Electrospun Generation of Lead-Free CsCu­2I3 Polystyrene Nanofibers

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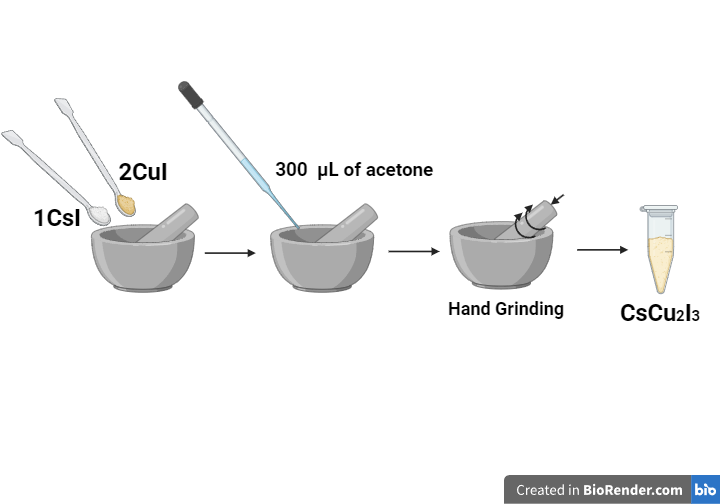
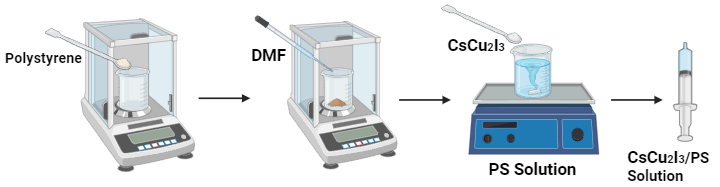
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**Abstract.** Lead-free CsCu2I3 perovskite nanocrystals showcase unique optoelectronic features. Nevertheless, their structural stability under harsh conditions and surface and defects of grain boundary diminish their optoelectronic efficiency due to non-radiative recombination. Incorporating a polymer matrix into perovskite structure presents an effective strategy to enhance its stability and decrease undesired defects. This study explores the formation of lead-free CsCu2I3 perovskite integrated with the polystyrene nanofibers (PS-NFs), examining their structural and vibrational characteristics through Fourier transform infrared (FTIR), X-ray diffraction (XRD) and Raman spectroscopy. PS facilitates the encapsulation of the CsCu2I3 perovskite surface, leading to an enhanced stability. CsCu2I3-PS nanofibers were prepared via post-encapsulation using the electrospinning technique, utilizing several parameters such as a flow rate of 0.4 mL/min, voltage of 15 kV, and humidity at 60 °C. FTIR analysis reveals the successful incorporation of CsCu2I3 into PS-NFs, evidenced by the emergence of characteristic absorption peaks at 439 cm-1, indicative of the existence of metal-halide bonds. The XRD spectrum presents an amorphous peak attributed to the polystyrene matrix within the CsCu2I3 perovskite structure. XRD analysis indicates the presence of both styrene and CsCu2I3 phases in the CsCu2I3-PS nanofibers, demonstrating a strong correlation with the original materials utilized. Raman spectra confirmed that CsCu2I3 was successfully embedded in polystyrene nanofibers. This encapsulation led to intensity reduction in the PS nanofibers Raman spectrum, indicating the strong binding with CsCu2I3, accompanied by the present of emerge peak in the 200 – 50 cm-1 range. This effort led to the advancement of hybrid materials towards the high-efficiency optoelectronic devices to come.

**INTRODUCTION**

All-inorganic metal halide perovskites are showing promise applications in solar cells, LEDs, and photodetectors due to their unique features such as adjustable distribution, flexible band gap, and long carrier diffusion [1]. Despite the common use of lead-based perovskites in optoelectronic devices, their toxic nature and stability issues limit their widespread adoption. Substituting Pb2+ with environmentally sustainable cations presents a potential solution to these issues. Recently, there has been significant interest in utilizing the copper(I) cation (Cu+) as a substitute for lead in the creation of all-inorganic lead-free halide emitters, denoted as (CsX)1-y(CuX)y (where X=I, Br, or Cl). This alternative is gaining attention due to its cost-effectiveness, widespread availability, and environmentally benign light emission properties. By examining the crystal structures of these compounds, a series of Cu(I)-based ternary halide materials can be derived, wherein the copper composition (y) is increased, resulting in the formation of Cs2CuX3, Cs3Cu2X5, CsCuX2, CsCu2X3, CsCu4X5, and CsCu9X10 (where X=Cl, Br, or I) [2,3]. Researchers are particularly interested in low-dimensional perovskites because of their high exciton binding energies from intrinsic quantum confinement and high photoluminescence quantum yields (PLQYs). Yang et al. (2020) have successfully developed one-dimensional CsCu2I3, which exhibits remarkable bright yellow emission and a high photoluminescence quantum yield (PLQY) of up to 12.3%. This material also emerges as a promising candidate for deep ultraviolet (DUV) photodetector applications, achieving impressive responsiveness (R), specific detectivity (D\*), and external quantum efficiency (EQE) values of 22.1 mA/W, 1.2 × 1011 Jones, and 10.3%, respectively, at a specific light density (0.305 mW/cm2) [4,5]. However, perovskite materials face challenges in thermal and optical stability, being prone to chemical degradation from environmental factors, which can compromise their optoelectronic performance [6].

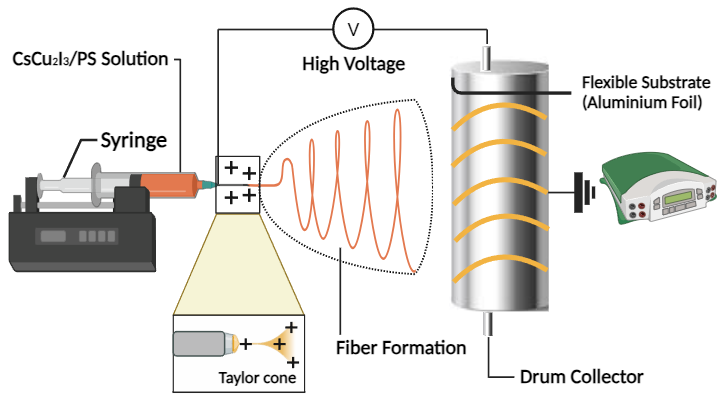
Perovskite nanowires with controlled dimensions can improve device performance by reducing grain boundaries and surface imperfections, thereby enhancing carrier flow and device efficiency [6]. Additionally, integrating perovskites with polymers that form nanowires can further enhance stability by protecting the perovskite surface and facilitating the formation of a solid film with excellent crystallinity [7]. Electrospinning is a technique used to produce nanofibers that adopt nanowire configurations by injecting a solution through a needle under high voltage, with fibers collected by a collector through potent electrostatic forces. Rigorous control over this process is crucial for achieving homogeneous nanofibers [8]. This study presents a novel approach by embedding CsCu2I3, a lead-free perovskite, within polystyrene nanofibers (CsCu2I3-PS NFs) using the electrospinning technique. Due to its versatility, low cost, and stability, polystyrene (PS) is chosen to encapsulate the perovskite film. Polystyrene can be used to coat the surface of perovskite films, migrating towards the grain boundaries during the crystallization process. This coating inhibits mass exchange between perovskite grains and the external environment. As a result, it enhances the stability of the chemical phase, morphology, and microstructure of the material under extreme conditions, while also protecting it from water corrosion [9, 10]. We utilized FTIR, XRD, and Raman spectroscopy to examine the material's vibrational and structural properties. Furthermore, we optimized the electrospinning process parameters by first fabricating polystyrene nanofibers and subsequently applying these optimized parameters to synthesize CsCu2I3-PS nanofibers. This research not only addresses the toxicity and stability issues associated with lead-based perovskites but also explores the enhanced stability and performance provided by the novel CsCu2I3-PS nanofibers.



**2) Preparation of CsCu2I3-PS Solution**

**3) Fabrication of CsCu2I3-PS Nanofibers**

**1) Synthesis of CsCu2I3**



**CsCu2I3-PS**

**Solution**

**FIGURE 1.** Schematic illustration of fabrication CsCu2I3-PS nanofibers.

**METHOD**

**Control Experiment, Cone jet image of Polystyrene (PS) Nanofibers**

This research conducted control experiments by producing polystyrene (PS) nanofibers with varying concentrations of polystyrene in a N,N-Dimethylformamide (DMF) solvent. These optimal parameters were subsequently employed to integrate polystyrene nanofibers with CsCu2I3 lead-free perovskite. The PS solution was prepared in a syringe for fabrication using the ILMI-N101 electrospinning method, which was adapted from previous studies with specific modifications for this research [11]. The experimental parameters consisted of a flow rate of 0.4 mL/h, a working distance of 15 cm between the syringe pump and the collector, and an applied voltage of 15 kV. The CsCu2I3-PS nanofibers were collected on aluminum foil mounted on a rotating drum collector. The electrospinning process was conducted continuously under controlled conditions with temperature around 25 – 40 oC and 60% relative humidity.

**Synthesis of CsCu2I3-Polystyrene (PS) Nanofibers**

A pre-synthesis technique is employed to fabricate CsCu2I3-PS nanofibers, as illustrated in the schematic diagram featured in Fig. 1. The CsCu2I3 material is synthesized first before being mixed into a polystyrene solution. The lead-free perovskite CsCu2I3 was synthesized using an all-solid-state mechanochemical synthesis method [12-14]. CsI and CuI were used as precursors in a molar ratio of 1:2, placed in a mortar, and ground for 10 minutes in 300 µL of acetone. The CsCu2I3-PS solution was prepared by dissolving 0.5 grams of PS and 1.0 gram of CsCu2I3 into 10 ml of DMF (15% w/v). Subsequently, the mixture was stirred until completely dissolved. The CsCu2I3-PS solution was placed into a syringe for nanofibers fabrication using the ILMI-N101 electrospinning method, employing the same parameters as those used for fabricating polystyrene nanofibers.

**Characterization**

The functional groups present in each material were characterized utilizing the ATR-FTIR Thermo Scientific Nicolet iS10 spectrometer, boasting a spectrum range spanning from 500 to 4000 cm-1, a spectrum resolution of 0.4 cm-1, and employing a fast recovery deuterated triglycine sulfate (DTGS) detector. Furthermore, the Raman spectra and morphology of the materials were analyzed employing the Thermo Scientific DXR3xi Raman Imaging microscope, featuring a spectrum range of 50 to 3000 cm-1, a laser power of 3 mW, an exposure time of 100 Hz, and conducting 1000 scans. Additionally, the Raman Imaging technique utilizing the peak height profile was employed in this experimental investigation. The material structure was measured with an x-ray powder diffractometer (XRD) (PANalytical AERIS, Malvern Panalytical, The Netherlands) with a Kα of 1.54 Å.

**RESULTS AND DISCUSSION**

**Unveiling the Impact of PS Solution Concentration on Nanofibers Morphology**

Electrospinning has been utilized as an effective method for producing simple and easily controllable polystyrene nanofibers morphology, catering to tissue engineering, sensors, composite materials, and filtration needs. The electrospinning process involves injecting a polymer solution through a metal needle under high voltage. As the polymer fluid exits the metal needle, it elongates gradually to form a "Taylor cone." When the electrostatic force exceeds the surface tension of the droplet, charged fluid emits and stretches straight into thin fibers, accompanied by solvent evaporation during the process. The compacted polymer nano/micro fibers are collected on a collector coated with a specific substrate [15, 16]. To achieve controlled morphology and fine fiber formation of polystyrene nanofibers, attention must be paid to parameters in the electrospinning process, especially polymer solution concentration. We analyzed cone Taylor images using a TFT color monitor and examined fiber morphology using an optical microscope in Raman spectroscopy to understand the influence of solution concentration. Nanofibers morphology is influenced by solution concentration, as it also impacts the conductivity and viscosity of the solution. This phenomenon can be attributed to the enhanced entanglement of polymer molecular chains within the solution. With increasing solution concentration, the number of polymer molecules present rises proportionally, leading to greater entanglement and consequently, a rise in viscosity. Conversely, the increased polymer concentration reduces the solvent content within the solution, hindering the mobility of ions and thereby decreasing conductivity [17, 18].

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| (c) | (d) |

**FIGURE 2.** Image of taylor cone captured from TFT color monitor (top side) and its illustration (bottom side) at different concentrations. (a) 5 wt.% PS, (b) 10 wt.% PS, (c) 15 wt.% PS, and (d) 20 wt.% PS.

We varied the concentration of polystyrene (PS) in DMF solvent across a range from 5% to 20% by weight. In Fig. 2, it is evident that distinct "Taylor cones" are formed at each concentration. At a concentration of 5wt.% PS, there is evidence of the formation of a short "Taylor Cone" that appears somewhat unstable as it doesn't shoot straight with a perfect cone shape. This instability arises from the excessively low concentration, resulting in a weakened cohesive force within the polymer solution, hence leading to an unstable jet formation. Conversely, concentrations ranging from 10% to 20% exhibit a stable cone shape, indicating that the cohesive strength of the polymer solution is sufficient to form a stable "Taylor Cone" [18]. As the concentration of the PS solution increases, the length of the "Taylor Cone" also increases. This occurs because as the solution concentration rises, the electric field force and the repulsive force of the surface charge of the jet decrease, resulting in a jet that is less prone to bending, with a longer Taylor cone length and a shorter trajectory towards the collector [19]. The appearance of polystyrene nanofibers product, as depicted in Fig. 3 and analyzed through optical microscopy from Raman spectroscopy, reveals a significant pattern: with an increase in the concentration of the polystyrene solution, the resulting fibers demonstrate a smoother structure without any bead-like formations. This phenomenon is attributed to the high viscosity of the concentrated solution, which allows the formation of smooth fibers with larger diameters due to the polymer chain entanglements overcoming surface tension. At the lowest concentration, 5 wt.% PS, irregular fiber formation is observed, with only scattered small particles visible. At a concentration of 10 wt. PS%, fiber formation begins with numerous beads. These phenomena arise from capillary breakup during spinning, influenced by surface tension and the viscoelastic properties of the solution. Solutions with lower concentrations exhibit higher surface tension, primarily from DMF, which counteracts coulombic forces, resulting in bead formation on the collector [20].

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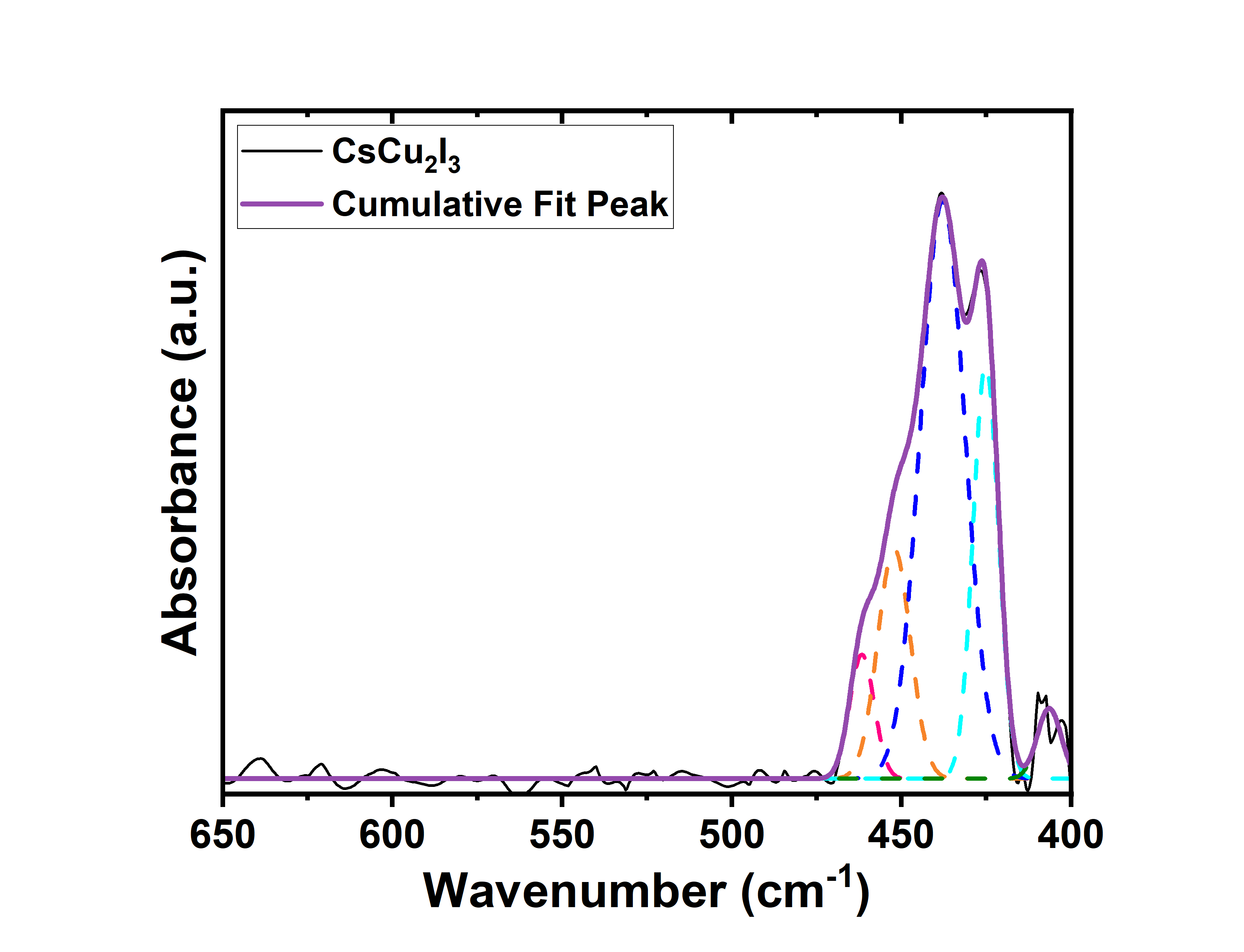
**FIGURE 3.** The morphology of polystyrene nanofibers at various concentration levels using optical Raman spectroscopy with magnification 50x (a) 5 wt.% PS, (b) 10 wt.% PS, (c) 15 wt.% PS, and (d) 20 wt.% PS.

**Fourier-Transform Infrared (FTIR)**

Fig. 4 illustrates the FTIR spectra and their analysis for PS nanofibers, CsCu2I3, and CsCu2I3-PS nanofibers materials. In Fig. 4a, the vibrational characteristics of polystyrene are identifiable through distinct peaks in both PS nanofibers and CsCu2I3-PS nanofibers. The FTIR spectra exhibit peaks at wavenumbers of 3025 cm-1 attributed to the vibration resulting from C-H stretching of the benzene ring. The molecular vibrations of C-H stretching from methylene are observed at 2924 cm-1 and 2866 cm-1 wavenumbers [21]. Three peaks at wavenumbers 1601, 1492, and 1452 cm-1 correspond to the vibrational stretching of aromatic C=C bonds, arising from deformation vibrations within the benzene ring. In addition, peaks at the wavenumbers of 755 and 696 suggest C-H out-of-plane bending vibrations, indicating the presence of a single substituent within the benzene ring [22]. The CsCu2I3 composite within polystyrene nanofibers exhibits reduced peak intensities in the fingerprint region compared to pure polystyrene nanofibers, indicating interactions between CsCu2I3 and polystyrene. Additionally, absence of characteristic DMF peaks at 1673, 1389, 1256, and 1096 cm-1 suggests complete evaporation of the DMF solvent during electrospinning [23, 24]. In the FTIR spectra of pure CsCu2I3, a prominent peak at 439 cm-1 signifies active infrared stretching vibrations of metal-halogen bonds within the inorganic complex, typically observed within of 500-200 cm-1 wavenumber range [14].

Fig. 4b shows the fingerprint region between 650-400 cm-1, where multiple peaks are detected within a large peak. In CsCu2I3-PS nanofibers, there's a shifting of peaks around 600-500 cm-1, splitting into several segments. Differences in peak patterns between 500-400 cm-1 also support the successful embedding of CsCu2I3 onto polystyrene nanofibers. On the other hand, pure CsCu2I3 absorption also reveals two peaks, characteristic of metal-halogen absorption in the metal complex. Further analysis involved deconvoluting of the FTIR spectra of each material. The deconvoluting results indicate that both CsCu2I3 (Fig. 4c) and polystyrene nanofibers (Fig. 4d) exhibit several absorption peaks within a single prominent peak, with 5 and 6 peaks identified and well-matched, respectively. In the deconvoluting of CsCu2I3 composited within PS nanofibers (Fig. 4e), significant differences from both main materials are evident. Shifts and splits in characteristic peaks of polystyrene occur, along with the emergence of new peaks scattered in the wavenumber range of 500-400 cm-1. These deconvoluting results suggest successful fabrication of CsCu2I3 within PS nanofibers, with characteristic CsCu2I3 peaks at 439 cm-1, and other peaks experiencing shifts due to interactions with polystyrene. This is further supported by the total number of peaks identified in CsCu2I3-PS nanofibers, which matches the sum of identified peaks from CsCu2I3 and polystyrene nanofibers.

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**FIGURE 4.** (a) FTIR spectra at 4000-400 cm-1, (b) FTIR spectra at 650-400 cm-1, (c) Deconvoluting FTIR spectra of CsCu2I3, (d) Deconvoluting FTIR spectra of PS nanofibers, (e) Deconvoluting FTIR spectra of CsCu2I3-PS nanofibers.

**Raman Spectroscopy**

Fig. 5 presents the Raman spectroscopy results, providing detailed information on the successful incorporation of CsCu2I3 perovskite into the polymer matrix. In Fig. 5a, the spectra for PS nanofibers, CsCu2I3, and PS/CsCu2I3 nanofibers are shown. The PS spectrum exhibits characteristic peaks at 3052 and 2906 cm-1, corresponding to aromatic C-H stretching and asymmetric CH2 stretching, respectively [25]. Additionally, peaks at 1602, 1031, 1001, and 612 cm-1 are attributed to C-C stretching vibrations within the benzene ring, C-H in-plane deformation, C-C stretching in the benzene ring (a hallmark peak for aromatic molecules), and V6bradial ring-stretching mode, respectively [26, 27]. The CsCu2I3 spectrum prominently features a peak at 121 cm-1, indicative of the I-Cu-I vibrational mode [1, 14]. In the spectrum of the PS/CsCu2I3 composite, the primary peaks associated with polystyrene are still observable but show reduced intensity, indicating successful embedding of CsCu2I3 in polystyrene. Furthermore, the emergence of a new peak in the 200-50 cm-1 range further substantiates the embedding of CsCu2I3 within the polymer framework. Fig. 5b shows the Raman spectrum in the 200-50 cm-1 range to confirm the successful embedding of CsCu₂I₃ in polystyrene nanofibers. The spectrum displays a vibrational mode at 119.72 cm-1 in the CsCu2I3-PS nanofibers, which corresponds to the characteristic I-Cu-I vibration mode observed in CsCu2I3. The shift observed in the I-Cu-I vibration mode peak signifies the interaction between CsCu2I3 and the PS matrix. Additionally, the lack of any peak in this spectral region for pure PS provides further confirmation that the emergence of the new vibration mode around 120 cm-1 directly correlates with the integration of CsCu2I3. We conducted Raman mapping analysis to ascertain the presence of CsCu2I3 within polystyrene nanofibers. This involved examining the height peak profile characteristic of CsCu2I3, employing a scanning frequency of five times and an area size of 80 µm with a pixel dimension of 0.5 µm. The mapping was conducted on regions exhibiting a uniform nanofibers surface under Raman optics. The mapping outcomes reveal a widespread distribution of CsCu2I3, with the minor existence denoted in green. Additionally, regions exhibiting dominant presence of CsCu2I3 are highlighted in red and yellow.

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| (c) | (d) | **350**  **300**  **250**  **200**  **150**  **100**  **50**  **0**  **-50**  **-100**  **-150**  **-200**  **-250**  (e) |

**FIGURE 5.** Raman spectroscopy measurements (a) Raman spectrum at 3400 – 50 cm-1, (b) Raman spectrum at 200 – 50 cm-1, (c) Optic region of CsCu2I3-PS nanofibers, (d) Mapping analysis of CsCu2I3-PS nanofibers, (e) Results mapping of CsCu2I3-PS nanofibers with scale bar.

**X-Ray Diffraction (XRD)**

The crystal structures of CsCu2I3, polystyrene, and CsCu2I3-PS nanofibers were identified using XRD, as depicted in Fig. 6. Fig. 6a illustrates the typical crystal structures of polystyrene and CsCu2I3. The diffraction pattern obtained from CsCu2I3 matches well with the diffraction pattern of CsCu2I3 reported in previous studies and with crystallographic databases for the orthorhombic structure with space group Cmcm of CsCu2I3 (PDF#45-0076). The diffraction peaks corresponding to 2θ 10.7º, 13.4º, 21.9º, 26.1º, 27.1º, 42.9º, and 44º respectively align with the (110), (020), (220), (221), (040), (350), and (440) planes [3, 28]. In contrast, the XRD pattern of PS exhibits a characteristic amorphous pattern, typical of polymer materials. The XRD pattern reveals polystyrene, evidenced by two main peaks in the range of 7-12º and 17-21º. The first peak is associated with polymerization peaks correlated with intermolecular backbone-backbone correlation and side group size, related to the polymer chain arrangement absent in structures with small molar mass like styrene and benzene. The second peak corresponds to an amorphous halo correlated with van der Waals distance [19, 29]. In the CsCu2I3 composite material, a unique diffraction pattern is observed, dominated by amorphous patterns, yet exhibiting one peak with significant intensity at 2θ = 26.13º. This pattern closely resembles the diffraction pattern of pure CsCu2I3 at 2θ = 26.1º with the highest intensity, indicating successful integration of CsCu2I3 into polystyrene nanofibers. Additionally, the diffraction pattern reveals low-intensity peaks, still dominated by the amorphous state, attributed to polystyrene as the polymer substrate and rapid phase transition from solution to solid nanofibers during the electrospinning process. We further analyzed the sample by conducting Rietveld refinement (Fig. 6b), simultaneously considering two phases: the styrene phase and the CsCu2I3 phase. The refinement results indicate a good fit with the presence of two phases as references, yielding Rwp = 78.8, and Chisquare = 2.96.

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| (a) | (b) |

**FIGURE 6.** (a) XRD patterns, (b) Rietveld refinement of CsCu2I3-PS nanofibers.

**CONCLUSION**

CsCu2I3 embedded in polymer nanofibers presents new development potential for optoelectronic device applications. We successfully integrated CsCu2I3 with polystyrene into a unified material in the form of nanofibers. The fabrication of these nanofibers using electrospinning techniques requires careful consideration of factors influencing the uniformity of the fiber shape. Concentration, as one of the key parameters, indicates that higher concentrations result in more uniform and smoother fibers, and influence the formation of the cone jet obtained. This success is indicated by a reduction in peak intensity upon integration with CsCu2I3 and the appearance of characteristic peaks from CsCu2I3 in FTIR, Raman, and XRD analyses. Raman mapping also confirmed the presence of CsCu2I3 within the polystyrene nanofibers. This material shows promise for the development of novel optoelectronic devices, providing enhanced stability due to the polymer's protective coating on CsCu2I3.

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