

**MOTOR – DRIVEN FABRICATION OF POLYPROPYLENE FIBER
BASED MULTI-LAYER MATERIALS USING CHITOSAN/GELATIN BINDER:
THICKNESS, WETTABILITY, AND SWELLING PROPERTIES**

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Abstract:

Polypropylene (PP) is a thermoplastic with many advantages: mechanical strength, lightweight, good heat resistance ($\approx 120\text{--}140^\circ\text{C}$), chemical resistance, and non-toxicity, making it widely used in medicine, pharmaceuticals, and food applications such as syringes, test tubes, infusion tubes, pill and powder containers, bottle caps, jar lids, etc.

To date, the method of fabricating multilayer polypropylene materials remains traditional manual, resulting in poor repeatability and scalability. In this study, PP-based multilayer materials using chitosan/gelatin (CS/Gel) binder were fabricated using a motor-driven fabrication system (CHITEC). Structures with 8, 16, and 32 layers were successfully fabricated and evaluated for thickness, wettability, and swelling capacity.

Thickness increases with the number of layers but exhibits a non-linear and uneven distribution, with greater accumulation in the central region due to binder displacement and the influence of compressive forces. Cold plasma treatment significantly improves wettability, reducing the contact angle from $127\text{--}129^\circ$ to $57\text{--}61^\circ$, indicating a transition from hydrophobic to hydrophilic surfaces. Swelling studies show rapid water absorption, with peak swelling rates of approximately $\sim 270\%$ (untreated) and $\sim 228\%$ (plasma treated), indicating improved structural stability after plasma treatment. This study demonstrates a scalable method for fabricating PP multilayer materials with tunable properties for advanced applications.

Keywords: *Polypropylene; CHITEC 3D equipment; Multilayer fiber; Thickness; Wettability; Swelling*

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1. Introduction

Multilayer polymer materials have attracted significant attention due to their versatile applications in fields such as food packaging, biomedical devices, filtration, and advanced composites. Their unique structure, consisting of stacked layers with tunable thickness and interfacial properties, enables the design of materials with enhanced mechanical strength, barrier performance, and functional surface characteristics (Wenyuan et al., 2023; Godaet et al., 2020; Arif et al., 2019; Wiktoria et al., 2023). In particular, multilayer systems based on polymer fibers offer advantages in flexibility, porosity control, and surface modification, making them promising candidates for next-generation functional materials (Holmberg et al., 2021; Terekhov et al., 2021).

Among synthetic polymers, polypropylene (PP) is widely used due to its low cost, chemical resistance, lightweight nature, and excellent mechanical properties. Nonwoven PP fibers, in particular, are extensively applied in filtration, medical textiles, and protective materials (Hossain et al., 2024). However, the inherent hydrophobicity and low surface energy of PP limit its interaction with hydrophilic substances and reduce its applicability in areas requiring wettability, adhesion, or functionalization (Yang et al., 2025). Therefore, the development of effective strategies to assemble

and modify PP-based multilayer materials remains an important research challenge.

To overcome these limitations, biopolymer-based binders such as chitosan and gelatin have been increasingly explored. Chitosan, a natural polysaccharide derived from chitin, exhibits excellent biocompatibility, film-forming ability, and antimicrobial properties, while gelatin provides favorable gelation behavior and adhesion characteristics. The combination of chitosan and gelatin has been reported to improve mechanical stability, interfacial bonding, and processing uniformity in multilayer systems (K. Li et al., 2019, Hassabo et al., 2022). In our previous work (Do et al., 2026), multilayer PP materials were successfully fabricated using a chitosan/gelatin adhesive system through manual layer-by-layer assembly. Although this approach demonstrated the feasibility of producing functional multilayer structures, it suffered from limitations in reproducibility, uniformity, and scalability due to manual processing.

To address these challenges, the development of automated or semi-automated fabrication systems is essential. Motor-driven systems offer precise control over material feeding, coating, and winding processes, enabling consistent layer formation and improved structural uniformity. Such systems also allow better control of processing parameters, including tension, coating thickness, and deposition

rate, which are critical for achieving reproducible material properties. However, the application of motor-driven fabrication techniques for producing polymer fiber-based multilayer materials remains limited.

2. Materials and Methods

Chemicals and reagents

Nonwoven polypropylene fibers (PP), analytical-grade gelatin derived from bovine hide (99.99, molecular weight: 50,000 Da), and chitosan ($\geq 75\%$ deacetylated, molecular weight: 190,000 Da) were purchased from PFNonwovens Group (Czech Republic) and Sigma-Aldrich (USA), respectively.

Equipment

The following instruments were used including an analytical balance (10^{-4} , model XA 82/220/2X) from RADWAG, a thermostatic bath (model KISS K12) and a heating plate from Huber – Germany,

a tensile testing machine (model Z010) with a data analysis software for tensile strength and elongation (testXpert) from ZwickRoell - USA, a Scanning Electron Microscope (SEM, model S-4800) with image processing software ImageJ from Hitachi-Japan, and a digital camera (model VIXIA HF M500) from Canon.

A motor-driven multilayer material fabrication system (CHITEC) was designed, self-fabricated in CEITEC, and presented in Figure 1. The CHITEC system consists of: spool 1 (driven by a stepper motor controlled by a computer) for the PP sheet; spool 2 for tensioning the PP sheet; a container holding the hydrogel solution (gelatin); spool 3 for tensioning the PP sheet after passing through the gelatin solution; and spool 4 (also driven by a computer-controlled stepper motor) for winding the gelatin-coated PP sheet to form the multilayer PP material.

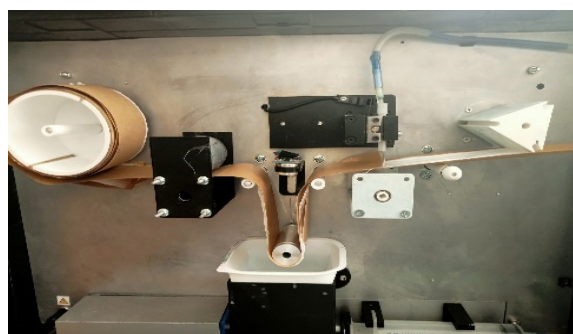
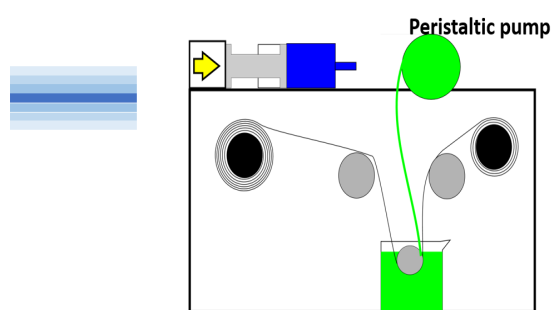


Figure 1. (a) Design of multilayer material fabrication equipment, and (b) equipment

PP/Chitosan/Gelatin multilayer material fabrication

In a previous work (Do et al., 2026), the PP multilayer fibers were successfully manually fabricated using a maximized 5% gelatin hydrogel. To fabricate multilayer materials from PP fibers, a chitosan/gelatin adhesive solution (PP-CS-Gel) was maximized as following: a 20% chitosan solution is first prepared, followed by the preparation of a 20% chitosan / 5% gelatin solution. The fabrication of PP/Chitosan/Gelatin multilayer materials (PP-CS-Gel) by the CHITEC equipment using a Chitosan/Gelatin hydrogel binder (CS-Gel) is carried out according to the following procedure: Step 1. Winding PP material onto spool 1: The polymer material is cut to an appropriate width and wound onto spool 1. Step 2. Coating the PP material with CS/Gel binder: A motor-driven system is used to regulate the rotation speed of the stepper motor driving spool 1, synchronized with the stepper motor driving spool 2. Spool 1 fed the PP sheet through spool 2 and into the hydrogel solution container. As the PP sheet passed through the solution, the hydrogel (gelatin) binder is coated onto both surfaces of the PP sheet,

which then moved upward and passed over spool 3. Step 3. Formation of PP multilayer materials: The PP sheet coated with hydrogel (gelatin) binder is pulled upward by spool 4 and wound onto it to form the first PP layer. The process is continued until the desired number of layers is achieved.

Stabilization of polymer components in multilayer materials by cold plasma treatment

The study was conducted according to the following procedure: Step 1. Cold plasma preparation: Install the cold plasma reaction sample holder (Fig 2a). Place the multilayer material sample from nonwoven PP fibers onto a glass plate that separates it from the reaction surface of the cold plasma sample holder. Step 2. Cold plasma treatment: Remove oxygen from the reaction chamber using nitrogen gas, verify with an alcohol lamp flame, and then gradually replace the nitrogen with hydrogen gas (Fig 2b). Turn on the plasma power supply and stabilize the material sample for 30 minutes (Fig 2c).



Figure 2. (a) Cold plasma equipment, (b) De-oxygen in the chamber, and (c) Cold plasma stabilization process
Wettability measurement by the sessile drop method

The contact angle between the sample surface and a water droplet provides information on wettability, i.e., the hydrophilic/hydrophobic nature of the sample. Among the various contact angle measurement techniques, the sessile drop method is considered the most effective and is widely used to evaluate surface wettability. When a liquid droplet can be easily placed on the sample surface, the wettability analysis is performed automatically using dedicated software. The apparatus used for the sessile drop method is a surface tension measurement system consisting of a camera and a light source. A 20 μL droplet of distilled water mixed with red food dye was deposited onto the sample surface. The contact angle was measured by capturing side-view images of the droplet on the material surface at time 0 s using a Canon VIXIA HF M500 digital camera. Measurements were conducted at 21 $^{\circ}\text{C}$. The

images were then analyzed using image analysis software to obtain accurate contact angle values. Each nonwoven polypropylene multilayer sample with gelatin was measured three times.

Swelling behavior

The swelling properties of multilayer materials are investigated using the gravimetric method. The samples are cut into square pieces with an area of 4 cm^2 (three specimens were prepared from each sample). The sample was weighed before swelling. The samples are then immersed in distilled water, ensuring complete submersion, for swelling at time intervals of 30 minutes, 1 hour, 3 hours, 5 hours, and 48 hours. After immersion, the surfaces of the samples are gently dried using Whatman filter paper. The samples were then weighed and the swelling degree was calculated. The percentage swelling (ΔG) is calculated using the following equation:

$$\Delta G = \frac{G_1 - G_0}{G_0} 100,$$

where: G_0 : mass of the sample before swelling (g) and G_1 : mass of the sample after swelling (g)

3. Results and Discussion

Adhesive chitosan/gelatin mixed solution preparation and motor-driven fabrication of PP multilayer materials

The dissolution of food-grade chitosan powder (up to 30 g) at mass ratios of CS: distilled water of 30:65, 20:75, and 10:85 showed that at a ratio of 30:65, the resulting chitosan solution was thick and highly viscous. At ratios of 20:75 and 10:85, the solutions were more dilute with lower viscosity, making them suitable for fabricating multilayer materials. However, due to the limited solubility of chitosan in hot water, the chitosan solutions, after vigorous stirring and standing, exhibited phase separation. The mass ratio of CS : distilled water of 20:75 (designated as 20% CS solution) was selected for subsequent experiments to achieve the highest

possible CS content in solution. A mixed chitosan/gelatin (CS-Gel) solution was prepared using the CS solution at a CS : distilled water ratio of 20:75, with an overall mass ratio of CS : Gel : water of 20:5:75, designated as 20% CS / 5% Gel solution. The results showed that the 20% CS / 5% Gel solution exhibited mechanical properties (viscosity and adhesion) comparable to the 20% CS solution, but without phase separation. The gel-forming property of Gel improved the uniformity of brush immersion and coating, resulting in more even application of the solution onto the material surface.

Using the CHITEC equipment, three PP multilayer materials based on nonwoven polypropylene fibers with a 20% CS / 5% Gel solution were fabricated and designated as follows: PP-CS-Gel GP-8L for PP 8-layers, PP-CS-Gel GP-16L for 16-layer material, and PP-CS-Gel GP-32L for 32-layer one as presented in Figure 2.

Thickness

The thickness of CS/Gel-bonded PP multilayer materials fabricated using the motor-driven CHITEC system was measured using a caliper, and the results are summarized in Table 1. The thickness increased with the number of layers; however, the relationship was nonlinear and exhibited clear spatial heterogeneity between the edge and central regions of the samples. For the 16-layer material (PP-CS-Gel GP-16L), the thickness ratio relative to the 8-layer sample (PP-CS-Gel GP-8L) was approximately 1.04 at the edge, but increased to about 1.62 at the center. A more pronounced difference was observed for the 32-layer material (PP-CS-Gel GP-32L), where the thickness ratio reached ~ 2.46 at the edge and ~ 3.87 at the center. These results indicate that the increase in thickness does not scale proportionally with the number of deposited layers, particularly at the edges. This non-uniformity suggests that the multilayer structure is strongly influenced by binder distribution and processing conditions. During fabrication, the CS/Gel solution may migrate toward the central region due to capillary forces and gravity, leading to higher binder accumulation

and reduced compaction in the middle of the sample (Yang et al., 2016). In contrast, the edge regions may experience lower binder retention and higher compressive forces during winding, resulting in a denser and thinner structure (Thomas and Valentin, 2006). Additionally, uneven pressure distribution during layer formation may further contribute to this effect, as higher localized pressure at the edges can enhance densification. Limited wetting near the boundaries, due to insufficient binder coverage, may also restrict effective layer buildup in these regions. Furthermore, minor misalignment or slippage between layers during the winding process could reduce the effective stacking efficiency, particularly at the sample edges (Evon and Reika, 2022).

Overall, although the motor-driven system enables controlled multilayer fabrication, the results highlight that thickness uniformity remains a critical challenge. Future optimization should focus on improving binder distribution, controlling tension and pressure during winding, and ensuring uniform coating across the entire material width to achieve more homogeneous multilayer structures.

Table 1. Thickness of motor-driven fabricated CS/Gel binder PP multilayer materials using a caliper

No	Sample	Layer ratio compared to 8-layer material (times)	Edge region		Middle region	
			Thickness (mm)	Thickness ratio compared to the 8-layer material (times)	Thickness (mm)	Thickness ratio compared to the 8-layer material (times)
1	PP-CS-Gel GP-8L	1	2,46 \pm 0,32	1	1,43 \pm 0,15	1
2	PP-CS-Gel GP-16L	2	2,55 \pm 0,27	1.04	2,31 \pm 0,2	1.62
3	PP-CS-Gel GP-32L	4	6,06 \pm 0,93	2.46	5,54 \pm 0,27	3.87

Cold plasma treatment and wettability

To evaluate the effect of surface modification, the multilayer material based on nonwoven polypropylene fibers with a 20% CS / 5% Gel binder (PP-CS-Gel GP) was subjected to cold plasma

treatment and denoted as PP-CS-Gel GP-Pla. Fig. 3 and Table 2 presented the surface wettability of both untreated and plasma-treated samples assessed using contact angle measurements.

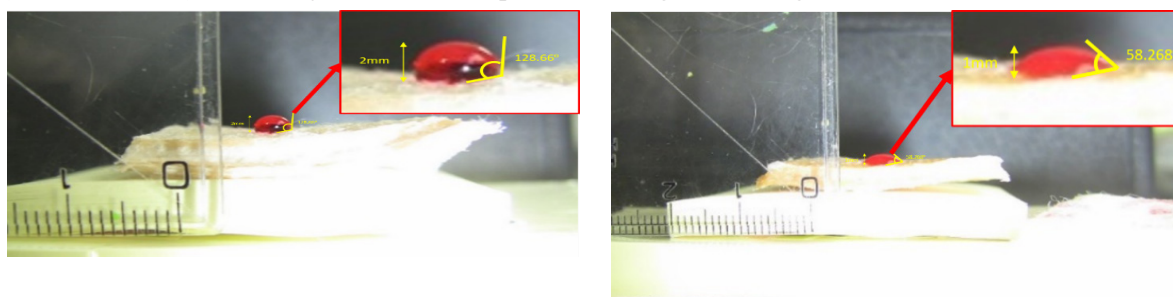


Figure 3. Contact angle images:(a) PP-CS-Gel GP, and (b) PP-CS-Gel GP-Pla

Table 2. Contact angle and wettability

No	Sample	Treatment	Contact angle	Wettability
1	PP-CS-Gel GP	No	127° - 129°	Hydrophobic
2	PP-CS-Gel GP-Pla	Cold plasma	57° - 61°	Good

The untreated PP-CS-Gel GP sample exhibited a high contact angle in the range of 127–129°, indicating pronounced hydrophobic behavior. This result is consistent with the intrinsic characteristics of polypropylene, which possesses a nonpolar structure and low surface energy. Although the presence of gelatin introduces hydrophilic components into the multilayer system, these components are likely embedded within the internal structure or insufficiently exposed at the surface, resulting in limited influence on surface wettability. In contrast, the plasma-treated sample (PP-CS-Gel GP-Pla) showed a significant decrease in contact angle to 57–61°, clearly indicating a transition to hydrophilic behavior and substantially improved wettability. This pronounced change confirms the effectiveness of cold plasma treatment as a surface modification technique for PP-based multilayer materials. The improvement in wettability can be attributed to several synergistic mechanisms induced by plasma treatment. Firstly, the incorporation of polar functional groups (e.g., –OH, –COOH) onto the polypropylene surface increases surface energy and enhances affinity toward water molecules. Secondly, plasma exposure removes surface contaminants and weak boundary layers, thereby activating the surface

and promoting better interaction with liquids. Thirdly, mild surface etching may increase micro-scale roughness, contributing to improved wetting behavior. Additionally, plasma treatment may alter the interfacial structure, facilitating greater exposure of the hydrophilic gelatin binder at the outer surface (Pantoja et al., 2013; Primc and Mozetič, 2024). The study showed that these combined effects lead to a substantial reduction in contact angle and a marked enhancement in wettability. From an application standpoint, such a transition from hydrophobic to hydrophilic behavior is highly desirable for applications requiring efficient liquid interaction, including filtration, biomedical materials, drug delivery systems, and absorbent structures.

Swelling behavior

The results summarized in Table 3 and illustrated in Fig. 5 showed the swelling behavior of the 16-layer multilayer materials before and after cold plasma treatment, denoted as PP-CS-Gel GP-16L and PP-CS-Gel GP-16L-Pla, was investigated using the gravimetric method. The mass showed negligible change compared to the initial mass; specifically, the initial mass and the mass after swelling and complete drying were 408,21g and 408,15g, respectively.

Table 3. Swelling behavior of 16-layer PP mats before and after cold plasma treatment

Sample		PP-CS-Gel GP-16L	PP-CS-Gel GP-16L-Pla
Initial weight (mg)		408,21	338,27
0.5 h	Weight (mg)	789,83	602,64
	Swelling ratios (%)	193,49	178,15
1 h	Weight (mg)	900,65	634,24
	Swelling ratios (%)	220,63	187,50
3 h	Weight (mg)	976,65	645,43
	Swelling ratios (%)	239,25	190,80
5 h	Weight (mg)	1004,94	656,67
	Swelling ratios (%)	246,18	194,13
48 h	Weight (mg)	1103,21	771,21
	Swelling ratios (%)	270,26	227,99

Both samples exhibited rapid water uptake during the initial immersion period, with the majority of swelling occurring within the first 0.5–3 hours. This fast swelling kinetics can be attributed to the porous structure of the nonwoven PP fibers and the presence of the hydrophilic chitosan/gelatin (CS/Gel) binder, which facilitates water diffusion into the multilayer network. After this initial stage, the swelling rate gradually decreased, approaching equilibrium over prolonged immersion (up to 48 hours). The untreated sample (PP-CS-Gel GP-16L) showed a higher swelling capacity, reaching approximately 270.26% after 48 hours. In comparison, the plasma-treated sample (PP-CS-Gel GP-16L-Pla) exhibited a reduced swelling ratio of about 227.99% under the same conditions. Despite this decrease, both materials maintained substantial water absorption capacity, confirming their suitability for applications involving aqueous environments. After immersion for 3 hours followed by complete drying (surface dried with filter paper and then air-dried under gentle airflow for 30 minutes), the mass of the PP-CS-Gel GP-Pla sample also showed little change compared to its initial mass; specifically, the initial mass and the mass after swelling and drying were 976,65g and 645,43g, respectively. The slight reduction in swelling behavior after cold plasma treatment can

be attributed to structural stabilization within the multilayer system. Plasma exposure may induce partial crosslinking or densification of the polymer network, including both the polypropylene fiber surface and the CS/Gel binder (Joshua et al., 2018). This results in a more rigid and compact structure, which restricts the penetration and retention of water molecules. Additionally, enhanced interfacial bonding between layers after plasma treatment may further limit the expansion of the material during swelling. Notably, both samples demonstrated good structural stability after swelling–drying cycles. The mass of the samples after complete drying remained nearly unchanged compared to their initial mass, indicating that no significant material loss or dissolution occurred during immersion. This suggests that the CS/Gel binder system provides sufficient integrity to maintain the multilayer structure under wet conditions. Overall, the results indicate that while cold plasma treatment effectively improves surface wettability, it slightly reduces the swelling capacity due to increased structural stability. This balance between wettability and swelling behavior is advantageous for tailoring the material toward specific applications such as filtration, biomedical scaffolds, and absorbent systems.

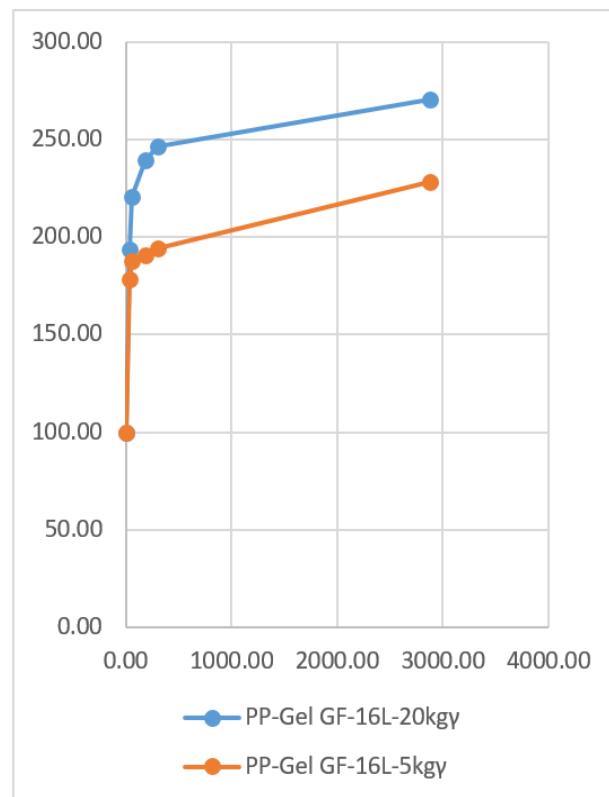


Figure 4. Swelling ratio–time plots of PP-CS-Gel GP (blue) và mẫu PP-CS-Gel GP-Pla (brown)

4. Conclusion

In this study, a motor-driven fabrication system (CHITEC) was successfully developed and applied for the preparation of polypropylene (PP) fiber-based multilayer materials using a 20% chitosan/5% gelatin (CS/Gel) adhesive system. Compared to previously reported manual fabrication methods, the motor-driven approach enabled improved process control, enhanced reproducibility, and the ability to produce multilayer structures with varying numbers of layers (8, 16, and 32 layers). The results demonstrated that the CS/Gel binder system provided suitable viscosity, adhesion, and coating uniformity for multilayer formation without phase separation.

Thickness analysis revealed that although the overall thickness increased with the number of layers (8-, 16-, and 32-layers), the growth was nonlinear and spatially heterogeneous, with significant differences between edge and center regions. This behavior highlights the influence of binder distribution, capillary flow, and mechanical compression during fabrication, suggesting that further optimization of processing parameters is required to achieve uniform structures.

Cold plasma treatment proved to be an effective post-processing method for modifying surface properties. A significant reduction in contact angle from $\sim 127\text{--}129^\circ$ to $\sim 57\text{--}61^\circ$ confirmed the transition from hydrophobic to hydrophilic behavior, attributed to surface activation, introduction of polar functional groups, and improved exposure of the gelatin component. This enhancement in wettability expands the potential applicability of the materials in areas such as filtration, biomedical devices, and absorbent systems.

Swelling studies indicated rapid water uptake within the first few hours, followed by stabilization. While cold plasma treatment slightly reduced the swelling capacity, likely due to structural stabilization or partial crosslinking, the overall swelling behavior remained substantial, demonstrating the material's ability to interact with aqueous environments.

Our study provides a scalable and controllable strategy for fabricating PP-based multilayer materials with tunable structural and surface properties. The integration of a motor-driven system with biopolymer binders and plasma surface modification offers a promising platform for the development of advanced functional materials. Future studies should focus on optimizing process parameters to improve thickness uniformity, investigating mechanical performance, and exploring application-specific functionalities.

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Statements and Declarations

Not applicable.

Declaration of Conflicting Interests

The authors declared no potential conflicts of interest with respect to the research, authorship, and/or publication of this article.

Declaration of AI Technology Usage

The authors declare that no Artificial Intelligence (AI) technologies or AI-assisted tools were utilized in any capacity during the writing and preparation of this article

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**CHẾ TẠO VẬT LIỆU ĐA LỚP TỪ SỢI POLYPROPYLENE
BẰNG HỆ THỐNG TRUYỀN ĐỘNG MOTOR
SỬ DỤNG CHẤT KẾT DÍNH CHITOSAN/GELATIN:
ĐỘ DÀY, ĐỘ THẤM ƯỚT VÀ KHẢ NĂNG TRƯỞNG NỞ**

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Tóm tắt:

Polypropylene (PP) là một loại nhựa nhiệt dẻo có nhiều ưu điểm: bền cơ học, nhẹ, chịu nhiệt tốt ($\approx 120-140^\circ\text{C}$), kháng hóa chất, không độc nên được ứng dụng rộng rãi trong y tế, dược phẩm và thực phẩm như bơm tiêm, ống nghiệm, ống truyền dịch, lọ đựng thuốc viên, thuốc bột, nắp chai, nắp lọ, v.v.

Đến nay, phương pháp chế tạo vật liệu Polypropylene đa lớp vẫn là thủ công truyền thống nên có độ lặp lại và khả năng mở rộng kém. Trong nghiên cứu này, vật liệu đa lớp nền PP sử dụng chất kết dính Chitosan/Gelatin (CS/Gel) được chế tạo bằng hệ thống chế tạo truyền động bằng motor (CHITEC). Các cấu trúc gồm 8, 16 và 32 lớp đã được chế tạo thành công và đánh giá về độ dày, độ thấm ướt và khả năng trương nở.

Độ dày tăng theo số lớp nhưng thể hiện sự phân bố không tuyến tính và không đồng đều, với sự tích tụ lớn hơn ở vùng trung tâm do sự dịch chuyển chất kết dính và ảnh hưởng của lực nén. Xử lý Plasma lạnh cải thiện đáng kể độ thấm ướt, làm giảm góc tiếp xúc từ $127-129^\circ$ xuống $57-61^\circ$, cho thấy sự chuyển đổi từ bề mặt kỵ nước sang ưa nước. Nghiên cứu trương nở cho thấy khả năng hấp thụ nước nhanh, với tỷ lệ trương nở cực đại khoảng $\sim 270\%$ (không xử lý) và $\sim 228\%$ (xử lý Plasma), cho thấy độ ổn định cấu trúc được cải thiện sau xử lý Plasma. Nghiên cứu này chứng minh một phương pháp có khả năng mở rộng để chế tạo vật liệu đa lớp PP với các tính chất có thể điều chỉnh cho các ứng dụng tiên tiến.

Từ khóa: Polypropylene; Thiết bị CHITEC 3D; Sợi đa lớp; Độ dày; Độ thấm ướt; Trương nở.

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