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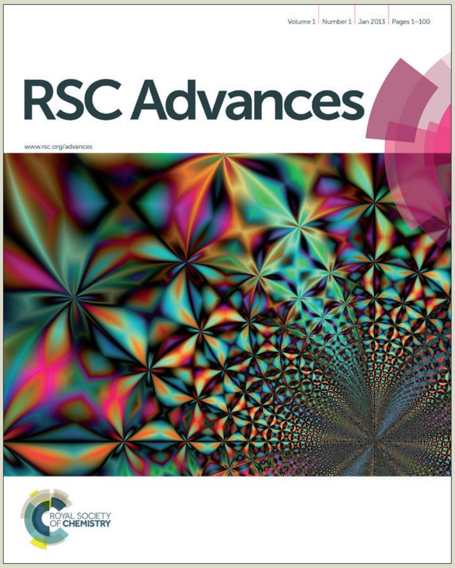

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Fabrication and Characterization of Continuous Silver Nanofiber/Polyvinylpyrrolidone (AgNF/PVP) Core–Shell Nanofibers Using Coaxial Electrospinning Process.

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In the present study, continuous and uniform Core–shell silver nanofiber/poly (vinyl) pyrrolidone (AgNF/PVP) nanofibers have been successfully fabricated via an efficient coaxial-spinneret electrospinning method with vertical configuration using PVP and $AgNO_3$ as a precursor solution. PVP polymer primarily used as a guide and provided an appropriate viscoelastic property by surrounding the $AgNO_3$ (core) solution to fabricate an aligned AgNF/PVP core-shell nanofibers. A series of $AgNO_3$ concentration were prepared by fixing concentration of PVP as shell fluid. The $AgNO_3$ concentration had a significant influence on the formation of continuous and uniform AgNF/PVP core-shell structure of nanofibers as well applied voltage had an effect on the formation of the compound stable Taylor cone. The (AgNF/PVP) core-shell nanofibers were formed via the stretching of the co-electrospinning jet and reduction temperature with in an appropriate time induced the silver nanofiber to be well aligned along the axis of the PVP-temple electropsun fiber. The structure and properties of the thus obtained core-shell nanofibers have been investigated thoroughly through optical microscopy (OM), transmission electron microscopy (TEM), Focused Ion Beam (FIB), X-ray diffraction (XRD) and selected area electron diffraction (SAED). Energy Dispersive spectroscopy (EDS) and X-ray photoelectron

spectroscopy (XPS) have also been employed to analyze the elemental composition of the core–shell nanofiber surface. On the other hand, UV-Vis spectrophotometry has been used to test the reduction of silver ions into metal silver. Moreover, electrical measurements were performed on (AgNF/PVP) core-shell nanofibers, which indicated the core-shell nanofiber became insulating due to embedded of highly conductive silver nanofibers by insulating PVP shell. Hence, coaxial electrospinning is a convenient and cost effective process for fabricating of continuous and uniform (metal/polymer) core-shell nanostructure fibers.

Introduction

The demand for novel nanostructures in different fields such as telecommunications, biotechnology and computing has increased enormously and has engaged the interests of scientists and engineers to create new and novel structures of nanofibers and efficient processes for producing them.[1, 2] Nanostructures of various morphologies, including nanorods, nanowires, nanofibers, nanotubes, nanocables and nano patterned materials have been synthesized using different methods. For example, template synthesis,[3, 4] self-assembly,[5] phase separation,[6] lithography,[7] and electrostatic spinning.[8]

Electrospinning is a simple and well-established methods for the preparation of ultrafine 1D-nanofibers with diameters ranging from the nano to micrometric scale.[9] The decrease of diameter into the nanometer scale provides increase to a set of favorable characteristics, including the increase of surface to volume ratio which will enhance the strength of the fibers.[10, 11] As compared to other nanofibers fabrication methods, electrospinning has gained an attention as this technique is more versatile, productive, cost-effective, and less demanding for processing conditions.[12, 13]

In recent years, several modifications have been made in the single electrospinning process in order to improve the functionality and enhance the quality of the resulting nanofiber structures. One such modification that holds great promise and has gained much attention in a variety of applications is fabrication of core-shell component nanostructure fibers using coaxial electrospinning process.[13-16] Coaxial electrospinning is an effective and simple method to fabricate metal -polymer nanofibers with uniform core-shell structures (micro/nanocapsules and coaxial fibers).[17] In this process, two unlike materials are delivered independently with a coaxial capillary and drawn to generate nanofibers in core-shell configuration. Increasing need for the fabrication of two material based nanostructures in which one is surrounded by the other at the nano or micro level, shows potential for a wide range of applications; such as: - separating an unstable component and minimizing its chances of decomposition under a reactive environment, supporting a material to improve its mechanical properties and serving as scaffold for engineering tissues in which a more biocompatible material is surrounded a less biocompatible materials.[18-21] Intense research is being carried out on 1D micro and nanostructures of inorganic/ organic core –shell composite fibers via different synthesis methods for several potential applications.[22, 23] For example, Several publications have been reported on core-shell composite nanofibers, which is made of metal and metal oxide in the core (such as, Au,[24-26] Ni,[27] Si,[28, 29] CuO,[30] and TiO_2,[31]) covered by silica and polymers.[32] Moreover, metal/polymer core-shell nanostructure fibers, such as silver nanowire/poly (vinyl) pyrrolidone (AgNW/PVP)[33] and Copper/Poly (vinyl alcohol) (Cu/PVA)[22] nanocables have been fabricated via single-spinneret electrospinning method. Similarly, different Silver (Ag)/poly (vinyl) pyrrolidone (PVP) electrospun nanofibers with different patterns (nonwoven, aligned, and crossed) and palladium/poly-(L-lactide) (Pd/PLA) core–shell nanostructured fibers have been fabricated via coaxial electrospinning.[11, 34] Also, a variety of synthesis methods have been employed to fabricate 1D silver nanostructures with polymers because of their unique properties,[35-39] which includes, hydrothermal method,[40] photo-reduction processes,[41] Sol-gel synthesis[42] and seed-mediated growth.[43] However, those methods have certain limitations such as; difficult to control the shape and size of product, need complicated synthetic procedure and difficult to achieve continuous production. In addition, the widely used Single electrospinning has a limitation to synthesis continuous metal nanofiber/polymer core-shell nanofibers from a metal salt and polymer solutions (i.e. metal salt and polymer, such as silver nitrate and poly vinyl pyrrolidone).[33] Hence, to the best of our knowledge there is no report on the fabrication of a continuous silver nanofiber/poly (vinyl) pyrrolidone) (AgNF/PVP) core-shell nanofibers using electrospun PVP polymer fiber as a guide and surrounds the core material via co-axial electrospinning process. To overcome those limitations, coaxial electrospinning process can be used to fabricate continuous and uniform AgNF/PVP core-shell nanofibers from inexpensive materials (i.e. silver nitrate and poly vinyl pyrrolidone).

Herein we describe the fabrication and characterization of continuous and uniform AgNF/PVP core-shell nanofibers from $AgNO_3$ (which is used as the core solution and a precursor of AgNF) and PVP (used as the shell) via coaxial electrospinning process. The PVP polymer primarily was used as a guide and provided an appropriate viscoelastic property by surrounds the core material to fabricate an aligned AgNF/PVP core-shell nanofibers. The effect of different $AgNO_3$ concentration was investigated on AgNF/PVP core-shell nanofibers formation by maintaining reduction temperature and time constant. The morphology, uniformity and dimensions of the core–shell AgNF/PVP nanofibers were performed by using optical microscopy (OM), Focused Ion Beam (FIB). The structure of the AgNF/PVP core-shell nanofibers was clearly identified by x-ray diffraction (XRD) and selected-area electron diffraction (SAED) pattern. Transmission electron microscopy (TEM) were characterized the size of the final and their morphology of core-shell nanofibers. On the other hand, UV-Vis spectrophotometry were used to test the reduction of Ag (I) ions into Ag (0) metals nanostructure. Moreover, the elemental composition of AgNF/PVP core-shell nanofibers surface were characterized by using Energy Dispersive spectroscopy (EDS) and X-ray photoelectron spectroscopy (XPS).

Experimental section

Materials

Poly (vinyl) pyrrolidone K-90 (PVP, Mw =1,300,000) was purchased from Sigma-Aldrich (USA). Silver nitrate ($AgNO_3$, >99%) was purchased from Sigma-Aldrich (UK). Ethanol (CH_3CH_2OH, >99.5%) was analytical grade and purchased from Aldrich Co. Ltd (USA). Water was double distilled (deionized water) before use. All chemicals were used without any further purification.

Methods

Preparation of Precursor Solution

The PVP powder and $AgNO_3$ salts were used to synthesis nanofibers precursors. Briefly, 1 g of PVP powder was dissolved in 11.5 g of ethanol (8% (w/w) of PVP) and vigorously stirred using magnetic stirrer until clear solution was obtained. Similarly, different concentration of $AgNO_3$ (20%, 30% and 40% (w/w) were mixed with deionized (DI) water and stirred gently using magnetic stirrer until a homogeneous solution was obtained.

Co-axial electrospinning Process

The process of coaxial electrospinning was much attractive, simple and effective top-down way to prepare core– shell ultrathin fibers with lengths of up to several centimeters on a large scale compared to other methods.[44] The general experimental set up adopted by most researchers is relatively similar to single electrospinning.[13] A change is made in the spinneret by inserting a inner (smaller) capillary that fits concentrically inside the outer (bigger) capillary to make coaxial configuration. The inner needle is attached to the reservoir containing the **core** fluids (a non-viscous liquid) and the outer needle is connected to the one holding the shell fluids (viscous liquid) form a core–shell compound jet in an electric field, which then solidifies to form core–shell nanofibers.[45] The solutions feeding rates are controlled using metering pumps.[46] The arrangement is vertical as illustrated in (**Fig. 1**).

Core-shell AgNF/PVP nanofibers were fabricated from the above precursors solution via co-axial electrospinning process as displayed in the (**Fig. 1**). Briefly, in the first step, 8% (w/w) PVP solution was added to a 10 mL syringe, which was connected to the outer capillary with a size of N18 (inner diameter: 0.96 mm, outer diameter:1.26 mm) and 20% (w/w) solutions of $AgNO_3$ in deionized water was filled with another 10 mL syringe connected to the inner capillary with a size of N25 (inner diameter: 0.26 mm, outer diameter: 0.5 mm). Secondly, these two liquids were fed at a fixed rate through syringe pumps (YSP-101, YSP series syringe pump). All parameters were identical with PVP solution flow rate of 0.4 mL/h, $AgNO_3$ solution flow rate of 0.1 mL/h and the fibers collected on a glass slide at a tip-to-collector distance of 20 cm under the same ambient Conditions (25±2°c) and relative humidity (20~25%). Then, in the range of 8-10 kv voltage was supplied and the electrospinning solution is drawn out from spinneret and forms a compound Taylor cone with a core-shell nanostructure of co-axial jet. The fibers with PVP shell and sliver ions core were produced in solid form, which can be collected on the collector.

Thirdly, the collected solid nanofibers were annealed with hot-stage at 180°c for 1h. Later on, the fabricated nanofibers were stored in dry environments and used for the further characterization.

Characterization

The morphology of AgNF/PVP core-shell electrospun (ES) nanofiber mats were analyzed using an optical microscopy (OM) (Olympus BX51 system microscope (x1000) fitted with a DP-12 digital camera), Dual Beam Focused Ion Beam (FIB) (FEI Quanta 3D FEG) with an Energy Dispersive Spectrometer (EDS). Transmission electron microscopy (TEM) (JEOL JEM-1200EX II (JEOL, Tokyo, Japan) was used for the detailed morphological investigation of the core–shell AgNF/PVP ES nanofiber mats as well as measurement of core and shell thicknesses. The elemental composition on the surface of nanofibers were cheracterized by X-ray photoelectron spectroscopy (XPS) (VG ESCA spectrometer system with Al Ka source) and Energy Dispersive Spectrometer (EDS). The UV-visible spectra of the AgNF/PVP core-shell ES nanofiber mats were recorded on Jasco V-670 UV−visible−near-infrared (UV−vis−NIR) spectrophotometer over the wavelength range from 200 to 800 nm. The structural properties of the obtained samples were further investigated by X-ray diffraction (XRD) on (a Bruker D8 DISCOVER SSS Multi-Function High Power XRD) X-ray Diffractometer using Ni-filtered Cu-Kα radiation (wavelength 1.54 Å) with a scanning rate of 1° min^{-1} at 40 kV and 200 mA from 2θ range of 20°-80°. Finally, electrical measurements were performed using a standard four-probe technique (Keith Link Technology, Taiwan).

Results and discussion

The main goal of this study was to fabricate and characterize continuous and uniform AgNF/PVP core-shell nanofibers via co-axial electrospinning process from inexpensive precursors of silver nitrate and polyvinylpyrrolidone solutions. The $AgNO_3$ solution does not form a continuous fiber like structures up on electrospinning due to their low molecular weight and lack of required viscoelastic properties[13] as shown in **Fig. 2A.** But obviously, fiber like structures can be obtained by co-electrospinning with PVP as shown in **Fig. 2B**. Since, the viscosity of the PVP (shell) solution was required to be such that the viscous stress imparted by it on the $AgNO_3$ (core) solution was much enough to overcome the interfacial tension between the two solutions and allows the formation of a compound Taylor cone and a jet from the latter.[47] Accordingly, viscosity of the PVP shell solution was critical and the shell PVP-polymer could be electrospinnable by itself to lend to core-shell structure formation.[46] The particular interest and advantage of co-axial electrospinning method was that nonelectrospinable material like $AgNO_3$ solution can be forced into 1D arrangements using electrospinable materials as shell like PVP polymer solutions. Hence, coaxial electrospinning technique should be very versatile for the processing of a wide variety of systems different from those presented here and will certainly foster new material design.

In order to fabricate continuous and uniform core –shell nanofiber, firstly we Optimized stable and uniform Taylor cone during co-axial electrospinning process, since stabilized compound Taylor cone and the initial jet are the requirements for the co-axial electrospinning.[48] Hence, the effects of applied voltage and different concentration of $AgNO_3$ were investigated. The effects of applied voltage (from 5-14 kV) was displayed in the **Fig. 3.** The result from **Fig. 3A, D and E** shows unstable Taylor cones, can cause the formation of irregular core-sheath structures and in separate fibers from the two solutions. Taylor cone will break when work voltage is too high (i.e. applied voltage > 10 kV) and in the other hand, the solution will drop on the collector when voltage is to low (i.e. applied voltage < 8kV). We observed the more stable Taylor cones for the applied voltage in the range between 8-10 kV, as shown in **Fig. 3B** & **C**.

Similarly, the effects of $AgNO_3$ were investigated by using various concentration of $AgNO_3$ (i.e., 20%, 30% and 40% (w/w)). Among the used concentrations, only 20% (w/w) $AgNO_3$ revealed good continuity and with uniform core-sheath structure as displayed in **Fig. 4A**. In contrary, as the concentration of $AgNO_3$ increased, nanofiber with poor continuity and uniformity was seen as shown in **Fig. 4B** & **C.** This is may be due to high conductive properties of $AgNO_3$ after dissociating to Ag^+ and NO_3^-, which bring about devastating effect on the morphology of core-shell nanofibers such as non-uniform and break of core-nanofibers.[49] Hence, in this study 20% (w/w) of $AgNO_3$ concentration as the basic matrix of core solution and 8-10 kV of applied voltage were selected throughout the experiment. Therefore, all the results and discussion in this paper were based on the above optimization.

The morphologies of was summarized in **Fig. 5**. Optical microscopes were used to observe the formation and size of AgNF/PVP ES nanofiber mats for initial examination. The optical microscope result showed that the nanofibers has relatively uniform size as displayed in the **Fig. 5A**. Similarly, FIB image result of AgNF/PVP core-shell ES nanofiber mats shows relatively uniform and smooth surface that found on the glass slide as illustrated in the **Fig. 5B.** This results revealed that, silver nanofiber well embedded in PVP polymer matrix. The TEM image of the annealed coaxial fibers clearly proved the AgNF/PVP core-shell ES nanofiber mats has a core-shell morphology as shown in **Fig. 5C** with the diameter of 480 nm and 110 nm for outer and core phase respectively. The corresponding selected-area electron diffraction pattern in **Fig. 5D**; revealed that, crystalline characteristics for the silver-metal, consistent with the distinct diffraction rings of the (111), (200), (220), and (311) planes. Moreover, the SAED image of

AgNF/PVP core-shell ES nanofiber mats further revealed that the d-spacing of 0.24 nm was attributed to the (111) plane of Ag, which is similar to XRD-data in **Fig. 6B**.

The structural properties of the pure PVP and AgNF/PVP core-shell ES nanofiber mats were also investigated by XRD as displayed in **Fig. 6A** & **B**, respectively. The XRD pattern of pure PVP ES nanofiber mats have revealed a broad bands located at $2\theta = 22^{\circ}$ those could clearly indicate the amorphous nature of the PVP [50] (as illustrated in **Fig. 5A**). Similarly, **Fig. 6B;** shows an XRD pattern of the AgNF/PVP core-shell ES nanofiber mats with the diffraction peaks at 2θ value 38.12°, 44.23°, 64.44°, and 77.40° indexed to the (111), (200), (220), and (311) planes of cubic crystal structure of silver with JCPDS Card (File No JCPDS # 04-0783). The XRD data confirms the presence of Ag in the AgNF/PVP core-shell ES nanofiber mats.

Fig. 6C & **D** Shows the UV-VIS-NIR spectrum of pure PVP and AgNF/PVP core-shell ES nanofiber mats, respectively. UV-VIS-NIR spectrum of Pure PVP ES nanofiber mats shows a weak light absorption below 300 nm and a strong absorption in the higher wavelength range, whereas, Silver nanostructures with different shapes exhibit surface Plasmon resonance (SPR) bands at different wavelengths. The three SPR peaks (at ~350,~380, and ~410 nm) belong to the optical signatures of silver which clearly seen on the UV-VIS-NIR spectrum of AgNF/PVP core-shell ES nanofiber mats, which proves the reduction of Ag (I) ions into Ag (0) metal nanostructures.[51, 52]

The elemental composition on the surface of AgNF/PVP core-shell ES nanofiber mats were also cherecterized by XPS and EDS as showed in the **Fig. 7A** & **B** respectively. From XPS spectrum, only peaks related to C, N, and O appeared in the spectrum. Very interestingly, the XPS data show that no silver was present on the surface of AgNF/PVP core- shell ES nanofiber mats. Since, elemental analysis of XPS can detect in the uppermost~100 Å in depth of an analyzed specimen,[38] it is reasonable to draw a conclusion that the absence of Ag signal in the PVP shell surface was at least deeper than 10 nm. The above results show that the formed AgNF/PVP core-shell ES nanofiber mats is corresponding with the ideal nanofiber model (the inset of **Fig. 1**).

Fig. 7B Shows EDS spectra for AgNF/PVP core- shell ES nanofiber mats. Only peaks related to C, N, O, Si and Pt appeared in the spectrum. From EDS data, C, N and O peaks should come from PVP polymer on the surface of AgNF/PVP core-shell ES nanofiber mats, whereas Si peaks originate from the slide glass that supports the sample, and Pt is present due to the sputtering step for sample preparation for FIB imaging. EDS data show that no silver was present at the surface of the AgNF/PVP core- shell ES nanofiber mats, which further supports that the AgNF was successfully incorporated into the PVP polymer matrix. By combining XPS and EDS data, it could be concluded that the AgNFs were completely embedded in the PVP nanofibers, at least deeper than 10nm, which was the probe depth for XPS. This implies that we have successfully fabricated AgNF/PVP core- shell nanofibers with the AgNF embedded inside as the core and PVP outside as the shell, as shown in the TEM image of **Fig. 5C.**

Furthermore, electrical measurements were performed on AgNF/PVP core- shell ES nanofiber mats, which indicated the AgNF/PVP core- shell ES nanofiber mats became insulating due to embedded of highly conductive AgNF by insulating PVP shell. To summerize, coaxial electrospinning is an attractive, simple and effective top-down way to fabricate continuous and uniform AgNF/PVP core-shell nanofibers compared with other methods. Therefore, this set up is a promising method to fabricate other continuous and uniform metal/polymer core– shell nanostructure fibers with lengths of up to several centimeters on a large scale for several applications such as electronics circuit, optoelectronics, and sensing devices.

Conclusions

In the present study, continuous and uniform AgNF/PVP core–shell nanofibers have been successfully fabricated via an efficient coaxial-spinneret electrospinning method with vertical configuration using PVP and $AgNO_3$ as a precursor solution. The Silver nanofibers to be well aligned along the axis of the PVP electropsun fibers by the stretching of the electrospinning jet and reduction temperature with in an appropriate time. The PVP polymer primarily used as a guide and provided an appropriate viscoelastic property by surrounding the non-viscous AgNO3 (core) solution to fabricate an aligned AgNF/PVP core–shell nanofibers. The formation of silver nanofiber in AgNF/PVP core–shell nanofibers is completely confirmed by XRD and UV-Vis characterization, and the FIB images have shown that uniform and smooth PVP nanofibers on the surface. By combining XRD and EDS results, it is also possible to confirm that the AgNFs were embedded in the PVP matrix, forming a core–shell nanofiber structures. The TEM images clearly assess and display a continuous and uniform core – shell structure of AgNF/PVP nanofibers. Furthermore, electrical measurements were performed on AgNF/PVP core–shell nanofiber mats, which indicated the AgNF/PVP core–shell nanofiber mats became insulating due to embedded of highly conductive Ag-nanofibers by insulating PVP nanofibers shell. To summarize, coaxial electrospinning is an attractive, simple and cost effective top-down way to fabricate continuous and uniform AgNF/PVP core-shell nanofibers compared with other methods. Therefore, this set up is a promising method to fabricate other continuous and uniform metal nanofiber/polymer core– shell nanostructure fibers with lengths of up to several centimeters on a large scale for several applications such as electronics circuit, optoelectronics, and sensing devices.

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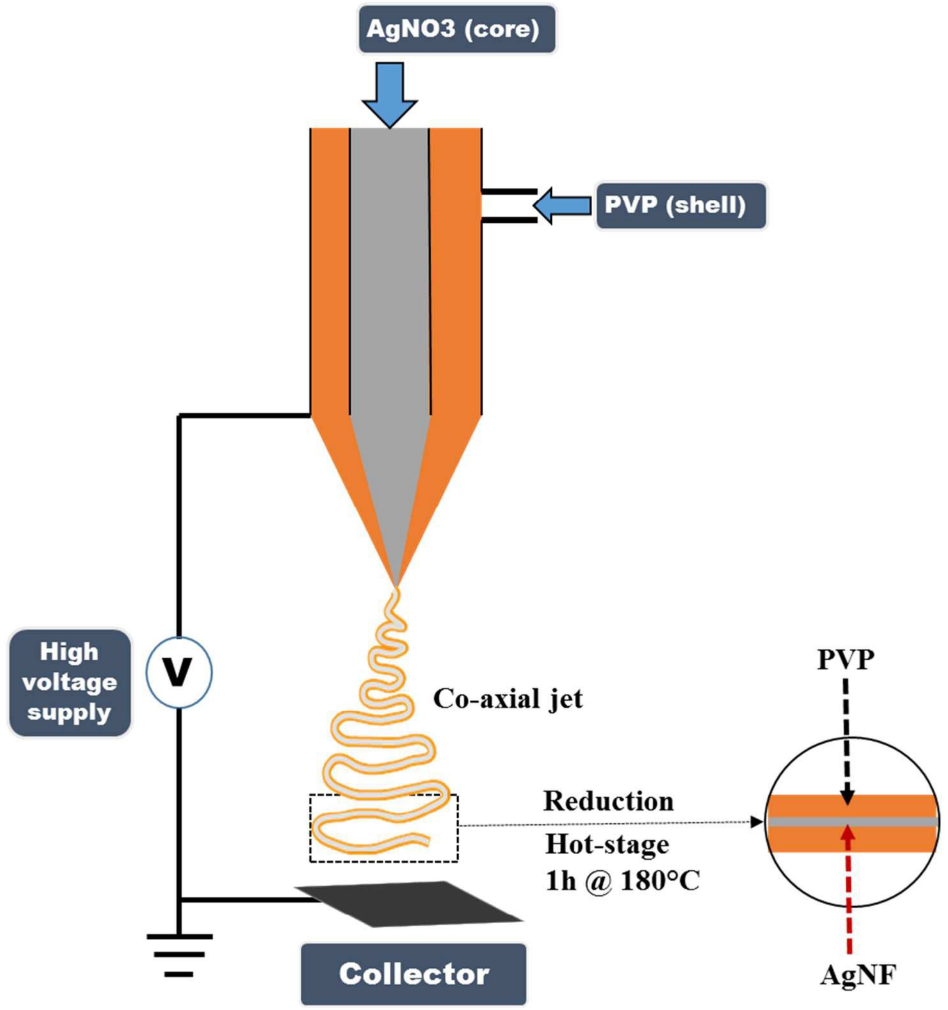

Fig. 1 Schematic illustration of AgNF/PVP nanofibers formation with core/sheath structures.

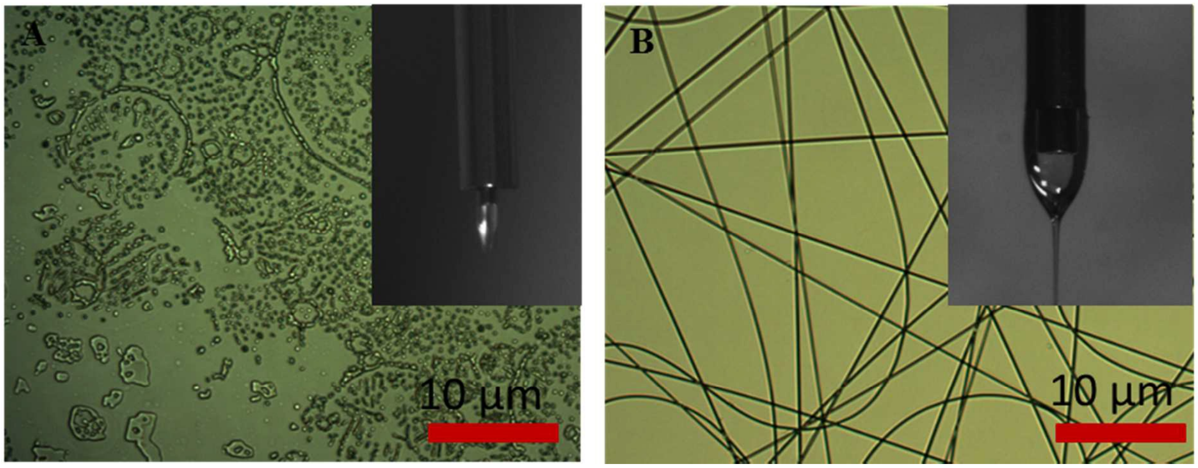

Fig. 2 Optical microscope image with Stable Taylor cone: (A) 20% (w/w) $AgNO_3$ in DI water on a glass slide (B) Coaxial electropsun of 20% (w/w) $AgNO_3$ (core) in DI water and 8% (w/v) PVP (Shell) in ethanol.

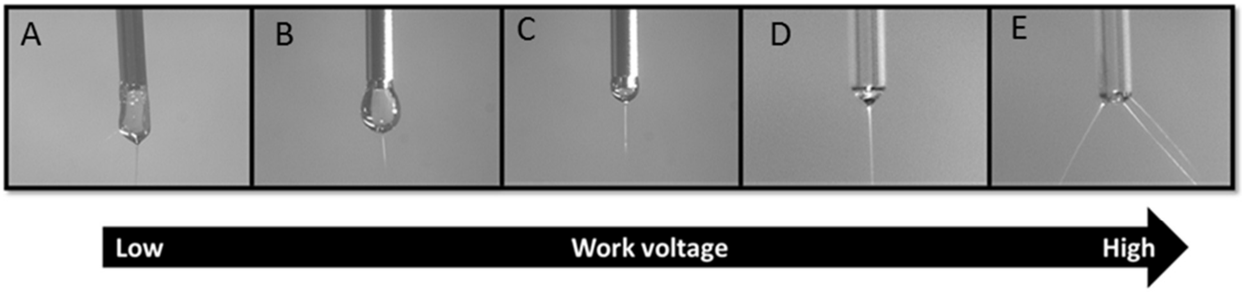

Fig. 3 The shape of Taylor cone is observed by self-assembled camera with a fixed flow rate of core (0.1mL/h) and shell (0.4mL/h) solution @ (A) 5 kV, (B) 8 kV, (C) 10 kV, (D) 12 kV & (E) 14 kV.

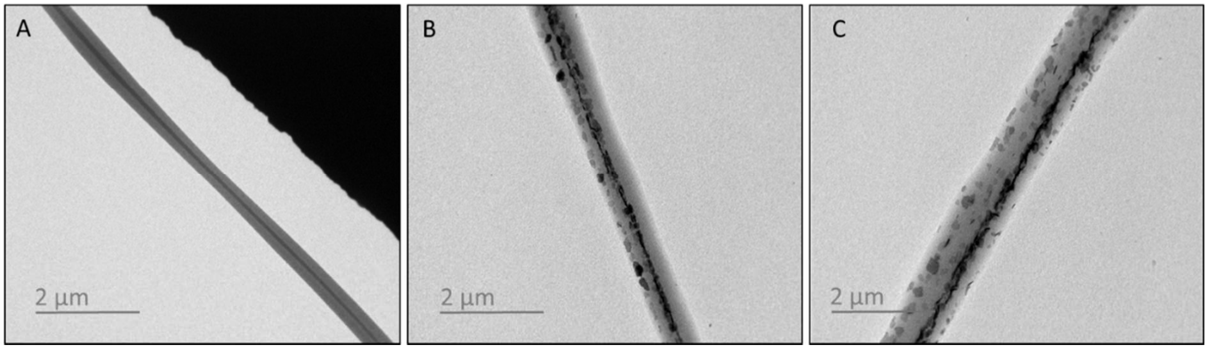

Fig. 4 TEM image of annealed (180°c/1h) AgNF/PVP core-shell ES nanofiber mats of 8% (w/w) PVP with (A) 20%, (B) 30 % and (C) 40 % (w/w) of $AgNO_3$ solution respectively.

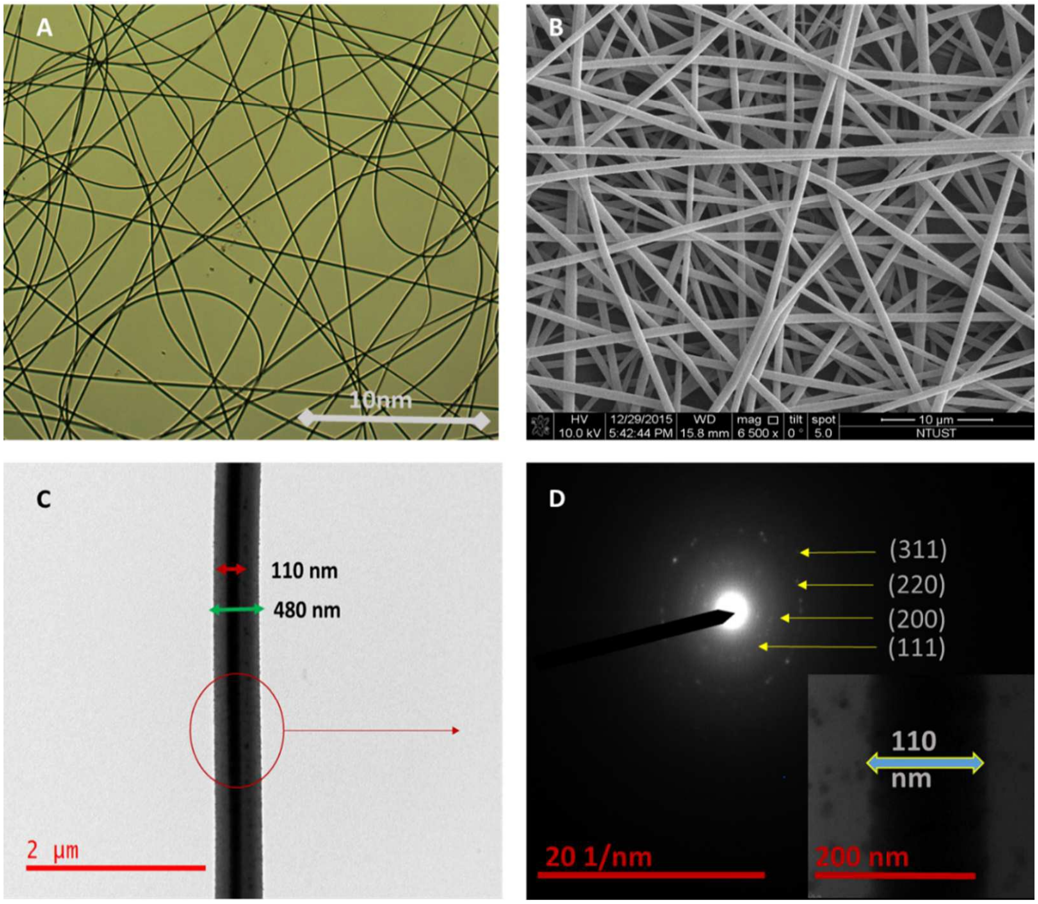

Fig. 5 The Morphology of AgNF/PVP nanofibers from 20% (w/v) $AgNO_3$ and 8% (w/w) PVP at a gap distance of 20 cm with an applied voltage of 8-10 kV (A) Optical microscope (OM), (B) FIB, (C) TEM & (D) Selected-area electron diffraction (SAED)

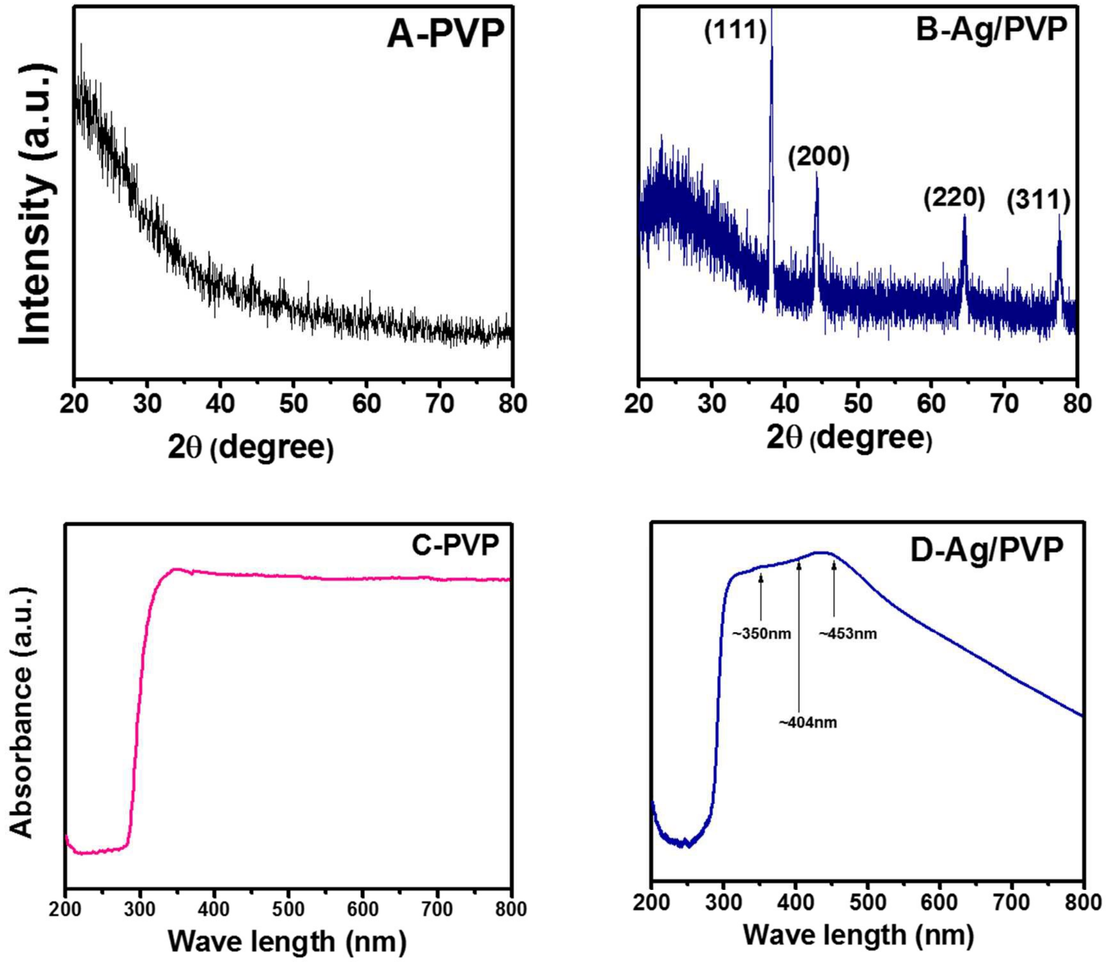

Fig. 6. XRD and UV-VIS-NIR spectrum of PVP and AgNF/PVP core-shell ES nanofiber mats @180°c for 1h. (A) XRD pattern of pure PVP, (B) XRD pattern of AgNF/PVP, (C) UV-VIS-NIR spectrum of pure PVP & (D) UV-VIS-NIR spectrum AgNF/PVP

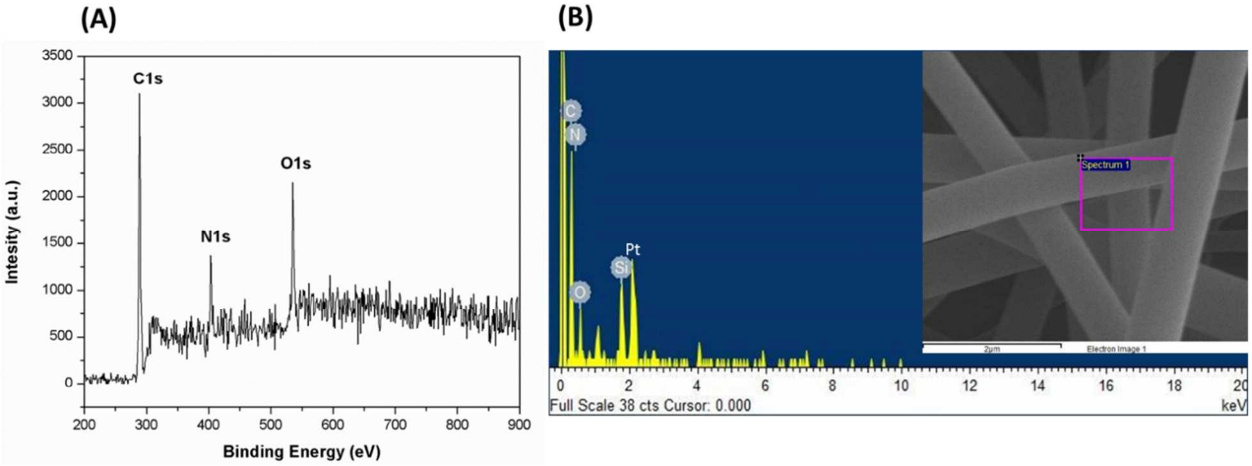

Fig. 7 Shows the elemental composition on the surface of AgNF/PVP core- shell ES nanofiber mats (A) XPS wide energy survey spectrum and (B) EDS spectrum.

Graphical Abstract

Core–shell silver nanofiber/polyvinylpyrrolidone (AgNF/PVP) nanofibers have been successfully fabricated via an efficient coaxial-spinneret electrospinning method with vertical configuration using PVP and $AgNO_3$ as a precursor solution.