Synthesis and morphological characterization of Li-Ti/PVP fibers as precursors for Li$_4$Ti$_5$O$_{12}$ towards its future use as anode materials in Li-ion fiber batteries by means of Electrospinning

N Castano$^{1,2}$, M A Cortes$^{1,2}$, E Garcia$^{1,2,3}$ and H V Martinez$^{1,2}$

1 Mechanical Engineering Department. Grupo de investigación sobre Nuevos Materiales (GINUMA), Universidad Pontificia Bolivariana, Medellin, Colombia
2 Mechanical Engineering Department. Grupo de investigación Energía y Termodinámica (GET), Universidad Pontificia Bolivariana, Medellin, Colombia
3 Nanotechnology Engineering Department, Universidad Pontificia Bolivariana, Medellin, Colombia

Abstract. The ever-increasing evolution and popularity of electronic devices, in hand with globalization, has led to a highly innovative market, always in movement. Novel applications such as smart textiles, where portable electronics are coupled to fabrics, are expected to become a worldwide trend when they overcome some limitations, especially with the energy storage systems used to power them. It is in this context where a flexible battery with the shape of a fiber is of importance. Electrospinning is a versatile synthesis technique where micro and nano fibers can be obtained. When these fibers are conducted onto a substrate it is possible to produce materials with different characteristics and morphologies. In this work, Li$_4$Ti$_5$O$_{12}$ (LTO) was synthesized as anode material for li-ion batteries and was further characterized. LTO is attractive as it presents characteristics such as high thermal stability, relatively high volumetric capacity and high cyclability. The synthesis of this material was developed in two steps. First, a precursor solution containing a spinnable polymer, and titanium and lithium salts, which were dissolved in a mixture of solvents, was subjected to a process of electrospinning. Then, the obtained fibers were calcined at temperatures between 650 and 850 °C for 7-10 hours in air or argon. Scanning electron microscopy (SEM) coupled to energy dispersive x-ray spectroscopy (EDS) was used to study its morphology and elemental composition. Meanwhile, thermogravimetric analysis (TGA) was performed to study the thermal stability.

1. Introduction
The ever-increasing degree of portability in electronic devices, where there is a constant requirement for reduction in size and weight, makes evident the need for more efficient energy storage systems to power these applications. Likewise, hyper connectivity in today’s society is not something temporary but, on the contrary, there is a worldwide trend moving towards an inter-networking of physical devices, vehicles and even buildings, which has been denoted as Internet of Things (IoT) [1][2]. This game changing idea calls for objects to collect and exchange data with the possibility that, using a cellphone,
different electronic devices can be controlled [1][3], i.e. physiological vital signs from a patient [4][5][6] or electrocardiogram signals from animals by means of Smart textiles [7][8] could be remotely monitored. The design of such monitoring systems is more common every day due to their potential application in medicine, sports and security [9]. Nevertheless, the performance and massive market penetration of these type of technologies depend critically on the development of energy storage systems with adequate configuration and geometry, which can be adapted for different applications [10][11][12].

In this regard, it is crucial to develop better materials to increase the efficiency of energy storage systems [13]. In particular, nanotechnology is one of the strategies people use for the improvement of materials for anodes, cathodes, electrolytes and other components, as the increase in surface area provided when dealing with materials at this scale offers more reaction and exchange sites [14], which allows for the design of electrodes with higher energy and power densities and stability upon cycling [15].

In Li-ion batteries, graphite is the material typically used for the anode but it has issues associated with mechanical stability upon cycling, operative safety and relatively slow kinetics during charge/discharge [16]. Different alternatives for graphite as anode material have been investigated. Among them, the spinel material Li4Ti5O4 (LTO) stands out in terms of stability upon successive lithium ion insertion/removal. It has a decent theoretical capacity of 175 mAh/g, at a voltage of 1,55 V vs Li+/Li [17] and is dubbed as a “zero stress” material as the phase change during lithiation/delithiation results in very small volumetric changes (≈0.2%) [18]. This feature makes it a material with an increased safety (it suppresses the formation of the solid electrolyte interphase -SEI- and impedes the formation of lithium dendrites), highly durable as it presents a good cycling life cycling rate and it is able to withstand high charge/discharge current rates [19][20][21]. It is therefore a strong candidate to circumvent several technical issues associated with batteries, both in stationary electricity storage and portable applications [22].

Electrospinning is a synthesis technique which permits the creation of fibers from a polymer solution. In it, a precursor solution is loaded into a syringe and is subjected to a high voltage and, depending on operation parameters of both the equipment (voltage, flow rate, distance between tip and collector) and the solution (concentration, viscosity, surface tension, conductivity), the formation of fibers, sometimes with diameters in the nano-range, can be achieved. These geometrical morphology could open doors in the development of flexible batteries with potential application in smart textiles [23][24][25].

This article describes the procedure to obtain a working precursor solution formulation for the production of Li4Ti5O12 fibers to be used as anode material for Li-ion fiber batteries.

2. Materials and Methods
2.1. Preparation of precursor solution.

The process of preparation of a working precursor solution was iterative and was initially based on formulations found in the literature [26],[27]. To obtain Li4Ti5O12 fibers, precursors for each component (i.e., lithium, titanium and carbon) were needed.

Following the formulation suggested by Bingkun Guo et al [28], isopropyl alcohol was used as solvent for polivinilpyrrolidone (PVP) as well as for lithium acetate and titanium butoxide precursor salts. However, the solution was not stable as it precipitated during the electrospinning process. On the other hand, other authors suggested a mixture of solvents such as acetic acid, ethanol, acetone and acetylacetone [29],[30]. Following this formulation, a stable solution was obtained for the timespan of the electrospinning process and, thus, two precursor solutions were prepared: in solution 1 (Sln 1), 7.5 g of ethanol, 1.5 g of acetic acid and 2.5 g of acetone were mixed during 10 minutes under constant stirring. Then, 0.5 g of lithium acetate, 3 g of titanium butoxide and 5 g of polivinilpyrrolidone (PVP, MW: 55000) were added under constant stirring during 24 h at 50 ° C. Solution 2 (Sln 2) was prepared by mixing 9 g of ethanol, 2.5 g of acetic acid under constant stirring for 10 minutes, followed by an addition of 0.5 g of lithium acetate and 3 g of titanium butoxide under constant stirring during 60 minutes. Finally, 5 g of polivinilpyrrolidone (PVP, MW: 55000) was added under constant stirring during 24 h at 50 °C. All chemicals used were purchased from Sigma-Aldrich.
2.2. Rheological characterization
The rheology of the precursor solutions described above, was analyzed using a DHR2 rheometer (Discovery Hybrid Rheometers) with a standard 25 mm diameter stainless steel flat plate geometry, 50.0 μm gap between plates, a tension of 2%, with an applied oscillatory strain of 10 Hz and temperature control at 25 °C. Pretreatment curves were done at a shearing rate of 10 1/s during 10 seconds, followed by flux scan in the range between 0.1-200 1/s point by point, with an average value of 10 s per point.

2.3. Thermal characterization
Thermogravimetric analysis (TGA) was done using a thermobalance (Mettler Toledo TGA/SDTA 851 E) between 30 - 800 °C with a heating rate of 10 °C/min under nitrogen. Two analyses were performed, one with PVP to have a comparison (blank) and the second with the Li-Ti/PVP precursor fibers.

2.4. Electrospinning setup
A schematic drawing of the setup used for the production of Li-Ti/PVP fibers is shown on figure 1 (right). It consists in a modular system with three main elements: (1) a high voltage source (5-30 kV); (2) a syringe pump, to deliver solutions in a controlled way (flow rate and volume); and (3) a rotating drum which acts as collector and that is grounded to close the circuit. Additional accessories such as (4) and (5) are a ventilation system and a high intensity lamp, respectively.

When the precursor solution is flowing at a specific flow rate, a droplet starts forming at the tip of the nozzle. When it is subjected to a high voltage, the forces acting in the droplet reach a point in which the so-called Taylor cone [31] is formed, mainly due to electrostatic effects, and a jet emerges from the apex of the cone. Inner forces such as surface tension prevent the breakage of the droplet and, thus, the jet becomes elongated forming a fiber. Depending on operation parameters of both the equipment (voltage, flow rate, distance between tip and collector) and the solution (concentration, viscosity, surface tension, conductivity), the formation of fibers with diameters in the micro/nano-range, can be achieved.

As it can be appreciated in figure 1, electrospinning is a simple and versatile technique, that is relatively easy to setup, which permits the production of fibers at the micro/nano scale for a large variety of applications, due in part for the wide range of polymers that can be used [32][33]. The morphology of the materials produced with this technique can be varied by tuning the parameters associated with the precursor solution (viscosity, concentration, molecular weight of the polymer, surface tension,
conductivity, solvent type), the setup (voltage, flow rate, distance from tip to substrate, geometry of the collector) and the environmental conditions (temperature and humidity) [34][35].

In this research, the setup parameters were fixed while the behavior of precursor solutions 1 and 2 were evaluated. Thus, both solutions were subjected to a voltage of around 20 kV (Gamma RS232 power source), a flow rate of 0.2 mL/h and a distance of 15 cm from tip to a rotating cylindrical drum covered with aluminum foil.

2.5. Thermal treatment.
The obtained electrospun fibers were pre-dried between 80-90 °C during 30 minutes. Then, the deposited film was scratched from the collector surface and was calcined at 750°C during 10 h in air, following similar reports in the literature [36].

2.6. Chemical and morphological characterization.
The morphology, size and size distribution of samples were evaluated using a scanning electron microscope (SEM, JEOL JCM-6000 Plus) coupled with an energy dispersive x-spectroscopy (EDS) detector for chemical composition analysis.

3. Results and Discussions
3.1. Rheological characterization.
The rheology of the precursor solutions is shown in figure 2. Both present a behavior similar to a dilatant fluid (i.e. a liquid that presents an increase in viscosity with the increase of shear rate) on the first points of the curve [37]. For Sln 1, this behavior goes until a shear rate of 20 1/s and for Sln 2 until 46.5 1/s.

![Figure 2. Rheological behavior of the precursor solutions](image)

After these inflection points, the behavior of Sln 1 tends towards a pseudo plastic liquid [38], while Sln 2 looks more like a Newtonian fluid with a viscosity of ~0.6 Pa.s. According to the dilatant behavior in both solutions, it seems convenient to operate the electrospinning at high voltages to guarantee a small range of viscosities during the process, as larger viscous forces affect the size of the fibers [39][40]. It is worth mentioning that to know the exact operating conditions during electrospinning, a correlation
between the shear rate and applied voltage should be considered. In this regard, it is known that for a liquid flowing through a cylindrical cavity there is a proportional relation between shear rate and flow rate \( \alpha \) [flow rate]. Also, it’s been experimentally observed that the diameter of the jet decreases with the increase on voltage. However, this correlation is not completely understood at present and is beyond the scope of this work.

3.2. Thermal characterization:
Thermogravimetric analysis (fig. 3) of both the precursor fibers obtained through electrospinning of Sn 2 and the polymer used as precursor (PVP, MW: 55000).

![Figure 3. Thermogravimetric analysis of PVP and as-obtained Li-Ti/PVP fibers](image)

A summary of the relevant thermal events present in the thermogram are shown in table 1. It is observed that for the precursor fibers there is loss of humidity between 46 and 107°C, corresponding to a 10.1% loss of mass. For the PVP, there is loss of humidity between 46 and 104°C, corresponding to 3.7% of its mass.

| Mass (mg) | Thermal event | \( T_i \) (°C) | \( T_f \) (°C) | \%M_i | \%M_f |
|----------|---------------|----------------|----------------|------|------|
| PVP      | 1             | 46             | 104.2          | 100  | 96.3 |
|          | 2             | 335.2          | 477.1          | 96.3 | 9.6  |
|          | 3             | 477.1          | 780            | 9.6  | 5    |
| Precursor Fibers | 1 | 46 | 107.1 | 100 | 89.9 |
|          | 2             | 135            | 500.7          | 89.8 | 22.2 |
|          | 3             | 500.7          | 780            | 22.2 | 20.8 |

In the second event for the PVP, between 335 and 477°C most of the carbon is eliminated, corresponding to 90% of its mass. Likewise, for the Li-Ti/PVP fibers, from 135 and 500°C carbon is eliminated, corresponding to 75% of its mass. After this, the mass of the precursor fibers remains almost constant suggesting that the reaction is over. Therefore, to obtain the Li4Ti5O12 composite a calcination temperature above 500°C is required. Similar reports in the literature suggest a range of temperature between 500-780°C [29][41].
3.3. Morphological and chemical characterization

Several attempts were made with Sln 1 to obtain fibers. Figure 4 shows the outcome of most of them and it can be clearly seen that no fibers were formed by using this solution. This result looks closer to an electrospray process where instead of fibers there is a generation of droplets or particles [42]. The average diameter of the obtained particles is 3.1 μm with a standard deviation of 1.0 μm. The main difference between solutions 1 and 2 are the mix of solvents used to dissolve the precursor salts and the polymer. This sole variation can change properties such as surface tension, boiling point and rheological behavior. The boiling point of the precursor solution is crucial as the solvent evaporation of the formed jet during the time of flight it takes to travel between the tip of the nozzle and the collector drum clearly defines the final morphology [34] [43]. Besides, the environmental conditions for this experiment, i.e 24.3°C and 70% relative humidity, were not favorable for the formation of PVP-based fibers, according to Pelipenko et al. [44].

![Figure 4. SEM micrograph of an electrospinning process with Sln 1 (with acetone)](image)

In figure 5, a set of micrographs of fibers obtained with Sln 2 are shown. Fig. 5A-B represent the Li-Ti/PVP precursor fibers, as-obtained with electrospinning, while fig. 5C-D represent the LTO fibers after calcination at 750°C in 10 hours in air.

The average diameter of Li-Ti/PVP fibers is 0.87 μm with a standard deviation of 0.63 μm, while the LTO fibers have a mean diameter of 1.23 μm and a standard deviation of 0.74 μm. The parameters used to obtain these fibers were: flow rate, 0.2 mL/h; voltage, 20 kV; distance, 15 cm; 19 G nozzle (external diameter, 1.07 mm and internal diameter, 0.69 mm); collector drum rotation speed, 20 rpm-clockwise; temperature, 26-27°C; and humidity, 40-50% (these environmental conditions were more favorable for the production of fibers [42][45]).

EDS spectrum of the LTO fibers (after calcination) are shown in figure 6. It reveals the presence of Ti and O, both of them crucial to form the Li4Ti5O12 stoichiometry and the spinel structure [46]. Quantitative analyses gave a 49.60% mass percentage and 74.66% atomic percentage for oxygen, while for titanium it gave 50.40% mass and 25.34% atomic. These results suggest a molar relation of 1:2.95 titanium to oxygen, respectively, and are very promising when looking at the molar ratio for LTO of 1:2.4 [47].
Figure 5. SEM images. (A-B) Li-Ti/PVP precursor fibers before calcination. (C-D) LTO fibers after calcination at 750°C for 10 h in air

Figure 6. Energy-dispersive X-ray spectroscopy (EDS) of electrospun LTO fibers (0.2 mL/h, 20 kV, 15 cm)
4. Conclusions
In this study, nanofibers based on lithium and titanium salts, together with PVP polymer were successfully produced by means of electrospinning. Moreover, upon calcination at 750°C, after finding a temperature window for this process using thermogravimetric analysis, the fibers maintained their morphology. After chemical composition analysis, the atomic percentage of oxygen to titanium in these fibers matched that of the Li$_4$Ti$_5$O$_{12}$ spinel.

Two different solutions were used, employing different solvent mixtures, while keeping all other parameters involved in electrospinning fixed. Fibers were not obtained using solution 1 (with acetone), contrary to solution 2, with the same equipment parameters (voltage, flow rate, distance). It is important to clarify that this electrospinning parameters are recommended for the specific precursors used here, since properties of the solution such as concentration, viscosity, surface tension and conductivity will vary and hence, the optimal electrospinning parameters will also change.

It is relevant to mention that this is a preliminary step on the synthesis of full LTO anode fibers. Finally, the applicability of these nanofibers as anode material will ultimately depend on their electrochemical performance.

This is an ongoing project and further work is needed.

Acknowledgments
The work carried out in this project has been financed by Universidad Pontificia Bolivariana and Patrimonio Autónomo Fondo Nacional de Financiamiento para las Ciencias, la Tecnología y la Innovación, Francisco José de Caldas.

References
[1] T. Saarikko, U. H. Westergren, and T. Blomquist, “The Internet of Things: Are you ready for what’s coming?,” *Bus. Horiz.*, vol. 60, no. 5, pp. 667–676, 2017.
[2] L. Atzori, A. Iera, and G. Morabito, “Understanding the Internet of Things: definition, potentials, and societal role of a fast evolving paradigm,” *Ad Hoc Networks*, vol. 56, pp. 122–140, 2017.
[3] J. M. Jornet and I. F. Akyildiz, “The Internet of Multimedia Nano-Things,” *Nano Commun. Netw.*, vol. 3, no. 4, pp. 242–251, Dec. 2012.
[4] S. Majumder, T. Mondal, and M. J. Deen, “Wearable Sensors for Remote Health Monitoring.,” *Sensors (Basel).*., vol. 17, no. 1, Jan. 2017.
[5] G. Appelboom, E. Camacho, M. E. Abraham, S. S. Bruce, E. L. Dumont, B. E. Zacharia, R. D’Amico, J. Slomian, J. Y. Reginster, O. Bruyère, E. S. Connolly, and Jr., “Smart wearable body sensors for patient self-assessment and monitoring.,” *Arch. Public Health*, vol. 72, no. 1, p. 28, 2014.
[6] “Biomedical monitor for smartphone,” Dec. 2012.
[7] A. Guidi, A. Lanata, G. Valenza, E. P. Scilingo, and P. Baragli, “Validation of smart textile electrodes for electrocardiogram monitoring in free-moving horses,” 2017.
[8] V. Koncar, “1 – Introduction to smart textiles and their applications.,” in *Smart Textiles and their Applications*, 2016, pp. 1–8.
[9] T. Yilmaz, R. Foster, and Y. Hao, “Detecting vital signs with wearable wireless sensors.,” *Sensors (Basel).*., vol. 10, no. 12, pp. 10837–62, 2010.
[10] C. Wang and G. G. Wallace, “Flexible Electrodes and Electrolytes for Energy Storage,” *Electrochim. Acta*, vol. 175, pp. 87–95, Sep. 2015.

[11] D. Miorandi, S. Sicari, F. De Pellegrini, and I. Chlamtac, “Internet of things: Vision, applications and research challenges,” *Ad Hoc Networks*, vol. 10, no. 7, pp. 1497–1516, Sep. 2012.

[12] V. L. Pushparaj, M. M. Shaijumon, A. Kumar, S. Murugesan, L. Ci, R. Vajtai, R. J. Linhardt, O. Nalamasu, and P. M. Ajayan, “Flexible energy storage devices based on nanocomposite paper,” *Proc. Natl. Acad. Sci. U. S. A.*, vol. 104, no. 34, pp. 13574–7, Aug. 2007.

[13] C. J. Brinker and D. Ginger, “Nanotechnology for Sustainability: Energy Conversion, Storage, and Conservation,” in *Nanotechnology Research Directions for Societal Needs in 2020*, Dordrecht: Springer Netherlands, 2011, pp. 261–303.

[14] A. K. Hussein, “Applications of nanotechnology in renewable energies—A comprehensive overview and understanding,” *Renew. Sustain. Energy Rev.*, vol. 42, pp. 460–476, 2015.

[15] G. Zhou, F. Li, H.-M. Cheng, L. Gao, W. P. Tang, J. Y. Xie, J. Zhi, X. M. Ju, C. Scherr, S. Kaskel, C. Wang, L. Hu, C. J. Yu, C. Chuang, T. I. Kim, T. Song, K. Shigeta, S. Kang, C. Dagdeviren, I. Petrov, P. V. Braun, Y. G. Huang, U. Paik, and J. A. Rogers, “Progress in flexible lithium batteries and future prospects,” *Energy Environ. Sci.*, vol. 7, no. 4, pp. 1307–1338, Mar. 2014.

[16] X. Sun, P. V. Radovanovic, B. Cui, C. H. Chen, W. W. Ge, V. Gariepy, K. Galoutov, P. Hovington, A. Mauger, H. Groult, and C. M. Julien, “Advances in spinel Li4Ti5O12 anode materials for lithium-ion batteries,” *New J. Chem.*, vol. 39, no. 1, pp. 38–63, Dec. 2015.

[17] L. Wang, Q. Xiao, Z. Li, G. Lei, P. Zhang, and L. Wu, “Synthesis of Li4Ti5O12 fibers as a high-rate electrode material for lithium-ion batteries,” *J. Solid State Electrochem.*, vol. 16, no. 10, pp. 3307–3313, 2012.

[18] T. Ohzuku, A. Ueda, and N. Yamamoto, “Zero-Strain Insertion Material of Li[Li1/3Ti5/3]O4 for Rechargeable Lithium Cells,” *J. Electrochem. Soc.*, vol. 142, no. 5, p. 1431, May 1995.

[19] B. Zhao, R. Ran, M. Liu, and Z. Shao, “A comprehensive review of Li4Ti5O12-based electrodes for lithium-ion batteries: The latest advancements and future perspectives,” *Mater. Sci. Eng. R Reports*, vol. 98, pp. 1–71, 2015.

[20] C. P. Sandhya, B. John, and C. Gouri, “Lithium titanate as anode material for lithium-ion cells: a review,” *Ionics (Kiel)*., vol. 20, no. 5, pp. 601–620, May 2014.

[21] N. Nitta, F. Wu, J. T. Lee, and G. Yushin, “Li-ion battery materials: present and future,” *Mater. Today*, vol. 18, no. 5, pp. 252–264, 2015.

[22] T. Yuan, Z. Tan, C. Ma, J. Yang, Z.-F. Ma, and S. Zheng, “Challenges of Spinel Li4Ti5O12 for Lithium-Ion Battery Industrial Applications,” *Adv. Energy Mater.*, p. 1601625, Jan. 2017.

[23] Y. Liu, S. Gorgutsa, C. Santato, and M. Skorobogatiy, “Flexible, Solid Electrolyte-Based Lithium Battery Composed of LiFePO4 Cathode and Li4Ti5O12 Anode for Applications in Smart Textiles,” *J. Electrochem. Soc.*, 2012.

[24] X. Pu, L. Li, M. Liu, C. Jiang, C. Du, Z. Zhao, W. Hu, and Z. L. Wang, “Wearable Self-
Charging Power Textile Based on Flexible Yarn Supercapacitors and Fabric Nanogenerators,”
Adv. Mater., vol. 28, no. 1, pp. 98–105, Jan. 2016.

[25] X. Wang, K. Jiang, and G. Shen, “Flexible fiber energy storage and integrated devices: recent progress and perspectives,” Mater. Today, vol. 18, no. 5, pp. 265–272, Jun. 2015.

[26] J. G. Kim, D. Shi, M. S. Park, G. Jeong, Y. U. Heo, M. Seo, Y. J. Kim, J. H. Kim, and S. X. Dou, “Controlled Ag-driven superior rate-capability of Li4Ti5O12 anodes for lithium rechargeable batteries,” Nano Res., vol. 6, no. 5, pp. 365–372, 2013.

[27] H. L. Zou, H. F. Xiang, X. Liang, X. Y. Feng, S. Cheng, Y. Jin, and C. H. Chen, “Electrospun Li3.9Cr0.3Ti4.8O12 nanofibers as anode material for high-rate and low-temperature lithium-ion batteries,” J. Alloys Compd., vol. 701, pp. 99–106, 2017.

[28] B. Guo, Y. Li, Y. Yao, Z. Lin, L. Ji, G. Xu, Y. Liang, Q. Shi, and X. Zhang, “Electrospun Li4Ti5O12/C composites for lithium-ion batteries with high rate performance,” Solid State Ionics, vol. 204–205, pp. 61–65, Dec. 2011.

[29] C. Liu, S. Wang, C. Zhang, H. Fu, X. Nan, and Y. Yang, “High power high safety battery with electrospun Li 3 V 2 ( PO 4 ) 3 cathode and Li 4 Ti 5 O 12 anode with 95 % energy efficiency,” Energy Storage Mater., vol. 5, pp. 93–102, 2016.

[30] H. Park, T. Song, H. Han, and U. Paik, “Electrospun Li4Ti5O12 nanofibers sheathed with conductive TiN/TiOxNy layer as an anode material for high power Li-ion batteries,” J. Power Sources, vol. 244, pp. 726–730, 2013.

[31] L. Rayleigh, “On the Capillary Phenomena of Jets,” Proc. R. Soc. London, vol. 29, no. 196–199, pp. 71–97, 1879.

[32] C. Wang, H. Koh, S. Ramakrishna, and S. Liao, Electrospinning for Tissue Regeneration. 2011.

[33] L. M. Duque Sánchez, L. Rodríguez, and M. López, “ELECTROSPINNING: LA ERA DE LAS NANOFIBRAS,” Rev. Iberoam. Polímeros Vol. Iber. Polímeros, vol. 14, no. 141, pp. 10–27, 2014.

[34] J. V. Patil, S. S. Mali, A. S. Kamble, C. K. Hong, J. H. Kim, and P. S. Patil, “Electrospinning: A versatile technique for making of 1D growth of nanostructured nanofibers and its applications: an experimental approach,” Appl. Surf. Sci., 2017.

[35] Z.-M. Huang, Y.-Z. Zhang, M. Kotaki, and S. Ramakrishna, “A review on polymer nanofibers by electrospinning and their applications in nanocomposites,” Compos. Sci. Technol., vol. 63, no. 15, pp. 2223–2253, Nov. 2003.

[36] Y. Wu, M. V Reddy, B. V. R. Chowdari, and S. Ramakrishna, “Electrochemical studies on electrospun Li(Li1/3Ti5/3)O4 grains as an anode for Li-ion batteries,” Electrochim. Acta, vol. 67, pp. 33–40, 2012.

[37] J.-F. Agassant, P. Avenas, P. J. Carreau, B. Vergnes, M. Vincent, J.-F. Agassant, P. Avenas, P. J. Carreau, B. Vergnes, and M. Vincent, “2 – Rheological Behavior of Molten Polymers,” in Polymer Processing, 2017, pp. 33–175.

[38] A. K. Sharma, A. K. Tiwari, and A. R. Dixit, “Rheological behaviour of nanofluids: A review,”
Renew. Sustain. Energy Rev., vol. 53, pp. 779–791, Jan. 2016.

[39] N. Okutan, P. Terzi, and F. Altay, “Affecting parameters on electrospinning process and characterization of electrospun gelatin nanofibers,” Food Hydrocoll., vol. 39, pp. 19–26, 2014.

[40] M. Chowdhury and G. Stylios, “Effect of Experimental Parameters on the Morphology of Electrospun Nylon 6 fibres,” Int. J. Basic Appl. Sci. IJBAS-IJENS, vol. 10, pp. 6–70.

[41] X.-Y. Wang, Y.-J. Li, C. Xu, L. Kong, and L. Li, “Synthesis and characterization of Li4Ti5O12 via a hydrolysis process from TiCl4 aqueous solution,” Rare Met., vol. 33, no. 4, pp. 459–465, 2014.

[42] M. Parhizkar, P. J. T. Reardon, J. C. Knowles, R. J. Browning, E. Stride, R. B. Pedley, T. Grego, and M. Edirisinghe, “Performance of novel high throughput multi electrospray systems for forming of polymeric micro/nanoparticles,” Mater. Des., vol. 126, pp. 73–84, 2017.

[43] D. Li and Y. Xia, “Electrospinning of Nanofibers: Reinventing the Wheel?,” Adv. Mater., vol. 16, no. 14, pp. 1151–1170, Jul. 2004.

[44] J. Pelipenko, J. Kristl, B. Janković, S. Baumgartner, and P. Kocbek, “The impact of relative humidity during electrospinning on the morphology and mechanical properties of nanofibers,” Int. J. Pharm., vol. 456, no. 1, pp. 125–134, 2013.

[45] T. Liang, M. Parhizkar, M. Edirisinghe, and S. Mahalingam, “Effect of humidity on the generation and control of the morphology of honeycomb-like polymeric structures by electrospinning,” Eur. Polym. J., vol. 61, pp. 72–82, 2014.

[46] B. Guo, Y. Li, Y. Yao, Z. Lin, L. Ji, G. Xu, Y. Liang, Q. Shi, and X. Zhang, “Electrospun Li4Ti5O12/C composites for lithium-ion batteries with high rate performance,” Solid State Ionics, vol. 204–205, pp. 61–65, 2011.

[47] Y. Zhang, Y. Zhang, L. Huang, Z. Zhou, J. Wang, H. Liu, and H. Wu, “Hierarchical carambola-like Li4Ti5O12-TiO2 composites as advanced anode materials for lithium-ion batteries,” Electrochim. Acta, vol. 195, pp. 124–133, 2016.