

Synthesis of polymeric fibers incorporated with pineapple peel with potential antioxidant and anti-inflammatory application in the cosmetic industry

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Abstract– In higher education, fostering an environment that encourages student engagement in real-world challenges is crucial for professional development. This principle underpins our collaborative effort with 8th-semester nanotechnology engineering students. Through innovative approaches like synthesizing polymeric fibers incorporating pineapple peel, we address environmental concerns and exploit the untapped potential of pineapple waste. The pineapple industry annually generates significant non-utilized waste, primarily stems, crowns, and peels, comprising up to 67% of the whole fruit. Pineapple peel, rich in bioactive compounds like polyphenols, holds promise for applications in the cosmetic industry, potentially enhancing products such as pull-off formulations, if they are incorporated and stabilized in suitable delivery systems. In the present work, Polylactic acid (PLA) and Polycaprolactone (PCL) fibers loaded with 10, 20, and 30 % Pineapple peel powder (PP) were synthesized using a commercial extrusion machine. Fourier Transform Infrared and Differential Scanning Calorimetry confirmed effective PP incorporation into the fibers due to the formation of new chemical bonds and interactions. The morphological characterization performed using Scanning Electron Microscopy (SEM) reveals that the cross-sectional lengths of the fibers range from 3.7 μm to 90.19 μm . High-Performance Liquid Chromatography and Folin-Ciocalteu method assessed phenolic compound content and release rates. PLA fiber with 20 % of PP showed the maximum retention of phenolic compounds, with $1243.69 \pm 234.14 \mu\text{g}$ compound/ g fiber), whilst the PCL fibers showed a rapid release, up to $95.79 \pm 5.94 \%$ in 24 hrs. These results show the feasibility of a commercial extrusion machine to synthesize polymeric microfibers with potential use in the cosmetic industry as a delivery system for phenolic compounds found in pineapple peel.

Keywords—Higher Education, Engineering Education, polymeric microfibers, pineapple polyphenolics, biopolymers, forspinning, cotton candy machine.

I. INTRODUCTION

Pineapple (*Ananas comosus*) is a fruit with high nutritional value. It is consumed fresh, as a dried snack, in juice, and in canned products. The result of processing this fruit generates waste such as the peel, stem, and crown [1], representing up to 315,000 tons of waste annually [2]. Pineapple peel is a

valuable source of bioactive compounds, such as Ferulic acid, Feruoyl hexoside, S-Coniferyl-L-cysteine, Gallic acid glucoside, and 4-Hydroxy-2,5-dimethyl-3(2H)-furanone (Furaneol) [3, 4]. These are organic compounds with antioxidant activity that have more than one phenol group in their structure and are naturally found in plants [2]. Phenolic compounds have been extracted from pineapple peel using sonication and centrifugation with methanol as the extraction solvent and analyzed by HPLC-DAD. These bioactive compounds found in pineapple peel have demonstrated antioxidant and anti-inflammatory activity. Polyphenols show anti-radical activity [5] which is a desirable characteristic in anti-aging or photo-protective cosmetic products [6]; also, they suppress histamine release, preventing inflammatory reactions in dermatological conditions, such as in allergic reactions [7, 8].

However, the use of these phenolic compounds as functional ingredients in cosmetic applications is hindered by their low solubility in water (i.e., 0.57 to 2.19 g/L in a range of 15 to 50 °C for ferulic acid [9]) and instability due to environmental factors such as light, heat, and presence of oxygen [10]. One proposed solution for their utilization is to incorporate them into controlled delivery systems to increase their stability and bioavailability in the body [11]. The utilization of fibers as delivery systems has garnered significant attention in recent years, primarily due to their versatility and effectiveness. These fibers, which can range from micro to nanoscale, offer a unique advantage in their ability to function as delivery systems. The high surface area, tuneable porosity, and customizable properties of these fibers make them ideal for controlled and targeted release applications. Whether used for drug delivery, release of bioactive compounds, or other functional materials, fibrous delivery systems provide a promising platform for enhancing the efficacy and precision of various therapeutic and industrial processes [13, 16].

Among the various synthesis methods for producing fibers, the classic electrospinning technique is the one that predominates in most current research [17, 18]. This technique employs electrostatic forces, whereas alternative methods like force spinning or rotary jet spinning utilize centrifugal force to produce fibers with diameters of up to 100 nm by

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manipulating polymer solutions [11, 12, 20]. Although classic electrospinning is known for its low initial costs [22], force spinning techniques offer several advantages, including high production yields of over 1 g/min at the laboratory scale [13, 23]. Furthermore, force spinning reduces production costs by operating at low voltages, typically between 0.5 to 3.0 V [24], compared to the higher voltages above 5 kV required for electrospinning [25].

An alternative method for producing fibers without the use of high voltages, is the use of a commercial extrusion machine, which is a technique based on the principle of rotary jet spinning method [26]. It involves a combination of hydrostatic pressure and centrifugation in a perforated tank that rotates at high speeds and propels the liquid polymer out of the tank orifice, resulting in the stretching, drying, and solidification of the polymer thread. One of the main advantages is the direct use of the polymer, which avoids the use of harmful solvents, thus protecting the environment and the production user. Furthermore, this type of technology allows for solvent-free structures that are easier to approve for commercialization, especially for medical applications [29]. The use of techniques such as electrospinning and forcespinning for fiber fabrication has proven effective. However, it is essential to evaluate the impact of the materials used to determine the resulting fiber size, which can vary from micro to nano scale depending on the polymers or bioactive compounds employed [13-16].

The use of biodegradable polymers such as polycaprolactone (PCL) and polylactic acid (PLA) have demonstrated biocompatibility and effective delivery of active compounds, providing the necessary safety and characteristics for their use as a delivery system in cosmetic industry [19, 20]. For instance, Miletic et al. demonstrated the correct incorporation of fatty oils in PLA nanofibers, showing ability to preserve antioxidant properties of the bioactive compound, enhancing their potential for cosmetic applications [21].

In the present work, the synthesis of biopolymeric PLA and PCL fibers loaded with pineapple peel powder (PP) using a commercial machine (i.e., cotton candy) was performed, to evaluate the matrix/PP combination that offers the best characteristics in terms of retention and concentration of polyphenolic compounds. The obtained fibers can be a potential option for delivery systems in the cosmetics industry, showing antioxidant and anti-inflammatory properties. This article aims to bypass the extraction step of phenolic compounds from pineapple peel by incorporating them directly as an ingredient in fiber formation, with the goal of assessing their effect on the size and type of fibers produced.

II. MATERIALS AND METHODS

A. Plant material

The pineapple peel waste was sourced from local shops, subsequently diced into small pieces, and placed into a lyophilization equipment (Labconco, Missouri, USA), under

high vacuum conditions (0.002 mBar) at -47°C . Following lyophilization, the pineapple peel underwent grinding utilizing a commercial coffee grinder, followed by sieving to achieve a uniform particle size of 250 microns. The resulting samples were then stored under refrigeration in sealed bags until needed.

B. Fibers synthesis

A commercial extrusion machine, i.e., cotton candy machine, (DISPLAY4OP, USA) was used for the experiments. The machine was heated to the required temperature based on the melting point of each polymer, which was monitored using an infrared thermometer. Specifically, for the PLA polymer, the temperature was maintained within the range of $160\text{-}180^{\circ}\text{C}$, as its melting point is documented at 173°C [30]. Conversely, for the PCL polymer, the temperature was set between $60\text{-}80^{\circ}\text{C}$, reflecting its lower melting point of $60\text{-}65^{\circ}\text{C}$ [31]. Subsequently, a PCL: PLA mixture was also explored and the temperature range of $180\text{-}200^{\circ}\text{C}$.

The synthesis of the fibers followed the experimental design detailed in Table I. A total weight of 2g, consisting of the polymer and pineapple powder mixture, was fed into the machine's nozzle. After initiating the synthesis process, ranging from 5 to 30 minutes, the resulting fibers were gathered, measured, and subsequently stored at room temperature.

TABLE I. EXPERIMENTAL DESIGN OF THE POLYMER COMBINATIONS AND PINEAPPLE PEEL POWDER.

	PLA	PCL	PCL: PLA 1:2	PCL: PLA 1:1	PCL: PLA 2:1
<i>Pineapple peel powder (%)</i>	0	0	0	0	0
	10	10	10	10	10
	20	20	20	20	20
	30	30	30	30	30
<i>Collecting time (min)</i>	15	5	5-7	5-7	5-7

C. Characterization of polyphenolic compounds

Triplicate samples of fibers with PP concentrations of 90:10, 80:20, and 70:30 w/w were homogenized with a 50:50 ethanol-water solvent in a solid-liquid ratio of 1:20 w/v. The samples were subjected to sonication for 30 minutes at room temperature and subsequently centrifuged for 5 minutes. The samples were weighed and transferred to a Genevac EZ-2 Plus (Ipswich, UK) for evaporation until dryness for a minimum of 24 hours. Finally, lyophilization under -70°C for 24 hours was done prior to analysis.

The analysis was performed according to Steingass et al. [32] using an Agilent Series 1260 high-performance liquid chromatography system (HPLC) coupled with a diode-array detector (DAD). The column used for the separation of the compounds Luna C18(2) Phenomenex™, 250×4.6 mm. HPLC grade water acidified with formic acid (A) and HPLC grade pure methanol acidified with formic acid (B) were used as mobile phase. The total run time for each sample was 65

minutes, at a flow rate of 0.8 mL/min, with an oven temperature of 30 °C and 262 bar pressure. For the quantification of polyphenols, concentration curves of the standards were utilized: ferulic acid, gallic acid, and coumaric acid, according to [4].

D. Fourier Transform Infrared Spectroscopy (FTIR) analysis of the integration of bioactive compounds.

The spectroscopic equipment employed was a Perkin Elmer Spectrum 400 coupled with Attenuated Total Reflectance (ATR). Each sample was positioned on the sample holder, and the resolution was fine-tuned until a stable spectrum was achieved. Scanning was conducted from 4000 to 380 cm⁻¹ at 25 °C. It is worth noting that the fiber samples underwent no pre-treatment for this analysis; they were solely stored under light- and moisture-protected conditions.

E. Differential Scanning Calorimetry analysis of bioactive compound integration in active-loaded fibers

The thermal analysis of the synthesized fibers was conducted by a Mettler Toledo Differential Scanning Calorimetry equipment (Thermo Fisher Scientific, USA). For each analysis, samples ranging between 1-3 mg of PCL, PLA and PCL: PLA fibers at different PP, along with their respective controls with no bioactive compound, were measured to obtain their profile during the fusion of the 25 fibers at final temperature. Depending on the polymer, the samples were heated from -80 °C (PLA from 25 °C) to a final temperature of 175 °C at a rate of 5 °C/min under a 50mL/min nitrogen flow in aluminum pans. The integration of the bioactive compounds into the fibers was analyzed based on the observed endothermic and exothermic peaks.

F. Fibers morphological characterization

A Phenom Pro x (Thermo Fisher Scientific, USA) equipment was used to obtain images that allow the observation of the network formed by the fibers, aiming to observe the incorporation of PP and determine diameters of the fibers. The samples were placed on 1/8" pins (Ted Pella, USA) using double-sided carbon tape for adhesion. The images were taken without coating at 5 KV on the selected samples. The average diameter of the fibers was determined from the SEM images using ImageJ 1.54 software (National Institutes of Health, Bethesda, MD, USA). 20 measurements were taken, 10 at a magnification around 100x and 10 at 500x.

G. Determination of release rate of polyphenols from fibers

To assess the release rate, we employed the Folin-Ciocalteu total phenol quantification technique [33]. For total retention quantification, a 50% (v/v) methanol extraction was conducted, following the aforementioned procedure utilized in the HPLC analysis. To measure polyphenol release, a 50% methanol solution was adjusted to a pH of 5.45 to simulate potential cosmetic application. Fibers were suspended at a ratio of 1:20 (w/v) and placed in a shaker at 37 °C and 100

rpm. Aliquots were collected at intervals of 15, 30, 60 (1 hour), 120 (2 hours), and 1440 minutes (24 hours). Each aliquot underwent a process where 20 µL was mixed with 100 µL of 10% (v/v) Folin reagent, left to stand in darkness for 5 minutes, and then neutralized with 80 µL of 7.5% (w/v) sodium carbonate solution. The mixture was homogenized and incubated for 1.5 hours at 25 °C in the absence of light. Absorbance was measured at 765 nm using a microplate reader (Biotek, Synergy HT, USA). A calibration curve was prepared using gallic acid ranging from 1 to 200 ppm, following the same procedure. Results are expressed as gallic acid equivalents (GAE) in µg/g of fiber.

H. Statistical analysis

The experimental data were statistically evaluated using the JMP program (SAS Institute Inc., North Carolina, USA). The results are presented as mean ± standard deviation. Significant differences were tested using the Tukey-Kramer HSD test and t-student test with a confidence level of 95 % (p ≥ 0.05).

III. RESULTS AND DISCUSSION

A. Fibers synthesis

The pure PLA fibers had the highest yield among the others, with 73.17 %, and the combination PCL: PP 1:1 had the lowest, with 30.34 %. Summary of yields and general physical characteristics of the fibers are presented in Table II and Figure 1. Fibers with 10 % pineapple had the highest yield with 62.07, 40.40, and 56.73 % for PLA, PCL, and PCL: PLA 2:1, respectively. In contrast, the fibers with lower yield were those with 30 % pineapple for PLA, and 20 % for PCL; this last one presented the lowest yield of all the loaded fibers with 3.81 %. Tendency to lower yields with larger quantities of pineapple may be attributed to burned pineapple peel, which caused the clogging of the extruder nozzle of the machine during the extrusion process. This may be attributed to the rapid decomposition of pineapple at elevated temperatures, stemming from its high sugar content and its pronounced hygroscopic nature, resulting in a highly sticky mixture [34].

TABLE II
YIELD AND OBSERVABLE CHARACTERISTICS OF SYNTHESIZED FIBERS.

Polymer	Polymer: PP ratio	Yield (%)	Main Characteristics
PLA	100:0	73.17	Flat, short.
	70:30	26.1	Thin, slightly darker.
	80:20	45.50*	Like cotton, yellow.
	90:10	62.07*	Flat, light yellow /brown.
PCL	100:0	42.00*	Fine, like cotton.
	70:30	5.00	Like cotton, yellow dots.
	80:20	3.81*	Like cotton, yellow dots.
	90:10	40.40	Like cotton, yellow dots.
PCL: PLA 1:2	100:0	49.27	Hard, laminar shape.
PCL: PLA 1:1	100:0	30.34	Short, fragile lamellas.
PCL: PLA 2:1	100:0	43.32	Spider-web form.
PCL: PLA 1:2	90:10	56.73	Flat, long, light yellow.
PCL: PLA 2:1	70:30	15.27	Thin, spongy, bright yellow.

*Average values of yield. Pineapple peel powder (PP)

Additionally, it was observed that the combination of PCL: PLA 2:1 with 30% pineapple exhibited a yield of 15.27%, which was four times higher than that of PCL alone, indicating that PLA enhanced the fiber yield. These results highlight that, overall, PLA fibers had higher yields when pineapple was added, while PCL yielded the lowest. Combining the polymers resulted in enhanced fiber yields containing PCL, possibly attributable to an increase in matrix elasticity with the PCL-PLA combination [35].

In general, macroscopic characteristics, PCL fibers tended to exhibit a softer texture akin to cotton, which is desirable for potential applications in the cosmetic industry. The authors attribute the difference in fiber morphology between PCL and PLA to their distinct properties, such as glass transition temperature (T_g), chain flexibility, and molecular weight. PCL has a lower glass transition temperature and higher chain flexibility compared to PLA, enabling it to form thinner and more flexible fibers [36]. For further comparisons, images of the synthesized fibers are presented in Figure 1, showcasing variations in colour depending on the proportion of pineapple, along with subtle differences in physical mat formation. All fibers containing pineapple displayed a colour shift to yellow/brown, attributed to the Maillard reaction, or exhibited speckles, indicating the presence of pineapple in the fibers qualitatively.

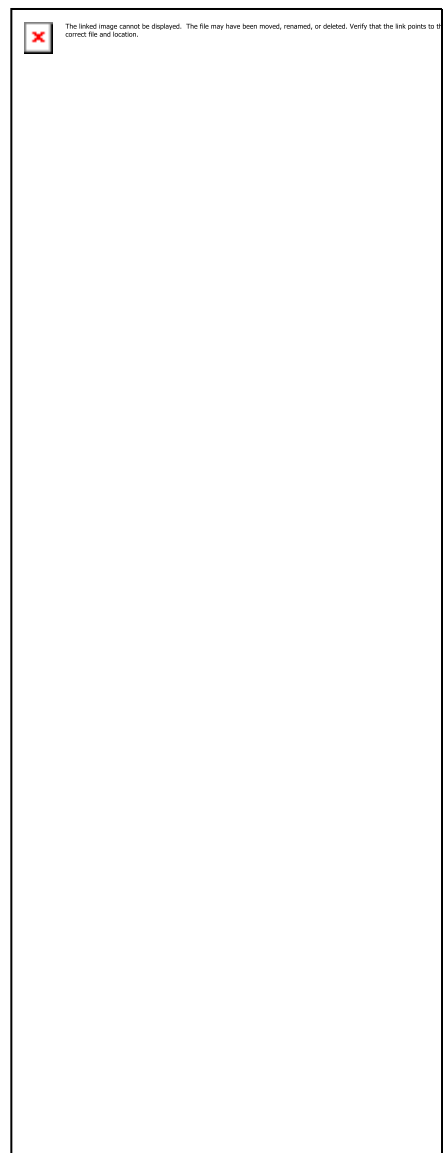


Fig. 1. Synthesized fibers A) PLA control, B) PLA: PP 90:10, C) PLA: PP 80:20, D) PLA: PP 70:30, E) PCL control, F) PCL: PP 90:10, G) PCL: PP 80:20, H) PCL: PP 70:30, I) PCL: PLA: PP 1:2:10, J) PCL: PLA: PP 2:1:30.

B. Characterization of polyphenolic compounds

Peaks eluted within the time range of 5.298 to 36.976 minutes (6 signals) and at 38.592 minutes, remained unidentified. Nevertheless, based on their respective wavelengths, these peaks are tentatively assigned to the polyphenol family [37]. Ferulic acid with a derivative thereof detected, were successfully identified, representing 14.7 % and 7% of the sample, respectively.

Conversely, peaks observed in the synthesized fibers exhibited minimal quantities of ferulic acid derivatives, alongside matching retention times as reported by Martínez-Alvarado [4]. The PCL: PLA: PP 1:2:10 and PCL: PLA: PP 2:1:30 combinations revealed peaks identified as coumaric acid [4], with concentrations of 11.92 and 81.79 $\mu\text{g/g}$ fiber

obtained for this compound at each composition. This phenomenon can be attributed to the fact that certain thermal processing methods, such as hot air drying, can have a detrimental impact on the levels of polyphenols and anthocyanins found in certain vegetables and fruits [39]. Similarly, other research has demonstrated that the loss of free polyphenols can vary widely, ranging from 15% to 60%, in thermal processes like pasteurization, and can reach levels as high as 60% to 90% in more intense thermal processes such as extrusion or baking [40]. This may elucidate why fibers subjected to higher temperatures exhibited fewer signals.

The statistical analysis revealed that among all the fibers compared, the PLA: PP 80:20 fiber exhibited a significant difference, with the PCL: PLA: PP 1:2:10 fiber following suit. Conversely, the PCL: PP 80:20 fiber did not demonstrate a significant difference, indicating considerable variability in its retention, like the PCL: PLA 2:1:30 fiber. Notably, the fiber with the lowest retention of polyphenolic compounds was the PCL 90:10. Synthesized fibers exhibiting higher retention of phenolic compounds were selected for further characterization techniques. Statistical analysis revealed significant differences in p-values for the 80:20 fibers of both PLA (0.0193 and 0.0205) and PCL (0.0116 and 0.0059). Consequently, PLA 80:20, with 878.34 $\mu\text{g/g}$, and PCL 80:20, with 632.16 $\mu\text{g/g}$ in terms of total phenolic compounds, were selected for subsequent analysis. However, for the PCL:PLA combination fibers, no favorable values were observed for making a selection ($p=0.9825$). Nevertheless, since quantifications of 663.69 $\mu\text{g/g}$ were obtained for PCL:PLA:PP 2:1:30 and 669.24 $\mu\text{g/g}$ for PCL:PLA:PP 1:2:10, it was deemed relevant to pursue in-depth analysis through qualitative characterization techniques with both combinations to determine the optimal choice.

C. Fourier Transform Infrared Spectroscopy (FTIR) analysis of integration of bioactive compounds.

FTIR spectra for PCL showed the characteristic bands (Figure 2), correspond to the asymmetric and symmetric vibrations of C-H at 2994 cm^{-1} and 2866 cm^{-1} , respectively. The stretching of carbonyl groups C=O is observed at 1722 cm^{-1} , and the stretching of C-O and C-C in the crystalline phase is observed at 1293 cm^{-1} . The asymmetric and symmetric vibrations of ether C-O-C are observed at 1239 cm^{-1} and 1165 cm^{-1} [46]. These wavelengths are also found in the spectrum obtained with PCL: PP 80:20, but a change in intensity and a slight shift in the C-H vibrations were observed at 2943 cm^{-1} and 2863 cm^{-1} , which is consistent with the work of Radusin et al. [47] explaining the interaction between both polymers.

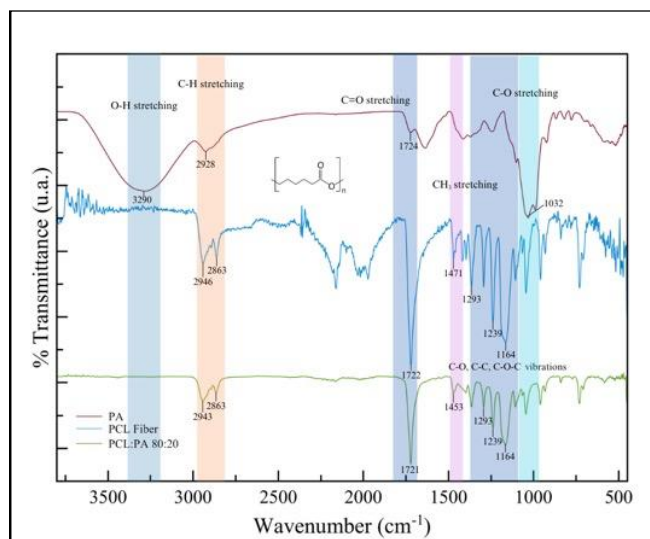


Fig. 2. FTIR spectra of pure PCL fiber compared to the result with the best retention in HPLC, PCL: PP (pineapple) 80:20.

The PCL: PLA 1:2 combination (Figure 3), for the control fibers, peaks at 2944 cm^{-1} , 2862 cm^{-1} , and 1721 cm^{-1} are observed, corresponding to the symmetric stretching of C-CH₃ in PLA, stretching of CH₂, and stretching of carbonyl group in PCL, respectively. Signals at 1295 cm^{-1} , 1238 cm^{-1} , and 1163 cm^{-1} correspond to symmetric stretching vibrations of C-O, C-C, and C-O-C [48]. In the fibers with PP, the two peaks corresponding to PLA at 2994 cm^{-1} and 2944 cm^{-1} are observed, while the peak at 2862 cm^{-1} from PCL disappears. This could be due to a lower amount of PCL in the analyzed sample. A shift from 1721 cm^{-1} to 1748 cm^{-1} is observed for C=O, and a peak at 1181 cm^{-1} coincides with the characteristic symmetric stretching vibration of C-O-C in PCL.

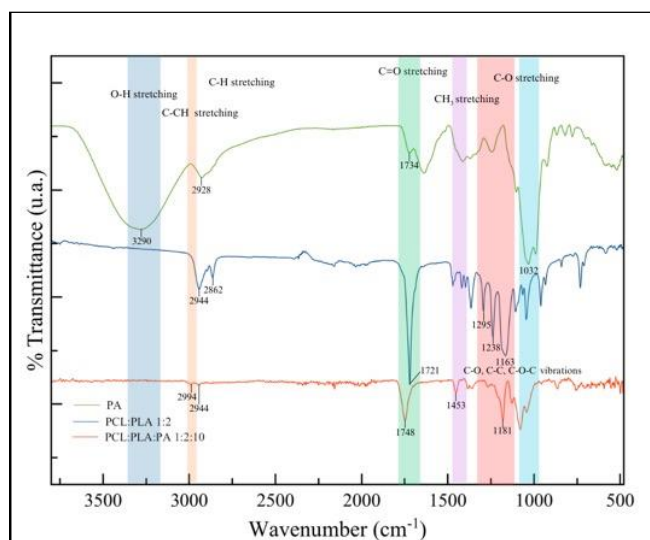


Fig. 3. FTIR spectra of PCL: PLA 1:2 fiber compared to PCL: PLA 1:2 PP (pineapple):10.

In the PCL: PLA 2:1 combination (Figure 4), there is a high similarity between the previous spectrum of the PCL: PLA 1:2 combination and its combination with pineapple. Signals at 2944 cm⁻¹ and 1458 cm⁻¹ are observed, which are characteristic of symmetric and asymmetric stretching of C-CH₃ and asymmetric bending of CH₃ in PLA. For PCL, signals at 2861 cm⁻¹ and 1721 cm⁻¹ correspond to stretching of CH₂ and stretching of carbonyl groups C=O, respectively [45], and signals at 1293 cm⁻¹, 1238 cm⁻¹, and 1178 cm⁻¹ correspond to symmetric stretching vibrations of C-O, C-C, and C-O-C [48]. Comparing the PCL: PLA 2:1 fiber with PCL: PLA: PP 2:1:30, a peak appears at 2994 cm⁻¹, which is characteristic of the asymmetrical stretching vibration of C-CH₃ in PLA. Similarly, the peak at 1721 cm⁻¹ shifts to a higher frequency of 1748 cm⁻¹. Furthermore, the characteristic bond at 1453 cm⁻¹ of PLA and signals at 1296 cm⁻¹, 1240 cm⁻¹, and 1181 cm⁻¹ representing PCL remain constant.

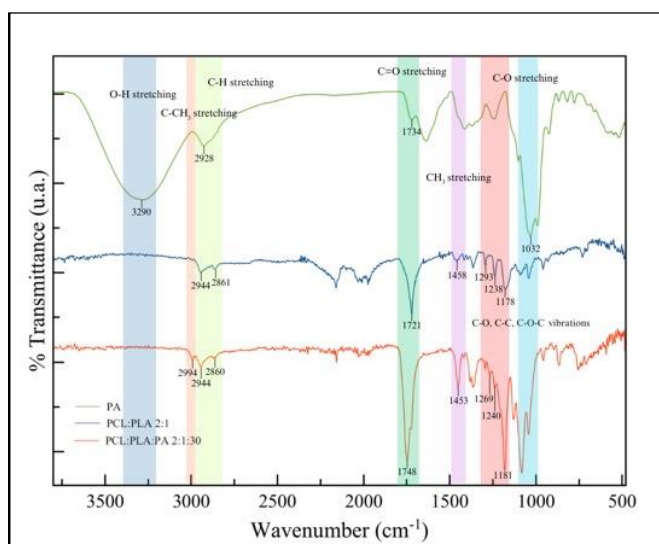


Fig. 4. FTIR spectra of PCL: PLA 2:1 fiber compared to PCL: PLA 2:1 (pineapple):30.

To confirm the presence of pineapple polyphenols in the fibers, the identification and comparison of bond types and functional groups present in the samples were performed. Across all three polymer types, a consistent disappearance of the band at 3600-3100 cm⁻¹, corresponding to the O-H groups present in the polyphenolic compounds of pineapple polyphenols, was observed. This initial indication of pineapple polyphenols presence into the fibers aligns with findings by Radusin et al. [47]. The behaviour is attributed to phenolic O-H groups capable of forming hydrogen bonds with PLA and PCL, which contain ester groups serving as moderate hydrogen bond acceptors [49]. In the case of PLA: PP and both PCL: PLA: PP combinations, a shift to a higher frequency in the C=O band was noted, indicating a potential change in the vibrational frequency of the carbonyl. This shift suggests stronger C=O bonds, likely due to newly formed

hydrogen bonds with incorporated pineapple polyphenols [50]. However, this behaviour was not observed with PCL, where no apparent shift occurred.

On the other hand, in the case of PCL: PP and PCL: PLA: PP 1:2, PCL: PLA: PP 2:1, a decrease in peak intensity was observed in the range of 1300 to 1100 cm⁻¹, corresponding to molecular vibrations of C-O, C-C, and C-O-C bonds characteristic of PCL. According to Zupančič et al. [49], weak interactions between polyphenols and PCL result in smaller spectral peak changes as the ratio of polyphenolic compounds to PCL increases. The weakening of hydrogen bonds within biopolymer structures, due to interactions between O-H groups forming new bonds with carbonyl groups [51], along with observed interactions, suggest the correct incorporation of pineapple peel through newly formed bonds and the appearance of characteristic organic compound bands.

D. Differential Scanning Calorimetry (DSC) analysis of integration of bioactive compounds.,

In the case of PLA (Figure 5a), the control PLA exhibits a T_g at 61.35 °C and a T_m within the range of 148-153°C. On the other hand, the PLA: PP 80:20 matrix shows a slight decrease in T_g to 59.34 °C, but its T_m remains in the same range as the control PLA. For the PCL fibers (Figure 5.b), melting temperatures (T_m) of 61.15°C and 59.95°C were obtained for the control and 80:20 fibers, respectively, indicating that the incorporation of PP caused a slight shift towards a lower melting temperature. Additionally, the glass transition peak is absent (T_g), which is consistent with literature reporting the synthesis of PCL fibers [52].

For the PCL: PLA control fibers (Figure 5.c and d), endothermic peaks corresponding to the melting temperatures of PLA and PCL are observed, indicating the presence of both polymers in the fibers and aligning with the previously reported theoretical temperatures. Upon incorporating PP, the appearance of a third exothermic peak is observed in both combinations, at temperatures of 106.87 and 105.21 °C for PCL: PLA: PP 1:2:10 and 2:1:30, respectively. This could be attributed to the restructuring of certain crystalline structures [53] and the amorphous state of the PLA polymer [54, 55].

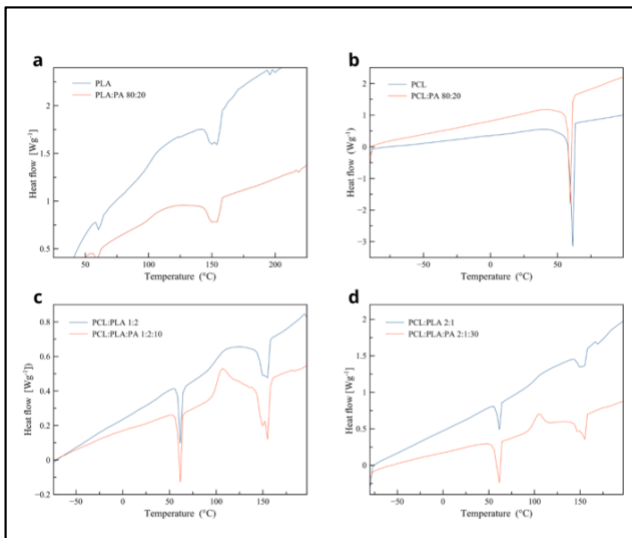


Fig. 5. Scanning Differential Calorimetry (DSC) curves for each fiber comparing its control with its pineapple-integrated form: a) PLA; b) PCL; c) PCL: PLA 1:2; and d) PCL: PLA 2:1.

E. Fibers morphological characterization

The determination of the cross-sectional lengths for each combination was measured. It is noteworthy that the minimum fiber size determined was $3.70 \mu\text{m}$ in the PCL: PLA: PP 2:1:30 combination, while the combination PCL: PLA: PP 1:2:10 obtained the smallest average size. Literature on PCL fibers manufactured by rotary jet spinning reports sizes ranging from $1.2 \mu\text{m}$ with dispersed sizes up to $30.1 \mu\text{m}$ [20, 59]. Therefore, the obtained sizes are within the expected range. On the other hand, the maximum sizes reached were $64.78 \mu\text{m}$ and $90.19 \mu\text{m}$ in the PLA fiber and the PCL: PLA: PP 1:2:10 combination, respectively, indicating that PLA produces larger fibers. The obtained microfiber sizes are within the same order of magnitude as similar works. Wongpajan et al., working with PLA microfibers manufactured by the cotton candy machine method, reported the tendency that higher temperatures resulted in smaller synthesized fibers, but with irregular sizes [28]. Similarly, Zamproni et al. reported average sizes of $7.28 \pm 4.23 \mu\text{m}$ using rotary jet spinning [60], suggesting that if they were working at higher temperatures, the size of the PLA fibers could have been reduced.

In Figure 6, the obtained images for each fiber are presented using SEM, allowing the observation of their interweaving and morphology. It is notable that unlike the PCL fibers (c) and (d), and PCL: PLA 1:2 and 2:1 fiber (e), (f), (g), and (h), the PLA fibers (a) and (b) do not exhibit the spheres corresponding to PP. This could mistakenly lead to the belief that these fibers failed to capture the pineapple polyphenols, when as reported in previous sections, these fibers had the highest levels of polyphenol retention. This suggests that the PP must have integrated into the fibers in a different way, such as being encapsulated within the fibers,

explaining their larger size and slower release compared to the fibers containing PCL.

Based on the gathered data, it is evident that the synthesized fibers exhibit characteristics more akin to microfibers. It appears that the pineapple polyphenols are encapsulated within the PCL fibers and in combinations containing this polymer, suggesting a trapping effect. This observation may corroborate the stronger interaction observed between PLA and PP fibers via FTIR analysis, indicating potential integration of pineapple polyphenols within the fiber structure.

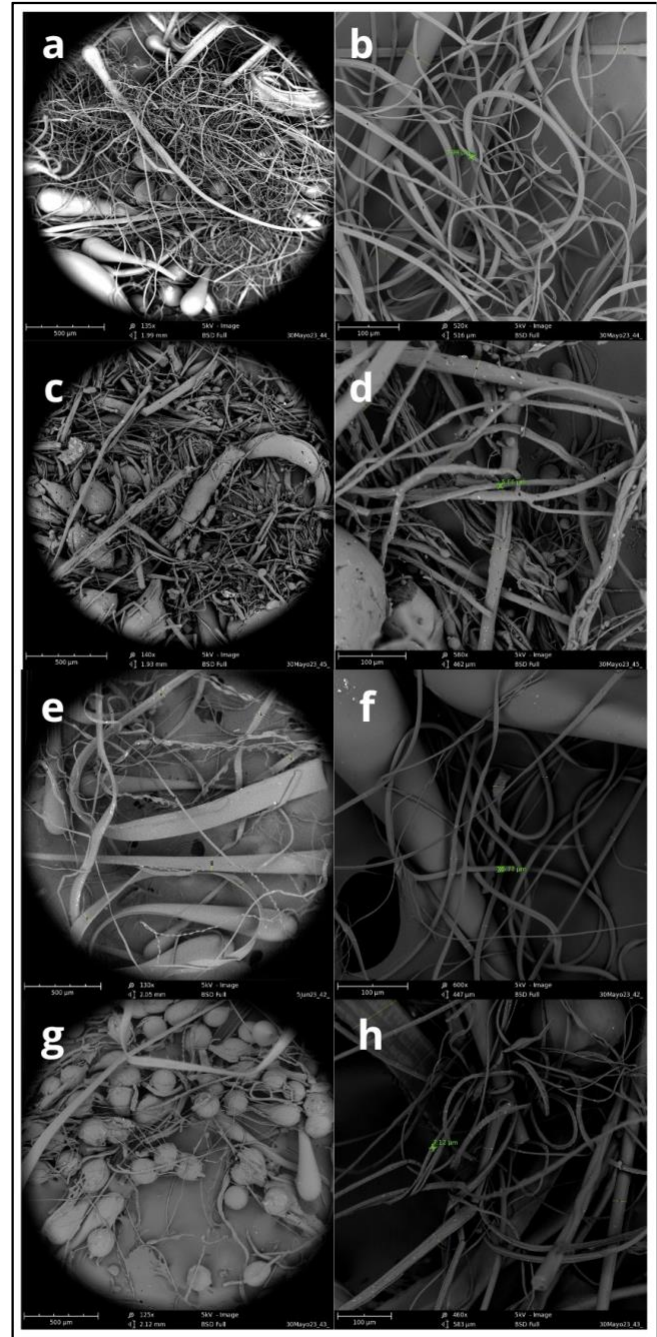


Fig. 6. SEM images of the fibers a) PLA global image 135x b) PLA 520x; c) PCL global image 140x, d) PCL 560x; e) PCL: PLA: PP 1:2:10 global image 125x, f) PCL: PLA: PP 1:2:10 600x; g) PCL: PLA: PP 2:1:30 global image 125x, h) PCL: PLA: PP 2:1:30 460x.

E. Release rate of polyphenols at biological conditions determined.

Different release profiles were evident among the different fibers. The PCL 80:20 microfiber exhibited a faster release, reaching a release of up to 69.46 ± 9.42 % at 30 minutes, compared to its PLA 80:20 counterpart, which only had a release of 27.010 ± 0.87 % at the same time. This is consistent with the work of Haroosh et al., which determined that PCL nanofibers exhibit rapid drug release in short periods, with up to 66 % compared to 33 % for PLA, both at 25 minutes [61].

It is interesting to note that the PCL: PLA combination microfibers show synergistic behaviors depending on the proportion of both polymers. With a higher level of PLA (PCL: PLA 1:2) greater retention is observed, almost comparable to pure PLA fiber in this study. While maintaining a low level of it and increasing the amount of PCL (PCL: PLA 2:1), also shows an increase in the incorporated pineapple powder but to a lesser extent.

Regarding the presence of PCL in the combined fibers, it increases the rate of release, surpassing that obtained with pure PLA. For PCL: PLA 1:2, a maximum of 95.79 ± 5.94 % was achieved at 24 hours (approximately 24 μg GAE), while PCL: PLA 2:1 showed a more prolonged release, reaching up to 65.402 ± 10.71 % at 24 hours with approximately 4.85 μg GAE release.

III. CONCLUSIONS

Polymeric microfibers composed of PCL, PLA, PCL: PLA and various ratios of pineapple peel (PP) were successfully synthesized using a cotton candy machine based on the functional principle of the rotary jet spinning method. Confirmation of pineapple peel incorporation into the fibers was achieved through FTIR and DSC techniques, with PLA fibers exhibiting stronger interactions. Notably, the PLA 80:20 w/w combination displayed the highest phenolic compound incorporation. The presence of PCL in the fibers facilitated faster compound release, particularly evident with PCL: PP combinations releasing up to 80.64 ± 12.56 % within the initial 60 minutes. Synergistic effects on both incorporation and release rates were observed with PCL: PLA: PP combinations, notably with 1:2:10, displaying significant incorporation (669.25 ± 149.24 μg compound/g fiber) and release (89.89 ± 10.6 % at 60 minutes). Given the desirable high retention and rapid release behaviour required for cosmetic applications like pull-off products, the PCL: PLA 1:2:10 combination fiber emerges as a promising candidate for further investigation into its antioxidant and anti-inflammatory activity *in vitro*, alongside studies assessing its stability under varying storage conditions. Moreover, this interdisciplinary project involving students from nanotechnology engineering illustrates the

application of innovative educational methods to tackle real-world challenges, fostering their development as future engineers capable of contributing to sustainable solutions in fields such as cosmetology, thus highlighting the relevance of educational innovation in addressing pressing global issues.

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