Bulgarian Academy of Sciences
Institute of Polymers
Laboratory of Bioactive Polymers

Electrospun poly(ε-caprolactone)/ascorbyl palmitate nanofibrous materials

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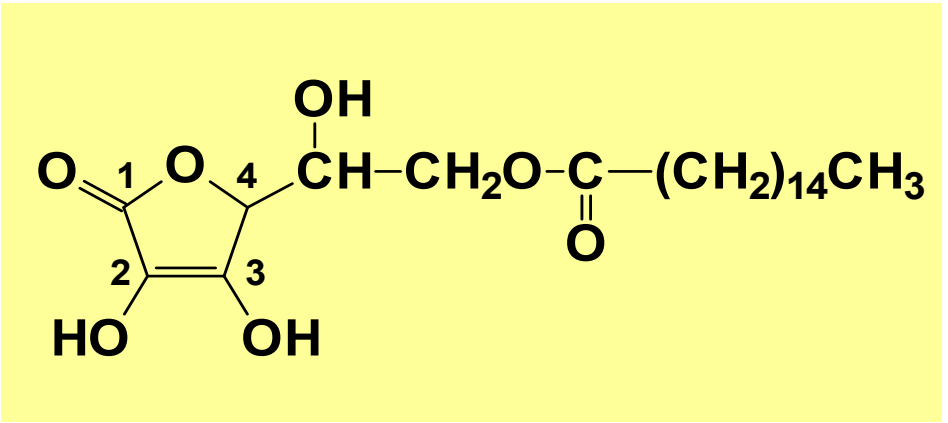
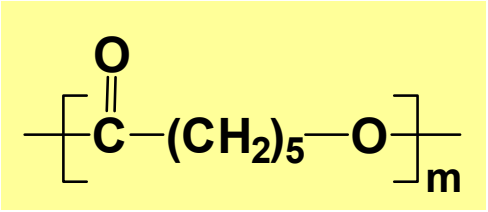
Regardless of the fact that vitamins are a major component of a number of polymer compositions intended for use in medicine, pharmacy, cosmetics, and food industry, until now investigations on vitamin incorporation in polymer fibrous materials by electrospinning are still scarce.

Ascorbyl palmitate (AP) is an amphipathic derivative of vitamin C that has found wide application as an antioxidant additive in food, pharmaceutical, medical and cosmetic products.

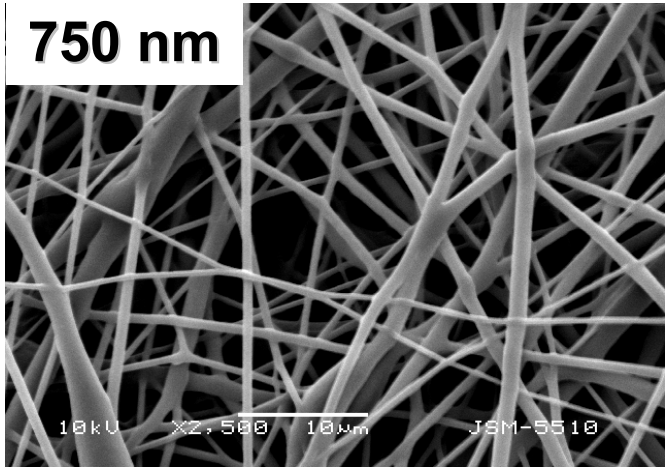


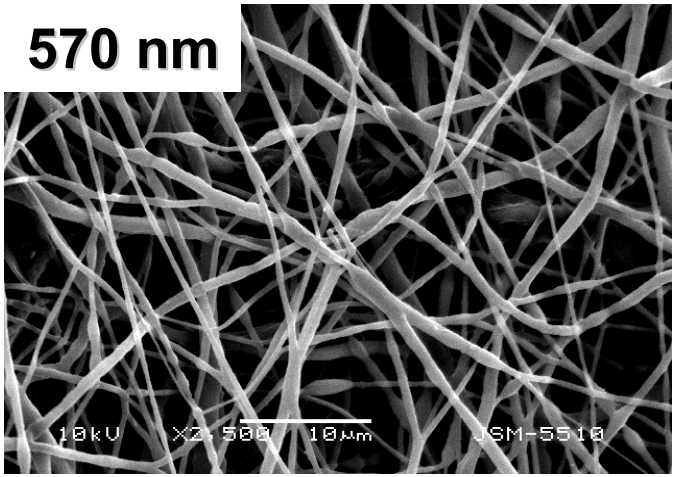
Despite the higher stability as compared to vitamin C, AP is still sensitive to oxidation thus the development of novel polymer materials able to incorporate AP while preserving its antioxidant properties is of interest.

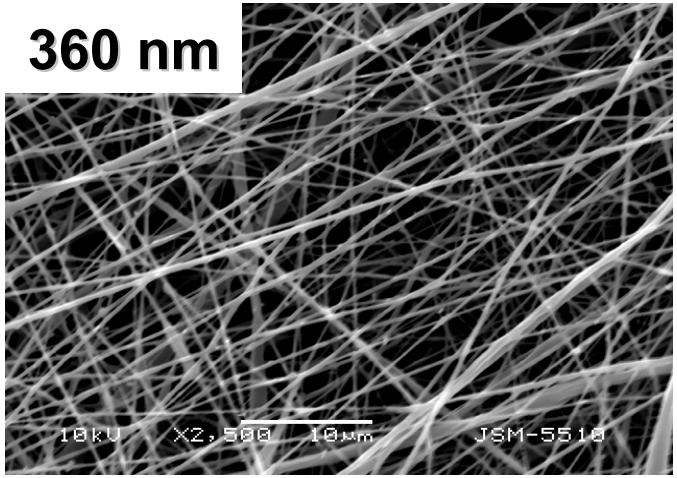


Poly(ε-caprolactone) (PCL) is a biocompatible and hydrolytically degradable polyester, which is regarded as an extremely promising candidate to design different polymer products that can find diverse applications.

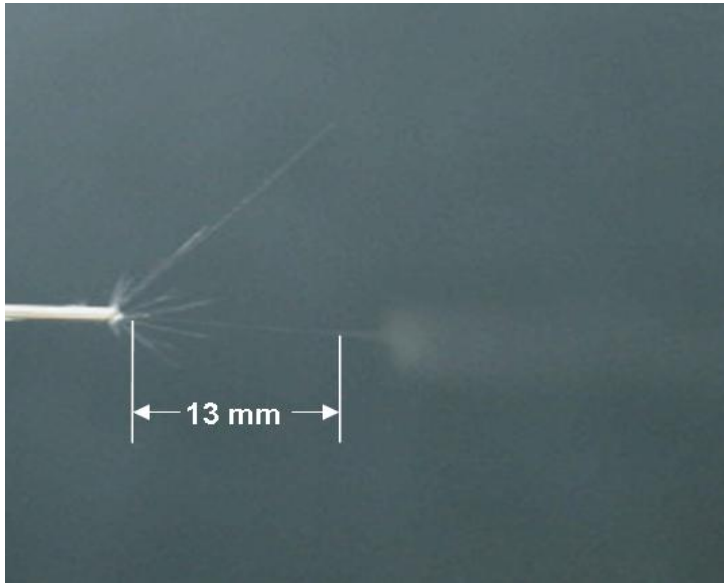
The presence of AP leads to electrospinning of thinner fibers



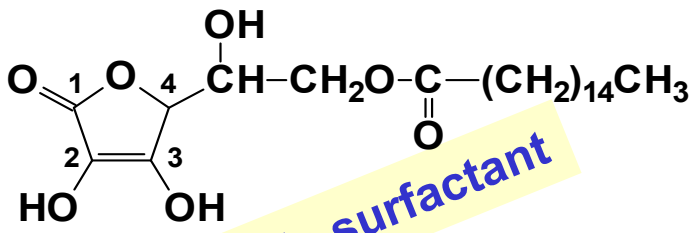




PCL

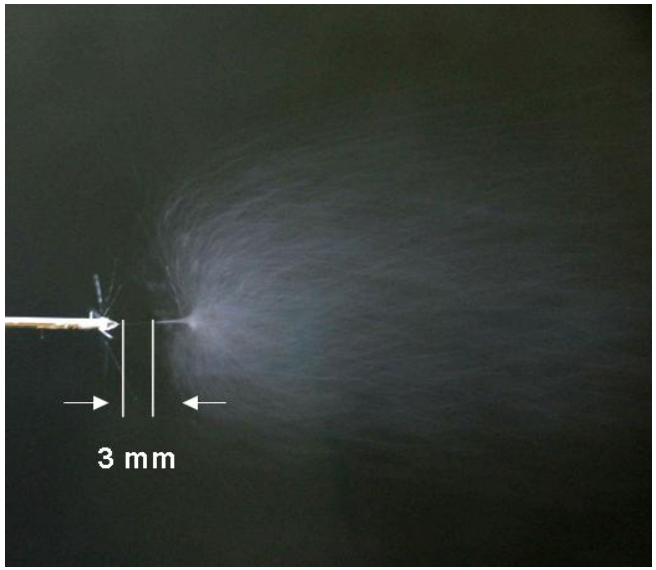


PCL/AP(9%)



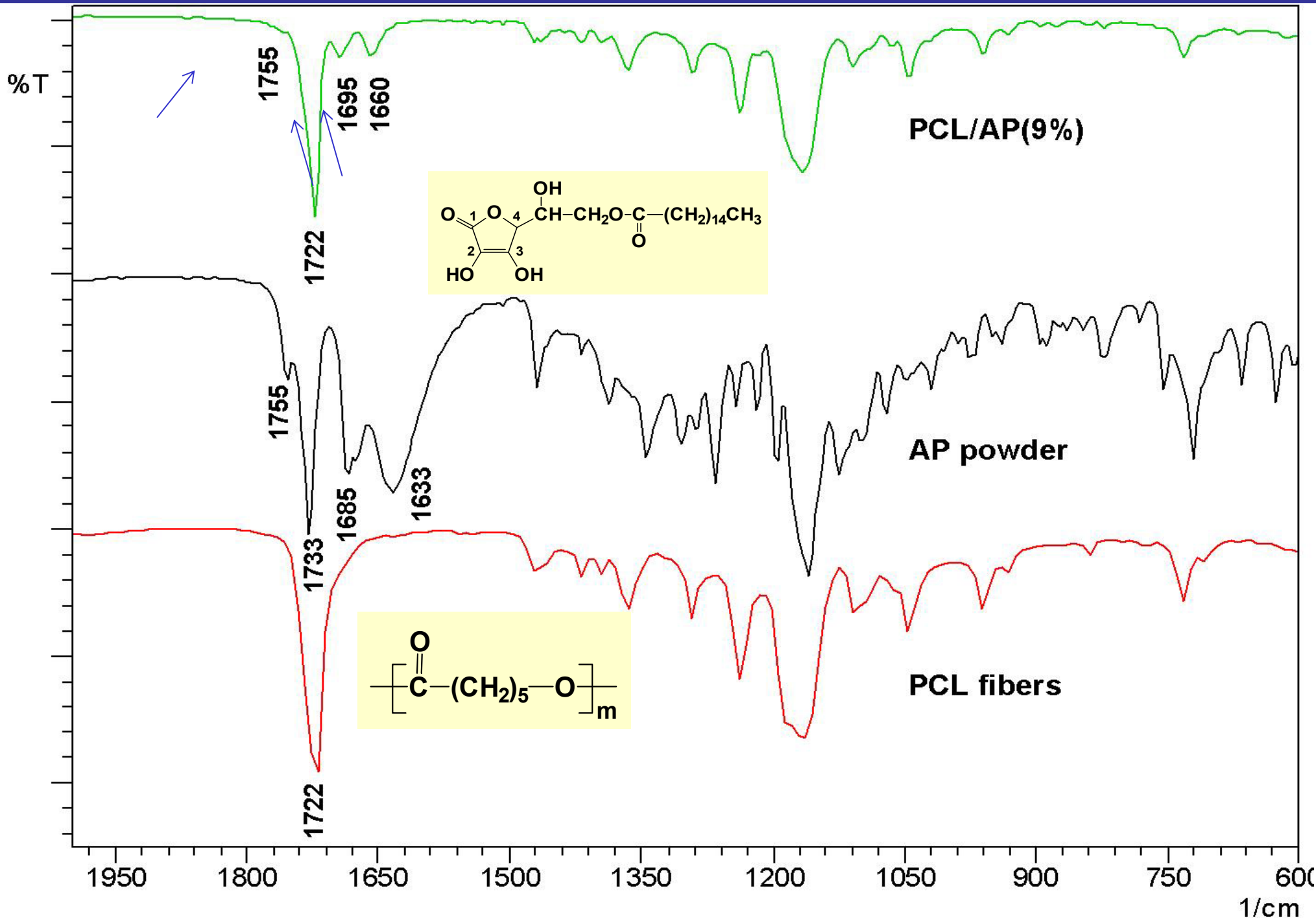
anionic surfactant

PCL/AP(30%)

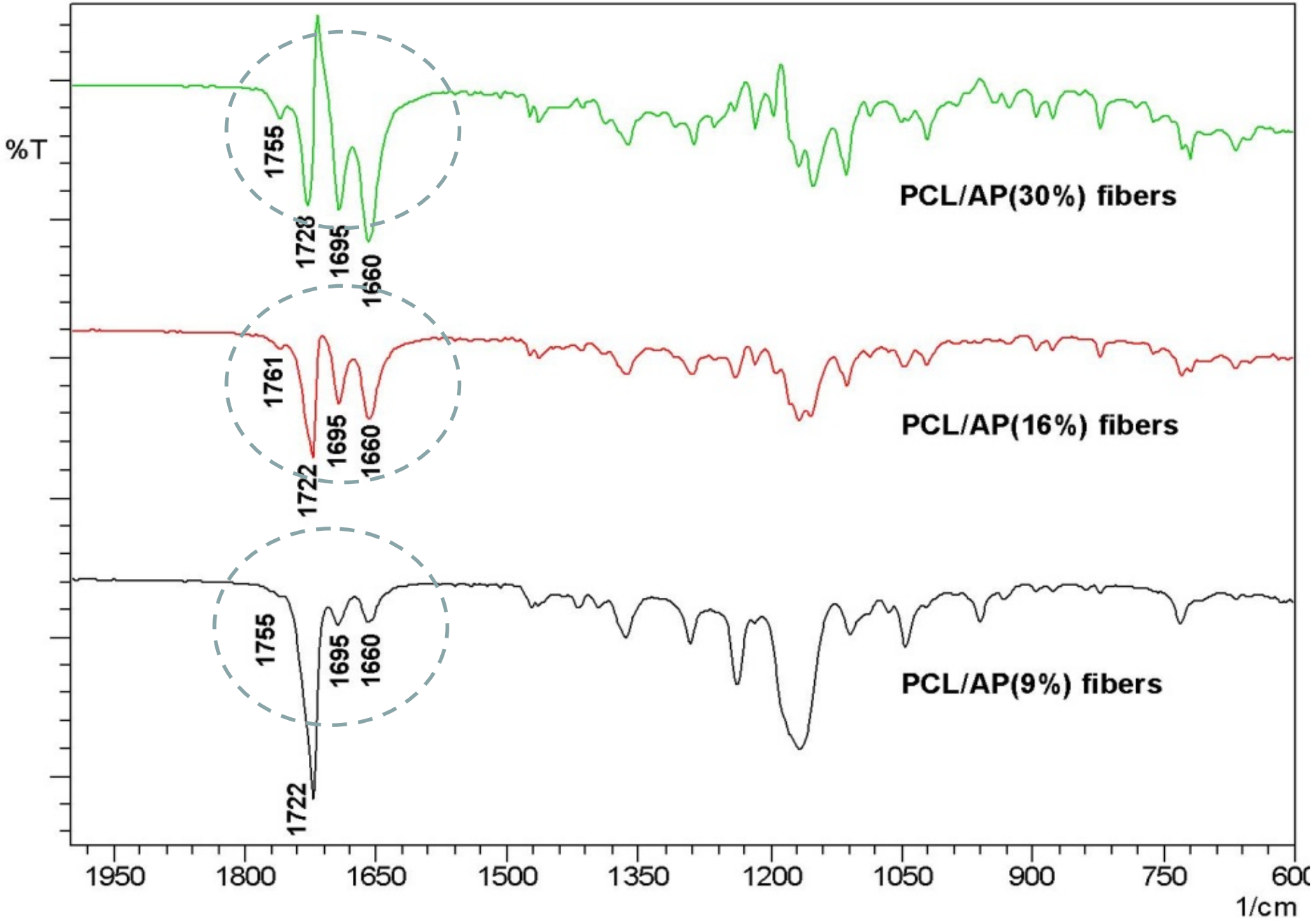


The decrease in the average diameters of the PCL/AP fibers can be attributed to shortening of the linear fragment of the jet and to an increase in the jet instability as a result of an increase in the conductivity and a decrease in the surface tension of the spinning solutions.

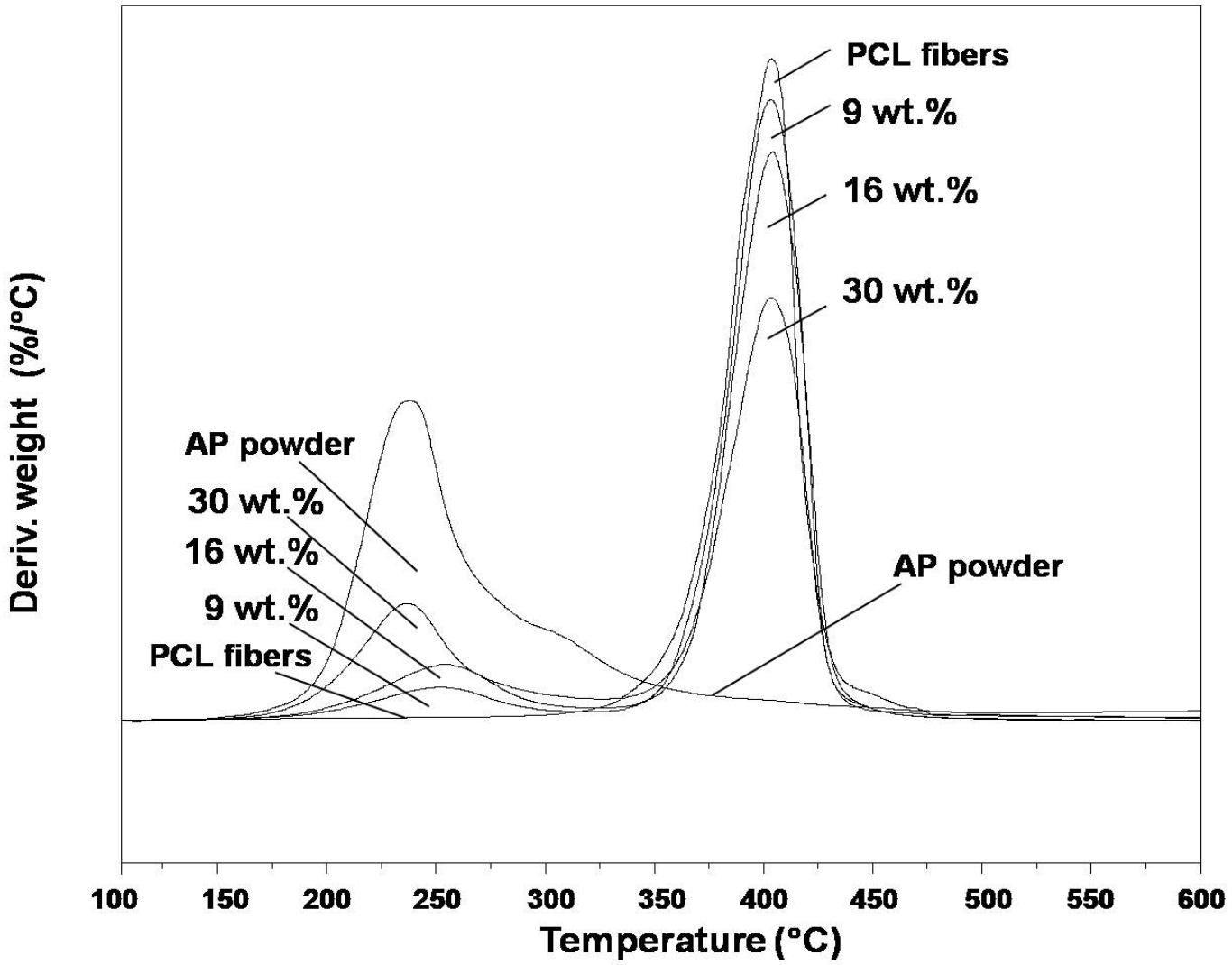
In the IR spectrum of the mat containing 9 wt.% vitamin characteristic of AP bands at 1660 and 1695 cm^{-1} and a shoulder at 1755 cm^{-1} are detected.



With the increase in the vitamin content the intensity of the bands at 1660 and 1695 cm^{-1} increases, and the shoulder which is observed at 1755 cm^{-1} at low AP content is replaced by a well defined band at vitamin content of 30 wt.%.

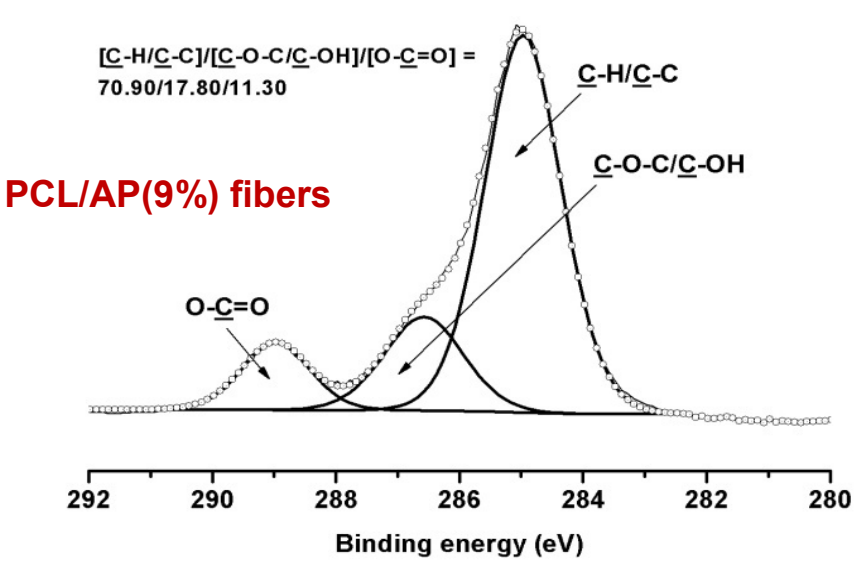


The amount of AP in the nanofibrous materials was estimated by thermogravimetric analysis (TGA)

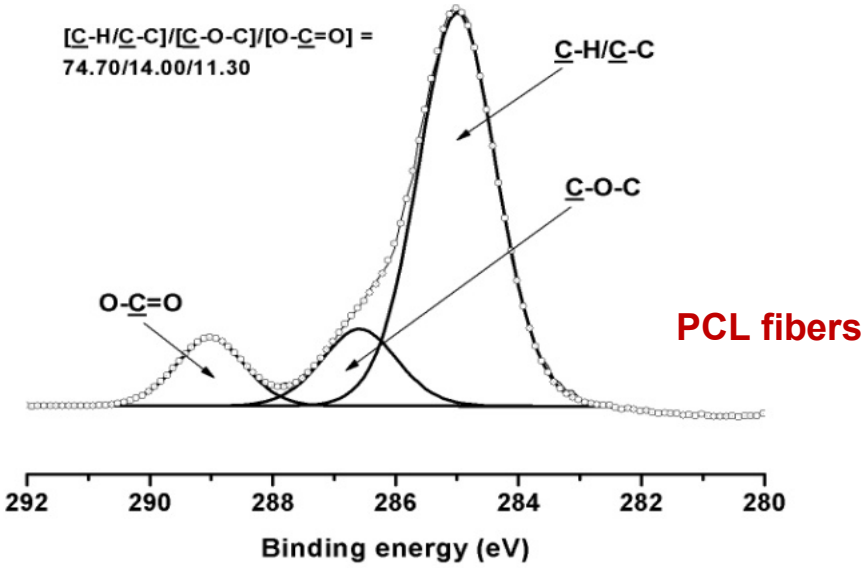


It was found that the amount of AP in the fibers is equal to that in the initial solution, i.e. the electrospinning of the PCL/AP system is an effective method for one-step preparation of nanofibrous materials containing the vitamin in the targeted concentration.

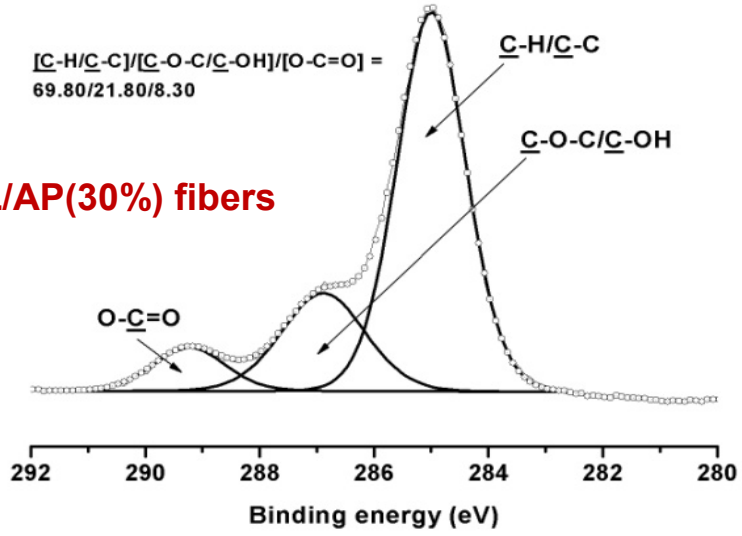
Surface composition as determined by X-ray photoelectron spectroscopy (XPS)



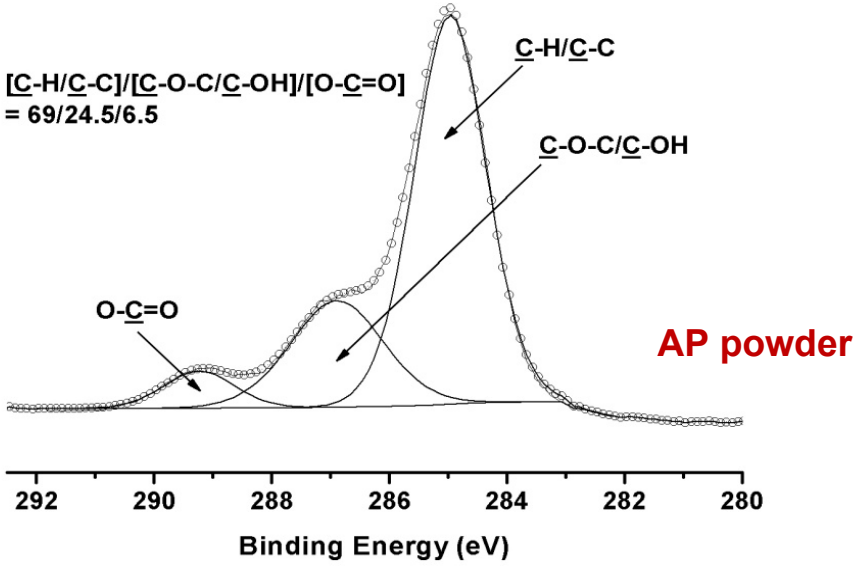
PCL/AP(9%) fibers



PCL fibers



PCL/AP(30%) fibers

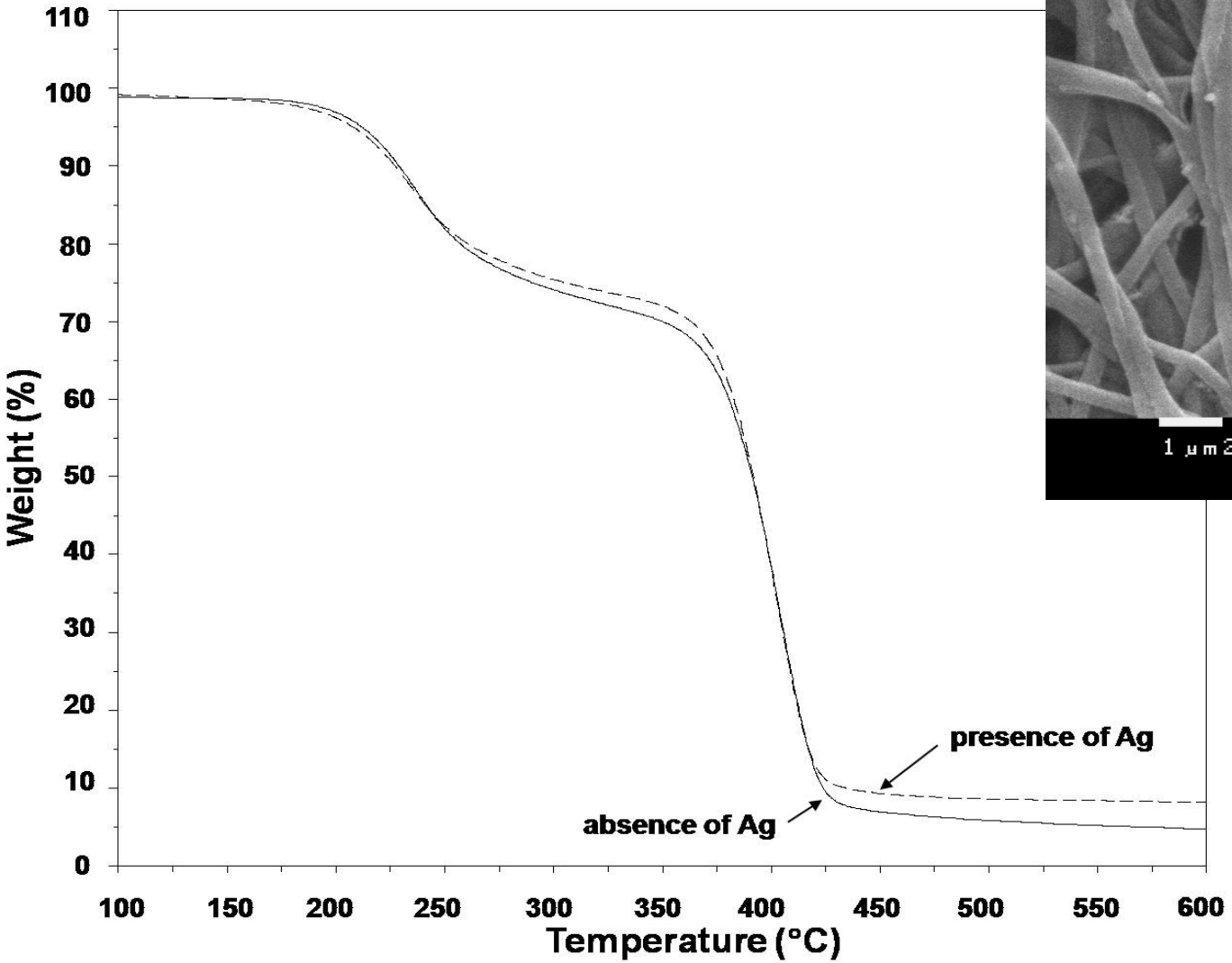


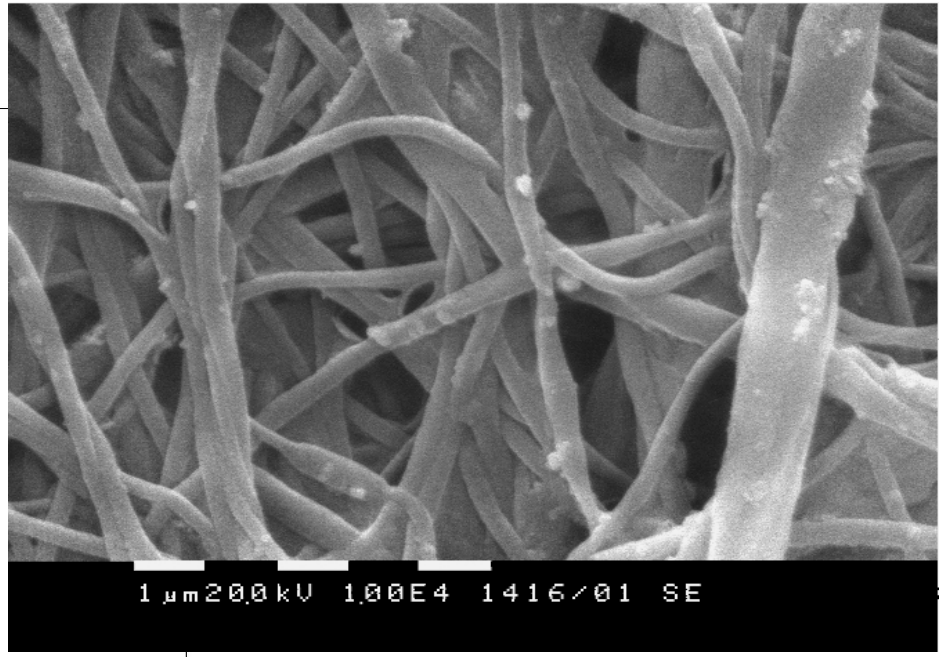
AP powder

With the increase of the AP content up to 30 wt.% the peak area for the C atoms engaged in C–O–C/C–OH bonds increases, which indicates that the higher the vitamin content of the fibers, the greater the amount of AP on the surface of the PCL/AP fibrous materials.

AP mildly reduces silver or gold ions to elemental silver or gold

$$2Ag^{+} + C_{22}H_{38}O_{7} \rightarrow 2Ag + C_{22}H_{36}O_{7} + 2H^{+}$$

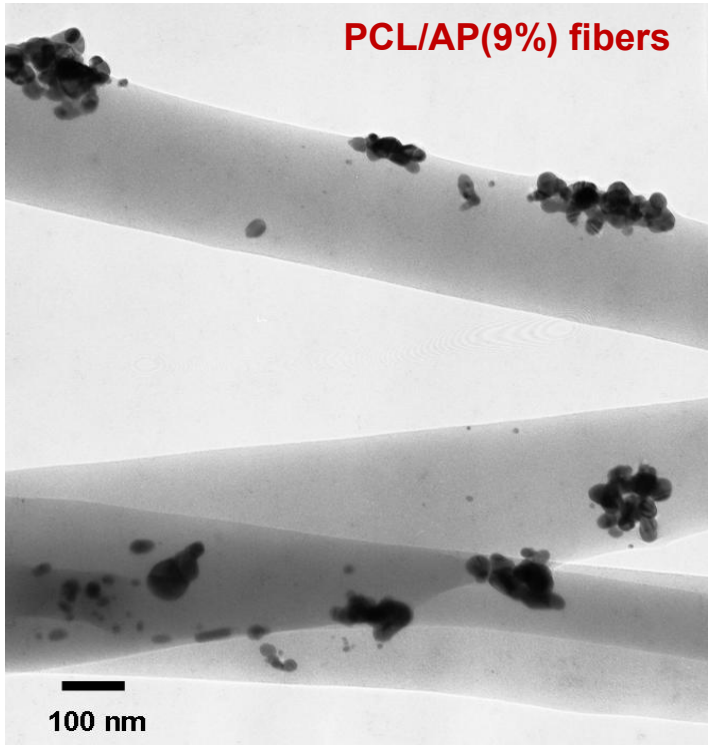


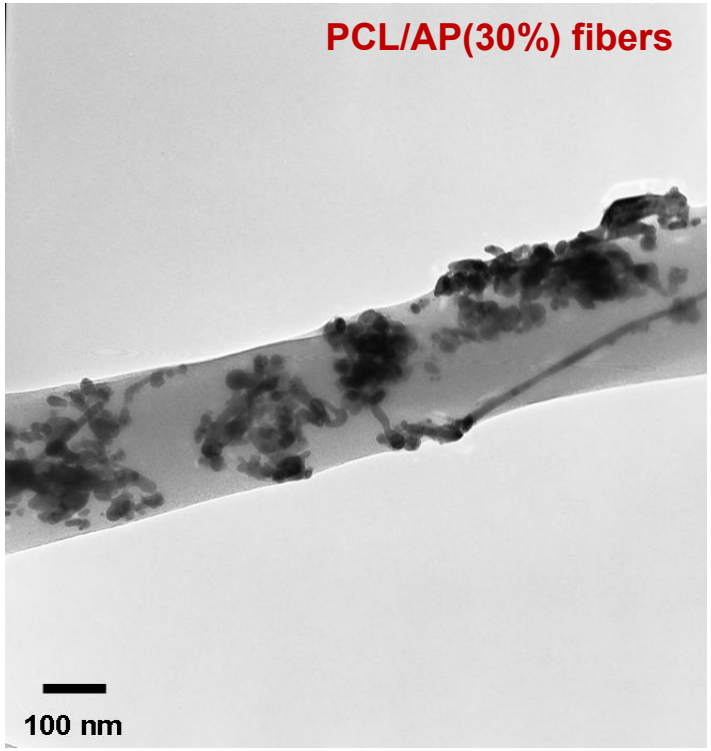


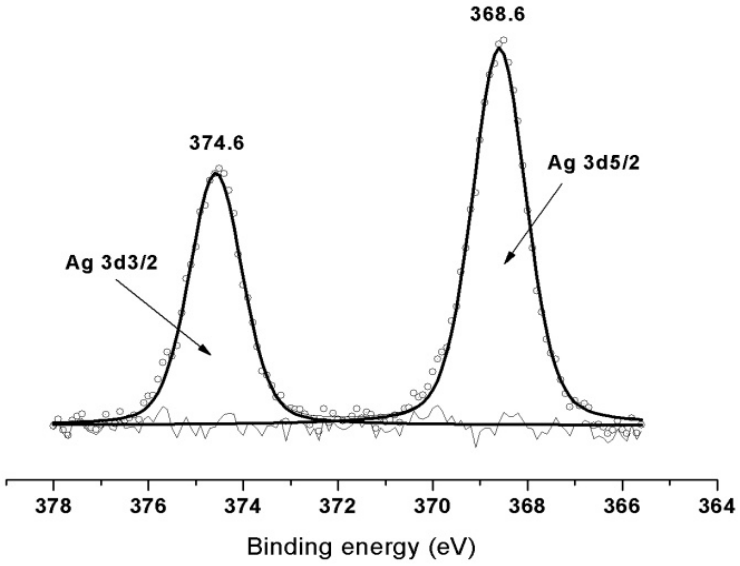
PCL/AP(30%) fibers after a 6-h stay in aqueous solution of $AgNO_3$

It was found that the silver content of the PCL/AP mats is ca. 4 wt.%.

With the increase in the AP content of the PCL/AP fibers the number of the aggregates on the fiber surface increases.



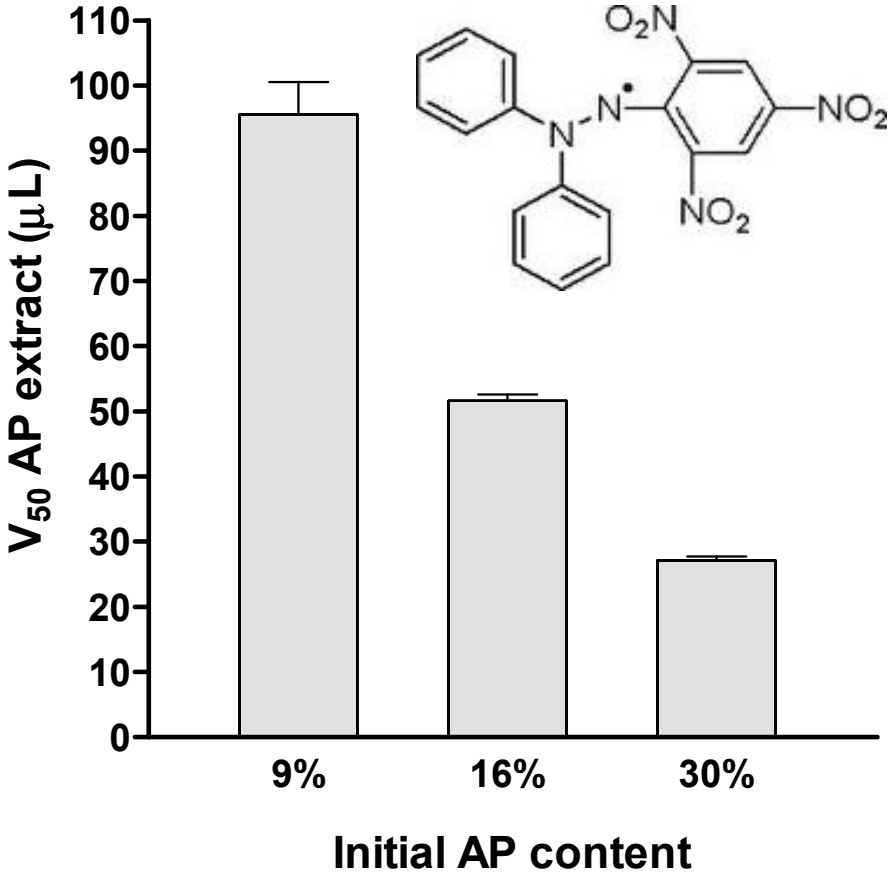


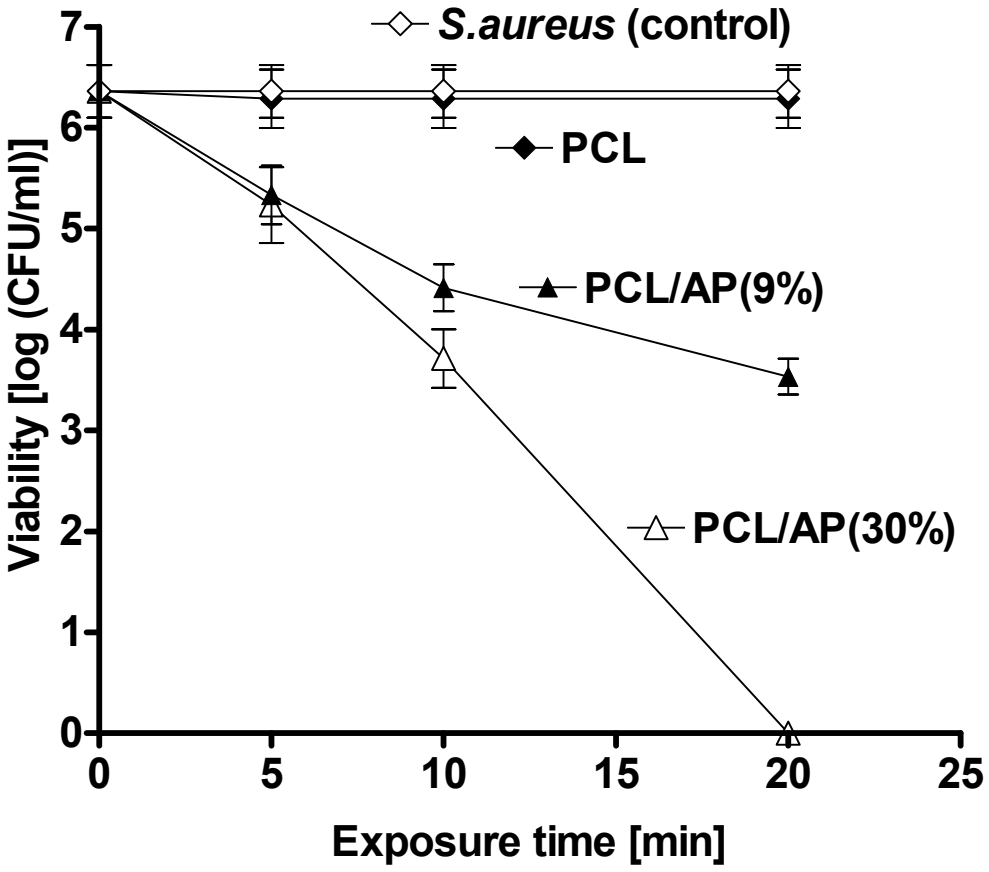


The formation of aggregates of silver nanoparticles and not of individual nanoparticles has also been evidenced by XPS analysis through the recorded peaks at 368 and 374 eV.

The incorporation of AP imparts antioxidant and antibacterial activity to the PCL mats

free 2,2-diphenyl-1-picrylhydrazyl (DPPH) radical



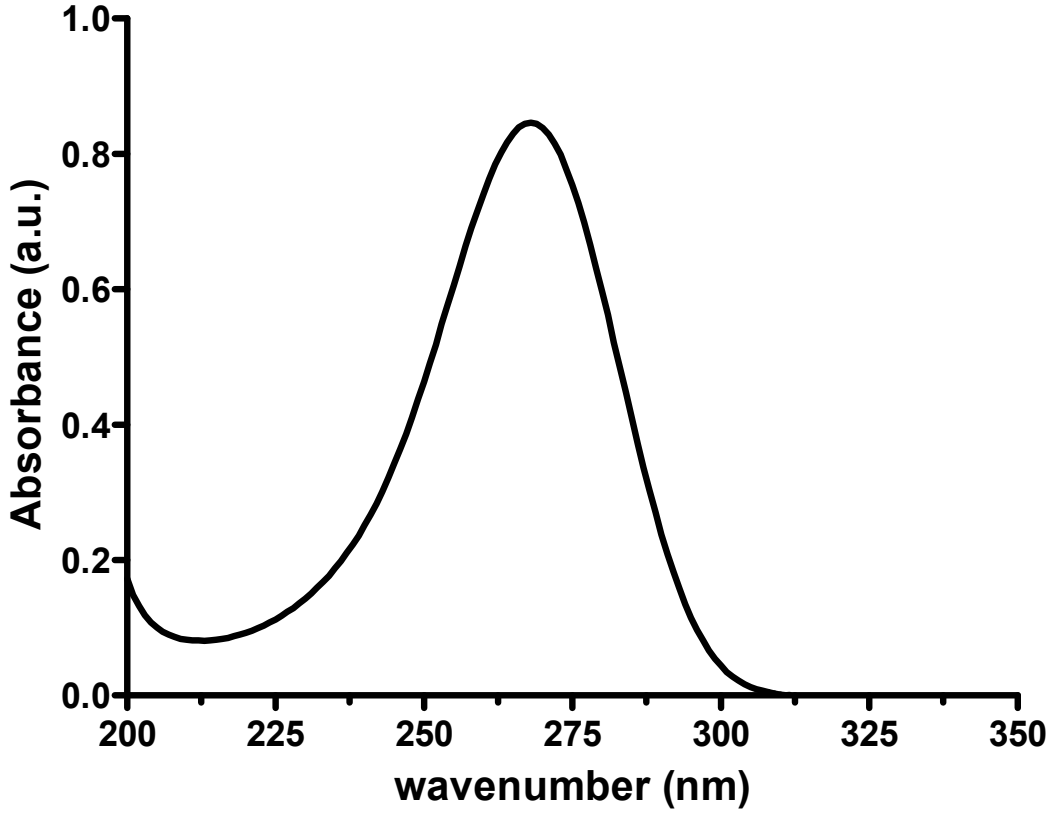


Dependence of the V_{50} value of methanol extracts of AP with regard to DPPH on the AP content of the nanofibrous mats.

Log of viable *S. aureus* cells versus the time of exposure to PCL, PCL/AP(9%) and PCL/AP(30%) fibrous materials.

UV spectroscopic analysis in absolute methanol at 268 nm have been performed in order to examine the stability of AP incorporated in the mats.

It was found that the methanol extracts absorbance value of 268 nm as well as its intensity are not altered even after a **4-month** storage of the mats in the air.



AP incorporated in the PCL mats by electrospinning does not undergo any structural changes even after a continuous storage period.

The antioxidant and antibacterial properties of the novel PCL/AP nanofibrous materials render them promising candidates for application in medicine and cosmetics.

Acknowledgments

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(Grant DO-02-333/2008) is gratefully acknowledged.

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