RESEARCH PAPER

Fabrication and Characterization of Polycaprolactone Microfibers via Forcespinning for Drug-Delivery Applications

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Abstract:
This paper reports the fabrication of the Polycaprolactone Microfibers using a modified Forcespinning Machine repurposed from a commercial cotton candy machine. Three PCL Homogeneous solutions were prepared using a solvent (DCM), dichloromethane: 63%, 65%, and 67% w/v were systematically evaluated on various morphological parameters that are uniformity, amount of fine fibers, globules(beads), and amount of fused fibers. Scanning Electron Microscopy(SEM) revealed that the 65% w/v formulation produced the most uniform fibers with fewer globules and fused fibers, but more fine fibers in comparison with 63% and 67% formulation microfibers. Therefore, 65% formulation is selected as the best optimized concentration for further subsequent studies. Drug-loaded fibers were optimized with curcumin and Ibuprofen, and separate fibers were prepared at 5% w/w drug-to-polymer ratio and characterized for morphological integrity. A bilayer dual-drug fiber patch and a combined drug-loaded fiber patch were also optimized with both active pharmaceutical drugs curcumin and Ibuprofen, and subsequently fabricated. Scanning Electron Microscopy(SEM), Fourier transform Infrared spectroscopy(FTIR), and UV-visible Spectroscopy were employed to assess the morphology, chemical integration, and drug concentration to analyse drug release of the resulting fibers. A Mathematical Modelling framework describing fiber formation dynamics, internal drug distribution, and drug release kinetics was developed to complement experimental findings. These results demonstrate the feasibility of a cost-effective optimized machine, scalable force-spun PCL microfibers, and controlled dual-drug delivery applications.

Keywords: Forcespinning, PCL microfibers, Curcumin, Ibuprofen, Scaffold

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Introduction

The development of fibrous sheets has gained attention in recent years due to their wide range of applications in engineering and biomedical fields. These fibre sheets are commonly used in filtration systems, composite materials, and tissue engineering scaffolds.[1-3] Their fibrous structure provides properties such as extensive area, interconnected porosity, and flexibility, which are important for both industrial and biomedical applications.[1]

In the tissue engineering field for bone regeneration, the scaffold materials must fulfil several essential requirements. A perfect scaffold should possess sufficient mechanical strength to support the tissue structure while also being biocompatible and biodegradable. These properties allow the scaffold to support cell attachment and growth, while gradually degrading inside the body without causing harmful effects.[2,3] Fiber-based scaffolds can closely resemble the natural extracellular matrix that supports cell biological tissues, which is why it is trending.

The fibrous materials are generally classified into nanofibers and microfibers on the basis of their diameters. Microfibers have a typical diameter range in micrometers. Microfibers exhibit improved mechanical properties and are easier to fabricate and process. Their large diameter provides acceptable load-bearing capability, which gives them mechanical strength and stability for their various applications.[1,4]

Controlled processing techniques are required for the fabrication of microfibers to produce fibers with uniform diameter and tailored morphology. The most commonly used techniques for fiber fabrication are Electrospinning, Centrifugal Electrospinning, and Forcespinning®.

Traditionally, the fabrication of microfibres has been done by the method of electrospinning.[2] Electrospinning uses electrostatic forces to draw charged polymer jets, forming ultrafine, uniform fibers. However, its reliance on high voltage equipment (10-30 kV) and low production rate limit its large-scale industrial application.[5]

Centrifugal Electrospinning utilizes the centrifugal force generated by high rotating speed for fiber formation. However, this produces non-uniform fiber diameter at high speeds and is sensitive to solution viscosity and rotation speed.

Forcespinning® is a mechanical fibre fabrication technique that works on a centrifugal spinning mechanism, driven purely by mechanical forces.

Forcespinning does not depend on the electrical conductivity of the polymer; therefore works for both conductive and nonconductive materials.[5] Due to its scalability, simplicity, and cost-effectiveness, forcespinning is being regarded as a promising technique for industrial-scale production.

One of the main advantages of this technique is its high fiber production rate. It fabricates the fibers from polymer solutions or melts.[4] In this technique, the fiber morphology can be controlled by adjusting the parameters such as its speed, solution concentration, nozzle size, solvent evaporation rate, and the distance between the spinneret and the collector.[5] Microfiber scaffolds closely resemble the fibrous structure of the natural extracellular matrix in tissue engineering, which supports cell growth in living tissues. The pore size distribution in microfiber mats promotes cell infiltration and vascularization, which are important for successful tissue regeneration. Additionally, microfiber mats exhibit better structural integrity and are less prone to collapse during handling and implantation.

2. Materials and Methods

2.1 Materials

Polycaprolactone (PCL) is a synthetic biodegradable polymer used for microfiber fabrication owing to its good biocompatibility and thermal stability, which makes it suitable for mechanical and structural (composite reinforcement, filtration, insulation) and biomedical applications.[5]

Dichloromethane (DCM) was chosen for its good solubility with PCL. It has a low boiling point (39.6°C), hence it evaporates rapidly and produces dry, unmerged fibres with minimal solvent residual.[6]

Curcumin is a natural compound derived from *Curcuma longa* (turmeric). Its anti-inflammatory, antimicrobial, and wound healing properties promote tissue regeneration.[7] When loaded into PCL microfibers, it exhibits controlled drug release, maintaining therapeutic concentration for several days. Additionally, its hydrophobic nature ensures good compatibility with solvents such as DCM.

Ibuprofen is a non-steroidal anti-inflammatory drug (NSAID). It possesses analgesic and anti-inflammatory properties and is commonly used to reduce pain, inflammation, and swelling.[8] It is hydrophobic and exhibits controlled drug release, making it suitable for polymer scaffolds. Combining curcumin and ibuprofen for dual drug delivery enhances effectiveness and accelerates healing.

2.2 Optimization of Cotton Candy Machine for Forcespinning®

A commercial cotton candy machine was customized to perform as a Forcespinning® setup. The heating element of the machine was removed, and the orifice size was modified to 1.5 cm to facilitate controlled delivery of solution. At a distance of 5 cm from the spinneret, a cylindrical fiber collector was placed, where the solution solidified to form continuous fibers, facilitated by the evaporation of DCM. All the operations were performed at room temperature (25°C).

2.3 Preparation of PCL Fibers

PCL was dissolved in DCM using a magnetic stirrer to prepare the polymer solution. At three concentrations: 63%, 65%, and 67% w/v, PCL of mass 12.6 g, 13 g, and 13.4gm respectively, was dissolved in 20ml of DCM. The three solutions were processed through the optimized Forcespinning® machine at 4000 rpm under room temperature to check the optimal solution concentrations. The resulting fiber morphology revealed that fibers produced by 65% w/v solution were optimal fibers with good yielding and uniformity.

2.4 Preparation of Drug- Loaded PCL Fibers

2.4.1 Curcumin-Loaded Fibers

Curcumin was dissolved in the polymer solution at 5% w/w, which is 0.65g with 13g of PCL and 20ml of DCM.[10,14] Curcumin-loaded fibers were produced using the Forcespinning® machine at 4000 rpm under room temperature.[1,14]

2.4.2 Ibuprofen-Loaded Fibers

Ibuprofen was dissolved in the polymer solution at 5% w/w, which is 0.65g with 13g of PCL and 20ml of DCM.[8,12] Ibuprofen-loaded fibers were produced using the Forcespinning® machine at 4000 rpm under room temperature.[1,5]

2.4.3 Fabrication of the Bilayer Drug Fiber Patch

A bilayer patch was fabricated by sequential deposition of both the drug-loaded fiber layers. First, an Ibuprofen-loaded PCL solution (5% w/w at 65% w/v PCL in DCM) was fabricated into a mat of fibers and then left to dry completely so that all the DCM could evaporate.[13]Without removing the first layer of Ibuprofen, a Curcumin-loaded PCL solution was poured at a constant rate into the spinneret to fabricate Curcumin-loaded fibers over the Ibuprofen-loaded fibers.[12,16] This results in a free-standing, self-cohesive bilayer patch fiber matrix, intended for independent release of two active pharmaceutical drugs.[18]

2.4.4 Fabrication of Dual-Drug Patch

A dual-Drug Loaded Patch was fabricated with both curcumin and Ibuprofen in a single patch of PCL fiber Matrix. This scaffold contains both curcumin and Ibuprofen mixed together with PCL and DCM. Both drugs have been incorporated in the ratio of 5% w/w with PCL in the solvent (DCM). [10,12,17]

2.5 Characterization

2.5.1 Scanning Electron Microscopy (SEM)

The morphological properties, such as surface, diameter distribution, degree of fusion of fibers, and any other structural defects, were assessed by Scanning Electron Microscopy. The SEM images showed the formation of fibers; however, as the solution concentration changed, variations in fiber uniformity and thickness were observed. Initially, the fibers were fabricated with different concentrations 63%- 67% w/v. At 63% w/v, it was observed that the fibers were fused, and globules were observed. At 65% w/v, finer fibers were observed with fewer globules. At 67% w/v, thick fibers with thick globules were observed in the patch. Drug-Loaded patches also fall under the category of microparticles at 5% drug-to-polymer formulation.

PCL Concentration(%)	PCL mass(gm)	Fiber Diameter	Globule Frequency	Fused Fibers	Uniformity
63	12.6	Thin	Moderate	High	Poor
65	13	Fine	Minimal	Minimal	Good
67	13.4	Thick	High	High	Poor

Table 1. PCL Concentration Analysis

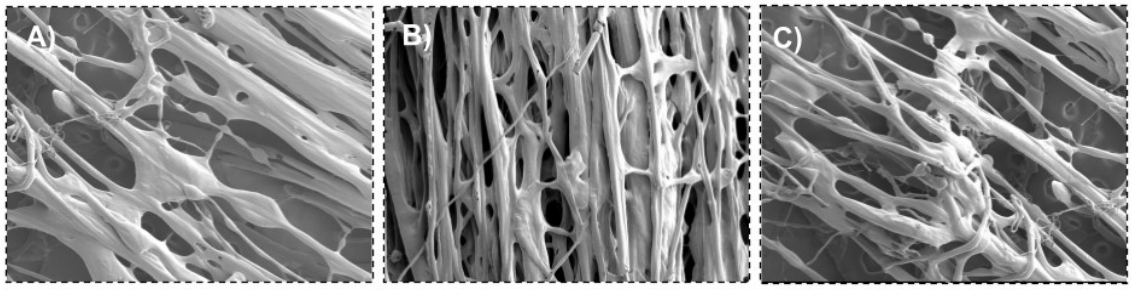


Fig 1: SEM micrographs of PCL fibers produced at different polymer concentrations: **(A)** 63 wt/v%, **(B)** 65 wt/v%, and **(C)** 69 wt/v%.

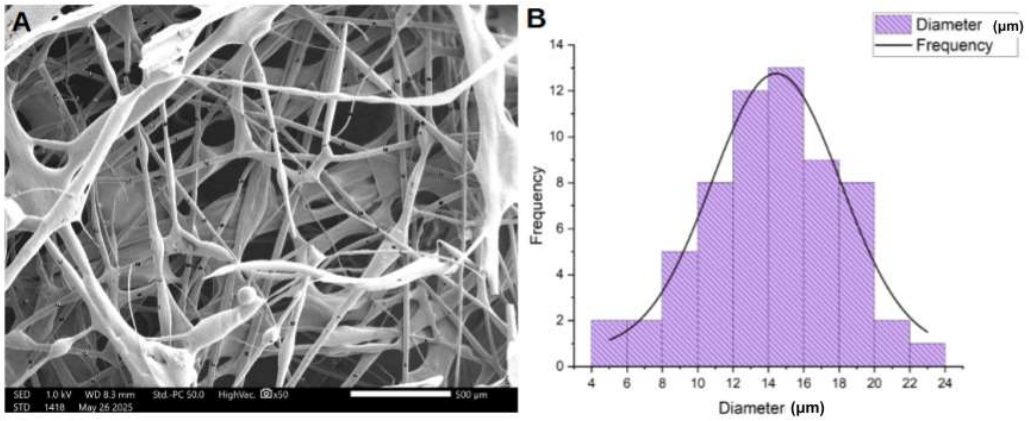


Fig 2: A) shows PCL fiber formation through optimized Forcespinning® method with minimal globules. **B)** shows the fiber distribution curve with maximum frequency of 14-16 µm diameter fibers.

2.5.2 FTIR

Fourier-transform Infrared Spectroscopy was developed to investigate the chemical structure. The FTIR spectra were acquired over 400-4000cm. [12]

The FTIR spectra of pure PCL fibers exhibit the lowest absorbance. A prominent peak was absorbed at 1720cm, corresponding to the ester carbonyl group. Then, the FTIR of Ibuprofen exhibits the highest overall absorbance profile,e followed by pure Curcumin, both

of which are consistent with its aromatic and carboxylic groups. [12,14,8] In all drug-loaded formulations, the characteristic absorption peaks of both DCL and drug were retained. The Ibuporfen+curcumin mixed fiber shows an intermediate absorbance profile, showing the contribution of both compounds.[13,16] The bilayer patch shows an overall absorbance profile across 1000-2500, likely attributed to the layered distribution of the drug across two distinct regions and the partial shielding of the inner drug layer.[18]

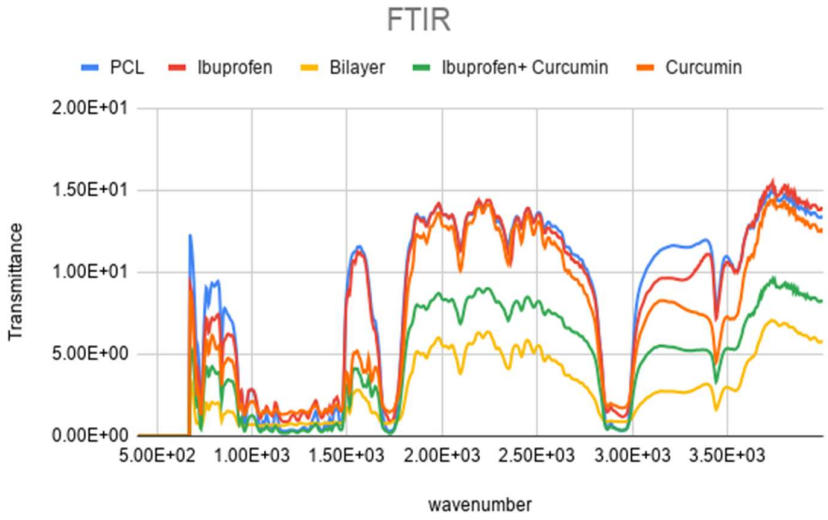


Fig 3: FTIR of patches

2.5.3 UV-Visible Spectroscopy

UV-Visible Spectroscopy was performed over 200-350nm wavelength to confirm the drug incorporation and assess optical interactions between drug molecules and the PCL matrix. [10,17]

UV-VIS spectra of drug-loaded fibers at 5% w/w exhibit absorbance peaks consistent with their respective pure form. Ibuprofen displayed a stable absorbance of 2.05-2.10AU across the range.[14] Pure Curcumin maintained absorbance of 1.85-2.00AU throughout the range. Ibuprofen+ curcumin mixed fiber exhibits the lowest combined absorbance 1.78-1.85 AU, which may reflect partial quenching or distribution of both the drugs, but in contrast, the bilayer patch exhibits the highest absorbance 2.30-2.40 AU across the entire range.[17,18]

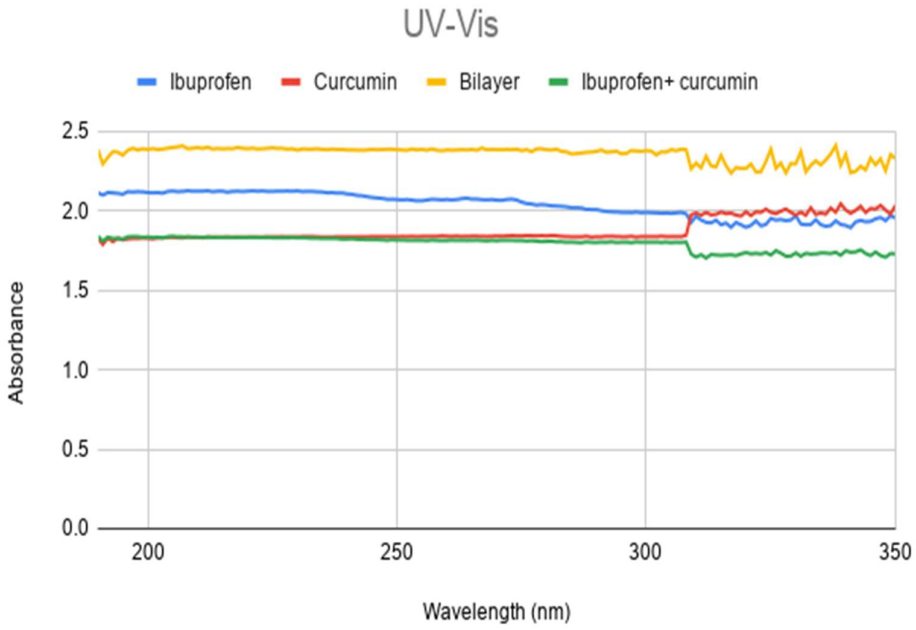


Fig 4: Uv-Vis of Patches

2.6 Mathematical Modelling

A modelling framework was established in this work to study the drug release process. The first stage is fiber formation, driven by centrifugal forces; the second stage studies the distribution of drugs within the fiber patch, and the third stage describes the drug release kinetics in the microfiber patches. By combining these altogether into a single approach, the final model concludes how the system of the drug-loaded microfibers behaves under different processing conditions. This system reduces the repetition of trial experiments, which can be time-consuming and costly. [20, 22]

The modelling approach in our work mainly considers the conditions such as rotational speed, solution viscosity, and polymer or drug concentration to the overall behavior of the system. [6, 22]

2.6.1 Fiber Formation

Microfiber formation by using the Forcespinning® technique is controlled by the balance between centrifugal and viscous forces, and the surface tension of the polymer solution. Curcumin and ibuprofen were incorporated into the solution of PCL (polycaprolactone) and DCM at a level of 5% w/w relative to the PCL [7, 8, 14]. For each formulation, 13g (w) of PCL was dissolved in 20ml (v) of DCM, and the required drug was added to the solution. The solution was made separately with each of the drugs one by one. The drug-loaded polymer solution of DCM and PCL rotates at a very high rotational speed of 4000 rpm; the centrifugal force stretches the solution in an outward direction, which creates the jet and stretches the solution into fibers [5,6, 33]. The centrifugal force acting on a fluid element of mass (m) at radial position (r) rotating at angular velocity (ω) is:

$F = m\omega^2 r$

To form the continuous fibres, centrifugal force must be strong enough to overcome the surface tension of the solution. If the force becomes weak, the liquid breaks into droplets [22, 33]. By combining the viscous-centrifugal force balance with mass conservation and jet acceleration scaling. The cubic scaling law [22,5] for the fiber diameter (d) as a function of process parameters is obtained:

$d \propto (\eta \cdot Q / \rho \cdot \omega^2 \cdot r)\ \hat{}\ (1/3)$

where (η) represents the viscosity of the polymer solution, (Q) represents the volumetric flow through the orifice, (ρ) is the density of the solution, (ω) is the angular velocity in radians per second, and (r) represents the radius of the spinneret. The strength of the force depends on the orifice size, which is set at 1.5 cm. The distance between the spinneret and the collector plate is set at 5 cm. This equation indicates that the higher rotational speed stretches the solution and decreases the fiber diameter, producing thinner fibers. Viscosity resists the deformation and slows the stretching process, due to which thicker fibers can be formed [6, 22]. These results can be seen from the results of SEM analysis presented.

2.6.2 Drug Distribution

Once the drug-loaded fibers are formed, the distribution of the drug within the patch becomes more considerable. Several assumptions were made in this approach. The drug was assumed to be uniformly dissolved in the polymer solution before fiber formation [10, 17]. During a jet flight, the DCM evaporates rapidly, allowing drug molecules to diffuse within the liquid jet before complete solidification [10,28].

As the jet travels from the spinneret to the collector, DCM evaporates rapidly, which causes the faster solidification of the solution at the surface region. Drug transport inside the cylindrical fiber can be described by Fick's second law [11] of diffusion in cylindrical coordinates [11, 28], where drug concentration varies with radial position and time, and the effective diffusion coefficient, which represents the drug mobility within the polymer solution. Fick's second law in cylindrical coordinates:

$\partial C/\partial t = D \cdot (1/r) \cdot \partial/\partial r\ (r \cdot \partial C/\partial r)$

Where (C) is the drug concentration at radial position (r) and time (t), (D) is the effective diffusion coefficient of the drug in polymer solution. This equation describes the movement of the drug molecules from a higher concentration to a lower concentration within the fiber. As DCM evaporates in ~30 seconds, the outer surface of the fibre solidifies early, which creates a concentration gradient. This creates a core-rich drug distribution, the drug concentration becomes higher near the centre of the patch and lower near the surface. This analysis is confirmed by the radial equation[17, 10] shown:

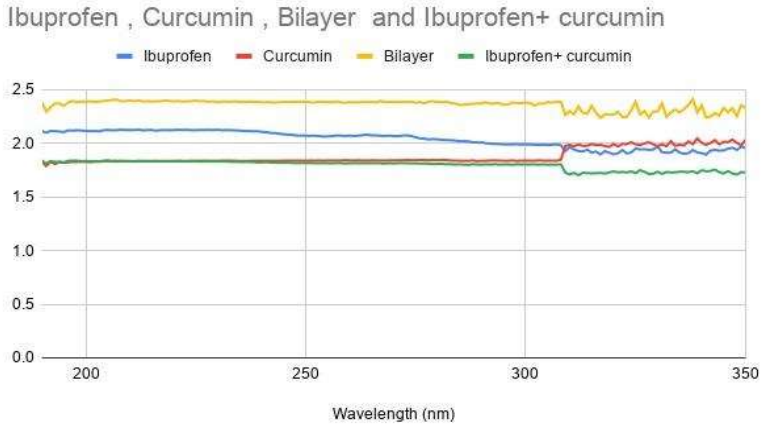
$C(r) = C_0 \cdot \exp(-\beta \cdot r^2)$

Where (C_0) is the central drug concentration and (β) is a parameter related to the evaporation and diffusion rate. The drug concentration was measured by using Beer-Lambert's law [7, 14].

$A = \varepsilon l C$

Where (A)is the absorbance of the solution, (ε) is the molar absorptivity, (l) is the length of the path of the cuvette, and (C) is the drug concentration.

To determine the drug concentration inside the fibre, UV-Visible Spectroscopy has been proposed to validate the model. In UV-Visible spectroscopy, to determine the drug concentration [17], a known mass of drug-loaded fiber mat is dissolved in DCM to extract the drug completely, and the absorbance of the solution is calculated. The graph is then obtained by UV-Visible spectroscopy, which shows the drug distribution within the fiber.



The UV-Visible spectra were recorded between 200 and 350 nm to verify the presence of drugs inside the fiber. Pure ibuprofen showed stronger absorption in the 220-280 nm, associated with the aromatic ring transition. Pure Curcumin showed absorption extending to the higher wavelengths due to its conjugated structure. This spectrum confirms that the drugs were uniformly incorporated into the fiber patches during fiber formation. In the bilayer and combined drugs patch, the absorption region confirms that each drug is successfully encapsulated within the fiber. The slight broadening and reduced intensity of the peaks indicate that the drugs are dispersed within the polymer matrix rather than existing as large crystalline. No additional peaks were observed, indicating that the processing condition did not cause chemical degradation.

When samples from different regions of the patch were analysed,d the middle region showed stronger absorbance than the outer region. According to Beer-Lambert's law, higher absorbance means higher concentration of drugs. And hence, the amount of drug is higher at the centre and lower near the surface.

2.6.3 Drug Release

When the drug-loaded patches were formed, the drug got distributed within the patch. The final stage is to analyse the release of the drug from the patch. In this system, the formulation of PCL fibers was done separately from the curcumin and ibuprofen drugs. The

drug release mainly occurs through the diffusion process [9, 7, 10, 12]rather than dissolution because the solidification of fibers starts before reaching the collector plate due to the rapid evaporation of DCM.
When the drug-loaded fibre patch is immersed in the aqueous solution of phosphate buffer saline (PBS pH 7.4), the drug located near the surface of the patch dissolves first and then diffuses rapidly into the solution [9, 10]. After the initial stage, the leftover drug travels through the polymer network and then reaches the fibre surface. To analyse the release behaviour with time, the Higuchi model has been proposed.

Experimental Drug Release Data
Using UV-Visible spectroscopy, the drug absorbance data is obtained over a 4-hr window in PBS (pH 7.4).

Time (Hr)	Time (Hr^0.5)	Absorbance (AU)	Absorbance (Avg.)	Drug Release (%)	Interpretation
0	0	0	0	0	No release
0.5	0.707	0.12-0.22	0.17	~19	Initial burst
1.0	1.000	0.22-0.38	0.3	~34.5	Early diffusion
2.0	1.414	0.38-0.55	0.465	~53.5	Diffusion start Dominating
3.0	1.732	0.55-0.70	0.625	~73.5	Slower release
4.0	2.000	0.70-0.95	0.825	~87.5	Controlled release

Higuchi Diffusion Model
The Higuchi model is one of the earliest and most widely used mathematical models for drug release behaviour from the drug-loaded microfiber patches [9, 27, 10]. When the patch gets immersed in the PBS solution, it is observed that the diffusion path increases with time, causing a gradual decrease in the release rate. The Higuchi model, derived from Fick's second law of diffusion, assumes uniform drug distribution within the patch, negligible polymer swelling, and constant diffusion coefficients [9, 27, 12]. It is observed through this equation that initially the drug release is fast and slows down over time. A high surface-to-volume ratio of fibres enhances the early diffusion process and produces the noticeable burst release in the initial hours.
The linear relation between cumulative drug release and the square root of time confirms the applicability of the Higuchi model [10, 13]. The Higuchi equation 9, 27] has been used to predict the behaviour:
$Mt = kH \sqrt{t}$ or $Mt / M\infty = kH \sqrt{t}$
Where (Mt) is the drug release at time (t), (M∞) is the total drug encapsulated, and (kH) is the Higuchi constant. A graph is obtained from this relation of drug release over time, which states that initially, drug release occurs at a fast rate and gradually decreases with time.
By the experimental data, a calibration curve of Absorbance and Time, Drug release vs Time (Hr^0.5) is obtained, shown in the figure.

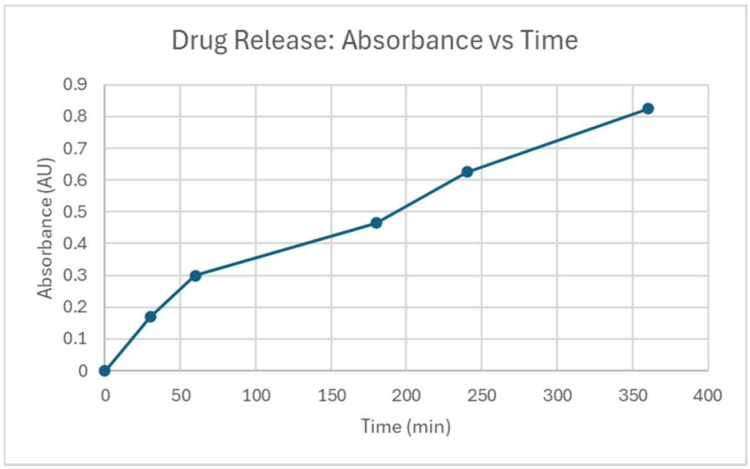


Fig 5. Showing behaviour of Drug Release

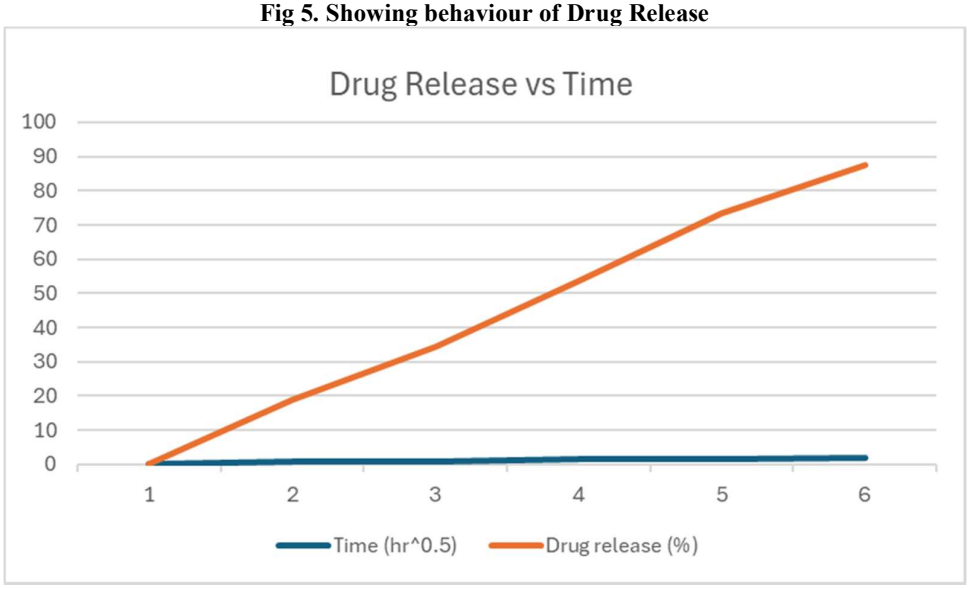


Fig 6. Higuchi Model: Drug Release vs Time

Conclusion

In this work, polycaprolactone (PCL) microfibers were fabricated with the DCM solvent using the Forcespinning® Method at three different concentrations: 63%, 65%, and 67%. After analysing all the results, it was observed that at 65%, fibers are fine with fewer globules or fused fibers. Drug-loaded PCL microfibrous scaffolds were successfully fabricated with a concentration of 5% drug-to-polymer ratio. Four types of drug-loaded patches were fabricated: Curcumin-loaded fibers(5% w/w), Ibuprofen-loaded Fibers (5% w/w), Mixed dual-drug fiber, and a Bilayer Dual- Drug fiber patch.

SEM confirmed the morphological superiority, including minimal globule formation, uniform diameter, and fewer fused fibers. FTIR analysis confirmed the retention of characteristics of all the functional groups peaks of PCL, Curcumin, and Ibuprofen. The graph of FTIR shows that Ibuprofen exhibits the highest absorbance, followed by curcumin, mixed dual-drug, bilayer, and PCL.

UV-Visible Spectroscopy characterizes the stable absorbance profiles of all formulations. The bilayer patch exhibits the highest absorbance because of its cumulative contribution of both drugs, followed by Ibuprofen, curcumin, and the mixed dual-drug. Time v/s absorbance graph shows the three-phase profile. Surface burst, fast early diffusion, and controlled release. This was demonstrated using mathematical modelling and the graph of time versus absorbance for the Higuchi Model.

In Conclusion, the PCL microfiber system was developed using the Forcespinning fabrication method; the fabricated PCL-drug-loaded fibers represent a chemically compatible, optically stable, and mathematically validated platform for controlled co-delivery of therapeutics.

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