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Effects of UV Irradation on Electrospun PLLA and PAN in the Production of Short Electropun Fibres Using Ultrasonication Method

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ABSTRACT

This work showed that exposure of ductile electrospun polymers, namely poly-*L*-Lactide acid (PLLA) and polyacrylonitrile (PAN) to UV-Ozone, leads to the embrittlement of fibres. Young's modulus for PLLA and PAN increased by 39% and 78%, respectively. Meanwhile, the ductility was reduced by 23% for PLLA and 40% for PAN. The SEM images show that the UV irradiation resulted in a surface pitted of PLLA and no changes in PAN surface morphology. The ATR-FTIR results indicate that this treatment did not change the chemical structure of the electrospun PLLA and PAN fibres. The as-spun polymers that failed to be scission directly using ultrasonication can now be fragmented into micron-length short fibres after the UV irradiation treatment. The minimum time to produce the short fibres is 18 mins for PAN and 29 mins for PLLA. It indicates ultrasonication is suitable for producing short electrospun fibres, even for ductile materials.

Keywords: Electrospun fibres, short fibres, ultrasonication

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INTRODUCTION

Electrospinning is a technique that employs electrical charge and polymer solutions to generate tiny filaments. In comparison to other conventional approaches such as drawing, template synthesis, phase separation, and self-assembly, this method has since been recognised as a quick and straightforward technique for producing continuous sub-micron to nano-sized fibres (Huang et al., 2003; Ramakrishna et al.,

2005). Natural polymers, synthetic polymers, polymer solutions, polymer melts, and biopolymers have all been employed to make fibre materials. Flexibility is one of the appealing characteristics of electrospinning. By adjusting the processing parameters, the morphology of the fibres can be easily altered with this technology, allowing the manufacture of solid, porous, and core-shell fibres (Bazilevsky et al., 2007; Sun et al., 2003).

Briefly, the electrospinning method uses the positive electric field to induce changes in the polymer solution, and once this charge build-up exceeds that of the surface tension, a Taylor cone is formed (He et al., 2008). A polymer jet erupts from the cone tip at this critical voltage, accelerating through the electric field towards the collector. The solvent evaporates as the jet travels towards the collector, and a solidified polymer fibre is collected. The electrospun fibres produced from this method have a wide range of applications, including tissue engineering, sensors, and composite reinforcement (Bhardwaj & Kundu, 2010; Huang et al., 2003; Li et al., 2019; Nakielski et al., 2022; Niemczyk-Soczynska et al., 2021; Ramakrishna et al., 2005; Zakrzewska et al., 2022).

There is little research on making individual, short nanofibres from commonly electrospun membranes. It would be useful to separate an electrospun membrane into distinct, short nanofibres because this would result in a method for making large amounts of these fibres. Short fibres are excellent for biomedical applications as injectable fibrous scaffolds for tissue engineering or drug-containment channels (Nakielski et al., 2022). Short electrospun fibres are produced quickly (in a few seconds to a few minutes) using ultrasonication (Niemczyk-Soczynska et al., 2021; Sawawi et al., 2013). Most laboratories utilise ultrasonic probes, which operate at a frequency of 20 kHz, for tasks including cleaning and mixing (Watmough, 1994). Bubbles in the fluid medium expand and contract during sonication, releasing energy as they do so. These bubbles begin with a diameter of approximately 1 μm and increase to approximately 50 μm under negative pressure (Suslick, 1988). The bubble expands under these sonication settings in 20 microseconds and collapses in nanoseconds. This technique has been used to help carbon nanotubes disperse in a solvent (Rennhofer & Zanghellini, 2021) and cause carbon nanotube scission (Hennrich et al., 2007).

There are varied degrees of success with several approaches to reinforced composite using the shortened, non-woven electrospun membrane. Mortar grinding (Deniz et al., 2011; Sancaktar & Aussawasathien, 2009) is a straightforward but unpredictable technique that is proven to work well for brittle electrospun membranes like carbonised polyacrylonitrile (Sancaktar & Aussawasathien, 2009). Better choices for scissioning electrospun membranes include rubber milling (Kim & Reneker, 1999) and cryogenic milling (Verreck et al., 2003), where the impactor of cycle and rate can be precisely regulated. Comparing the methods is challenging because the features of the resulting short fibres were not always accurately described. Short magnetic composite fibres such as methyl methacrylate-vinyl acetate

copolymer/superparamagnetic cobalt nanoparticles are created through the application of other mechanical techniques, such as razor blade cutting under nitrogen (Kriha et al., 2007). Another technique for creating distinct fibres from the electrospun membrane includes producing short nylon electrospun fibres using 1 μm step-sliced cryomicrotoming. However, it requires time-consuming, repetitive steps (Mark et al., 2008). Reduced poly(butadiene) electrospun fibre mats were made utilising UV light to degrade polymers. The UV light was shone through masks with well-defined slit diameters and inter-slit distances. The mesh is covered with mats, which result in short fibre bundles rather than single, short fibres (Stoiljkovic & Agarwal, 2008).

In this work, treatment of ductile polymers such as PLLA and PAN using UV irradiation. The electrospun membrane of these polymers will be exposed to UV irradiation, and the materials' surface morphology and chemical bonding will be investigated. Subsequently, ultrasonication of the as-spun electrospun and UV-irradiated membranes will be discussed. The ability of the ductile polymers such as PAN and PLLA to be directly scission using ultrasonication will open a venue for the methodology to produce individual short electrospun fibre in a short time and high quantities, which can be used in various applications.

MATERIAL AND METHODS

Poly(*L*-lactide) Acid (PLLA, Inherent viscosity = 0.9 - 1.2 dL/g) was purchased from Lactel, USA. Polyacrylonitrile (PAN, Mw = 120,000) and dodecyl trimethyl ammonium bromide (DTAB) were purchased from Sigma Aldrich, Australia. Chloroform, dimethylformamide (DMF) and acetone were bought from Merck Pty Ltd, Australia. Deionised water was obtained from the Direct-Q3 water purification system, Millipore.

Electrospun PLLA webs were made using chloroform/acetone (3:1) in 1 mM DTAB. Using 13.7 wt/v% concentration, DTAB was added to 10 mL chloroform/acetone to make a 0.1 M stock solution. A magnetic stirrer dissolved PLLA-solvent overnight. As-received PAN was mixed with DMF to make PAN solution at the concentration of 10 wt/v%. Electrospinning was done utilising a lab-built electrospinner with a Gamma High Voltage Research (USA) high voltage supply and a Razel Scientific Instruments, Inc. syringe pump (USA). The PLLA solution was spun at the feed rate of 1.6 ml/hr, a voltage of 20kV, a needle size of 18G and a working distance of 16 cm. PAN solution was prepared using the same electrospinning parameter except for the working distance, which was at 8 cm. After several optimisation processes, the electrospinning parameter was chosen to obtain a beadles fibre with a similar submicron diameter. All polymers were spun onto a flat collector (aluminium foil) and stored in a desiccator under a vacuum before use.

An ultrasonicator probe with a diameter of 13 mm (Vibracell 750W with 20kHz from Sonics & Materials, Inc, USA) is used for the ultrasonication experiment. A 1 cm^2 section

of the electrospun membrane with a ca 100 – 200 µm thickness was cut and placed in a 25 mm glass vial containing 15 ml of MiliQ water at room temperature. The sonicator was set to 80% amplitude with a 2 s ON/2 s OFF (2/2) cycle. The sonication time was halted until visible fibre breakup. After sonication, a drop of short fibres in suspension was deposited on a carbon-tape-covered stub. The material was dried prior to FESEM imaging (JEOL 7001FEG SEM),

UV-Ozone irradiation treatment was conducted using UV Ozone ProcleanerTM UV PC 220 (Bioforce, USA). The electrospun membrane was irradiated for 12 min at 14.75 mW/cm^2 prior to the sonication experiment.

Field Emission SEM (JEOL 7001FEG SEM) characterised fibre morphology. Before imaging, the sample was platinum-coated at 1 nm thickness (Cressington 208HR, UK). Image J programme (National Institute of Health, USA) was used to measure the diameter and length of 50 short fibres.

The mechanical testing was done with an Instron 5848 microtester (USA) at 5 mm/min and 10 N load. The 0.3-mm-thick electrospun membrane was sliced with a dogbone cutter with a gauge length of 20 mm. Five independent samples of each polymer were tested for ultimate tensile strength (UTS), Young's modulus (E), and per cent strain to failure (ductility).

The samples were further characterised by Attenuated Total Reflectance Fourier Transform Infrared (ATR-FTIR) spectroscopy (Thermo Scientific Nicolet 6700) with a scanning range of 4500 to 400 cm^{-1} and 4 cm^{-1} resolution.

RESULTS AND DISCUSSION

SEM images in Figure 1 show the untreated (as-spun) and the UV-ozone irradiated PLLA and PAN membrane at different magnifications.

Figure 1a shows that the as-spun PLLA fibre is beads-free and randomly oriented with a smooth surface (Figure 1b). Whilst the UV-Ozone treatment has changed the surface morphology of PLLA electrospun webs, where etching on the surface of the fibre can be observed (Figure 1c and d). In contrast, the PAN electrospun webs' surface morphology remains the same as for the untreated and the UV-Ozone treated webs (Figure 1f to i). The effect of UV-Ozone irradiation depends on the chemical nature of the polymer and the UV wavelength. It was reported that the carbonyl group (C=O) bond can be cleaved at the wavelength of 225 nm (Sakai & Tsutsumi, 2010), whilst the UV-Ozone cleaner used in this work has a wavelength of about 254 nm. In contrast, homolysis of alkane (C≡C) PAN fibres occurs at wavelengths greater than 300 nm, indicating that the UV wavelength used in this experiment is insufficient to cause chain scission.

The as-spun PLLA and PAN average fibre diameter is calculated to be 770 ± 240 nm and 740 ± 150 nm, respectively, with the fibre size distribution shown in Figure 2.

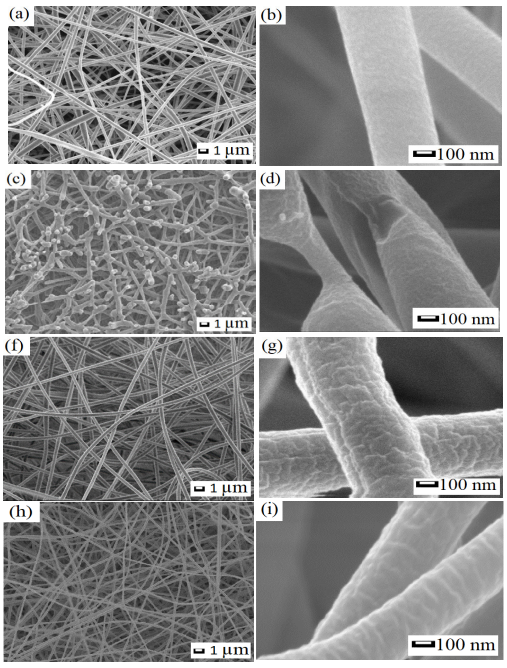

Figure 1. SEM images (a and b), untreated PLLA fibre (c and d), UV-Ozone irradiated electrospun PLLA fibre (f and g), untreated PAN fibre and UV-Ozone irradiated electrospun PAN (h and i)

It shows that the diameter of both fibres is broadly similar.

Tensile testing of the UV-Ozone irradiated and untreated electrospun PAN and PLLA was performed to study the effect of UV-Ozone on the mechanical properties. Figure 3 shows the stress-strain curves of the untreated, and UV-Ozone irradiated electrospun webs, and a summary of the average mechanical properties taken from 5 samples are presented in Table 1.

Figure 3 and Table 1 found that the UV-Ozone treatment increased Young's modulus of both electrospun PLLA from an average of 11.1 GPa to 15.4 GPa, which is about a 39% increment. It is also similar to PAN fibres, where the modulus increased from an average of 12.2 GPa to 21.7 GPa (78% difference). The tensile strength of PAN

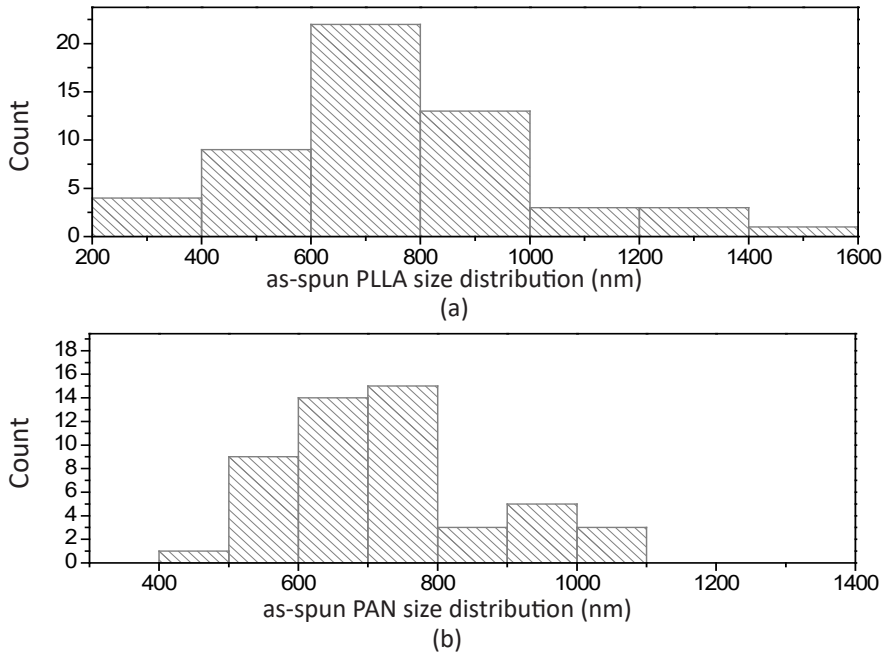

Figure 2. Size distribution of as-spun fibres (a) PLLA, (b) PAN

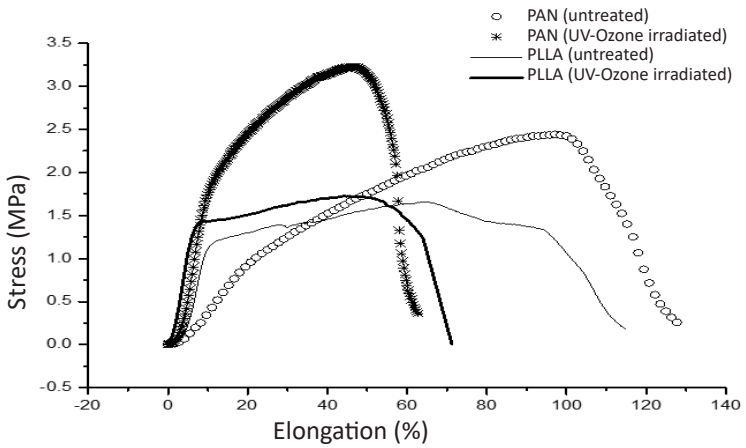

Figure 3. Stress-strain curve of the untreated and UV-Ozone irradiated PAN and PLLA

Table 1

Mechanical properties of the untreated and UV-Ozone irradiated electrospun PLLA and PAN (n=5)

Material	Young's modulus, E (GPa)	Tensile strength, σ (MPa)	Ductility (%)
PLLA (untreated)	11.1± 2.6	1.6 ± 0.2	79.2 ± 17.9
PLLA (UV-Ozone irradiated)	15.4 ± 2.8	1.2 ± 0.3	56.1 ± 16.9
PAN (untreated)	12.2 ± 7.7	2.5 ± 0.1	86.3 ± 22.3
PAN (UV irradiated)	21.7 ± 6.9	3.2 ± 0.3	50.2 ± 13.5

webs increased by 29% (from 2.5 MPa to 3.2 MPa) and showed a reduction in ductility of about 40% (from 86.5 to 50.2%). However, for PLLA, the tensile strength and ductility decreased by 23 % (from 1.6 MPa to 1.2 MPa) and 30% (from 79.2 to 56.1%), respectively. It was reported that the effect of UV irradiation varies depending on the polymer exposed (Guillet, 1972). The effect of UV irradiation on polymers includes discolouration, scission of the main chain, photoinduced crystallisation and crosslinking (Guillet, 1972; Sakai & Tsutsumi, 2010; Stowe et al., 1974). The UV-Ozone treatment of PLLA is likely to degrade the PLLA macromolecules, as it reduces its strength and the elongation to failure, initiating the degradation of polymer molecules and reducing the molecular weight (Yixiang et al., 2008). It could occur at the surface (since introducing surface flaws through exposure would change mechanical properties) and within the bulk. The increase in modulus observed could be due to cross-linking. Most importantly, it can be seen from these results that the UV-Ozone treatment leads to a decrease in ductility, resulting in the desired electrospun fibre embrittlement.

In this study, we investigated the impact of UV-Ozone treatment on the ultrasonication of PLLA and PAN electrospun webs. Specifically, we performed ultrasonication on webs that had been previously irradiated. Our previous work (Sawawi et al., 2013) discussed that the untreated PLLA and PAN fail to be scission using ultrasonication directly. Here, the UV-ozone-treated webs were sonicated in water at 2/2 lapsed ON/OFF time and 80% amplitude. It was found that the sonication time for the electrospun webs to break up fully is 29 min for PLLA and 18 min for PAN. The untreated electrospun webs for PLLA and PAN were also sonicated simultaneously for comparative purposes. The SEM images of the sonicated webs are shown in Figure 4.

Untreated PLLA and PAN webs (Figures 4a and 4b) could now be fractured into short fibres by ultrasonication. After 29 min of sonication, the PLLA fibre's average length is 5.2 ± 5.4 µm (Figures 4c and 4d). PAN-treated webs can be scission in 18 minutes with a 10.6 ± 5.1 µm average fibre length (Figures 4h and 4i). As with untreated PLLA webs, UV-Ozone-treated sonicated fibres produced a rougher, pitted surface than the pre-sonicated sample.

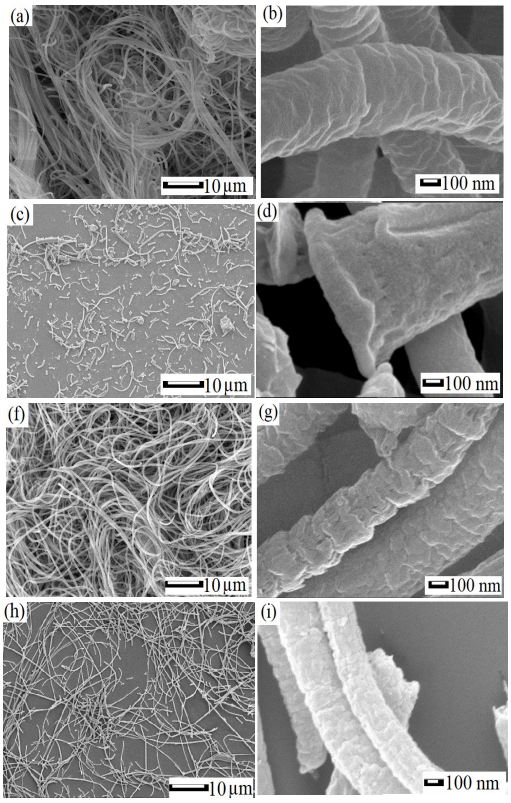

Figure 4. SEM images of sonicated (a and b), untreated PLLA t = 29 min (c and d), UV-Ozone irradiated PLLA t = 29 min (e and f), untreated PAN, t = 18 min and UV-Ozone irradiated PAN, t = 18 min (g and h)

This surface morphology change is also observed in sonicated untreated PAN (Figure 4f) compared to sonicated UV-Ozone treated PAN (Figure 4h). It supports the hypothesis that ductility represents the most important mechanical properties of the electrospun nanofibres in terms of the ability of sonication that cause scission. It is most likely that it is the manifestation of this ductility, which is reduced due to pitting and tensile and bending deformations that lead to the final failure.

The chemical characterisation of the untreated, pre and post-sonication UV-Ozone irradiated webs was performed using reflection mode by Attenuated Total Reflectance Fourier Transform Infrared spectroscopy (ATR-FTIR) with the spectra as shown in Figure 5.

It was observed that the as-received PLLA, UV-Ozone treated electrospun PLLA

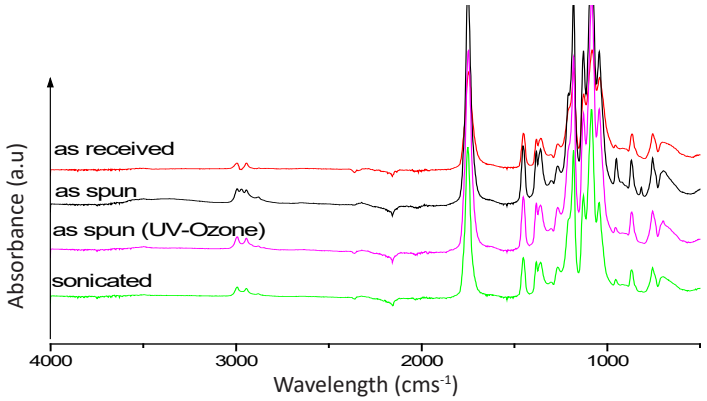

Figure 5. ATR -FTIR spectrum for as received, as-spun, as-spun with UV-Ozone treatment and sonicated PLLA (UV-Ozone treated web) in water

and sonicated UV-treated webs show a similar symmetric and asymmetric bending vibration of methyl groups at 1383 and 1456 cm^{-1} with ester linkages appeared at 1747, 1180, 1126 cm^{-1} which is corresponding to C=O, C-O-C and O-C-C vibration for all types of PLLA sample. However, the untreated PLLA webs showed a new band of a methyl group at 2973 cm^{-1}, whilst only symmetric and asymmetric stretching vibrations of the methyl group at 2947 and 2997 cm^{-1} were seen for other conditions. This new peak was probably due to the solvent residue of chloroform, $CHCl_3$. It was noted that chloroform peaked at 2976 cm^{-1}, which appeared in the untreated spectrum. After UV-Ozone treatment, this peak disappears, which is likely due to the removal of the residual solvent during UV-Ozone exposure.

The ATR-FTIR spectra of the PAN fibre before and after UV-Ozone treatment are shown in Figure 6.

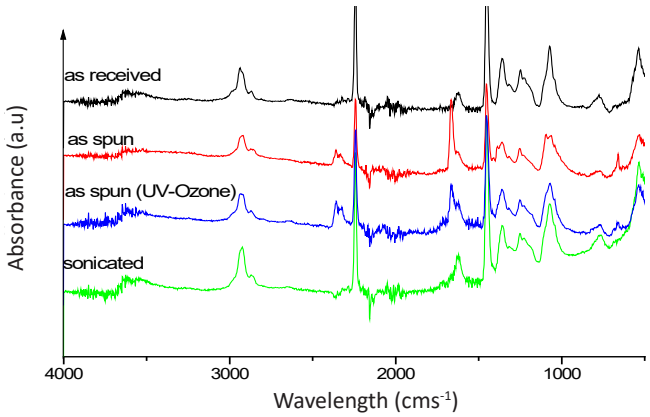

Figure 6. ATR-FTIR Spectra of PAN as received, as-spun, as-spun with UV-Ozone irradiation and sonicated PAN (UV-Ozone treated web) in water

Figure 6 shows that the as-received and sonicated electrospun webs PAN have a similar peak, where the methylene group stretching vibrations appear at 2923 and 2867 cm^{-1}. The strong peak at 2240 cm^{-1} is the characteristic vibration of the nitrile group (-CN). The stretching vibrations of carbon-carbon double bonds are shown at 1660 and 1630 cm^{-1}. The peaks at 1452 cm^{-1} and 1361 cm^{-1} are assigned to the bending vibrations of the methylene and methane groups, respectively. The 1253 cm^{-1} peak is due to the vibration of the methane group. However, an additional weak band at 2356 cm^{-1} and 2321 cm^{-1} appears in the as-spun, and as-spun (UV-Ozone) treated webs spectra due to CO_2 (could be originated from environment and contaminant) and can be neglected. UV-Ozone irradiation for PLLA and PAN did not seem to change the chemical structure of the surface and bulk polymer.

CONCLUSION

This work has shown that the UV-ozone irradiation to the surface of the electrospun fibres leads to a lower ductility resulting in embrittlement compared to the spun membrane. The embrittlement results in the ability for PLLA and PAN to be scission into short fibres down to about 5 μm using ultrasonication was unable to be achieved prior to the treatment. The ATR-FTIR results indicate no significant changes to the chemical structure of the electrospun fibres, which shows that this method can produce short electrospun fibres for other types of ductile polymers.

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