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Photo-Rechargeable Electrochemical Supercapacitors Based on Two-Dimensional Hybrid Halide Perovskites

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ABSTRACT

Two-dimensional HP has recently emerged as new materials for future energy storage gadgets because of their superiorphotoactivity and mixed electronic-ionic conductivity. Due to the growing demand of self-powered and mobile energy, the suitability of the layered 2D HPs (Arg2MA2Sn3Cl10) for photo-rechargeable electrochemical supercapacitors was investigated in this study. The HPs were synthesized by inverse temperature crystallization method and analyzed by UV-visible spectroscopy, XRD and FESEM to verify the crystallinity, optical bandgap (3.67 eV) and morphology (layered morphology) of as-prepared HPs. Electrochemical property was examined in dark condition and illumination condition under CV and GCDmeasurement. The material displayed the photo-rechargeable ability considering the specific capacitanceof 49.09 F/g under illumination and 43.63 F/g in the dark at the scan rate of 10 mV/s. In addition, The densities of power and energyreached 20.55 Wh/kg and 80 W/kg respectively. The experimental results demonstrate the dual functionality of the 2D HPs as both promising energy storage material and photo-absorbing layer that make it suitable for photo-rechargeable SSC applications. This work opens the way for further investigation of hybrid halide perovskites for the purpose of combined systems for collecting and storing energyfor sustainable and self-sufficient power devices for portable electronics and IoT devices.

Keywords: *Hybrid Halide Perovskites, Two-Dimensional Materials, Photo-Rechargeable Supercapacitors, Energy Storage*

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1. INTRODUCTION

Autonomous wearable electronics without external power output connections are getting enormous demand due to the portability and autonomy required in applications. Energy conversion and energy storage is a core enabling technology for off-grid power supply, allowing

the energy demand-supply curve (commonly known as the duck curve) to be handled more effectively (Kumar, Ramesh, et al., 2022). The duck curve describes the mismatch between energy supply and energy demand (Dong, Liubing, et al., 2016 & Kanaujiya, Neha, et al., 2021). Energy storage and harvesting systems are typically decoupled in existing technologies for energy conversion and utilization, so that many demands have been made for technologies which merge these functions into a single unit (Wang, Liqun, et al., 2021). Supercapacitors have drawn the most interest of all energy storage devices due to their high density of power, high charge-discharge rate, and extended cycle lifetimes (Olabi, Abdul Ghani, et al., 2022). Although these are beneficial, their application in autonomous and portable systems is limited because they must be recharged often by an external power supply. To overcome this limitation, new techniques of energy harvesting directly into supercapacitors have been investigated, which led to electrochemical supercapacitors that can be photo-recharged (Chini, M. K. (2024). Quantum capacitance devices are a promising candidate for revolutionizing energy storage systems by removing the need for an external power supply and thus are suitable candidates for wearable, Internet of Things (IoT) and other portable electronic applications. Two-dimensional (2D) hybrid perovskites (HPs) are a new and class of materials with unusually synergistic optical and electrical features that make them interesting for photo-rechargeable applications. For optoelectronic applications (like solar cells), these materials are very promising because of their substantial light absorption coefficients, high charge carrier mobility, &tunable electronic properties (Yukta, et al., 2021 & Chini, Mrinmoy Kumar, et al., 2020). The interlamellar structure of 2D HPs provides better stability and allows separation of photogenerated charge carriers that are needed to achieve high efficiency for the photo-rechargeable devices (Kim, Eun-Bi, et al., 2021). Moreover, their tunability in thickness and composition at the time of synthesis allows for a desired set of properties to be selected that can meet a set of specified requirements for a given application (Sandhu, A., & Chini, M. K. (2024). The growth of two-dimensional (2D) HPs to be incorporated into supercapacitors provides a potentially promising option for the combining the dual power features that combine energy harvesting and storage into one device. This technology is a major step toward the next generation of energy systems that store solar energy efficiently, in a vertically integrated mode that delivers both services. Energy conversion devices can be a disruptive technology that can shift the state-of-the-art of self-sufficient and lightweight high energy source for those applications where conventional power supply mechanisms cannot be used (Shin, Joobee, et al., 2021). For example, energy dense power cells for wearable and Internet of things (IoT) systems are one of the key technology areas which can benefit from such new technology (Chini, M. K., & Chatterjee, S. (2017). From the materials perspective, the two-dimensional HPs have a list of properties which are suitable for use in the photo-rechargeable supercapacitor. Due to the layered structure, they have high optical absorption coefficient, high light harvesting efficiency, and large surface area for charge transfer and ion adsorption. In addition, the photoactivity of the 2D HP can be optimized based on their device application requirement, since their bandgap is tunable (Fu, Xifeng, et al., 2024). Recently, the combined excellent light and dark performances of this type of materials have been demonstrated using specific capacitance and energy density, which has shown its potential as a next-generation energy storage material (Kumar, Tanuj, et al. , (2023). Synthesis and characterization of 2D-HPs is also an extremely active area of research. High quality two-dimensional (2D) HPs with controlled morphology and composition have been prepared by inverse temperature crystallization techniques and from solution (Kong, Lingmei, et al., 2022). Importantly, these experimental studies help to understand the mechanism of their performance in photo-rechargeable systems. Besides the intrinsic properties, 2D HPs, are interesting materials that can be combined with additional resources to improve their functionality. For example, it is demonstrated that the insertion of conducting polymers and/or carbon-based materials leads to enhancement of the stability and the charge transfer property (Zhang, Fan, et al., 2023). Among these hybrid systems, the best characteristics of each component are combined to obtain better performance or enhanced traits in terms of specific capacitance, cycle life, and photo-recharge ability (Namsheer, K., & Rout, C. S. (2021). The idea of having energy storage and harvest within one device is not an entirely novel concept. Early attempts were made to integrate photovoltaic cells with batteries, but such systems tended to be bulky and inefficient, because the materials and processes were mismatched (Kumar, Tanuj, et al., 2023). The introduction of 2D HPs has resolved most of the above-mentioned challenges, and provided a small footprint and high-performance solution for integration of these functions. It is therefore now possible to design supercapacitorswhose self-charging recharged to a high extent when exposed to light, greatly mitigating the need for external power sources for charging (Chen, Dongdong, et al., 2017). Despite promising properties, issues of practical application of 2D HP based photo-rechargeable supercapacitors are left unsolved. Operational stability is an important topic, because two-dimensional HPs are prone to degradation when exposed to heat, moisture, and ultraviolet light (Lv, Jian, et al., 2018). Plasmonic devices of PSCs also suffer from stability issues, and therefore strategies including surface passivation, encapsulation, and lead-free perovskite materials are being pursued to improve their stability (Kumar, Tanuj, et al., 2023). Further, scalability and cost are important considerations that need to be addressed for commercial implementation of this technology (Cheng, P., Han, K., & Chen, J. (2022). Among them, the synthesis of 2D hybrid halide perovskites for photo-rechargeable supercapacitors holds great significance to widen the scope of energy storage technology (Chen, Tse-Wei, et al., 2020). Having these two functions the energy storage and harvesting built into the materials creates opportunities for new, more efficient, sustainable and portable energy conversion systems. Hence, future studies should address the issues of stability and scalability during explorations of novel material compositions and device architectures for enhanced device performance. Such a new field holds a great promise for revolutionizing the way we design energy systems which opens new perspective in cutting-edge applications of wearable electronics and internet of things (IoT) devices, etc. (Wang, Liqun, et al., 2021) & (Yang, Mingrui, et al., 2024). We shall discuss the synthesis,

morphological, and electrochemical characterizations in this studyof 2D hybrid halide perovskites ($Arg_2MA_2Sn_3Cl_{10}$) and their future applications in photo-rechargeable supercapacitors. We are exploring the dual functionality of this class of materials by using advanced fabrication techniques (inverse temperature crystallization followed by extensive electrochemical characterization in dark and light) to better understand the device behavior. In this study, we optimize the electrode composition and evaluate the energy storage and photo-rechargeability of the device. In this work, we aim to address fundamental challenges in the integration on a single platform of both energy harvesting and storage, and provide fundamental information on the performance, stability and feasibility of 2D HP-based photo-rechargeable supercapacitors.

2. EXPERIMENTAL

2.1 Chemicals

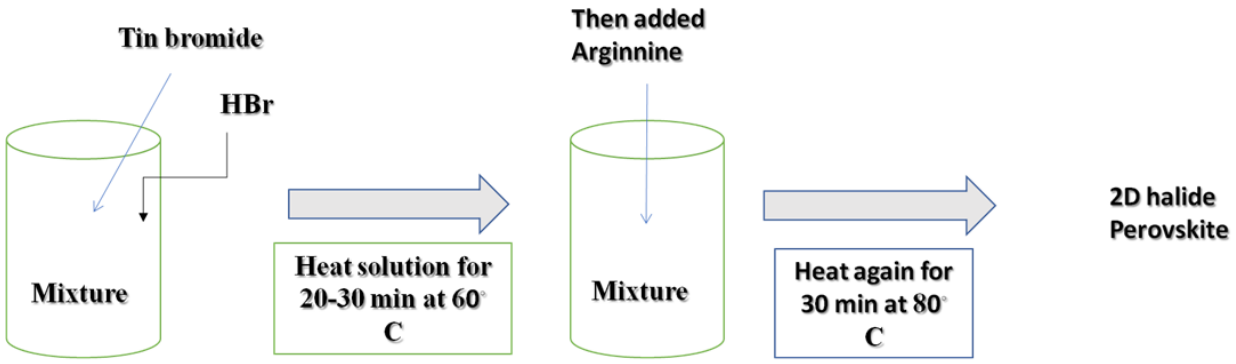
Photo-rechargeable materials used in this work include: i) arginine, ii) methylammonium chloride (CH3NH3Cl, 99.7%, TCI Chemicals), iii) tin chloride (SnCl2, 99.9%, Sigma-Aldrich) and iv) dimethylformamide (DMF, anhydrous 99.9%, Sigma-Aldrich). These are key precursors for the synthesis of the 2D hybrid halide perovskites ($Arg_2MA_2Sn_3Cl_{10}$). Also polyvinylidene fluoride (PVDF; Sigma-Aldrich) was used as a binder, and N-methyl-2-pyrrolidone (NMP; Sigma-Aldrich) was used as a solvent for the preparation of electrodes. Every chemical utilized was of analytical quality and didn't require any additional purification.

2.2 Electrode Preparation and Electrochemical Characterization

The 2D hybrid halide perovskite ($Arg_2MA_2Sn_3Cl_{10}$) was prepared by inverse temperature crystallization method. The method was accomplished by dissolving stoichiometric amounts of arginine, the methylammonium chloride and tin chloride in an anhydrous DMF solution under controlled conditions. Resulting solution heated gradually for crystallization of the crystals. After drying and filtering, the crystals were powdered into homogeneous powder for 15 - 20 min. This powder was used as an active material in the fabrication of the electrodes that follow. The electrodes were prepared by slurry method by mixing 70% of perovskite synthesized material with 15% activated carbon and 15% PVDF binder in NMP solvent. The mixture was kept under magnetic stirring continuously for 12 h to prepare a uniform viscous slurry. After the slurry had reached the desired consistency, the slurry was applied uniformly on the graphite sheets which served as the current collectors. The coated sheets were further dried in a vacuum at 70°C for about 12 hours to eliminate residual solvent and enhance adherence to the electrodes. A typical three-electrode arrangement was employed for electrochemical characterization. A platinum (Pt) electrode served as the counter electrode, while an Ag/AgCl electrode served as the reference electrode. The coated perovskite graphite sheet served as the working electrode. Dichloromethane was used to dissolve 0.1 M tetrabutylammoniumtetrafluoroborate solutions to create the electrolyte solution.The efficiency of the fabricated electrochemical cell was analyzed by CV and GCD measurements. To clarify the photo-rechargeable characteristics of 2D hybrid halide PSCs, both the dark measurement and illumination measurement were performed. The CV tests provided details on the electrochemical response and capacitive behaviour of a material, while the GCD tests provided details on the charge-discharge behaviours and energy storage efficiency of a material. In order to explain the dual performance of the synthesized 2D perovskites for both harvesting and storage, a comparative analysis was conducted between performance metrics as a function of different light irradiation conditions.

3. RESULTS AND DISCUSSIONS

3.1 Quantum Confined and Hybrid-state Optical Spectroscopy:



Scheme 1.Schematic for the synthesis of 2D HPs.

Quasi-two-dimensional HPs (2D HPs) $Arg_2MA_2Sn_3Cl_{10}$ (n = 3) (Arg_2DPs), which contain the bulky organic ligand, arginine shown in Scheme 1, were synthesized. The optical and morphological properties of these synthesized materials were verified by using UV-Visible spectroscopy, XRD and FESEM. According to Figure 1(a) and the corresponding onset absorption band is observed at 338 nm from the UV-Visible spectrum, It is established that the gap between the optical bands is 3.67 eV calculated by the following formula:

$$Eg = \frac{1240}{\lambda max}$$

This band gap agrees with values estimated for quasi-2D perovskites; verifying successful growth of these halide perovskites. The UV-Vis spectroscopic measurements were conducted in an anhydrous dimethylformamide (DMF) solvent, ensuring the stability of the sample during the study. Further structural characterization through XRD analysis (Figure 1(b)) confirmed the crystalline nature of the synthesized material. The XRD patterns exhibited sharp diffraction peaks at 2θ values of 30.1°, 34.5°, 37.6°, 48.1°, The minimum d-spacing of 1.59 Å indicates a well-ordered crystalline structure. However, the presence of minor peaks suggests a residual amount of unreacted reactants or a mixture of bulk (3D) perovskites along with the quasi-2D structure. These findings highlight the layered crystalline structure of the material, with predominant 2D characteristics. The FESEM images in Figure 1(c) provide a direct visual confirmation of the morphology of the synthesized $Arg_2MA_2Sn_3Cl_{10}$. The images distinctly display the layered architecture of the material, indicative of its quasi-2D structure. These layers are crucial for ensuring effective ion transport and interaction with the electrolyte during electrochemical applications. Overall, the structural and optical studies establish the successful synthesis of $Arg_2MA_2Sn_3Cl_{10}$ with desired properties suitable for energy storage applications.

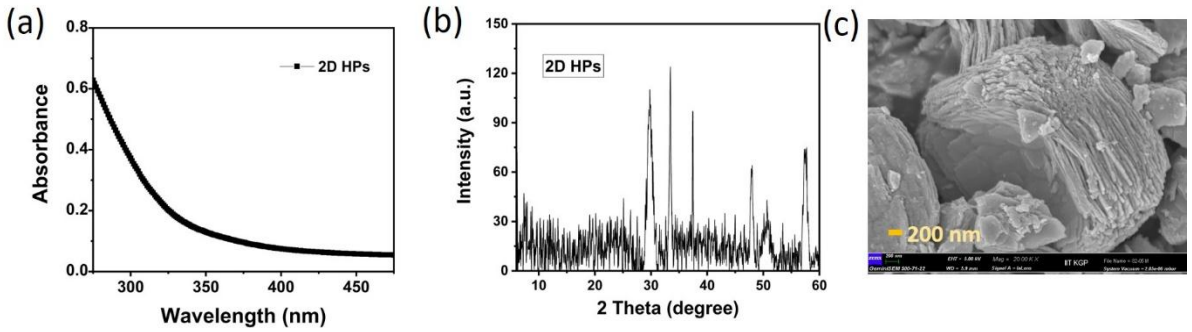


Figure 1. (a) UV-Visible spectrum, (b) PXRD and (c) FESEM images of $Arg_2MA_2Sn_3Cl_{10}$

3.2 Electrochemical Cell Performance

The electrochemical performance of the quasi-2D HPs was assessed under dark and light circumstances utilizing CV and GCD methods.

3.2.1. Cyclic Voltammetry Analysis

The CV experiments were carried out with the working electrode being the perovskite electrode at scan speeds between 10 and 100 mV/s. Figure 2 (a) illustrates the CV curves in the absence of light. With an increase in scan rate, the current correspondingly elevates, signifying the superior electrocatalytic activity of the material. The quasi-rectangular configuration of the CV curves indicates a combination of diffusion-limited faradaic reactions and surface redox mechanisms, indicative of pseudo-capacitive behavior. In Figure 2 (b), CV curves under dark as well as light conditions are presented a scan rate of 10 mV/s. Both conditions exhibit quasi-rectangular shapes, confirming the involvement of an EDLC mechanism along with pseudo-capacitance. Importantly, the CV curves under illumination show enhanced current density, signifying a photo-rechargeable response. This enhancement is attributed to the 2D perovskite structure's photogenerated charge carriers, which contribute to improved electrolyte-ion transport and interaction. Specific capacitance (Cs) values were calculated from the CV curves using the below mentioned formula:

$$Cs = \frac{\int IdV}{Vvm}$$

The capacitance values under varying scan rates are summarized in Table 1. Under dark conditions, the specific capacitance values decrease from 43.63 F/g to 11.36 F/g as the scan rate rises from 10 to 100 mV/s, reflecting limited increased scan rates for ion diffusion. Under light conditions, the specific capacitance at 10 mV/s increases to 49.09 F/g, further confirming the photo-responsive nature of the material. Figure 2 (c) illustrates the relationship between specific capacitance and scan rate. The declining trend in capacitance with increasing scan rate highlights the reduced interaction between the electrode material and electrolyte ions at higher scan rates, which is typical for such systems.

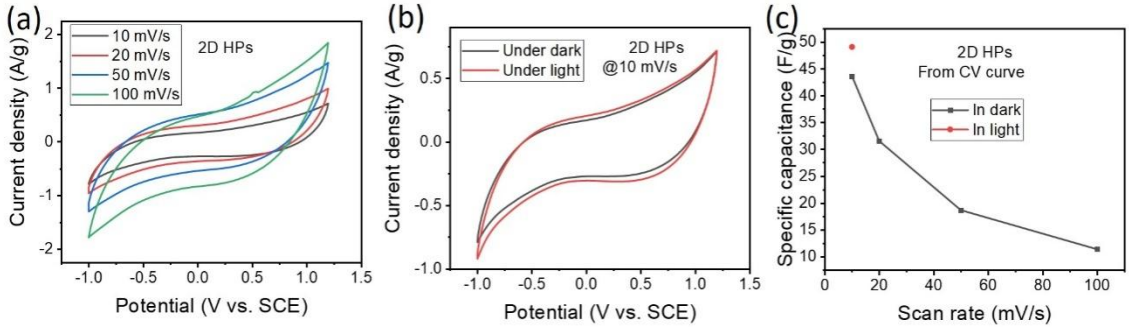


Figure 2.(a) 2D HPs' CV curves at various scan rates. (b) CVs of 2D HPs at 10 mV/s under dark &light conditions. (c) Calculated specific capacitance Vs Scan rates.

Table 1.Calculated specific capacitance values from CV

Condition	v (mV/s)	Area (AV/g)	Cs (F/g)
Under Dark	10	0.96	43.63
	20	1.39	31.54
	50	2.05	18.63
	100	2.50	11.36
Under Light	10	1.08	49.09

3.2.2. Galvanostatic Charge-Discharge Analysis

The quasi-2D HPs' electrochemical activity is further confirmed by the GCD measurements. The GCD curves at different current densities in the dark are displayed in Figure 3(a).The symmetric triangle shapes of the curves indicate high reversibility and minimal resistive losses, which are essential for supercapacitor performance. Figure 3 (b) compares the GCD curves at a current density of 0.1 A/g in both light and dark situations.The discharge time under illumination is significantly longer than that under dark conditions, resulting in an increased specific capacitance. This behavior reinforces the photo-rechargeable nature of the 2D HPs. Specific capacitance values were calculated from the GCD curves using the below equation and the results are tabulated in Table 2:

$$Cs = \frac{I * dt}{m * dV}$$

Under dark conditions, the specific capacitance values decrease from 49.38 F/g to 21.88 F/g as as the density of current rises from0.1 A/g to 0.5 A/g. Under light conditions, the capacitance at 0.1 A/g increases to 57.81 F/g, demonstrating a substantial improvement due to photo-induced charge generation. The relationship between current density and specific capacitanceis plotted in Figure 3 (c), which mirrors the trend observed in the CV analysis. At higher current densities, the reduced capacitance indicates weaker interaction between the electrode material and electrolyte ions, a common limitation in supercapacitor materials.

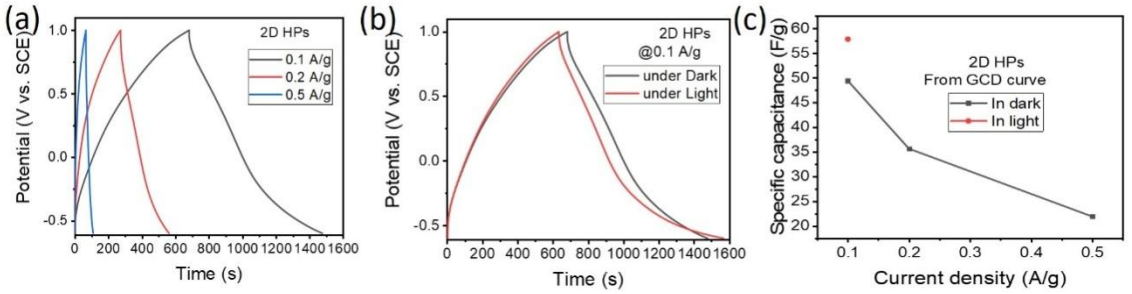


Figure 3.(a) Galvanostatic charge-discharge curves of 2D HPs at various current densities, (b) Charge-discharge curves under dark and light condition, and (c) Calculated specific capacitance with current densities.

Table 2.Calculated specific capacitance values from GCD

Condition	I (A/g)	Tc (s)[#]	Td (s) [#]	Cs (F/g)	Ŋ (%)[#]	ED (Wh/kg)	PD (W/kg)
Under Dark	0.1	680	790	49.38	116.18	17.55	80
	0.2	272	285	35.63	104.78	12.66	160
	0.5	65	65	21.88	107.69	7.78	400
Under Light	0.1	635	925	57.81	145.67	20.55	80

[#]Where, Tc and Td is charging and discharging time, Ŋ (%) is efficiency and equals to 100*(discharge time/charge time).

Alongside capacitance, energy density and power density were computed utilizing the GCD data. Under dark circumstances the energy & power densities are 17.55 Wh/kg & 80 W/kg, respectively, with a current density of 0.1 A/g.Under illumination conditions, the energy density was increased to 20.55 Wh/kg, which implies the improved energy storage ability of the material under illumination. The fact that electrochemical studies confirmed that the quasi-2D HPs show a very good capacitive behaviour for both EDLC and pseudo-capacitance components. At light condition, the material exhibits enhanced capacitance and energy density with a good photo-rechargeable response. This unique behavior combined with layered morphology and good crystallinity of the material makes this material contender for the upcoming generation of energy storage devices. Moreover, $Arg_2MA_2Sn_3Cl_{10}$ exhibits impressive efficiency in terms of power density, energy density, and specific capacitance, which can clearly show that $Arg_2MA_2Sn_3Cl_{10}$ is an excellent supercapacitor material. The continued evolution of the composition and morphology of the materials is expected to provide even superior electrochemical performance, and allow us to move from laboratory scale research to real world deployment for energy storage applications.

4. CONCLUSION

This paper, a systematic study of 2D HPs, whose structures are natural candidates for electrode materials in photo-rechargeable electrochemical supercapacitors, is presented. The lamellar $Arg_2MA_2Sn_3Cl_{10}$ compound was synthesized and characterized successfully to confirm the crystallinity, the optical properties and the morphology. With an estimated optical bandgap of 3.67 eV, the material has high absorptivity of light and therefore is suitable for photo-rechargeable applications. Good results of electrochemical characterization were obtained with specific capacitance of 49.09 Fg under light and 43.63 Fg under darkness at scan speedof 10 mV.s using CV. The quasi-rectiformalCV &symmetrical curves of charge and dischargeof the samples suggest the coexistence of faradaic pseudocapacitance and EDLC as the charge storing mechanism in the samples. In addition, the material showed good ion transport and stability characteristics as indicated by the preservation of specific capacitance with increasing scan speeds and current densities. Galvanostatic (current density 0.1 A/g) charge-discharge measurements of the composite electrodes verified the measured Power density and energy density of 20.55 Wh/kg or 80 W/kg, respectively, for the composite electrodes, demonstrating the potential of the use of 2D HPs as high-performance energy storage devices. Of particular interest, we find from the photo-rechargeable behavior observed under illumination, that 2D HPs exhibit a dual role as both light absorbers and energy storage materials. The promise of 2D HHPs as a novel class of materials for integrated and sustainable energy storage solutions is highlighted by this work. By combining energy harvesting and storage functionalities, these materials hold significant promise for advancing portable electronics, wearable devices, and IoT applications, where lightweight, efficient, and self sufficient power sources are essential. Improving the material composition should be the main goal of future research, exploring alternative electrolytes, and scaling up device fabrication to further enhance the practical applicability of these systems. We affirm that this manuscript is an original work, has never been published before, and no other journal is presently considering it for publication, or conference proceedings. All authors has been reviewed and approved the manuscript, and the order of authorship has been mutually agreed upon..

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