

Structural and Physicochemical Analysis of Polycaprolactone Electrospun Membranes for Biomedical Applications

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Research Article

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Abstract

Polymeric electrospun membranes have attracted considerable interest for various applications in filtration, adsorption, and medical fields due to their large surface area and unique properties. Polycaprolactone (PCL), a biocompatible polymer approved by the FDA, is well known for its gradual degradation, non-toxicity, and outstanding processability, rendering it appropriate for a range of medical uses, including drug delivery systems, sutures, wound dressings, and tissue scaffolds. This research work involved the successful fabrication of PCL membranes by electrospinning method utilizing a solvent system of dichloromethane (DCM) and dimethylformamide (DMF). The manufactured membranes were methodically analyzed with scanning electron microscopy (SEM), Fourier-transform infrared spectroscopy (FTIR), and mechanical testing to assess their morphology, chemical structure, and mechanical characteristics. Furthermore, contact angle measurements and thermogravimetric analysis (TGA) were utilized to evaluate wettability and thermal stability, respectively. The findings revealed the development of uniform, smooth, and bead-free fibrous structures exhibiting desirable mechanical strength, moderate water affinity, and excellent thermal stability. The lack of residual solvent signatures in FTIR validated the purity of the produced membranes.

1. Introduction

Electrospun polymeric membranes can fulfill numerous roles in biomedicine, such as systems for drug delivery, tissue engineering, dental applications, bone repair, wound healing, catheters, and medical implants. Polycaprolactone (PCL) is a synthetic polymer that is biodegradable and utilized in regenerative medicine [1]. PCL, as a commercial material, provides numerous advantages, such as its FDA approval enhancing its credibility for human use, biodegradability, compatibility with a variety of polymers, excellent processability, high thermal stability, affordable pricing, and versatility in applications including sutures, wound dressings, fixation devices, contraceptive devices, dentistry, drug delivery, and tissue engineering for scaffolds, bone, cartilage, tendons, ligaments, cardiovascular systems, blood vessels, skin, and nerves [2–4].

In this research, the electrospinning method was used to create a continuous, smooth PCL electrospun membrane. Electrospinning is a method for producing fibers by applying a high-voltage electric field to a polymer solution or melt, resulting in extremely fine fibers ranging from nano to micro scale, which are gathered as a nonwoven membrane. The electrospun membrane obtained was characterized through various physicochemical techniques, including scanning electron microscopy (SEM), Fourier-transform infrared spectroscopy (FTIR), hydrophilicity, tensile strength, and TGA, to evaluate its potential for biomedical applications, including the creation of biodegradable devices and utilization in biomedical areas like porous scaffolds, micro- and nanocarriers, wound dressings, and implantable systems[5]. The findings of the research show the creation of uniform polycaprolactone (PCL) nano/microfibrous membranes possessing excellent hydrophilicity, tensile strength, and thermal stability. This suggests that PCL electrospun membranes could be suitable for use in the biomedical sector, especially as tissue scaffolds. Overall, biodegradable polymers are essential in biomedical and environmental sectors, aiding

in the creation of sustainable materials and applications that enhance both human health and ecological well-being without endangering health or the environment [6].

2. Experimental

2.1 Materials, Membrane Production

Polycaprolactone (PCL, $M_w = 80,000$), dimethylformamide (DMF), and dichloromethane (DCM) were obtained from Sigma-Aldrich and utilized as provided. A 13 wt% PCL solution was created by dissolving PCL pellets in a DMF/DCM solvent blend (1:4, w/w) with continuous stirring for 24 hours at 27°C until a uniform viscous solution was achieved.

A 10 mL syringe fitted with a multi-nozzle spinneret was loaded with the solution and electrospun via a syringe pump regulated by E-spin software. Electrospinning was conducted with an applied voltage of 17 kV, a flow rate of 1.3 mL h⁻¹, and a distance of 18 cm between the tip and collector. Fibers were gathered on a spinning drum collector functioning at 500 rpm for 6 hours at 25°C. The resulting membrane was air-dried and then vacuum-dried for 24 hours to eliminate remaining solvents. The PCL electrospun membrane obtained was named 'PE'.

2.3 Characterization

The structure of PF was analyzed through scanning electron microscopy (SEM, ZEISS Gemini). The chemical structure was examined using Fourier-transform infrared spectroscopy (FTIR, Bruker Alpha II). Thermal stability was assessed through thermogravimetric analysis (TGA, EXTSTAR TG/DTA 6300, SII Nano Technology, Japan) ranging from 35 to 450°C with a heating rate of 10°C min⁻¹ in a nitrogen environment. Mechanical characteristics were assessed using a BOSE ElectroForce 3200 Series III (USA) fitted with a 225 N load cell at a crosshead speed of 0.6 mm s⁻¹. Specimens (15 × 5 mm²) were evaluated at ambient temperature with a gauge length of 2 mm, and findings are presented as the average ± standard deviation from a minimum of four samples. Surface wettability was evaluated through static water contact angle measurements with an Acam NSC 10 instrument.

3. Results and Discussion

3.1 Physicochemical Analysis

SEM images of PCL electrospun membrane (**Figure. 1a-c,**) exhibited a uniform, smooth, and continuous nano/microfibrous network. The nano/microfibers were homogeneously distributed with minimal bead formation, indicating stable jet formation and efficient solvent evaporation during the electrospinning process. The fiber diameter distribution, presented in **Figure. 2** ranged from approximately 200 nm to 2 μm with mean diameter of 1.03 μm and a well-defined hierarchical structure. This confirms successful fabrication of nano/microscale electrospun fibrous architecture, which is highly favorable for biomedical applications due to increased surface area and porosity with a complete evaporation of the solvent.

The PE exhibited typical elastic–plastic behavior with high tensile strength and significant elongation at break (Fig. 3), indicating a balanced combination of strength and flexibility. Its ability to withstand higher stress and deform without immediate failure reflects good mechanical stability and structural integrity. These properties are highly desirable for biomedical applications, where materials must endure mechanical stresses while maintaining flexibility. The good elongation and strength of the PE membrane support its potential use in wound dressings, tissue engineering scaffolds, and drug delivery systems, where durability, elasticity, and reliability are essential.

Thermogravimetric analysis (TGA), shown in Fig. 4, revealed a distinct stage in the weight loss of the PE membranes exhibited nearly identical weight loss patterns at higher temperatures, starting approximately 350°C. This sharp weight loss for electrospun membranes is likely attributable to the simultaneous elimination of solvent, coupled with the decomposition of chemical components. Moreover, the major weight losses are observed in the range of 350°-430°C. This may be correspondent to the structural decomposition of the polymer blends. These results indicated that there are distinguished in the weight loss, thermal stability and heat resistance capacities of as fabricated PE membranes without significant degradation of structural integrity. The absence of progressive weight loss in the PE membrane indicates that DCM and DMF was not successfully incorporated into the polymeric matrix. This observation is further supported by the FTIR results, which revealed no additional peaks corresponding to functional groups of DMF and DCM present in PE membrane.

As shown in the Fig. 5, the PE membrane exhibited strong characteristic peaks of C = O, stretching of the carbonyl group at 1726.3 cm^{-1} . It also showed strong peaks of C–O–C as well as stretching of the ester entity at 1170 cm^{-1} . Additionally, the asymmetric stretching peak of the aliphatic C–H bond is observed at 2942.1 cm^{-1} , along with the symmetric stretching peak at 2862 cm^{-1} which are frequently identified in the PCL spectrum (PE) [7, 8]. This shows that there is no solvent remains in the final PE membrane which is good option to use it as tissue scaffold and towards biocompatibility.

Figure 6 illustrates images capturing water drops situated on the surfaces of PE membranes. The alterations in the appearance of water drops on each PE membrane surface serve as indicators of the wettability and hydrophilicity of these electrospun membranes. The examined PE membrane exhibits moderate contact angle, signifying hydrophilicity. This is good because hydrophilic dressings have the ability to retain moisture, due to which natural moisture spreads evenly across the wound surface, maximizing contact and creating a moist environment thereby helping to accelerate the healing process compared to traditional dry wound management techniques [6, 9].

The combined mechanical strength, thermal stability, and controlled surface wettability of the fabricated PCL electrospun membranes suggest strong applicability in biomedical engineering. Their fibrous architecture closely promoting cell attachment and proliferation [10, 11]. Therefore, these membranes are promising candidates for wound dressings, tissue scaffolds, and controlled drug delivery systems.

4. Conclusion

In this research, PCL electrospun membranes were effectively produced using a DCM/DMF solvent combination. The membranes displayed consistent morphology, strong mechanical properties, thermal stability, and moderate hydrophilicity. The PE membrane's mechanical characteristics showed its capacity to endure stress while retaining flexibility, crucial for biomedical uses. The findings demonstrate that the created PCL electrospun membranes possess considerable potential for applications in tissue engineering, wound care, and drug delivery systems, emphasizing their importance as efficient and sustainable biomaterials in the biomedical sector.

Declarations

CRediT authorship contribution statement

Dinesh Shah: Investigation, Writing – original draft, Validation, and Visualization. review & editing. Software, Data curation and formal analysis.

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Ethics Declaration

Not applicable. This study did not involve human participants, human tissues, animals, or any procedures requiring ethical approval.

Conflict of Interest

The author declares no competing interests.

Data availability

Data will be made available on request.

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Figures

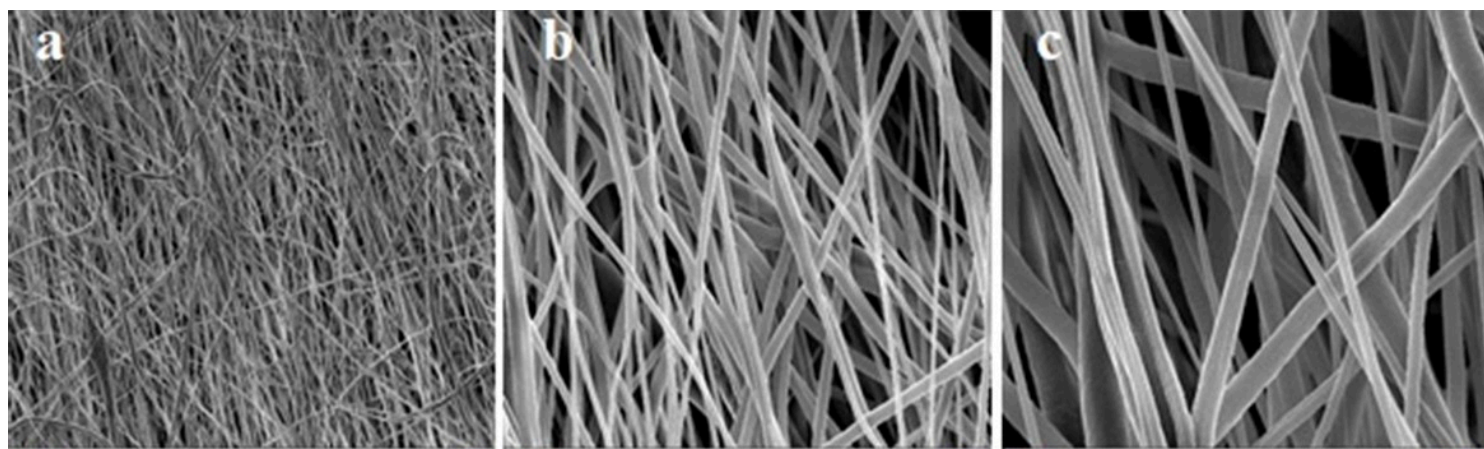


Figure 1

SEM images of PCL electrospun (PE) membranes at different magnification

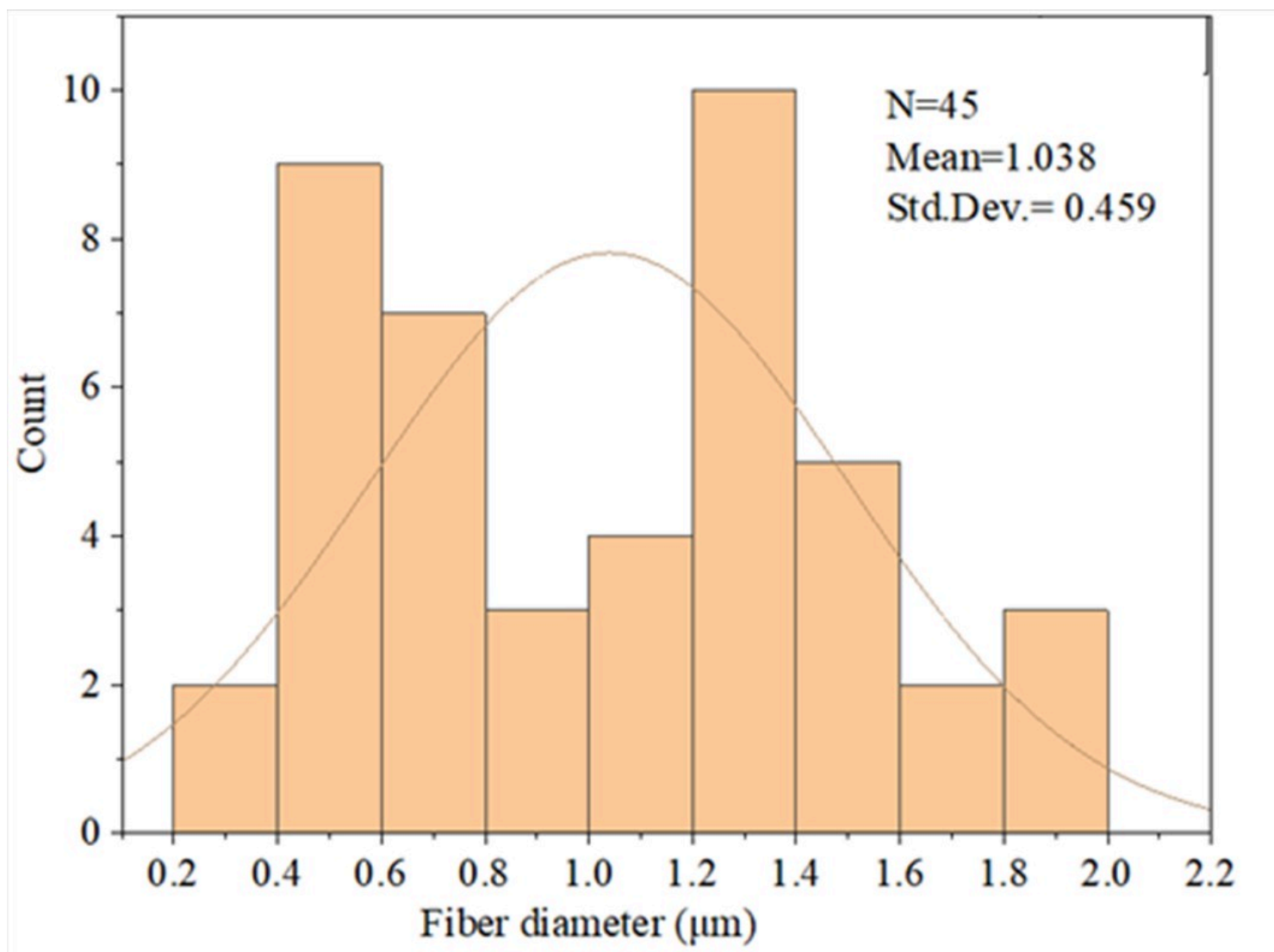


Figure 2

Histogram graph of PCL electrospun membrane

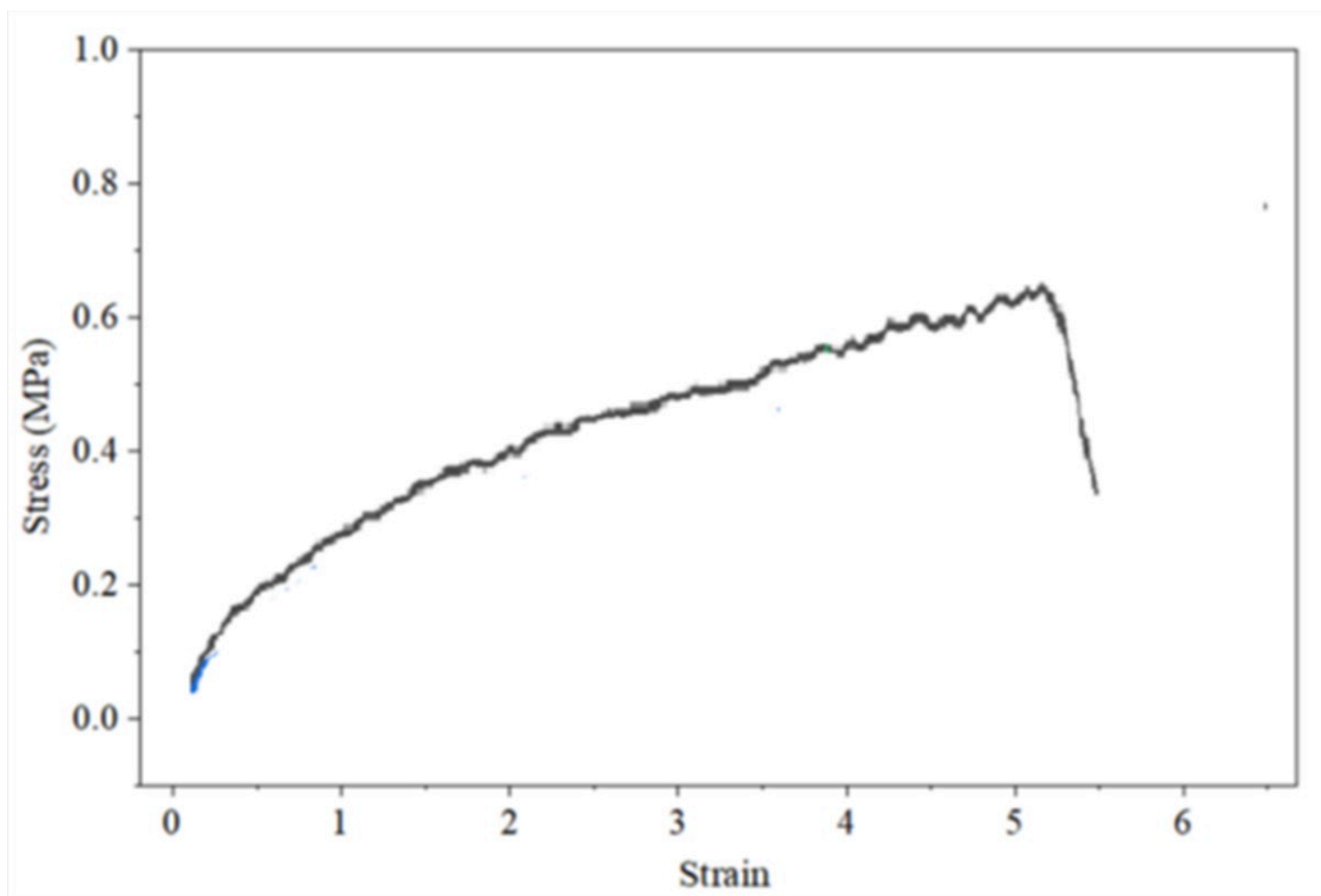


Figure 3

Stress-strain curve of PE electrospun membrane

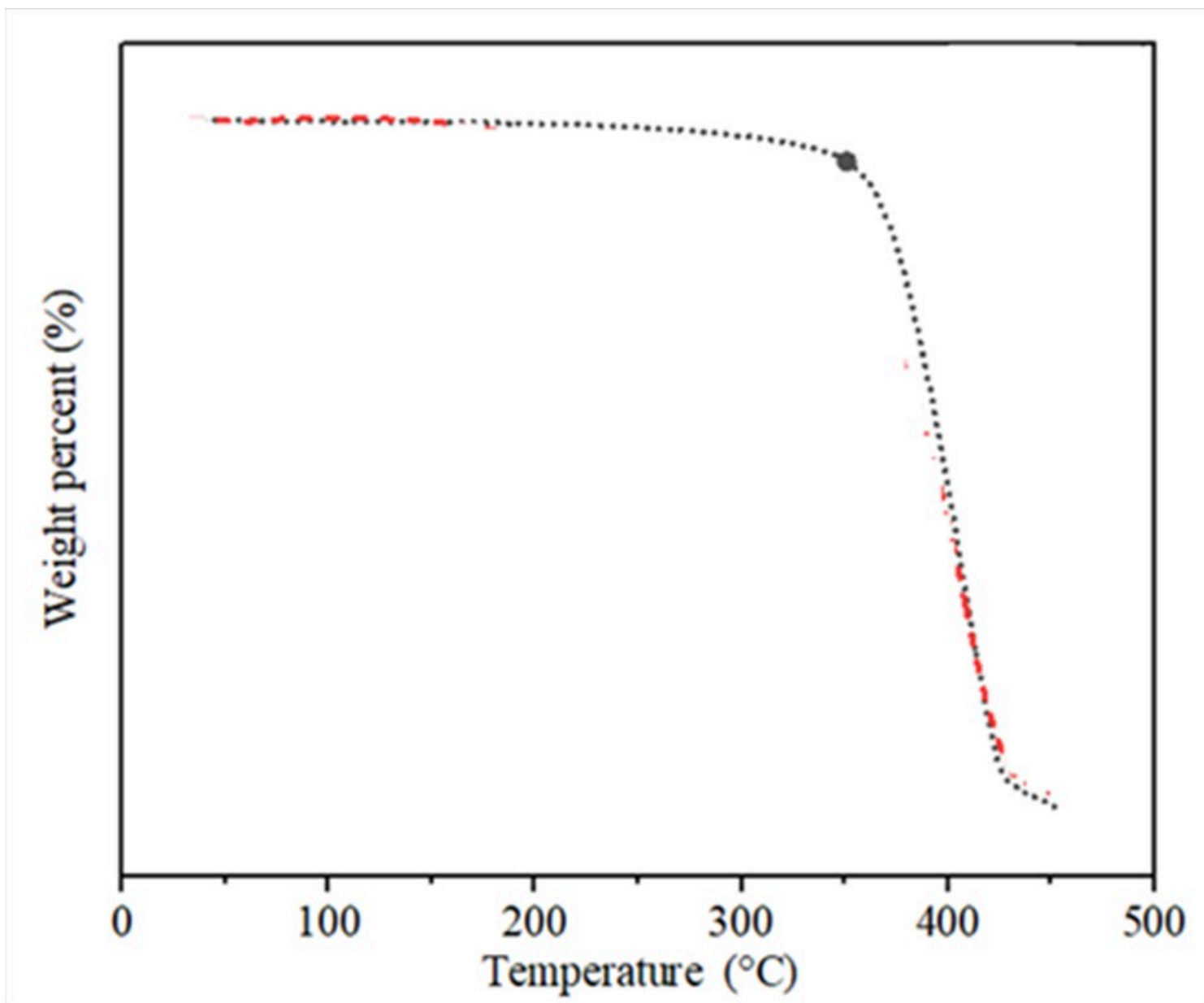


Figure 4

TGA thermogram of PE membrane

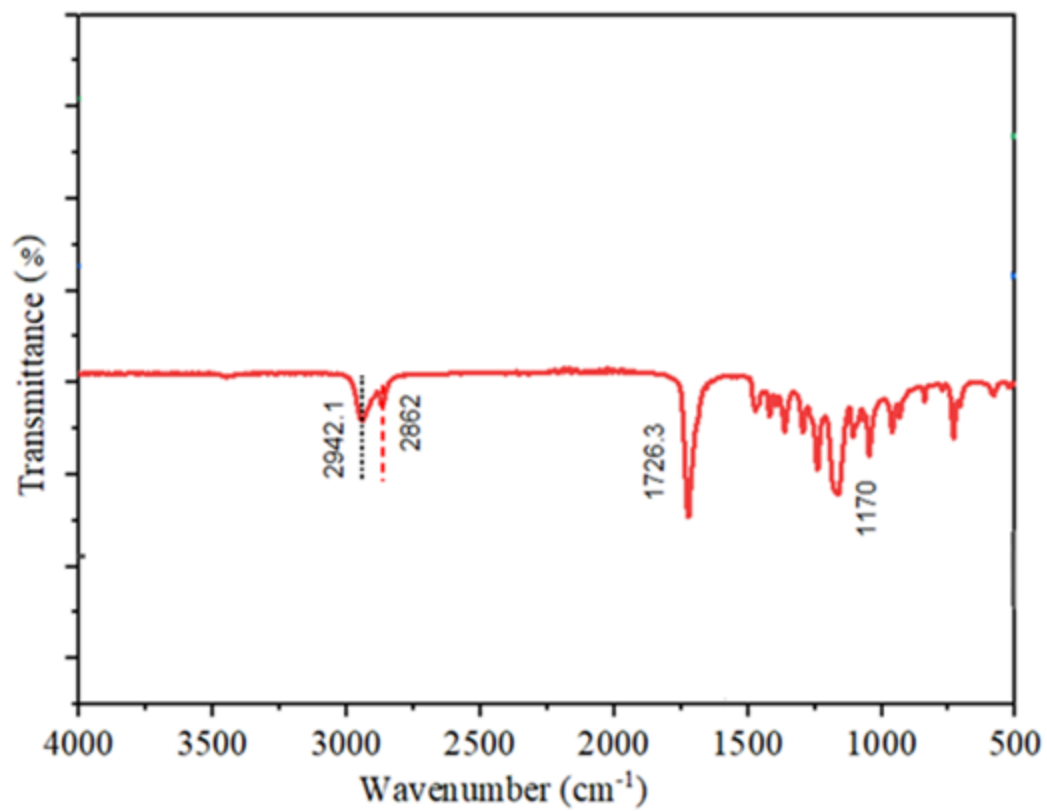


Figure 5

FTIR spectrum of PE membrane

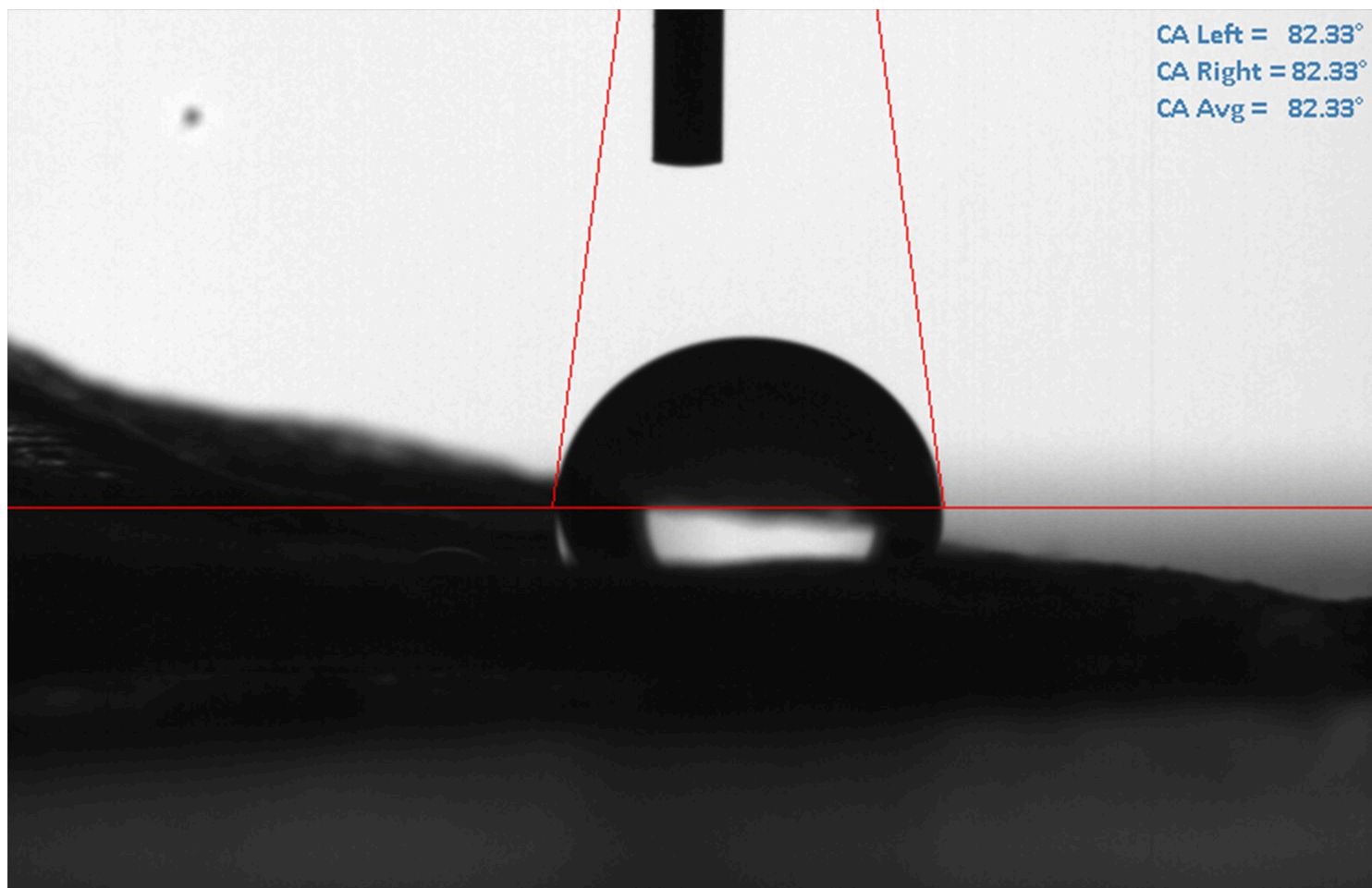


Figure 6

Water contact angle image of PE membrane.