

Introduction

- Electrospinning techniques have attracted interest as versatile, low cost techniques to manufacture sub-micron fibres and nanofibers from polymer solutions or polymer melts [1].
- High surface area and ease of incorporation of active ingredients have prompted some researchers to investigate using electrospun nanofibers in agricultural application [2].
- However, most of the reports have focused on using electrospun nanofiber delivery systems for the encapsulation of drugs and food materials [3-4], and there are few studies have reported the encapsulation of agrochemical materials into electrospun nanofibers [2].
- Flutriafol is a commonly used fungicide in plant protection in Australia, and in this study, for the first time, we aimed to encapsulate flutriafol fungicide into poly(L-lactide) (PLLA) nanofibers matrix by optimizing electrospinning conditions.

Materials and Methods

- PLLA with a average molecular weights (MW) of 282,000 g/mol was purchased from Vorina Biomaterials, Ireland (CAS Number: 33135-50-1).
- Solvents, Chloroform (CF); anhydrous, $\geq 99\%$; and Acetone (AC) for HPLC, $\geq 99.8\%$ were obtained from Sigma Aldrich.
- Flutriafol PESTANAL (C₁₆H₁₃F₂N₃O) with molar mass of 301.29 g/Mole was purchased from Sigma Aldrich.
- The schematic setup for nanofiber electrospinning is shown in Figure 1. It consists of a high voltage (HV) power supply, model 73030, DC input 30 kV @ 1 mA, from Genvolt, Ireland, and a New Era NE-300 "Just Infusion" syringe pump. A metal frame 14cm x 16cm with attached aluminium foil was located 15cm from the syringe needle to collect nanofibers. The positive terminal of the HV was connected to the needle and the ground was attached to the collector (metal frame). The electrospinning process occurs between the needle tip and the aluminium collector, and nanofibers are gathered on the surface of the foil.
- PLLA solution with optimum concentrations of 5% (w/w) in chloroform-acetone (75–25 v/v) was prepared and mixed with 10% (w/w) flutriafol powder relative to the weight of PLLA. The PLLA/flutriafol mixture was fed with a syringe to a basic electrospinning setup. Optimum electrospinning conditions were observed at 32 °C with a flow rate speed of 1mL/ h, and 12kV high voltage.

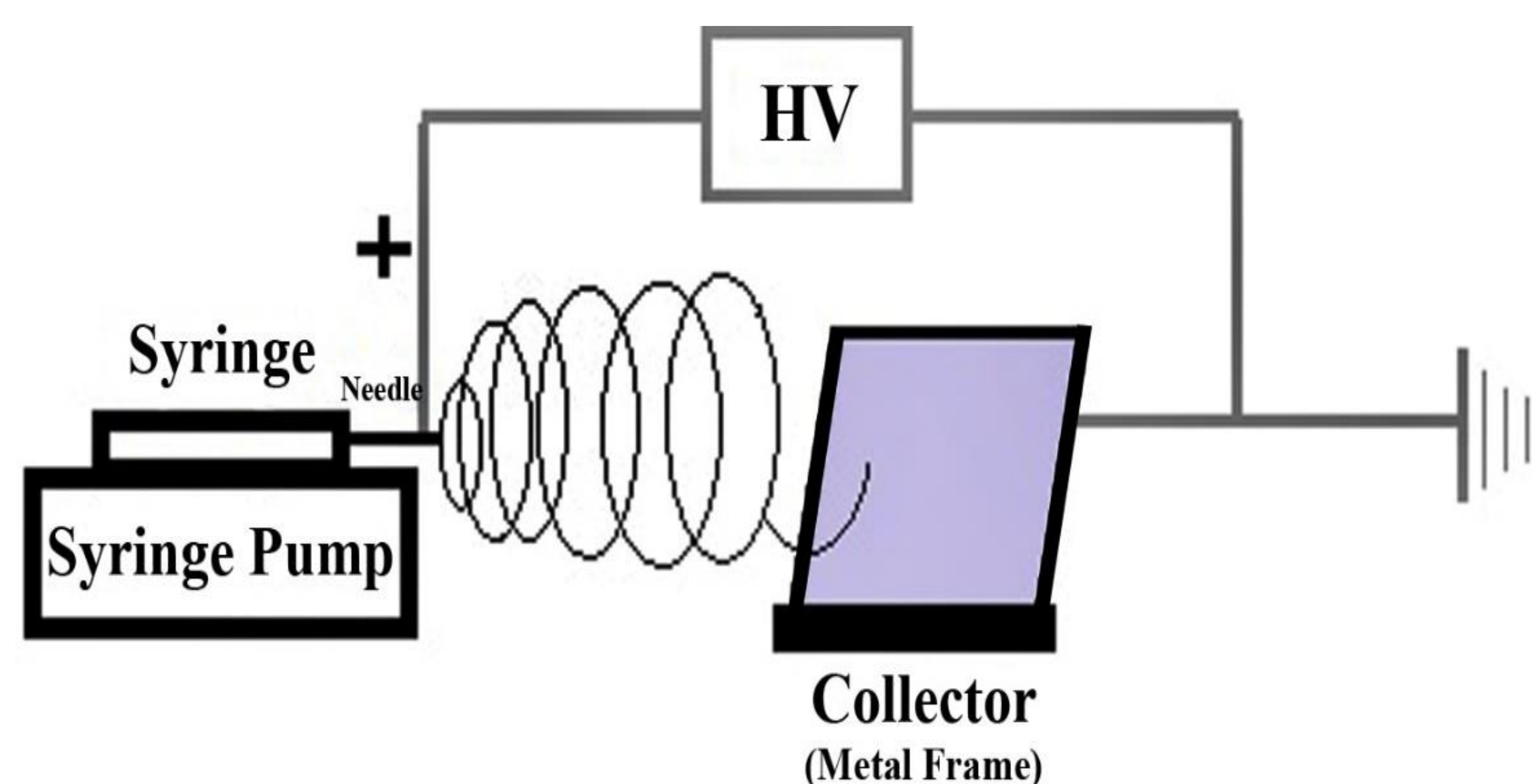


Figure 1: Schematic diagram of the electrospinning setup

Results and Discussion

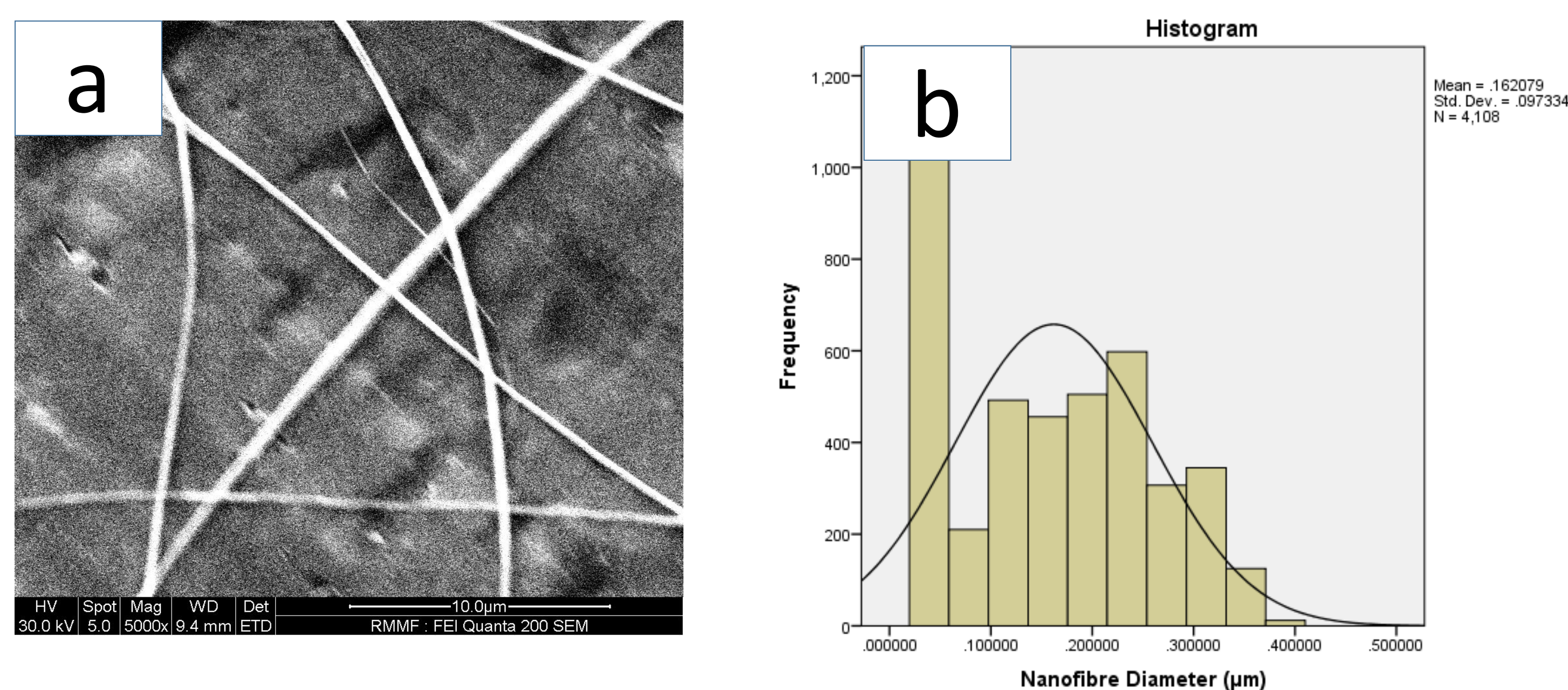


Figure 2: SEM image and related diameter histogram of the PLLA electrospun nanofibers containing 10% flutriafol

Nanofiber Characterization:

- SEM (FEI Quanta 200 SEM 2002) at the RMIT Microscopy and Microanalysis Facility (RMMF) with attached Energy-dispersive detector (EDS) was used to determine the morphology of electrospun nanofibers and identify successful encapsulation of flutriafol into PLLA nanofibers. Image J processing software was used to measure PLLA nanofiber diameters from high magnification SEM images.
- In the case of effect of flutriafol concentration on nanofiber morphology, SEM image of PLLA electrospun nanofibers loading flutriafol with related histogram of nanofibers diameter distribution are shown in Figure 2 (a, b), and nanofiber diameters are displayed in Table 1. This effect was consistent in the present study, the diameter of PLLA electrospun nanofibers decreased from 496.183nm to 162.079nm with adding 10% flutriafol to the electrospinning solution (Figure2; Table 1).
- By adding some ionic organic/inorganic compounds such as flutriafol to the PLLA polymeric solution, the increased number of electrical charge causes an increase in the elongation capacity of the solution, favoring the formation of smooth fibers, with smaller diameter [5].

Table 1. Effect of flutriafol concentration on nanofiber diameter

Flutriafol Concentration (% w/w)	Nanofiber diameter (nm)	**CV%
0	496.183	22.0%
10	162.079	9.7%

*% w/w based on amount of PLLA used, **Coefficient of Variation (CV).

EDS Characterization:

A typical SEM micrograph and EDS spectrum analysis of PLLA nanofiber containing 10% flutriafol are illustrated in Figure 3(a, b). The EDS shows the amount of nitrogen, and fluorine elements in spectrums 10, and 11 based on wt. % ratio to PLLA mass amount. The percentage amount of identified elements is shown in Table 2.

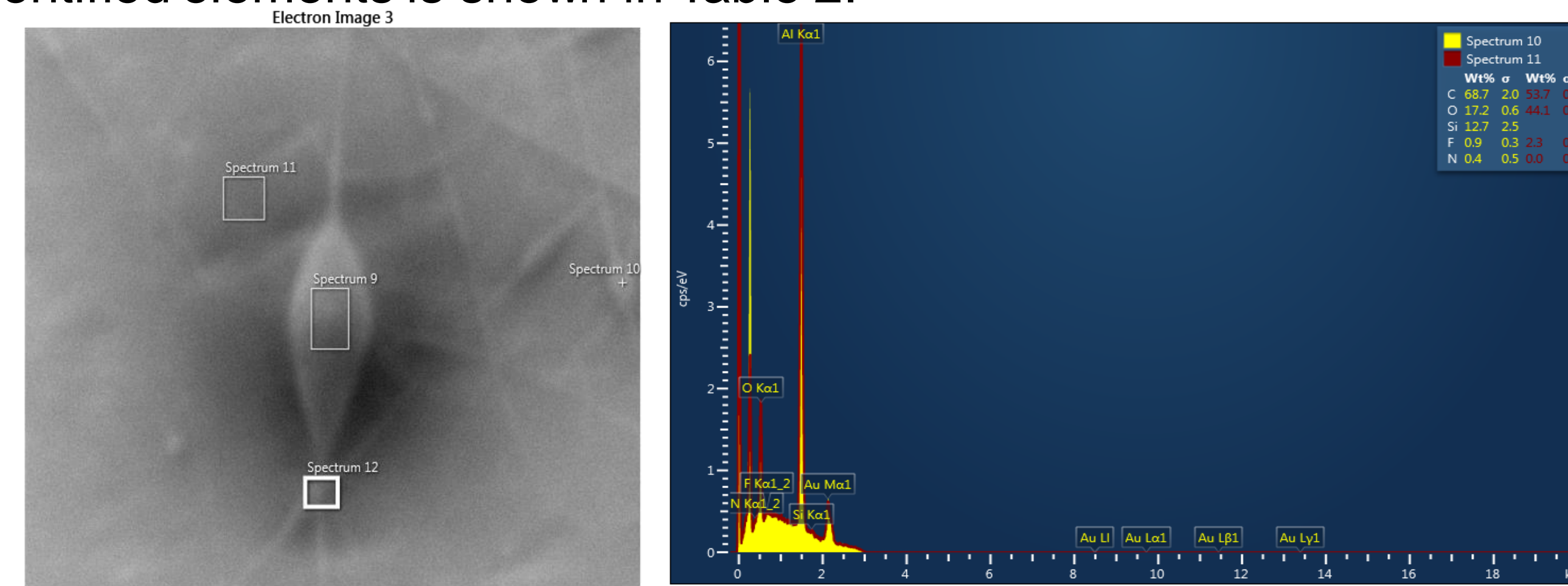


Figure 3: (a) SEM micrograph, and (b) EDS analysis of PLLA nanofiber containing 10% flutriafol.

Table 2. Percentage of identified elements in PLLA nanofibers.

Statistics	C	N	O	F
Max	72.63	1.30	44.06	2.27
Min	53.57	0.40	24.42	1.36
Average	63.10	0.85	34.24	1.81
Standard Deviation	13.40	0.74	12.76	0.64

Conclusion

- EDS analysis of PLLA nanofibers proved that flutriafol compound elements have been successfully encapsulated into PLLA nanofiber structures.

Acknowledgment

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References

1. Subbiah, T. et al. (2005) 'Electrospinning of Nanofibers', Journal of Applied Polymer Science, 96, pp. 557–569.
2. Noruzi, M. (2016) 'Electrospun nanofibers in agriculture and food industry: A review', Journal of the Science of Food and Agriculture, 96(14), pp. 4663–4678.
3. Kenawy, E-R. et al. (2002) 'Release of tetracycline hydrochloride from electrospun poly(ethylene-co-vinylacetate), poly(lactic acid), and a blend' Journal of Controlled Release 81, pp. 57–64.
4. Fernandez, A. et al. (2009) 'Novel route to stabilization of bioactive antioxidants by encapsulation in electrospun fibers of zein prolamine', Food Hydrocolloids, 23, pp. 1427–1432.
5. Amariei, N. et al. (2017) 'The Influence of Polymer Solution on the Properties of Electrospun 3D Nanostructures', IOP Conf. Series: Materials Science and Engineering, 209, pp. 1–7.