Production and Characterization of

Electrospun Polymer Nanofibers

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ABSTRACT

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Electrospinning is a process of generating polymer fibers by accelerating a polymer solution through an electric field. The polymer solution is released at a designated rate through a syringe; once the droplet enters the high voltage region, it whips throughout the chamber, landing upon a grounded collector. This procedure results in fibers with a range of diameter from several nanometers to a few micrometers. These fibers can be used in a variety of applications, including drug delivery, filter media, material substrates, optical media, tissue scaffolds, and wound dressing.

For my senior thesis, I established the most successful method of creating nanofibers and documented the qualities and characteristics of these fibers using optical microscopy. I investigated creating polymer fibers using different solutes and experimented with gold nano-particles additives. In my future experiments, I want to generate nanofibers with other additives such as carbon and quantum dots and measure the physical characteristics, both optically and electrically.

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BACKGROUND:

Electrospinning is a process of generating polymer fibers by accelerating a polymer solution through an electric field.¹ These fibers are threadlike structures that consistently range from a few micrometers to several nanometers in diameter. It is one of the most straightforward and economically sound methods of producing nanomaterials.²

Nanotechnology is an emerging technology concerning relevant processes that occur on the nanoscale.² Fiber production on this scale is of interest because of their small size and the high surface to volume ratio. Nano fibers with volume equal to that of fibers on the millimeter scale have surface areas several factors of ten greater.³ Because these fibers have nanoscale surface characteristics, the modes of interaction with other materials differ greatly from larger scale objects. The molecular structure of these fibers are highly oriented with few defects, approaching the theoretical maximum strength of these fibers.⁴ Through the perfection of the creation of these nanoscale units, we are given the possibility to "design and create new materials with unprecedented flexibility and improvements in their physical properties."⁴

Because of these novel characteristics, these fibers can be applied in a variety of fields, including but not limited to: drug delivery, filter media, material substrates, optical media, tissue scaffolds, and wound dressing.

POLYMERS:

A polymer is a large molecule consisting of multiple repeated structural units, or monomers. These monomers unfold and polymerize, or connect into a long chain of connected monomers. This polymer can vary in size, depending on the number of repeated monomers.

In order to create the polymer solution, a polymer powder is combined with a solvent. The solvent can be varied, but for the purpose of our experiment, we investigated one specific polymer with multiple solvents, polyethylene oxide, a polyether compound.

SOLVENTS:

For the purpose of this experiment, four solvents were used: toluene, chloroform, water, and methanol. Here, the volatility, dielectric constant, solution conductivity, and surface tension of the solvents come into play.⁴ Adding polyethylene oxide to these solutions may cause some changes to the nature of these characteristics. Due to the properties of these liquids, there were varying levels of success with dissolving the polymer powder into the solvent. In toluene, the polymer only dissolves when heat is applied, and the mixture becomes heterogeneous once heat is removed. With chloroform or water, the solution remains homogenous, yet slightly viscous. The methanol solution also remains homogenous and is fluid.

SOLUTION ADDITIVES:

Carbon:

The element carbon, symbol C and atomic number 6, is nonmetallic and tetravalent: it has four electrons available to form covalent bonds. A powder form is used in black printer ink. Carbon particles were added to a polymer solution to attempt to create electrically conductive nanofibers.

Quantum Dots:

Quantum dots (QD) are nano-sized semiconductors.⁵ Because these dots are on the nano-scale, quantum mechanical effects allow for the decoupling of certain material properties. For example, thermal and electrical conductivity are inherently coupled in all known natural semiconductors.⁵ QDs have high electrical conductivity with relatively low thermal activity. This decoupling leads to an ideal semiconductor, holding many advantages over semiconductors on the macro-scale.

QDs display very interesting optical properties. These dots absorb a wide range of wavelengths of light, yet they only emit a very narrow range of wavelengths. Due to the quantum confinement effect, the wavelengths that a QD emits correspond to its size. Lower energies are emitted from larger dots, while higher energies are emitted from smaller dots.⁵ QDs were added to a polymer solution to create a fiber with an isolated QD. Then, this QD fiber could be used as a "point" light source for optics experiments.

APPARATUS:

Our apparatus consists of a syringe pump, a 10mL syringe connected to a tube and a 35μ L syringe, a grounded collector, and high-voltage DC-power supply. The polymer solution is loaded into the 10 mL syringe, connected to the tube that connects to the 35μ L syringe with a 0.321mm diameter needle tip. The 10mL syringe is filled with the polymer solution, which fills the tube and small syringe and is dispensed from the needle. The 10mL syringe is placed into a syringe pump. This pump applies pressure on the syringe, releasing solution at a designated rate. The needle of the 35μ L syringe is placed at the top of a chamber, encased by a plexiglass tube to prevent any outside interference. High voltage is to the needle; the collecting base at the bottom of the chamber is grounded. The solution is released from the needle and travels through the chamber across the high voltage area to collect on the grounded base. The grounded base contains a small SEM stub, used to easily observe the collected samples.

LEAVING THE SYRINGE:

When no voltage is applied, the solution forms a droplet when leaving the tip of the syringe. However, when voltage is applied across the tip of the needle to the grounded collector, the electrostatic force caused by the high voltage applied overpowers the surface tension of the droplet. The droplet is pulled into a thin strand from the tip of the needle, known as a Taylor Cone.²

A Taylor Cone is governed by the following formula:

$$V_{C}^{2} = \frac{4H^{2}}{L^{2}} \left[\ln \left(\frac{2L}{R} \right) - \frac{3}{2} \right] \cdot [1.3\pi \cdot RT] \cdot [0.09]$$

where V_c is the critical voltage, H is the distance between the needle and the grounded collector, L is the length of the small syringe, R is the radius of the small syringe, T is the surface tension, and the factor of 0.09 is the conversion to kV.³

At critical voltage, the polymer solution leaves the needle, and instead of collecting in a droplet, it forms roughly a 50 degree angle with the tip of the needle.³ Here, the electric force is at its greatest, and the material is pulled from the needle into a thin strand. This can easily be observed using a laser, pointed at the tip of the needle.

The thin strand of polymer solution remains straight at the 50 degree angle for a short distance (~3cm). Then, the strand enters what is known as the whipping region. This area is appropriately named –the high-energy interactions between the electric field and the strand causes the strand to quite literally whip throughout the chamber chaotically.¹ Using a laser, one can observe the strand at the critical voltage angle, and then see that the strand is chaotically whipped throughout the chamber. It is easy to spot the whipping strand close to the tip of the needle; however, once the strand travels further towards the grounded collector base, it is growing smaller in size and is not as easily observed. This process of jet thinning can be described in two stages: the thinning of the straight jet and the thinning due to the whipping region.⁴

APPARATUS:

Our apparatus consists of a syringe pump, a syringe connected to a tube and a smaller syringe with a needle tip, a collector, and high-voltage DC power supply. The 10mL syringe is filled with the polymer solution; this syringe is connected to a tube from Churchill Medical Systems Inc. with approximately 1.7mL volume. The tube connects the 10mL syringe to a 35µL syringe. This smaller syringe is a Micro-Fine IV Needle, produced by Insulin Syringe Lo-Dose. The needle at the tip of the syringe is 12.7mm long and 0.321mm in diameter. This needle releases a small amount of material at a constant rate, controlled by the syringe pump. This needle is placed at the top of a 6" ID and 1/4" thick plexiglass tube that serves to shield room air currents. Positive high voltage is applied to the needle and the collector is kept at the ground potential. This voltage causes an electric field strong enough that the electrostatic force is greater than the force of surface tension of the droplet; the droplet becomes a stream of material that eventually collects at the grounded plate. The syringe pump is manufactured by Fisher Scientific: Fisher No. 14831200, Model No. 78-0100l, and Serial No. 114679. The high voltage DC power supply is manufactured by Glassman High Voltage Inc.: series EL, Model PS/EL40P01.0, Serial N147446-01KG060316.



Figure 1: A sketch of the electrospinning apparatus.

- A: Syringe and syringe pump
- B: Needle
- C: HV Power Supply
- D: Whipping Region
- E: Grounded Collector

PARAMETERS VARIED:

In order to achieve the best possible electrospinning results, I changed certain variable parameters involved. One parameter I varied is the height of the collector base. To create fibers on the nano-scale, the solution must travel a relatively significant distance. However, it is often difficult to obtain results if the collector is too far from the syringe. When initiating this project, I placed the collector base much closer to the syringe, about 20cm away, especially when troubleshooting problems with the apparatus. However, once the procedure was running smoothly, I lowered the collector base until it was 40cm from the tip of the syringe. This created nicely structured fibers on the nano-scale.

Another parameter I varied is the rate at which I dispel the polymer solution. For the first syringe apparatus I used, a 5ml syringe, 0.10 mL per hour was the most efficient rate. However, upon using the new plastic apparatus, a 10ml syringe, I found since the syringe was twice the volume, I needed to halve the rate. Though I have experimented with raising and lowering this rate, I found 0.05mL/h most consistently produces nano-scale fibers.

I wished to apply the minimum possible voltage in order to induce electrospinning. This parameter is easily adjusted; by observing the released solution as I apply more voltage, I could see when the droplet formed a Taylor cone from the amount of voltage applied.

Finally, I varied the amount of time I ran the experiment for. I need to be able to locate a single fiber in order to accurately assess its characteristics. To do so, I had to produce enough fibers that I would be able to locate groups on the SEM stub,

but few enough that I could still locate a single fiber. I kept track of the time from the second the voltage reached 20kV. Keeping the 0.05mL/h rate a constant, I found the most effective time to run the experiment for was 5 to 10 seconds, depending on the amount and orientation of the fibers I was looking to collect.

METHODS OF OBSERVATIONS:

Due to the small scale of the fibers, microscopy is used to observe the samples. I used an SEM stub to collect the fibers, which fit nicely into the grounded collecting base. The surface of these stubs is reflective, and small scratches often look like possible fibers. To more easily distinguish the fibers from the topography of the stub, I used a TEM grid, placed on top of a carbon sticking coating the stub. Then, the fibers would collect in squares of the grid and could be easily located using a microscope.

To look more closely at and document the nature of these fibers, I primarily used an optical microscope. I collected samples on SEM stubs with TEM grids placed on top, and observed the small grid-area with different magnifications. The camera attached to the microscope allowed me to document the fibers created in each sample run. Through this method, I could observe the fibers on very small scales without disturbing the integrity of the fibers.

I tested observing my samples using a scanning electron microscope. Here, I would theoretically be able to look much more closely at the fiber structure and determine the size more accurately. However, while observing the fibers on a closer

scale, the constant barrage of electrons from the SEM caused the fibers to melt. The nature of the fibers was too fragile to observe with such an invasive method.

Though I could observe the fibers using the optical microscope, using a different type of microscopy may provide alternative views of the fibers. The AFM provides topographical information about a sample; through this method I could determine the diameter of the fibers and look more closely at the intersections between fibers.

SOLUTIONS:

To create a polymer solution, I used polyethylene oxide (PEO). Alfa Aesar produces this polyethylene oxide powder, sold in 100g jars, stock # 43678, lot # F20Q35, and CAS # 25322-68-3. I add 0.2g of this PEO into 15mL of a particular solvent. These mixtures are stored in small glass containers at room temperature.

I found two effective methods of dissolving the mixtures. One way is adding stirring bars to the solution. The small containers are placed on the magnetic heating pads with temperatures several degrees Celsius below the boiling point of the solvent; the stirring bars mix the solution at a designated rate. Secondly, I use an ultrasound cleaner, a Ney UltraSonik 2Q/H : the samples are placed in a small amount of water, enough to cover 80% of the PEO-solvent container, and the ultrasound cleaner applies vibrations and low heat to the samples. One method may prove more effective, depending on the solute and solvent in question. For samples needing a specific applied heat applied, the heating pad method is more easily controlled.

Water:

My first samples were obtained using a solution of 0.2g of PEO dissolved in 15 mL of deionized water. To create the water-based PEO solution, deionized water is first added to the small vial; then 0.2g of PEO are added. The PEO clumps together rather than dissolving. The most effective method for dissolving the PEO in water is with the stir bar. The stir bar should complete 400 revolutions per minute, and around 90 degrees C should be applied, not to boil the water. Then, after approximately twenty minutes of stirring, the PEO was almost completely dissolved. Once dissolved, the PEO remains in solution, even once it is cooled to room temperature. The solution is not completely clear, and is slightly viscous.

With the water-based PEO solution, fibers were produced. In some cases, these fibers were viable. Examination with an optical microscope revealed that many fibers had beading, where droplets of solution collect along the fiber strands.

Chloroform:

To avoid this beading and to create better fibers, I switched to a solution using 0.2g of PEO dissolved in 15 mL of chloroform. To create this solution, I first added 15mL of chloroform to the small vial and then added the 0.2g of PEO. Here, the PEO power dispersed into the solvent almost instantly. However, the solution was not completely homogenous. To completely integrate the PEO, the solution was placed on the heating pad with the stir bar at 400 revolutions per minute. The boiling point of chloroform is 61.2 degrees C, so 50 degrees C was applied to

completely dissolve the solution. The solution is not completely clear, and is very viscous. It remained a homogenous solution when left overnight.

Again, fibers were spun. The collector distance was 40 cm, and 20kV were applied as the solution was dispensed at 0.05 mL/h. However, these fibers were much larger in scale, and the solution was much more viscous. A few times the syringe tip was clogged from the nature of the solution. The resulting fibers were larger than expected, and certain cases had similar beading results.

Toluene:

Next, I created a toluene-based solution. I added 15mL of toluene to the small vial and then added 0.2g of PEO. The PEO power reacted differently; it seemed to almost crystalize on the sides of the container. One vial was placed in the ultrasound box with low heat; the other was placed on the heating pad with the stir bar at 400 revolutions per minute. The boiling point of toluene is 110.6 degrees C, so the heating pad was set at 90 degrees C. The ultrasound box method was completely ineffective. The PEO remained attached to the sides of the vial, and very little dissolved. The heating pad completely dissolved the PEO after approximately thirty minutes. However, when left overnight, the PEO would recollect on the bottom of the vial and the solution would once again become heterogeneous. Once the heat was reapplied and the stir bar added, the solution would homogenize after approximately twenty minutes. The solution is almost completely clear, and is not viscous when homogenous.

When applying this solution to electrospinning, the toluene-based solution had some success creating fibers. However, heavy beading occurred. Due to the inconsistent nature of the solution, I did not find toluene to be an ideal solvent.

Methanol:

Next, I tested the merit of methanol as a solvent. I dissolved 0.2g of PEO in 15mL of methanol in a small glass vial. The PEO dispersed into the methanol very quickly. To completely homogenize the solution, I added a stir bar at 350 revolutions per minute with no heat applied. This solution was viable after approximately twenty minutes of stirring. The solution is almost completely clear, and is not viscous. It remained a homogenous solution when stored at room temperature.

When examining the fibers created with this solution, I saw I had successfully created fibers with diameters on the micro- and nano-scale without inducing beading. The fibers were created consistently, and the pattern with which they were oriented on the grounded collector implied a consistent spinning method. The solution and process reliably and effectively created nano-scale polymer fibers.

ADDITIVES:

I wanted to examine the properties of these fibers if other particles were introduced to the solution, specifically, carbon and quantum dots. Theoretically, carbon particles added to a nano-fiber could make the fiber conductive. This would allow us to measure the conductivity of the nano-fiber. Somewhat similarly, if a

single quantum dot could be isolated in a nano-fiber, the dot could be used to emulate a point source of light. Because of diffraction, it is not an option to shine a larger light source through a small hole to examine a nano-scale source. However, one quantum dot on a fiber would be a mobile model of a nano-scale light source.

Carbon:

To add carbon, I used carbon found in an InkJet Printer cartridge. These carbon particles were added to the 0.2g PEO/15mL methanol solution, specifically, 0.3g of carbon into the PEO methanol solution. The carbon particles seemed to be dissolved into the solution, however when the solution was electrospun, the carbon particles completely separated from the fibers. The carbon particle solution was evidently not entirely homogenous, and could not be used.

Quantum Dots:

For the quantum dots experiments, I used a solution of quantum dots dissolved in toluene, specifically, Fort Orange Core Shell EviDots. These quantum dots are created by evident technologies, lot # GBO04DCS. When first experimenting with the quantum dots, I added a small amount ($\sim 200 \ \mu$ L) of the quantum dots solution to a water-based PEO solution. However, because toluene is not miscible with water, the solutions did not mix; instead the quantum dots clumped together and were not present in the fibers. In order to accurately integrate the quantum dots into a polymer solution, I attempted a toluene-based solution.

First, I added 100µL quantum dot solution to 5900 µL of a toluene based PEO solution (0.2 g PEO dissolved into 15mL of toluene). Here, fibers were present. Some fibers created were much larger; the toluene solution was very viscous, causing visibly large fibers to collect around the very edge of the collecting base. Micormeter size fibers were created, much larger than the fibers consistently created with the methanol solution. However, there were too many quantum dots present in the solution to be able to distinguish a single quantum dot. Instead, the fibers glowed from groups of the quantum dots.

In order to more easily control the amount of quantum dot solution added to the polymer solution, I added 100µL of quantum dots to 300µL of toluene. This less concentrated solution could easily be diluted or strengthened and added to a toluene-based polymer solution.

The 400 µL toluene and quantum dot solution was added to 6mL of the toluene-based PEO solution. These solutions mixed very well, though the toluene-based PEO solution had to be heated to dissolve and then lowered back to room temperature. When I added this solution to the syringe and prepared it for electrospinning, the PEO solidified on the inside of the syringes and the tubes. It clogged the tip of the small syringe several times. The high viscosity of the solution prevented effective electrospinning.

IDEAL PARAMETERS:

After varying the parameters involved in electrospinning, I determined the optimal variables to create the smallest possible fibers.

Parameter Varied	Optimal Result
Distance Between Needle and	40cm
Collector Base	
Applied Voltage	20kV
Rate Dispensed	0.05mL/H
(from a 10mL syringe)	
Time Run	5-10s

Table 1: Results for the optimal electrospinning parameters.

METHODS OF OBSERVATION:

Optical microscopy, SEM, and AFM portray nanofibers with high accuracy and varying levels of invasiveness. However, for the purpose of my thesis, optical microscopy was the best available option. There is an optical microscope available in close proximity to the electropsinning set up; this way, I could take images of the fibers immediately after they were created. An optical microscope is easy to use, so I could produce several images very quickly. Because optical microscopy is the least invasive technique of the available options, I could image and then re-image fibers without worry that the nanofibers may become damaged or destroyed.

SOLUTIONS:

I electrospun fibers from polymer solutions with four different solvents: water, chloroform, toluene, and methanol, imaging each of the results. Note that for water, chloroform, and toluene, the optimal distance between the needle and collector base is not used. This is because when electrospinning these solutions, I could not create fibers if the distance between the needle and the collector base was any greater. With these three solutions, the resulting fibers are much larger than desired.

Water:

I first achieved electrospinning results with water. Here, 20kV were applied, the solution was dispensed at 0.05 μ L/h from a 10mL syringe, and the collector was roughly 25cm from the tip of the needle. A wire grid was placed upon the collector base; the fibers collected in the intersections between the wires.



0.5mm

Figure 1: Electrospinning results achieved with a water polymer solution. These fibers are on the micrometer scale.

These fibers resembled spider webs – they were thin, wispy structures that seemed to lack any sort of rigid definition. They often grouped together in small bundles.



0.1mm

Figure 2: A more magnified glance at fibers created with the water solution. Here, you can clearly see the fibers grouped together.

Chloroform:

The chloroform results look very similar to results achieved with the water solution, with slightly larger fibers. Here, 20kV were applied, the solution was dispensed at 0.05 μ L/h from a 10mL syringe, and the collector was roughly 25cm from the tip of the needle. Again, a wire grid was placed upon the collector base; the fibers collected between the wires.



0.5mm

Figure 3: Electrospinning results achieved with a chloroform polymer solution. These fibers are on the micrometer scale.

The chloroform electrospinning results were very similar to the results achieved with water. The fibers were soft without defined structure, and often clumped together, especially around intersections between the wires.

Toluene:

With toluene, the fibers looked slightly different from the water and chloroform results. Again, 20kV were applied, the solution was dispensed at 0.05 μ L/h from a 10mL syringe, and the collector was roughly 25cm from the tip of the needle.



0.5mmFigure 4: Electrospinning results achieved with a toluenepolymer solution. These fibers are on the micrometer scale.

The toluene solution results differed greatly from the results achieved with water and chloroform. Here, the fibers were more wire-like, and much more rigid. They collected less around the wire intersections; instead they formed across two wires. These fibers were slightly larger than the water and chloroform fibers, and also formed in groups of fibers. Methanol:

With methanol, results could be achieved with a greater distance between the needle and the collector base, resulting in much smaller fibers. Here the optimal parameter variables were used: 20kV were applied, the solution was dispensed at 0.05μ L/h from a 10mL syringe, and the collector was 40cm from the tip of the needle.



20µm

Figure 5: Electrospinning results achieved with a methanol polymer solution. These fibers are on the nanometer scale. The methanol solution achieved the greatest electrospinning success.

With methanol, I could increase the distance between the tip of the needle and the collector base, resulting in much smaller fibers. Here, the fibers have a distinct orientation: they consistently fall in coils upon the collector. Once the methanol solution was perfected, I could consistently create fibers of this orientation and size.



50µm

ADDITIVES:

Carbon:

To make the carbon particle solution, I added 0.3g of carbon particles to a methanol polymer solution. The optimal parameter variables were used: 20kV were

applied, the solution was dispensed at $0.05 \ \mu$ L/h from a 10mL syringe, and the collector was 40cm from the tip of the needle. At first look, the carbon particles looked as if they were dispersed within the fibers. The black/blue line of coiled fibers in the image below shows the supposed carbon nanofibers.



0.1mm

Figure 7: The black/blue lines consist of many coiled fibers; the methanol based solution led to this orientation pattern. Though their coloring seems to indicate carbon particles dispersed in the solution, closer investigation shows otherwise.

At first look, the carbon particles looked as if they were dispersed within the fibers. The black/blue line of coiled fibers in the image below shows the supposed

carbon nanofibers. Though the carbon particles seemed to be dissolved in the methanol polymer solution, the solution was not homogeneous. The carbon particles completely separated from the created fibers when the solution was electrospun, more easily seen when the image was more closely magnified.



20 µm

Figure 8: Upon closer examination, it can be seen that the carbon particles completely separated from the fibers; the fibers are collected in the bottom left corner of the image and the carbon particles in the top half.

Because the fibers did not disperse throughout the fibers, the carbon particle

solution could not be used to test conductivity. Instead, a substance that can

completely dissolve in a polymer solution must be used.

Quantum Dots:

For the QD solution, I added 100 μ L of QDs suspended in toluene to 23mL of the chloroform polymer solution. The optimal variable parameters were used: 20kV were applied, the solution was dispensed at 0.05 μ L/h from a 10mL syringe, and the collector was 40cm from the tip of the needle.

The resulting fibers were larger than those produced through the methanol solutions, and in many cases, the fibers grouped together. Because of the chloroform base, these fibers did not have the small diameter and rigid orientation found when using the methanol solution.



50µm

Figure 9: Two chloroform fibers with embedded QDs. The QDs cannot be seen in this image.

A group of QD was located, but it occurs in a larger mass of fibers. Though smaller fibers surrounded the large mass, no QDs are seen in any of the smaller fibers. The QDs are easily located by its glowing orange color.



Figure 10: A group of quantum dots located in a large fiber. 0.1mm

However, finding an isolated QD was much more difficult. Still, one was located, again in a group of fibers, but this time a much smaller group. The glow from the QD is much smaller and less colored.



Figure 11: A group of fibers with an isolated quantum dot.

50µm

Here, the single QD is obviously very small and is located upon one of the fibers in the group of fibers. However, no QDs occurred on single isolated fibers, only in instances where the fibers grouped together. This could be related to the properties of the chloroform solution; the QD is suspended in toluene, and the two solutions may not have completely homogenized on the micro-scale.

FURTHER RESEARCH:

To continue this project, one may continue to experiment with the additives to the solution. The carbon particles did not become dissolved in the solution, but another particle may become homogenous in the solution. Then, the solution could be used to test the conductivity of a single fiber.

For QDs, further QD experimentation is needed. Though results were found using a chloroform solution, the best electrospinning results have been produced using a methanol solution. Because the toluene that the QDs are suspended in and methanol are not miscible, the QDs cannot be used in a methanol solution. However, if similar electrospinning results to that of methanol could be achieved, a much smaller fiber could contain a single QD.

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