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Research on Fabrication and Physico-Chemical Properties of Polypropylene Fiber-Based Multilayer Materials

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ABSTRACT

This study presents the fabrication and physicochemical characterization of polypropylene (PP) fiber-based multilayer materials using a gelatin hydrogel as a binder. A simple manual multilayer fabrication method was developed to assemble controlled nonwoven PP 8-, 16-, and 32-layer fibers into structured composites. The effect of fabrication parameters, particularly the volume of 5% gelatin solution, was optimized, with 150 μ L per layer identified as suitable for achieving uniform adhesion and relatively thin structures. The 3 resulting PP multilayer materials were evaluated in terms of thickness distribution, wettability, and swelling behavior. Thickness measurements revealed non-linear scaling with layer number and noticeable structural non-uniformity between edge and central regions, attributed to binder migration and manual processing conditions. Surface wettability of the PP 16-layer fibers showed that untreated samples exhibited hydrophobic behavior (contact angle $>90^\circ$), while cold plasma-treated samples displayed significantly improved wettability (contact angle $<90^\circ$), indicating enhanced surface hydrophilicity due to plasma-induced functionalization. Swelling studies demonstrated rapid water uptake within the first hours, followed by stabilization. The overall swelling capacity of the materials remained high at 442,33% by stabilization, with minimal differences between untreated and

plasma-treated samples after prolonged immersion, suggesting that plasma treatment did not compromise structural integrity, although a slight reduction in swelling was observed due to increased network stabilization. The study showed that the combination of PP fibers, gelatin binder, and cold plasma treatment enables the fabrication of multilayer materials with tunable physicochemical properties. These materials show promising potential for applications in filtration, biomedical systems, packaging, and absorbent materials.

Keywords: Polypropylene, Multilayer fiber, Cold plasma, Thickness, Wettability, Swelling

1. INTRODUCTION

Single-layered films based on biopolymers have many operational drawbacks. As the biological materials with a single-layer structure, compared to multi-layer ones, have a faster decomposition time, the production of multi-layered materials is a specific answer to minimize these limitations (Alias et al., 2022). In recent decades, multilayer polymer-based materials have attracted significant attention due to their versatile structure and tunable physicochemical properties, enabling wide-ranging applications across advanced engineering, healthcare, textiles, and packaging industries (Wenyuan et al., 2023; Godaet al., 2020; Arif et al., 2019; Wiktorina et al., 2023). Particularly, fiber-reinforced multilayer systems combining microfibers, nanofibers, and functional binders have demonstrated superior mechanical strength, lightweight characteristics, thermal insulation, and environmental resistance compared to conventional single-layer materials (Holmberg et al., 2021; Terekhov et al., 2021). These advantages make them promising candidates for next-generation functional materials in both industrial and biomedical contexts.

Among various polymeric materials, polypropylene (PP) fibers have been extensively utilized due to their low cost, chemical stability, hydrophobicity, and favorable mechanical properties. However, their inherently low surface energy and limited interfacial adhesion often restrict their integration into high-performance composite systems (Hossain et al., 2024). Nonwoven PP fabrics, in particular, provide a flexible and porous structure that can serve as an effective substrate in multilayer assemblies (Yang et al., 2025).

The incorporation of hydrogel-based binders such as gelatin, alginate, and chitosan has emerged as a promising strategy to enhance interlayer bonding, improve wettability, and introduce additional functional properties such as biocompatibility and swelling capacity (K. Li et al., 2019). Gelatin is a high-molecular-weight, water-soluble protein that is abundant, low-cost, biocompatible, and highly compatible with synthetic polymers. Owing to its excellent film-forming ability, gelatin along with other proteins and polysaccharides has attracted considerable interest as a promising material for packaging applications. Furthermore, its intrinsic gelling, thickening, emulsifying, and encapsulating properties enable the formation of high-quality biodegradable films (Hassabo et al., 2022).

The stability of polymer components within multilayer systems remains a critical challenge, particularly under environmental or mechanical stress. Techniques such as cold plasma treatment, beta irradiation, and chemical crosslinking have been explored to stabilize polymer structures and improve long-term durability (Heidemann et al., 2019; Karthik et al., 2024). Despite growing interest, existing studies have largely focused on conventional fabrication methods, such as manual lamination, which often fail to achieve the stringent

technical requirements for advanced applications. These include high tensile strength, lightweight structure, thermal and acoustic insulation, flame resistance, and environmental durability (Maha et al., 2025). Therefore, this study aims to fabricate multilayer materials based on polypropylene fibers using hydrogel binders through a controlled manual assembly approach, and to comprehensively evaluate their physicochemical properties. The work focuses on optimizing fabrication parameters, enhancing interfacial stability, and characterizing key properties such as morphology, mechanical strength, wettability, swelling behavior, thermal stability, and chemical structure. The findings are expected to contribute to the development of advanced multilayer polymer materials with improved performance and broader applicability in healthcare, agriculture, and industrial sectors.

2. MATERIALS AND METHODS

Chemicals and reagents

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

Equipment

The following instrumenty 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, and a digital camera (model VIXIA HF M500) from Canon.



Figure 1. (a) Self-made manual multilayer material fabrication kit (MMF), and (b) PP layer covered with gelatin binder.

One self-made manual multilayer material fabrication kit (MMF), including a base and mold with a window size of $2.3 \times 3.5 \times 0.2$ cm was constructed as in Fig. 1, and a stainless steel tweezers, a baintbrush, a single-sided adhesive tape, and a petri dish were used to fabricate the manually prepared multilayer PP materials.

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. Place the multilayer material sample made 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. Turn on the plasma power supply and stabilize the material sample for 30 minutes.

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 μ L 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 °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 cm² (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

Manually fabricated PP multilayer material fabrication

Using the MMM equipment, multilayer materials based on nonwoven polypropylene fibers with a gelatin solution (5 wt%) were fabricated. Visual assessment indicated that gelatin exhibits good adhesion, effectively bonding the nonwoven polypropylene fibers to form multilayer structures. Investigation of the volume of analytical-grade gelatin solution used as the binder showed that 100 μL per layer was insufficient to fully wet the nonwoven polypropylene fibers. When using 150 $\mu\text{L}/\text{layer}$ and 200 $\mu\text{L}/\text{layer}$, multilayer materials could be successfully fabricated by the manual method. A comparison of the thickness of two multilayer materials prepared with gelatin solution volumes of 150 $\mu\text{L}/\text{layer}$ (right) and 200 $\mu\text{L}/\text{layer}$ (left), as shown in the figure below, indicates that the material fabricated with 200 $\mu\text{L}/\text{layer}$ is thicker.

Based on these results, a volume of 150 $\mu\text{L}/\text{layer}$ of analytical-grade gelatin solution was selected to obtain thinner multilayer materials while still ensuring uniform adhesion. This corresponds to a gelatin solution usage of 26.10 $\mu\text{L}/\text{cm}^2$ per layer. With a gelatin solution (5 wt%) volume of 150 μL per layer, three PP multilayer materials based on nonwoven polypropylene fibers were successfully fabricated and designated as follows: PP-Gel GP-8L for 8-layer polypropylene fiber, PP-Gel GP-16L for 16-layer material, and PP-Gel GP-32L for 32-layer one as presented in Figure 2.

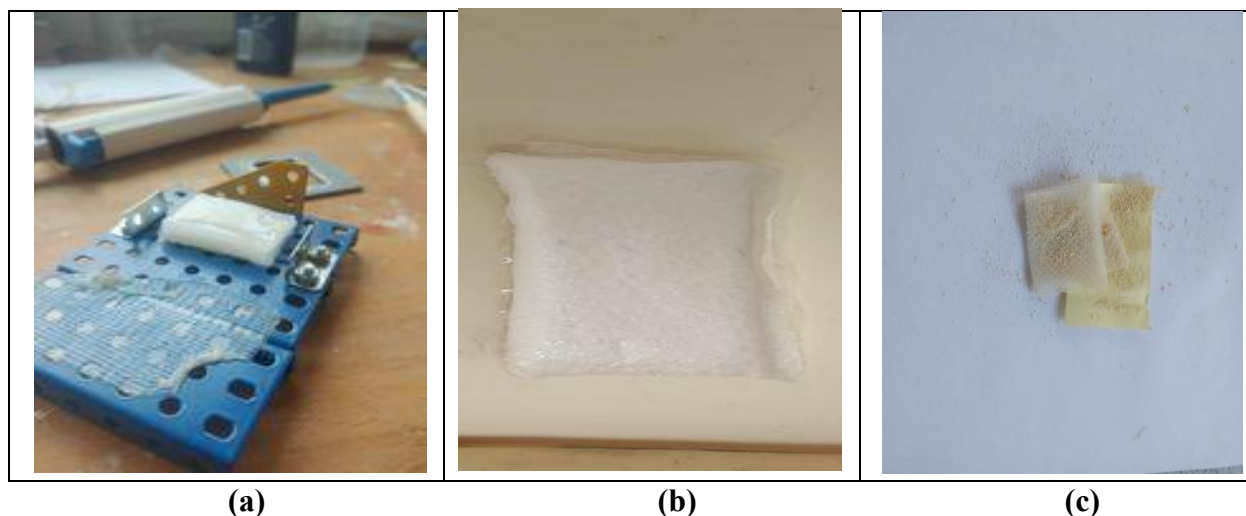


Figure 2. Manually fabricated PP multilayer material fabrication: **(a)** 16-layer PP mat (PP-Gel GP-16L) on the MMF, **(b)** PP-Gel GP-16L mat after several days, and **(c)** 32-layer PP mat (PP-Gel GP-32L)

Thickness

Thickness of gelatin-bonded PP multilayer materials measured using a caliper is presented in Table 1. The results showed that the thickness ratio of the 16-layer material PP-Gel GP-16L compared to the PP-Gel GP-8L is approximately 1.15 times at the edge, but it is about 2.9-3.0 times at the center. One of the PP-Gel GP-32L compared to the PP-Gel GP-8L is about 2.9-3.0 times at the edge, and it is only about 3.8–3.9 times at the center.

Table 1. Thickness of gelatin binder PP multilayer materials using a caliper.

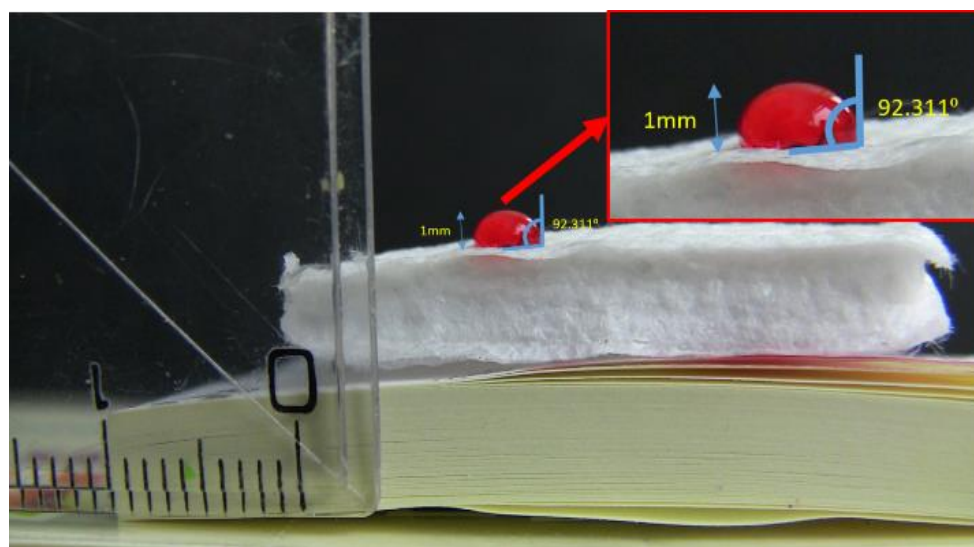
No	Sample	Layer ratio compared to 8-layer material (times)	Edge region		Middle region	
			Thickness (mm)	Thickness ratio compared to the 8-layer material (times)	Thickness (mm)	Thickness ratio compared to the 8-layer material (times)
1	PP-Gel GP-8L	1	1,24±0,34	1	0,83±0,02	1
2	PP-Gel GP-16L	2	1,43±0,38	1.15	1,30±0,05	1.57
4	PP-Gel GP-32L	4	3,74±0,44	3.02	3,20±0,15	3.86

The thickness results reveal a clear non-uniformity in the multilayer structure, with significant differences between the edge and the center of the samples. First, for PP-Gel GP-16L, the thickness ratio relative to PP-Gel GP-8L is only ~1.15 at the edge but increases to ~2.9-3.0 at the center. This indicates that the additional layers do not contribute proportionally to thickness at the edges. The most likely explanation is the loss or redistribution of gelatin binder toward the center during fabrication, leading to insufficient bonding and compaction at the edges. As a result, the edge region becomes more compressed and thinner. A similar trend is observed for PP-Gel GP-32L, where the thickness ratio is ~2.9-3.0 at the edge and ~3.8–3.9 at the center. Although increasing the number of layers does increase the overall thickness, the growth is still nonlinear and spatially heterogeneous. The center region accumulates more material and binder, while the edges remain relatively compacted. The results also show that thickness does not scale linearly with the number of layers. Several factors may contribute to this behavior. At first, during manual fabrication, the gelatin solution may migrate inward due to capillary forces and gravity, leading to higher binder concentration in the center due to the binder flow and accumulation (Yang et al., 2016). Secondly, with the uneven pressure distribution, the manual pressing or stacking may apply higher pressure at the edges, causing greater densification and reduced thickness (Thomas and Valentin, 2006). Third reason is the limited wetting at edges, the applied gelatin volume (150 µL/layer) may not fully and uniformly impregnate the entire surface, particularly near the boundaries. And finally, due to the layer slippage or misalignment, the misalignment during stacking could reduce effective thickness buildup at the edges (Evon and Reika, 2022).

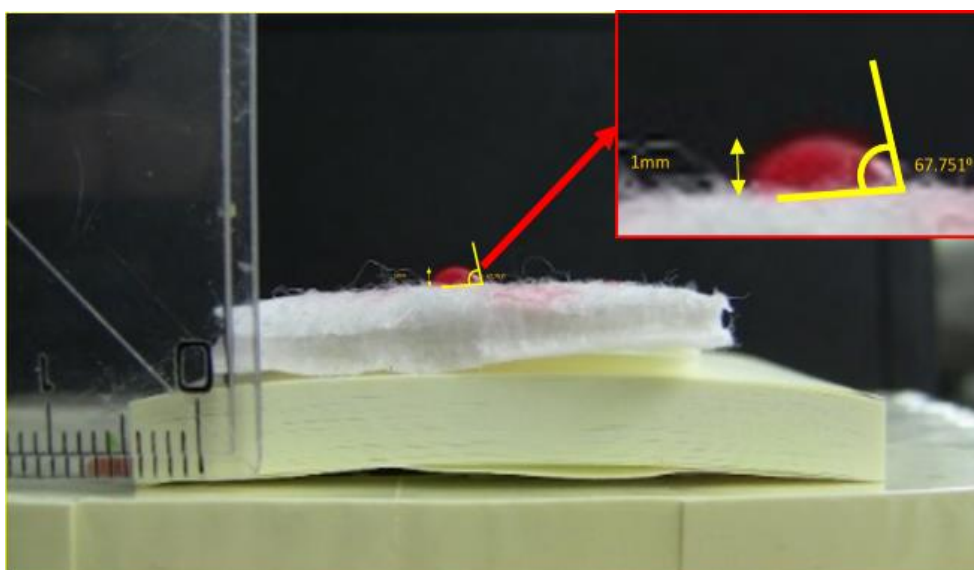
Cold plasma treatment and wettability

A multilayer material sample made from nonwoven polypropylene with a 5 wt% gelatin solution binder, PP-Gel GF-16L, was stabilized by cold plasma treatment and denoted as PP-Gel GF-16L-Pla. Both the PP-Gel GF-16L and plasma-treated PP-Gel GF-16L-Pla samples

were subjected to contact angle measurement to investigate changes in surface properties. Image analysis of the contact angle measurements in Fig. 3 and Table 2.



(a)



(b)

Figure 3. Contact angle images:(a) PP-Gel GF-16L, and (b) PP-Gel GF-16L-Pla.

Table 2. Contact angle and wettability.

No	Sample	Treatment	Contact angle	Wettability
1	PP-Gel GF-16L	No	92° - 108°	Hydrophobic
2	PP-Gel GF-16L-Pla	Cold plasma	67° - 87°	Good

The results showed that the PP-Gel GF-16L sample has a contact angle greater than 90°, indicating low wettability (hydrophobic behavior). The cold plasma stabilized sample, PP-Gel GF-16L-Pla, exhibits a contact angle of less than 90°, indicating good wettability. Thus, after stabilization of the polypropylene polymer fibers by cold plasma treatment, the PP-Gel GF-16L sample shows improved wettability. The contact angle results clearly demonstrate a significant change in surface wettability after cold plasma treatment, indicating an effective modification of the surface properties of the multilayer material. The untreated PP-Gel GF-16L sample exhibits a contact angle greater than 90°, which is characteristic of hydrophobic behavior. This can be primarily attributed to the intrinsic nature of polypropylene, a nonpolar polymer with low surface energy. Although gelatin is a hydrophilic material, its distribution within the multilayer structure may be limited or partially embedded between fiber layers, resulting in insufficient exposure at the outer surface. Consequently, the surface remains dominated by the hydrophobic polypropylene fibers. After cold plasma treatment, the PP-Gel GF-16L-Pla sample shows a contact angle lower than 90°, indicating a transition to hydrophilic behavior and improved wettability.

This enhancement can be explained by several mechanisms associated with plasma surface modification such as the introduction of polar functional groups by the oxygen- or nitrogen-containing functional groups (e.g., –OH, –COOH) onto the polypropylene surface; the surface cleaning and activation by removing surface contaminants and weak boundary layers, exposing more reactive sites and improving interaction with water molecules, the surface roughness modification by slightly etching the surface, increasing micro-scale roughness, and the improved exposure of gelatin binder by modifying the interfacial structure, allowing hydrophilic gelatin components to be more accessible at the surface (Pantoja et al., 2013; Primc and Mozetič, 2024). These combined effects lead to a substantial decrease in contact angle and thus improved wettability. From an application perspective, this transition is particularly important for fields requiring enhanced liquid interaction, such as filtration, biomedical materials, drug delivery systems, or absorbent layers.

Swelling behavior

The swelling properties of the 16-layer materials before and after cold plasma treatment, PP-Gel GF-16L and PP-Gel GF-16L-Pla, were evaluated. The swelling results are presented in Table 3 and Fig. 4.

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

Sample		PP-Gel GF-16L	PP-Gel GF-16L-Pla
Initial weight (mg)		707,80	453,83
0.5 h	Weight (mg)	1209,58	894,34
	Swelling ratios (%)	170,89	197,06
1 h	Weight (mg)	1437,64	1130,63
	Swelling ratios (%)	203,11	249,13

3 h	Weight (mg)	1753,67	1442,60
	Swelling ratios (%)	247,76	317,87
5 h	Weight (mg)	2018,80	1590,37
	Swelling ratios (%)	285,22	350,43
48 h	Weight (mg)	3130,80	2040,72
	Swelling ratios (%)	442,33	449,67

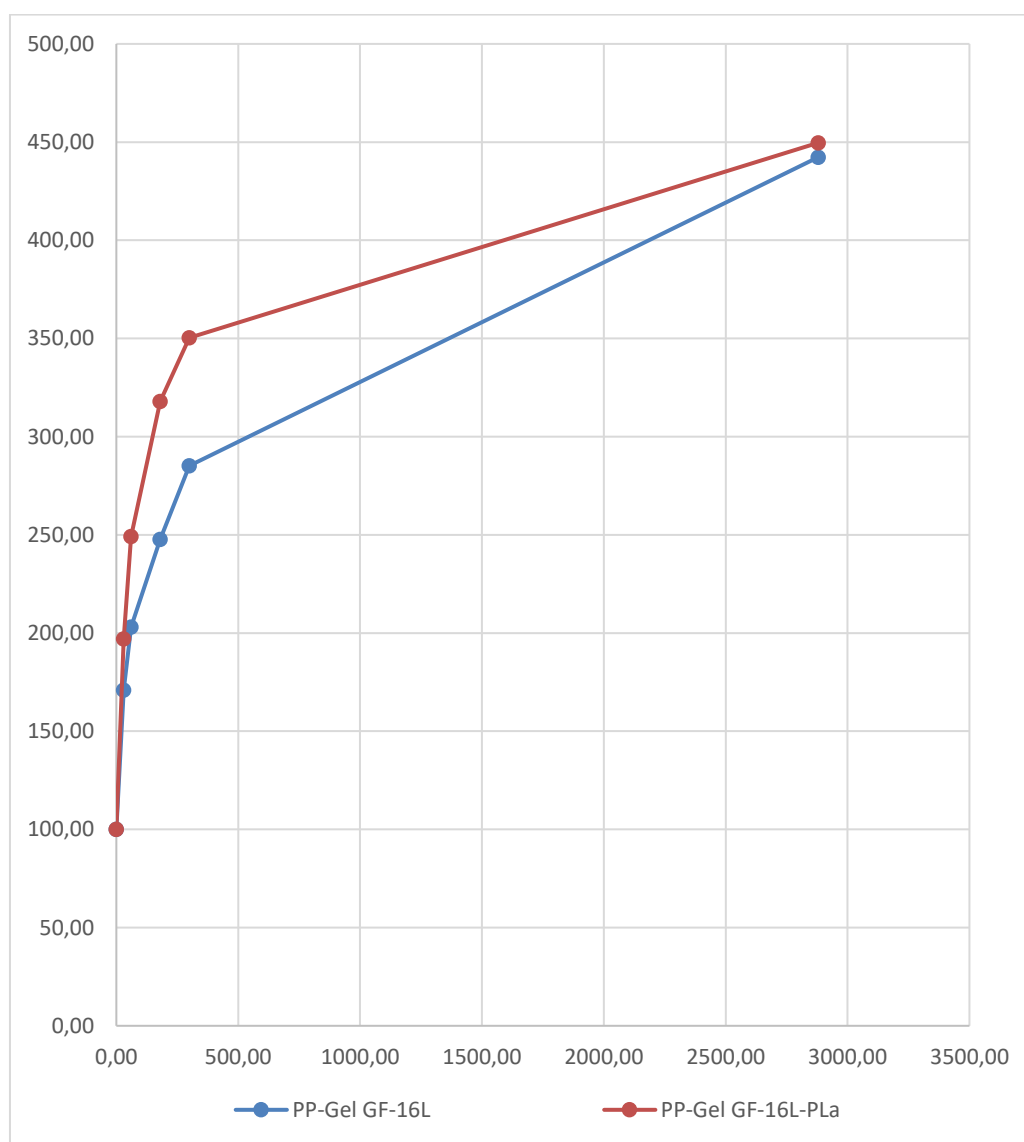


Figure 4. Swelling ratio–time plots of PP-Gel GF-16L (blue) and PP-Gel GF-16L-Pla (brown).

The results show that most of the swelling of the PP-Gel GF-16L sample occurred rapidly within the first 30 minutes, 1 hour, and 3 hours. After immersion for 3 hours, the sample was dried completely (surface dried with filter paper and then air-dried under gentle airflow for 30 minutes) and weighed again. The mass showed negligible change compared to the initial mass; specifically, the initial mass and the mass after swelling and complete drying were 707.80 and 708.26, respectively. Similarly, most of the swelling of the PP-Gel GF-16L-Pla sample occurred rapidly within the first 30 minutes, 1 hour, and 3 hours.

The cold plasma stabilization process did not produce a significant effect on the swelling behavior of the material, as the swelling ratios after 48 hours were 442.33% for PP-Gel GF-16L and 449.67% for PP-Gel GF-16L-Pla. 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-Gel GF-16L-Pla sample also showed little change compared to its initial mass; specifically, the initial mass and the mass after swelling and drying were 453.83 and 458.40, respectively. The observed reduction in swelling behavior of the PP-Gel GF-16L-Pla sample after cold plasma treatment (approximately twofold compared to the untreated sample) may be attributed to the stabilization and possible crosslinking or hardening of one or more components in the multilayer material, including polymer fibers and/or the gelatin binder (Joshua et al., 2018). This results in a more rigid network structure and consequently reduces water absorption capacity.

4. CONCLUSION

In this study, PP fiber-based multilayer materials were successfully fabricated using a simple manual assembly method with a 5 wt% gelatin concentration and an optimized volume of 150 μ L per layer as a hydrogel binder with the effective interfacial adhesion, relatively thin and uniform multilayer structures. Materials with different layer numbers (8, 16, and 32 layers) were obtained with their thicknesses increasing from 1.24 ± 0.34 mm to 1.43 ± 0.38 mm, and 3.74 ± 0.44 mm, respectively, confirming the feasibility of controlling structural thickness through layer stacking, although non-uniform thickness distribution between edge and center regions was observed due to binder migration and manual processing limitations.

The cold plasma treatment significantly improved wettability, reducing the contact angle from hydrophobic ($>90^\circ$) to hydrophilic ($<90^\circ$) behavior, which is attributed to the introduction of polar functional groups and enhanced surface activation. This surface modification of the PP multilayer materials is particularly beneficial for applications requiring improved liquid interaction. Swelling studies revealed that both untreated and plasma-treated materials exhibited rapid water absorption within the first 4.0-4.5 hours, followed by a plateau after 7.5 hours up to 48 hours. While plasma treatment slightly influenced the swelling kinetics, the overall swelling capacity after prolonged immersion remained comparable, indicating that the structural integrity of the multilayer system was preserved. Our study confirms that combining nonwoven PP fibers with gelatin binders and applying plasma treatment enables the fabrication of multilayer materials with tunable physicochemical properties. Despite some limitations in structural uniformity inherent to manual fabrication, the developed materials show strong potential for applications in filtration, biomedical materials, packaging, and absorbent systems. Future work should focus on improving fabrication uniformity, scaling up the process, and further enhancing interfacial stability and functional performance.

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