphous-like PET while preserving the core polyester chemistry and reorganizing the surviving crystalline framework. These results demonstrate that structurally well-defined track-etched membranes can be produced from locally available PET film, supporting their practical and economic potential as a domestic alternative to imported membranes for gas and liquid separation, medicine, and biotechnology. Surface wettability and membrane transport behavior were not measured in the present work and are identified as directions for follow-up study.

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Introduction

In recent years, the depletion of freshwater resources, increasing industrial waste generation, and the growing demand for high-purity process water have intensified scientific and technological interest in membrane-based separation processes [1]. Compared with conventional treatment technologies, membrane systems offer several advantages, including lower energy consumption, compact process configurations, reduced chemical requirements, and the ability to operate over a broad range of pH and temperature conditions [2]. Developing stable membrane materials with high separation efficiency for microfiltration, ultrafiltration, nanofiltration, reverse osmosis, membrane distillation, and electrodialysis therefore remains an important research priority [3].

Among polymeric membranes, track-etched membranes based on polyethylene terephthalate (PET) have attracted considerable attention for their precisely controlled pore

architecture. These membranes are typically fabricated by irradiating polymer films with energetic heavy ions and then selectively etching the radiation-damaged tracks [4]. This approach allows pore diameter, density, geometry, and orientation to be controlled with substantially greater precision than conventional phase-inversion techniques [5]: whereas phase-inversion membranes generally exhibit a broad pore-size distribution and heterogeneous morphology, track-etched membranes can contain cylindrical, non-interconnected pores of relatively uniform diameter [6]. This well-defined architecture makes them attractive for water purification as well as gas separation, biosensing, filtration of medical solutions, cell-culture systems, controlled drug delivery, and use as templates in nanotechnology [7].

The structural and functional properties of track-etched membranes are governed by several processing parameters, including ion species and energy, ion fluence, irradiation angle, film thickness, and the alkaline etching conditions, temperature, and duration [8]. Increasing the etching time generally produces larger pore diameters and higher porosity, but excessive etching can compromise mechanical integrity [9]; optimizing track-etched membranes therefore requires balancing pore size, permeability, mechanical stability, and surface hydrophilicity [10]. Irradiation of PET films with heavy ions such as krypton (Kr), xenon (Xe), and bismuth (Bi) has been shown to generate micro- and nanoscale channels over a broad size range [11], providing an opportunity to tailor membrane selectivity and mass-transport behavior for specific applications [12].

Track-etch technology is also being extended beyond conventional microfiltration membranes toward ion-selective materials [13]. Following heavy-ion irradiation, prolonged ultraviolet exposure combined with extraction of the radiolysis and photolysis products can produce ion-conducting channels in the subnanometer regime [14], while carboxyl groups formed on the internal pore walls can enhance surface hydrophilicity, ion-exchange capacity, and selective cation transport [15]. The resulting ion-selective behavior depends strongly on the energy loss of the incident ions and on the extraction medium's pH and temperature and on the ion fluence [16]; at sufficiently high fluence, neighboring tracks can approach or partially overlap, producing significant changes in membrane conductivity and transport behavior [17].

A major challenge for the industrial implementation of membrane technologies is fouling — the accumulation of unwanted substances on the membrane surface or within its pores [18]. Adsorption of proteins, polysaccharides, colloidal particles, organic compounds, and microorganisms reduces water flux, alters separation performance, and shortens membrane service life [19]; during microfiltration, fouling can develop through surface-deposit formation, pore-mouth blockage, internal pore-wall adsorption, or cake-layer formation [20]. Membrane surface properties — particularly hydrophilicity, charge, roughness, and chemical functionality — therefore play a critical role in fouling resistance [21].

A range of surface-engineering strategies has been investigated to mitigate fouling, including chemical oxidation, plasma treatment, graft polymerization, incorporation of hydrophilic monomers, and functionalization with nanomaterials [22]. Modification of PET track-etched membranes with hydrogen-peroxide-based oxidative systems has been reported to improve surface hydrophilicity and protein-fouling resistance [23]; however, graft polymerization with hydrophilic monomers such as acrylic acid or N-vinylimidazole does not always produce a net benefit, since the grafted chains can interact strongly with proteins and partially obstruct the pores, reducing permeability [24]. Effective surface modification therefore requires more than simply increasing hydrophilicity — preserving the pore architecture and controlling surface charge and chemical compatibility with the feed medium are equally important.

PET-based track-etched membranes are therefore promising materials for water purification, biofiltration, and ion-selective separation, owing to their precisely controlled pore structure, potential for high selectivity, and versatility for chemical surface modification. However, their practical performance depends on a complex interplay among pore morphology, hydrophilicity, mechanical stability, ion transport, and antifouling behavior. Accordingly, the present study aims to evaluate the fabrication conditions of PET-based track-etched membranes

and to characterize the resulting structural, chemical, and surface-morphological properties as a function of etching time, providing a basis for future work on their transport and antifouling performance following surface modification [25].

2. Materials and Methods

2.1. Materials

Foreign produced polyethylene terephthalate film (grade PET-6312), 12 μm thick and 0.32 m wide, was used as the base polymer. Sodium hydroxide (NaOH) and hydrochloric acid (HCl) were used for chemical etching and subsequent washing, respectively.

2.2. Ion irradiation

PET-6312 films were irradiated with krypton (Kr) ions using the IC-100 heavy-ion accelerator at the Laboratory of High Energy Physics, Joint Institute for Nuclear Research (JINR), Dubna, Russian Federation. The ion energy was 2.0 MeV/nucleon, the irradiation angle was 90° (normal incidence, so that the resulting channels are oriented perpendicular to the film plane), and the ion fluence was 10^9 cm^{-2} — low enough that individual latent tracks remain well separated and are not expected to overlap once etched. Under these conditions, the energy loss of the ions in the PET matrix ranged from 5–6 MeV/ μm , within the regime in which Kr ions are known to produce continuous, etchable latent tracks in PET.

2.3. Chemical (track) etching

Latent ion tracks in the irradiated PET films were converted into open pores by chemical etching in a 2.2 M NaOH solution at 82–84 $^\circ\text{C}$, followed by rinsing in HCl-acidified water. This process selectively removed radiation-damaged material along the ion trajectories, forming cylindrical pores and yielding the PET track-etched membrane (PETetch). To follow the time-dependent evolution of the surface morphology by SEM, separate film samples were etched under otherwise identical conditions for 150, 160, 170, and 180 s.

The selectivity of this process rests on the difference in chemical reactivity between the latent track and the surrounding, unirradiated polymer. As an energetic Kr ion traverses the film, it deposits energy predominantly through electronic excitation and ionization along a narrow trajectory, cleaving ester ($-\text{C}(=\text{O})-\text{O}-$) and backbone C–C bonds and leaving a cylindrical zone of reduced molecular weight, elevated chain-end (carboxyl and hydroxyl) density, and locally disordered chain packing — the latent track. Because these scission products and disordered segments are hydrolyzed by hot alkali considerably faster than the intact, more ordered bulk polymer, immersion in NaOH dissolves material along the ion path at a much higher rate than it dissolves the surrounding matrix, consistent with the general track-etch mechanism described for ion-irradiated polymer films. The ratio between this track etch rate and the bulk etch rate governs pore diameter, wall smoothness, and how faithfully the resulting channel reproduces the ion's original trajectory.

2.4. Characterization

The crystal structure of the pristine and etched PET films was examined by wide-angle X-ray diffraction (XRD) using a MiniFlex 600 diffractometer (Rigaku, Japan) in reflection mode with Cu-K α radiation, over a 2θ range of 5–48 $^\circ$ at a scanning rate of 2 $^\circ/\text{min}$ (generator voltage 22 kV, current 12 mA). Structural parameters were calculated from the diffractograms using SmartLab Studio II software (version 4.3.200.0, Rigaku, Japan).

Changes in the molecular structure and functional groups were examined by Fourier-transform infrared (FTIR) spectroscopy using an Invenio-S spectrophotometer (Bruker, Germany) over the range 500–4000 cm^{-1} .

The morphology of the pores formed during etching — including their shape, size, distribution, and the associated surface morphology — was examined by scanning electron microscopy (SEM). Together, XRD, FTIR, and SEM were selected to probe three complementary aspects of the same etching process: bulk crystalline order, chemical/functional-group identity, and surface topography, respectively, so that the structural picture built in Section 3 does not rest on any single technique.

3. Results and Discussion

The fabrication of a polyethylene terephthalate (PET) track-etched membrane is based on two consecutive stages: heavy-ion irradiation and subsequent chemical etching. Initially, the PET film is irradiated with high-energy krypton ions (Kr). As the ions penetrate the polymer matrix, they transfer energy along their trajectories, inducing localized radiation damage within the PET macromolecular structure. Consequently, latent tracks are formed along the ion trajectories. Within these regions, partial cleavage of polymer bonds and disruption of the macromolecular chains occur, resulting in increased chemical susceptibility of the irradiated zones.

In the subsequent stage, the irradiated PET film is subjected to chemical etching in an aqueous NaOH solution. Under alkaline conditions, the radiation-damaged regions exhibit a considerably higher etching rate than the undamaged PET matrix. This difference in chemical reactivity enables the selective dissolution of the latent tracks, while the non-irradiated polymer matrix remains comparatively intact. As the etching proceeds, the latent tracks are progressively opened, forming cylindrical pores that extend through the entire thickness of the PET film. Thus, the krypton-ion irradiation determines the spatial distribution and orientation of the potential pores, whereas the subsequent chemical etching converts the latent tracks into well-defined through-pores. The pore diameter and morphology can be controlled by adjusting the main etching parameters, including the NaOH concentration, temperature, and etching time. As a result, a PET track-etched membrane (PET-TEM) with a defined pore geometry, pore density, and preferential pore orientation is obtained. The unique porous architecture and tunable surface properties of PET-TEM make this membrane a promising material for membrane separation, selective ion transport, ion capture, and industrial wastewater treatment applications.

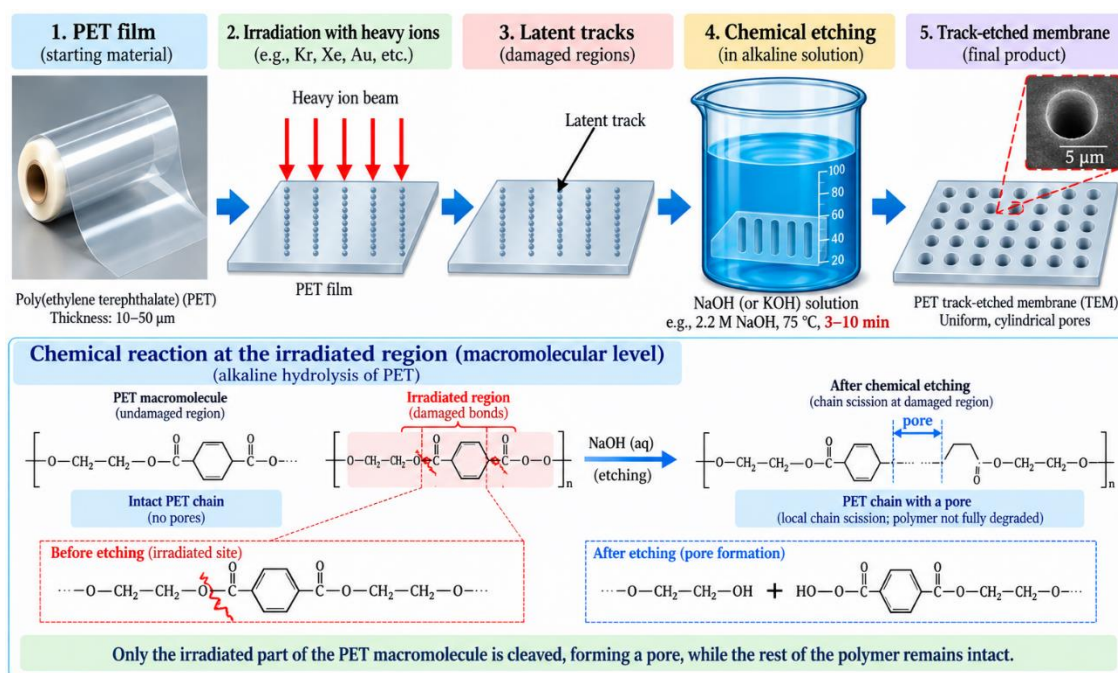


Figure.1. Preparation of PET track etched membranes (schematic picture).

3.1. X-ray diffraction analysis

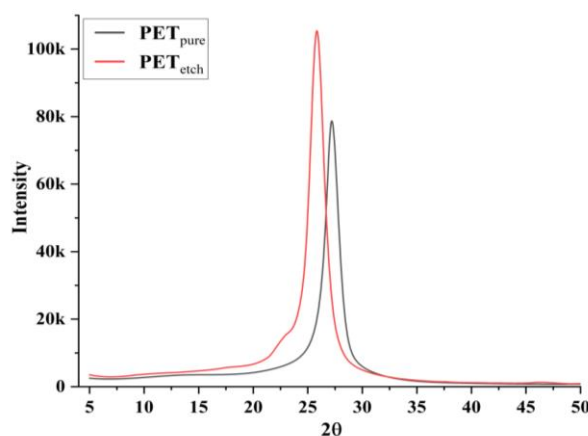


Figure 1. XRD patterns of pristine (PETpure) and etched (PETetch 180 s) PET films

XRD patterns of the pristine (PETpure) and etched (PETetch 180 s) PET films. As a semicrystalline polymer, PET exhibits an amorphous halo together with diffraction maxima associated with its crystalline phase; according to the literature, the reflections near $2\theta \approx 16\text{--}18^\circ$, $22\text{--}23^\circ$, and $25\text{--}27^\circ$ correspond to the (010), (110), and (100) crystallographic planes, respectively, with the $\approx 25\text{--}27^\circ$ reflection representing the principal crystalline peak of PET.

Both samples display a pronounced maximum at $2\theta \approx 25\text{--}27^\circ$, confirming that ordered crystalline domains are present both before and after etching. Etching, however, produced three consistent changes in this reflection (Table 1): its intensity increased from $\approx 7.8\text{--}8.0 \times 10^4$ to $\approx 1.05 \times 10^5$ a.u.; its position shifted from $2\theta \approx 27^\circ$ to $2\theta \approx 25.8\text{--}26^\circ$; and the peak broadened relative to the pristine film.

Applying Bragg's law,

$$d = \lambda / (2 \sin \theta) \quad (1)$$

with Cu-K α radiation ($\lambda = 1.5406 \text{ \AA}$), the interplanar spacing increases from $d \approx 3.30 \text{ \AA}$ ($2\theta = 27^\circ$) to $d \approx 3.45 \text{ \AA}$ ($2\theta = 25.8^\circ$), an increase of approximately 4–5%. According to the Scherrer equation,

$$D = K\lambda / (\beta \cos \theta) \quad (2)$$

where D is the mean crystallite size, K the shape factor, and β the full width at half maximum (FWHM) of the peak, the observed broadening is consistent with a decrease in the average crystallite size, or equivalently, with increased lattice disorder.

Taken together, these observations indicate that etching does not simply increase or decrease the overall crystallinity of PET; rather, it reorganizes the crystalline domains — increasing the interplanar spacing, most plausibly through chain rearrangement or the buildup of lattice strain, while reducing the effective crystallite size or long-range order. Because diffraction intensity is influenced not only by crystallinity but also by sample thickness, molecular orientation, and surface morphology, the intensity increase alone cannot be read as a quantitative increase in crystallinity. A reliable crystallinity index would require deconvolution of the crystalline and amorphous components and comparison of their integrated areas,

$$X_c = I_c / (I_c + I_a) \times 100\% \quad (3)$$

ideally cross-validated against differential scanning calorimetry (DSC). This distinction is consistent with previous reports showing that plasma or chemical etching of PET, which acts

primarily on the near-surface layers, can leave the bulk crystallinity essentially unchanged even as the diffraction peak shape and position are altered, while other studies attribute peak broadening and shifting under chemical etching specifically to disruption of the ordered domains. Within the mechanism outlined in Section 2.3, this pattern is consistent with the preferential removal, by alkaline hydrolysis, of the more disordered, radiation-damaged and amorphous-rich material: as that material is dissolved away, the diffraction signal increasingly reflects the surviving, reorganized crystalline domains, whose slightly expanded and less perfectly ordered lattice produces the observed shift and broadening. The present XRD results therefore confirm that ion-track etching structurally reorganizes the PET crystalline lattice, but a quantitative crystallinity value requires the additional deconvolution/DSC analysis outlined above.

Table 1

Summary of the principal XRD reflection before and after etching

Parameter	PET (pristine)	PET (etched)
2 θ of main reflection	$\approx 27^\circ$	$\approx 25.8\text{--}26^\circ$
Interplanar spacing, d (Bragg)	$\approx 3.30 \text{ \AA}$	$\approx 3.45 \text{ \AA}$
Relative peak intensity (a.u.)	$\approx 7.8\text{--}8.0 \times 10^4$	$\approx 1.05 \times 10^5$
Peak width (FWHM)	narrower	broadened

3.2. FTIR spectroscopic analysis

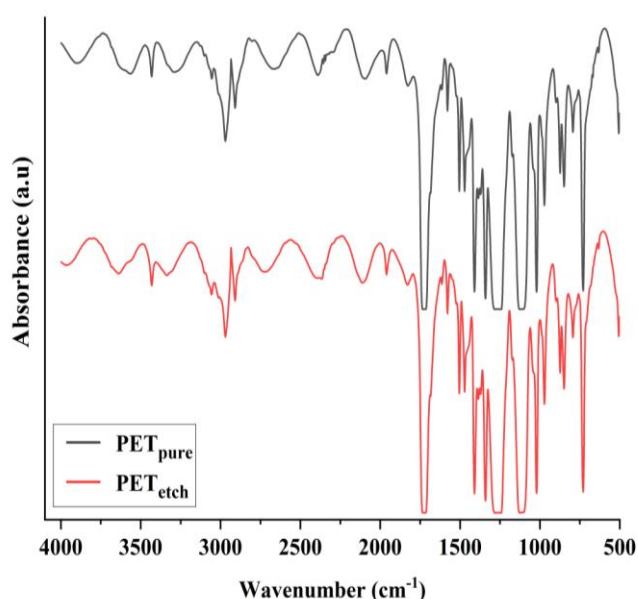


Figure 2. FTIR spectra (500–4000 cm^{-1}) of the irradiated PET film before (PET_{pure}) and after (PET_{etch} 180 s) chemical etching

Figure 2 shows the FTIR spectra of the irradiated-only PET film and the PET film subsequently etched in 2.2 M NaOH at 82–84 °C. All of the characteristic PET absorption bands are present in both spectra (Table 2), confirming that the polymer's chemical backbone was preserved throughout ion irradiation and chemical etching.

The strong band at $\approx 1715 \text{ cm}^{-1}$ is assigned to C=O stretching of the ester carbonyl group, the primary diagnostic signal for the terephthalate ester linkage in the PET backbone. Weak bands

at 3000–2800 cm^{-1} correspond to aliphatic C–H stretching of the ethylene-glycol $-\text{CH}_2-$ units, while the band at $\approx 1471 \text{ cm}^{-1}$ arises from CH_2 bending in the same segment. The doublet at 1370–1340 cm^{-1} , associated with CH_2 wagging, is particularly informative: the $\approx 1340 \text{ cm}^{-1}$ component is generally assigned to the *trans* (ordered) conformer and the $\approx 1370 \text{ cm}^{-1}$ component to the *gauche* (amorphous-like) conformer, so the relative intensity of this pair is commonly used as a qualitative indicator of PET crystallinity. Bands at 1245 cm^{-1} and 1090–1100 cm^{-1} , assigned to ester C–O stretching, together with the 1715 cm^{-1} carbonyl band, confirm the ester functionality; the 1017, 970, 873, and 845–850 cm^{-1} bands, associated with aromatic-ring and ethylene-glycol vibrations, constitute the PET fingerprint region and match values reported for both virgin and recycled PET.

The close correspondence between the peak positions of the two spectra shows that etching preserved the fundamental chemical structure of PET — direct spectroscopic support for the picture developed in Section 2.3, in which alkaline etching acts by dissolving and physically removing damaged material rather than by chemically transforming the polymer that remains. The observed intensity differences therefore more likely reflect changes in molecular conformation, crystallinity, orientation, or surface state than degradation of the polymer backbone, consistent with ATR-FTIR comparisons of virgin and recycled PET, in which the 844, 968, 1340, and 1470 cm^{-1} intensities varied with crystallinity while the overall spectral profile remained unchanged. A quantitative comparison of crystallinity or chemical degradation between the two samples would require baseline-corrected, normalized absorbance data and calculation of peak-intensity or peak-area ratios (e.g., 1340/1370 or 973/1018 cm^{-1}) rather than a qualitative comparison of raw spectra. If the membranes are subsequently exposed to thermal, mechanical, or UV stress, the 1715 cm^{-1} carbonyl region and the possible emergence of new carbonyl components near 1680–1700 cm^{-1} should be monitored as indicators of chain scission and terminal carboxyl-group formation.

3.3. Surface morphology (SEM) analysis

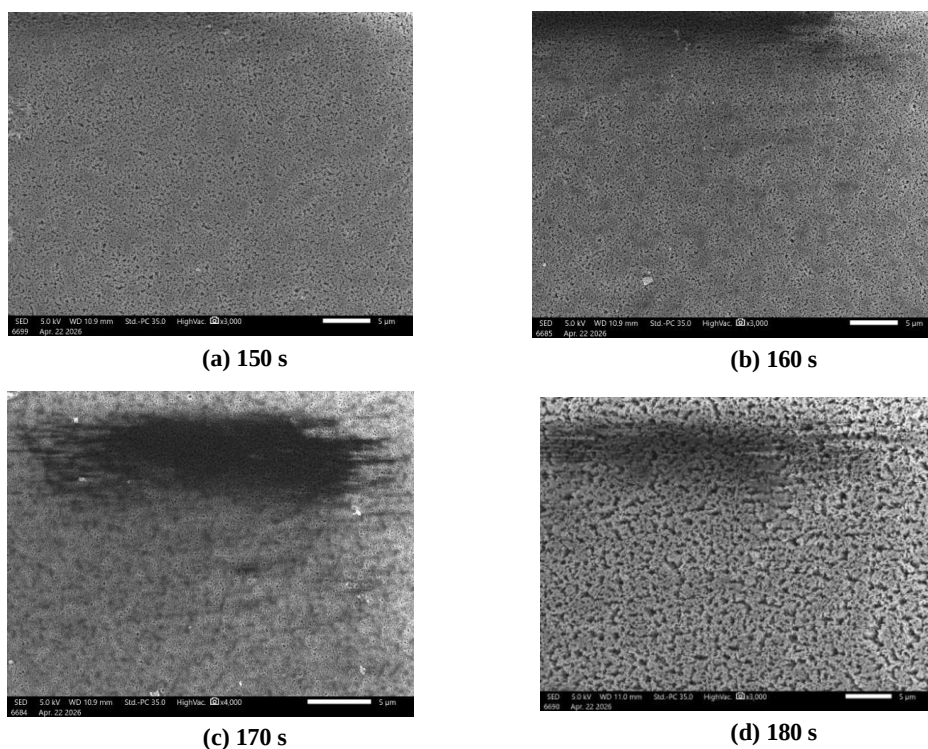


Figure 3. SEM images of the PET film surface after etching for (a) 150 s, (b) 160 s, (c) 170 s, and (d) 180 s

Figure 3 shows SEM images of the PET film surface after etching for 150, 160, 170, and 180 s, revealing a clear, progressive evolution of surface morphology with etching time. After 150 s, the surface is relatively compact and uniform, with only fine, shallow, point-like irregularities and no evidence of large pits or a well-developed porous texture — consistent with etching having modified only the outermost surface layer at this stage. By 160 s, the density of small pits and porous features increases measurably, and the surface texture becomes visibly rougher. At 170 s, surface heterogeneity intensifies further, with pits, irregularities, and microrelief features clearly visible in place of the initially compact morphology. After 180 s, the surface shows the most extensive modification of the series, with numerous, in places closely spaced, pits and porous features producing a rough, well-developed morphology.

Taken together, the 150–180 s series shows a consistent, stepwise morphological evolution: from a comparatively compact surface at 150 s, through increasing microporosity and roughness at 160–170 s, to the most extensively roughened and porous morphology at 180 s. This trend is the direct surface-level signature of the selective-hydrolysis mechanism described in Section 2.3: as etching proceeds, alkaline attack progressively dissolves radiation-damaged and structurally disordered material both along the ion tracks and inward from the free surface, and the resulting erosion accumulates visibly as roughness and porosity.

This surface-selective erosion is also consistent with the semicrystalline nature of PET, whose amorphous and crystalline regions are not expected to etch at equal rates; preferential removal of the more accessible amorphous, structurally disordered regions is a plausible additional origin of the pits and roughness observed here, in agreement with previous reports linking PET surface heterogeneity to differential etching of amorphous versus crystalline domains. The resulting increase in surface roughness and effective surface area is also relevant to the membrane's functional behavior, since comparable topographic changes in PET have been associated with altered wettability and surface energy in other physicochemical surface-modification studies.

It is important to note that SEM alone cannot quantify the degree of crystallinity: it provides a visual assessment of surface morphology, roughness, pits, and porous structure, but not a quantitative crystallinity value, so the present images should not be interpreted as evidence that crystallinity “increased” or “decreased.” A quantitative assessment of crystallinity requires XRD/WAXD, ideally combined with DSC, with surface morphology and crystallinity treated as related but distinct parameters, as is standard practice in PET characterization studies.

SEM results show that increasing the etching time from 150 to 180 s produced a clear, progressive transformation of the PET surface — from a compact initial state to an increasingly porous, rough morphology — with the 180 s sample exhibiting the most extensive surface erosion within the conditions examined.

3.4. Water Contact Angle – WCA

The water contact angle (WCA) measurements demonstrate a clear change in the surface wettability of the PET membranes after treatment. Pristine PET exhibited a WCA of **67.7°**, indicating a moderately hydrophilic surface. After treatment, the WCA decreased to **59.4° at 150 s** and **57.4° at 160 s**, showing improved water spreading. A further decrease to **52.1° at 170 s** indicates a progressive increase in surface wettability with treatment time. The lowest WCA of **47.6° was obtained after 180 s**, representing the most pronounced hydrophilic character among the investigated samples. Compared with pristine PET, the 180 s treatment reduced the WCA by approximately **29.6%**. This decrease suggests that the treatment substantially modified the surface properties of the PET membrane. The enhanced wettability may be associated with an increase in polar surface functionality and/or changes in the surface morphology. Such changes can increase the interaction between water molecules and the modified PET surface. The gradual reduction in WCA from 67.7° to 47.6° also indicates that treatment duration plays an important role in controlling surface wettability. Importantly, all measured contact angles remained below **90°**, confirming that the investigated PET surfaces retained predominantly hydrophilic behavior. The relatively small difference between the 160 s and 170 s samples suggests that the wettability

response becomes less pronounced at intermediate treatment times. Nevertheless, the further reduction observed at 180 s indicates that prolonged treatment can produce additional changes in the surface characteristics.

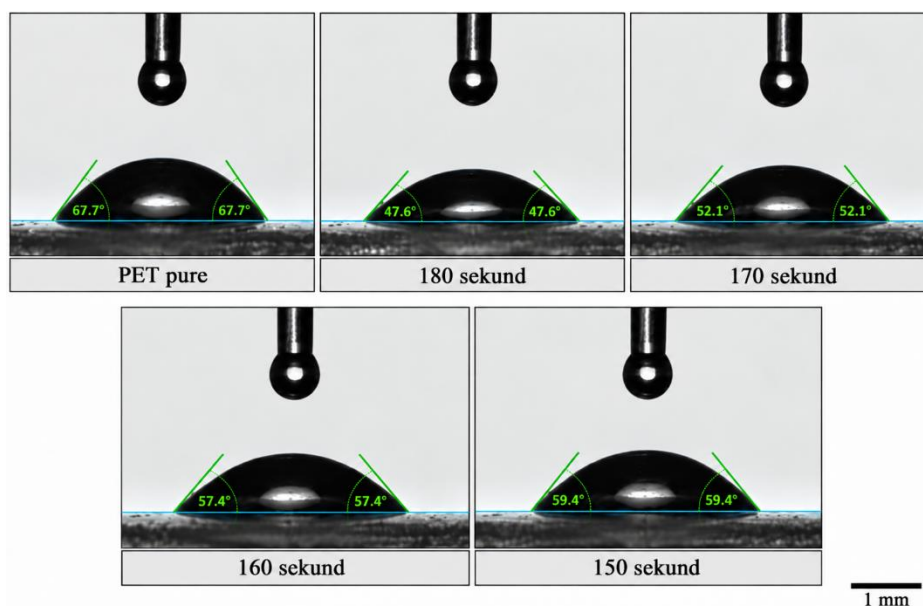


Figure 4. Water Contact Angle – WCA

Conclusions

The track-etching method was successfully applied to modify the surface of locally produced PET films (12 μm thick) through irradiation with Kr ions (2.0 MeV/nucleon, fluence 10^9 cm^{-2}) followed by chemical etching in a 2.2 M NaOH solution at 82–84 °C. XRD analysis showed that etching shifts the principal PET reflection from $2\theta \approx 27^\circ$ to $\approx 25.8\text{--}26^\circ$ and broadens it, corresponding to an $\approx 4\text{--}5\%$ increase in interplanar spacing and a Scherrer-consistent decrease in apparent crystallite size; because diffraction intensity alone cannot quantify crystallinity, these changes are best interpreted as a reorganization of the crystalline domains rather than a simple increase or decrease in crystallinity. FTIR spectroscopy confirmed that all characteristic PET absorption bands ($\approx 1715, 1471, 1370\text{--}1340, 1245, 1090\text{--}1100, 1017, 970, 873, \text{ and } 845\text{--}850 \text{ cm}^{-1}$) were retained after etching, demonstrating that the polymer's chemical backbone remains intact and that the observed spectral differences reflect conformational or crystallinity-related changes rather than chemical degradation. SEM imaging showed a clear, etching-time-dependent evolution of surface morphology, from a comparatively compact surface after 150 s to a progressively rougher, more porous morphology at 160, 170, and 180 s. Considered jointly, these three observations converge on a single mechanistic picture — selective alkaline dissolution of radiation-damaged, amorphous-like PET along the ion tracks and near-surface region, leaving the core polyester chemistry intact and reorganizing the surviving crystalline framework.

Collectively, these results demonstrate that well-defined, structurally characterized track-etched membranes can be produced from locally available PET film using a straightforward Kr-ion irradiation and alkaline-etching route, supporting the feasibility of domestic production as an alternative to imported track-etched membranes. Future work should quantify crystallinity by XRD peak deconvolution combined with DSC, determine the pore-size distribution and surface wettability (e.g., by water contact angle measurement), and evaluate the transport and antifouling performance of the resulting membranes under realistic filtration conditions.

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