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Optimizing Methylammonium Lead Iodide Perovskite Synthesis for Solar Cells via Two-step Spin Coating with Mixed Solvents

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ABSTRACT

Methylammonium Lead Iodide (MAPbI3) has often been known to have a perovskite content and is widely employed in Perovskite Solar Cells (PSCs). However, PSCs suffer from rapid degradation due to their low stability and sensitivity to light, heat, humidity, and air. Hence, this project proposes a two-step spin coat method to synthesize MAPbI3 perovskites in an ambient condition using two different types of solvent (DMF only and a mixture of DMF and DMSO) which were prepared to examine the quality of the perovskite layer. The thin films were characterized to study the structural, optical properties, and surface morphology using XRD, UV-Vis spectroscopy, and SEM. The XRD results show that the perovskite layer prepared using a mixture of DMF and DMSO has a better crystallinity than DMF, even after the sample was kept for two days as the principal peaks confirm the complete formation of MAPbI3 perovskite film. UV-Vis showed that the perovskite layer prepared with the mixture of DMF and DMSO as the solvent has a higher absorbance than the perovskite layer prepared with DMF. The surface morphology of the MAPbI3 films consists of the formation of cubical crystals of uniform size with high compactness, as confirmed by the SEM analysis. However, after two days, both samples showed a degradation providing feedback on the occurred transition to PbI2 due to the contact with moisture and thermal stability. Moreover, the better stability of perovskite films was observed by a two-step than one-step process was demonstrated with comparatively good reproducibility.

Keywords: *Perovskite solar cell, DMF, DMSO, Two-step spin coat, $MAPbI_3$, Crystallinity, Ambient synthesis*

1. INTRODUCTION

Global energy consumption is on the rise due to a growing population. The depletion of non-renewable resources like fossil fuels is causing environmental issues. As a solution, there's a rising emphasis on renewable energy sources [1]. Solar energy is a crucial renewable resource that meets global energy demands and addresses fossil fuel depletion [2]. It offers numerous advantages, including abundant sunlight, low emissions compared to nuclear energy, and decentralized generation [3]. Solar cells, also known as photovoltaic cells, convert light into electricity via the photovoltaic effect. These cells have evolved into three generations, with perovskite solar cells (PSCs) being the third and rapidly advancing with high-power conversion efficiency. Their reported efficiency has increased from 3.8% to 22.1% in less than a decade [4, 5]. PSCs offer a significant advantage over traditional inorganic solar cells due to their low-cost, low-temperature fabrication using techniques like spin coating or dip coating [6, 7]. However, their stability requires an inert environment like a nitrogen-filled glove box, as exposure to light, heat, humidity, and air can degrade the perovskite material, reducing system efficiency [8–10].

Two methods for depositing $MAPbI_3$ thin films are commonly used: one-step spin coating and two-step spin coating. The two-step method provides better control over crystal development, resolving issues with stoichiometry and crystallinity compared to the one-step method [6]. This two-step spin coat approach is preferable for synthesizing perovskite thin films, as reported in [6 - 13]. For the two-step process, comprehensive conversion of the PbI2 layer to perovskite often requires a lengthy response time of over an hour. Some innovative methods involve manipulating the PbI2 film's shape to create a macroscopically porous scaffold, enhancing perovskite production efficiency [13, 14].

Various techniques, such as vapor-assisted optimization, solvent management, and thermal treatment interdiffusion, have been explored to enhance perovskite's intramolecular interaction and reactivity [13, 14]. The solvent design technique, as seen in studies by Han et al.

[15] and Seok et al. [16], has shown promise in improving perovskite transformation and device functionality [15 - 17]. In this study, we investigate the influence of two different solvents (DMF only and a mixture of DMF and DMSO) on the synthesis of $MAPbI_3$ thin films using a two-step spin coat method in an ambient condition. The samples are characterized using X-ray powder diffraction (XRD), UV-Vis spectroscopy, and Scanning electron microscopy (SEM) to examine their morphological, structural, and optical characteristics. The findings reveal that the DMSO/DMF solvent mix supports the directed development of intermediates into a porous "cubic-like" precursor film, leading to improved perovskite crystallinity and film quality. This study offers insights into producing high-performance PSCs using a two-step spin-coating method and understanding the solvent control mechanism for perovskite crystallization [17].

2. METHODOLOGY

2.1. The Perovskite Thin Film Preparation

Two different types of solvent, DMF (Method 1) and a mixture of DMF and DMSO (Method 2) were used for the preparation of the precursor solution for $MAPbI_3$. The thin films of $MAPbI_3$ were deposited on the glass substrate using the two-step spin coat method.

2.2. Chemicals

Lead 2 Iodide (PbI_2) CP, HmbG, Methylammonium iodide (MAI) 98%, Sigma Aldrich, N,N-Dimethylformamide (DMF), anhydrous≥99.8% Sigma Aldrich, Iso Propyl Alcohol (IPA), Dimethyl sulfoxide (DMSO), anhydrous, ≥99.9% Sigma Aldrich. The chemicals were used without any further purification.

2.3. Method 1 (Using DMF only)

The precursor solution was prepared by dissolving 1844 mg of PbI_2 in 4 ml of DMF and stirred for 3 hours at a temperature of 80 ºC to obtain a clear yellow solution. Next, 70 mg of MAI was dissolved in 4 ml of IPA and stirred for 3 hours to obtain a clear solution. The illustration of the preparation of precursor solutions using a two-step method is shown in Figure 1.

The PbI2 solution was heated at a temperature of 80 ºC until it completely dissolved before starting the deposition process. On the other hand, the glass substrate was heated at a temperature of 100 ºC for 15 minutes. To reduce a temperature drop during the spin coat process, the preheated stage is crucial. Then, the hot PbI2 solution was immediately dropped on the hot glass substrate and spin-coated at 4000 rpm for 40 seconds. The glass of PbI2 thin film was then dried for 10 minutes at 80 ºC on a hot plate. Subsequently, the MAI solution was dropped and spun at 4000 rpm for 40 seconds on the PbI2 thin film to form the perovskite thin film. Finally, the thin film is dried at 100ºC for 10 minutes on a hot plate to form a perfect perovskite film. All the preparation and deposited processes were entirely conducted in ambient air conditions. Figure 2 shows the summary of the deposited process of $MAPbI_3$ using the two-step spin coat method.

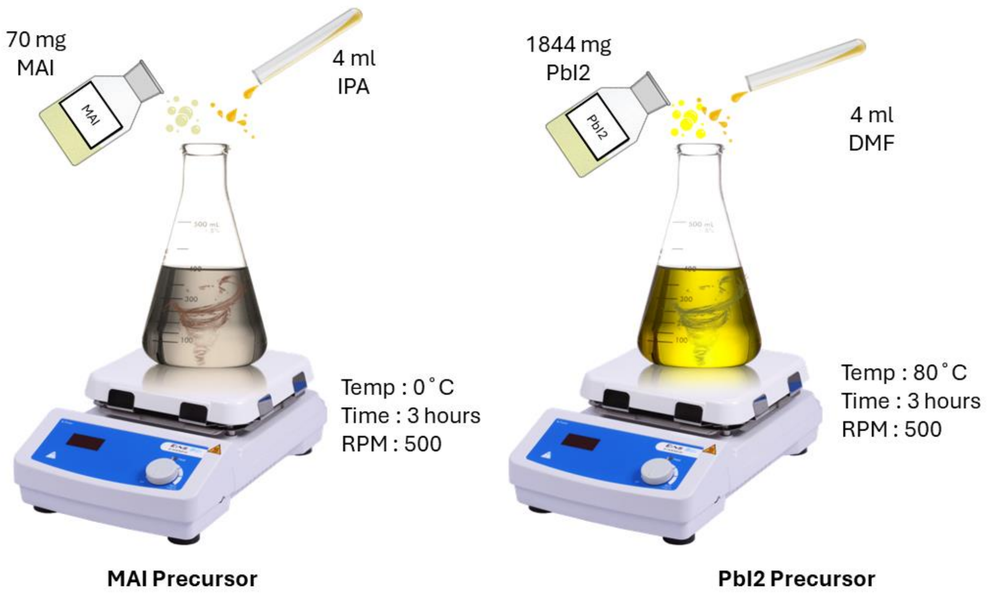


Figure 1. Steps to prepare the precursor solution of PbI2 and MAI using Method 1

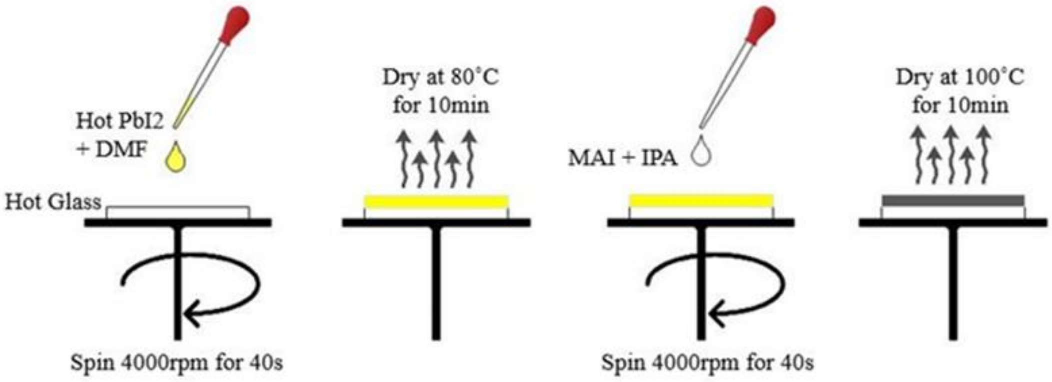
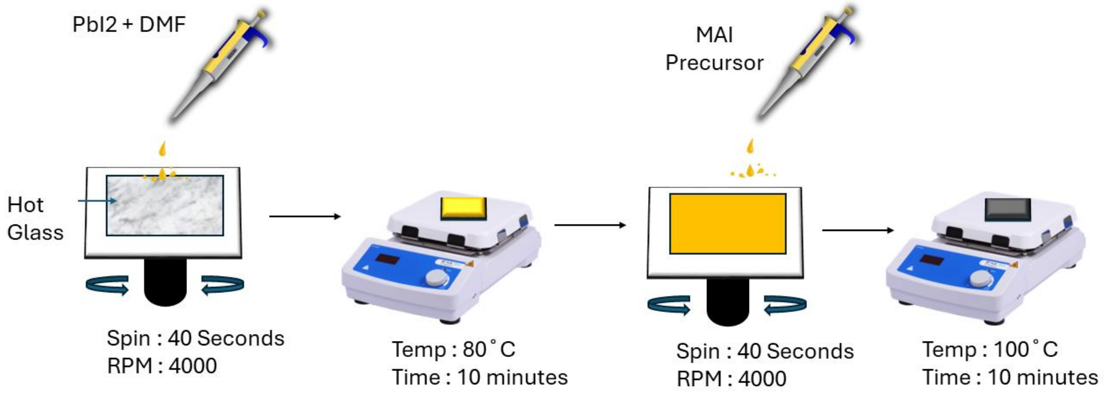


Figure 2. The deposition process of $MAPbI_3$ using the two-step spin coat method on a glass substrate

Method 1 (Using DMF only)



Method 2 (Using a mixture of DMF and DMSO)

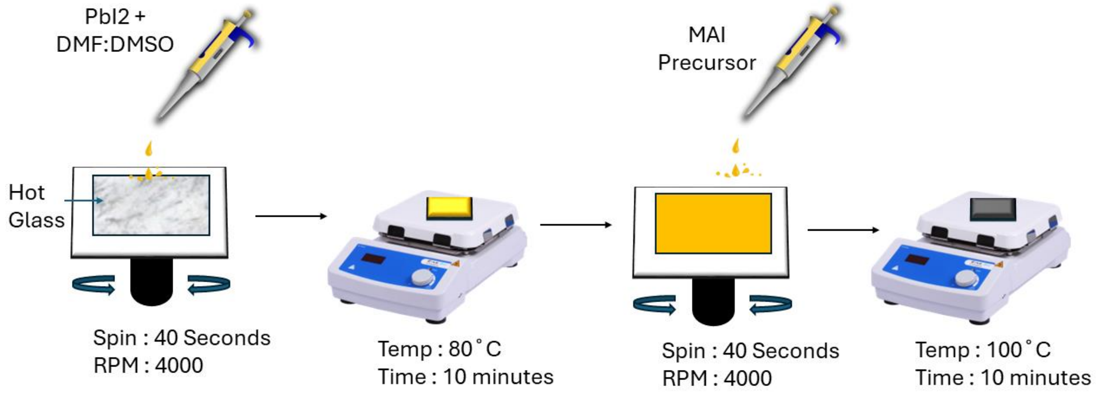


Figure 3. Steps to prepare the precursor solution and deposition process of $MAPbI_3$ using Method 1 and Method 2

2.4. Method 2 (Using a Mixture of DMF and DMSO)

This method dissolved a precursor solution of 70 mg of MAI in 4 ml of IPA. In addition, 1844 mg of PbI_2 was dissolved in 3.2 ml of DMF and 0.8 ml of DMSO. The ratio of DMF:DMSO is 4:1 for a 4 ml solution. The PbI_2 solution in DMF and DMSO will also become clear yellowish after stirring for 3 hours with a temperature of 80 ºC. The deposition process of $MAPbI_3$ using the two-step spin coat method on a glass substrate for this method is the same as the process in method one.

2.5. Comparison of Method Deposition

Table 1. J-V parameters of the $MAPbI_3$ with different volumes of DMF by two-step spin coat method [24]

	Jsc(mA/cm2)	Voc(mV)	FF	PCE (%)
w/o DMF	20.36	1051.2	0.642	13.743
1% DMF	23.15	1070.2	0.776	19.231
2% DMF	23.72	1075.4	0.788	20.108
4% DMF	18.73	885.7	0.466	7.724

Table 2. The lifetime of charge carriers for perovskite films prepared with different amounts of DMF additives by the double exponential fitting of PL data. τ1 is for fast transient and τ2 is for slow transient [24]

	w/o DMF	1% DMF	2% DMF	4% DMF
τ1(ns)	32.8	26.2	35.6	5.6
τ2(ns)	91.0	118.5	182.1	49.2

2.6. Sample Deposition of $MAPbI_3$ Thin Films

The two-step spin coat technique was used to implement the perovskite thin films. The prepared solution mentioned in Method I and Method II was dropped onto a clean glass substrate. Then, the sample is directly heated after deposition of PbI2 or indirectly, where the sample is heated after letting for two minutes after depositing PbI2. The sample is then divided into two following the time to store the sample, either one or two days. Table 3 shows the summary of the deposition process of perovskite thin films.

Table 3. Sample deposition of $MAPbI_3$ thin films using the two-step spin coat method

Sample	Precursor solution	Direct/Indirect after deposit PbI_2 solution	Time to store the sample
1	MAI + IPA PbI_2 + DMF	Direct	1 day
2		Direct	2 days
3		Indirect	1 day
4		Indirect	2 days
5	MAI +IPA PbI_2 + DMF+DMSO	Direct	1 day
6		Direct	2 days
7		Indirect	1 day
8		Indirect	2 days

3. CHARACTERIZATION RESULTS

X-ray diffraction analysis (XRD) was used to determine the crystallographic structure of $MAPbI_3$ perovskite material. Scanning Electron Microscope (SEM) was used to observe the surface morphology of the materials. Lastly, UV-Vis Spectroscopy was used to measure the optical properties and band gap of $MAPbI_3$ perovskite.

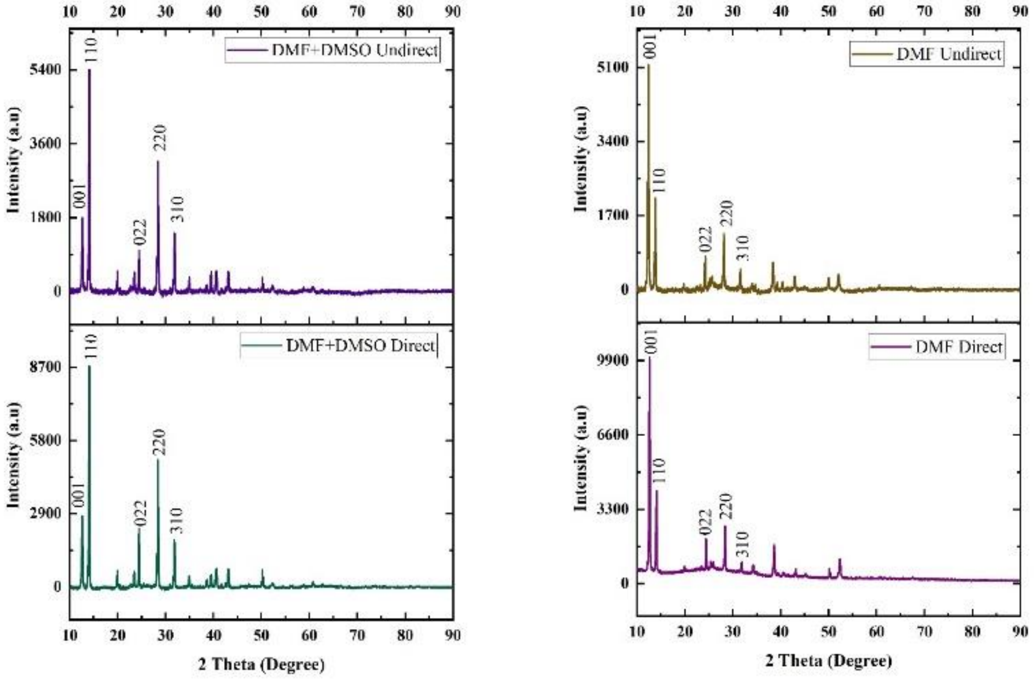
3.1. XRD Measurements

The XRD measurements with λ= 1.54A were done on the synthesized $MAPbI_3$ thin films to verify the formation of $MAPbI_3$ perovskites. The intensity data were collected over a range of 2θ from 10º to 90º. Figure 4(a) shows the XRD results of $MAPbI_3$ thin films using a DMF and DMSO solvent mixture after one day for both indirect and direct methods. As can be seen from Figure 4, both samples have principal peaks of $MAPbI_3$ at 2θ of 14.08º (110), 28.37º (220), and 31.81º (310). These three principal peaks confirm the complete formation of $MAPbI_3$ perovskite film as reported by P. Basumatary et al. [18]. The higher intensity at the sample with a mixture of DMF and DMSO Direct shows a complete crystallization of the sample and high crystalline size when directly heated after depositing the PbI2 solution. The heated process should be done immediately in order to have high crystallization perovskite, as reported by Y. Cheng et al. [19]. In addition, both samples have a small number of PbI2 phases, with the peak at 12.73º corresponding to the PbI2 001 lattice plane. This indicates that a few PbI2 conversions are incomplete during the deposited process [20].

Figure 4(b) shows the XRD results of $MAPbI_3$ thin films using a DMF solvent after one day for both indirect and direct methods. From the figure, the existence of the principal peaks of $MAPbI_3$ at 2θ of 14.08º (110), 28.37º (220), and 31.81º (310) confirm the complete formation of $MAPbI_3$ perovskite film. However, the highest intensity was at 12.73º (001), which shows a high number of PbI2 exists in the sample. Therefore, only a little complete perovskite formation happens when using DMF solvent as a precursor solution. The high intensity of the graph DMF Direct shows that the sample has high crystallinity if the sample is directly heated after the deposited process. Therefore, it is crucial to directly heat the sample after the first step deposited process to have high crystallinity.

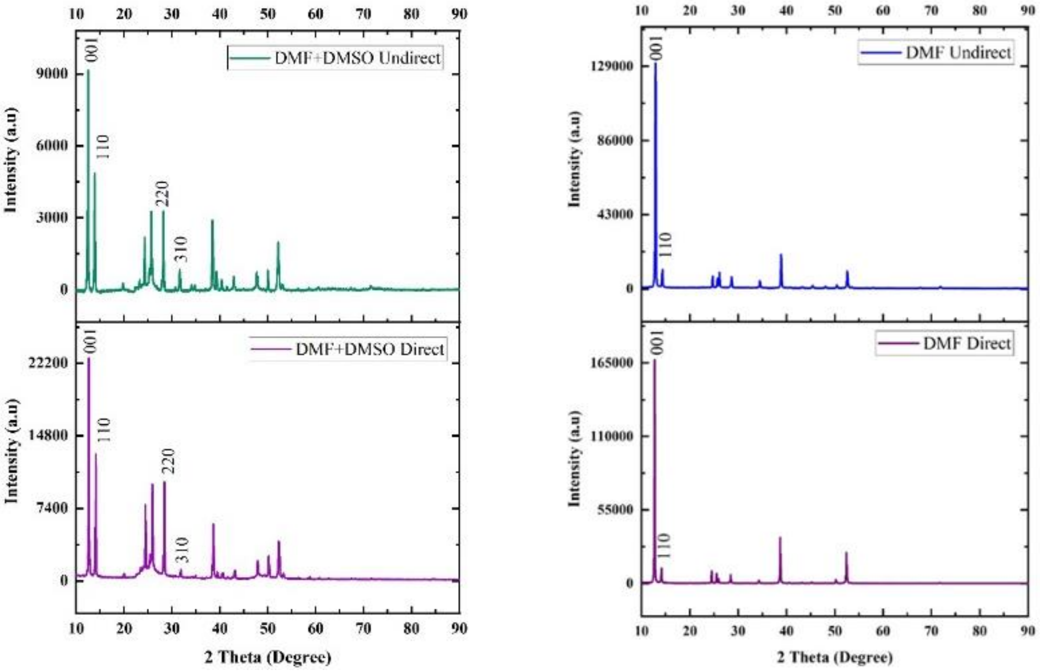
Figure 5 (a) shows the XRD results of $MAPbI_3$ thin films using a DMF and DMSO solvent mixture after two days. As can be seen from Figure 5 (a), the principal peaks of $MAPbI_3$ at 2θ of 14.08º (110), 28.37º (220), and 31.81º (310) still exist, which confirms the complete formation of $MAPbI_3$ perovskite film. However, both samples have a high number of PbI_2 phases, with the peak at 12.73º corresponding to the PbI_2 001 lattice plane. This indicates that the sample provides feedback on the occurred transition to PbI_2. This means the sample is already degraded. When MA-based perovskites were exposed to the environment, the principal degradation mechanism was attributed to the catalytic reduction of compounds in

PbI_2, CH_3NH_2, and HI [1]. Figure 5 (b) shows the XRD results of $MAPbI_3$ thin films using a DMF as solvent after two days. Only one principal peak of $MAPbI_3$ at 2θ of 14.08º (110) can be seen in the figure. This indicates that there is no formation of $MAPbI_3$ perovskite left in the sample. The sample is degraded to PbI_2 as there is a high intensity of PbI_2 001 lattice plane at 2θ of 12.73º.



(a) (b)

Figure 4. X-ray diffraction patterns of $MAPbI_3$ thin films after one day (a) using a mixture of DMF and DMSO solvent (b) using DMF only



(a) (b)

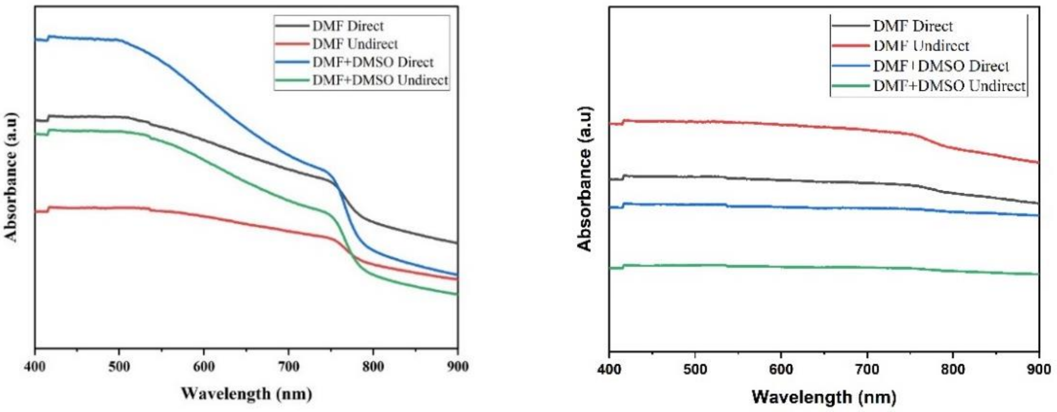
Figure 5. X-ray diffraction patterns of $MAPbI_3$ thin films after two days (a) using a mixture of DMF and DMSO solvent (b) using DMF only

3.2. UV-Vis Spectroscopy Analysis

The optical properties of the $MAPbI_3$ thin films were determined using UV-Vis analysis. The region was taken at wavelength 400-900 nm as in the previous paper [18]. Figure 6(a) shows the UV-Vis absorption spectrum of the $MAPbI_3$ thin films after one day. All the samples have a sharp absorption edge at ~760 nm wavelength, similar to MAPbI3 thin films reported in P. Basumatary et al. [18]. The absorption edge nearly occupied the full visible range, which corresponds to the basic property of $MAPbI_3$ perovskite [22]. Figure 6(b) shows the optical properties of the $MAPbI_3$ thin films after two days. The region was also taken at wavelength 400-900 nm. The samples deposited by using DMF have a sharp absorption edge at ~760 nm wavelength, similar to $MAPbI_3$ thin films reported in P. Basumatary et al. [18]. Meanwhile, the $MAPbI_3$ thin films deposited using a mixture of DMF and DMSO seem to be straight lines without any absorption edge. There is no observation found in any previous studies showing the same spectrum.

3.3. SEM Analysis

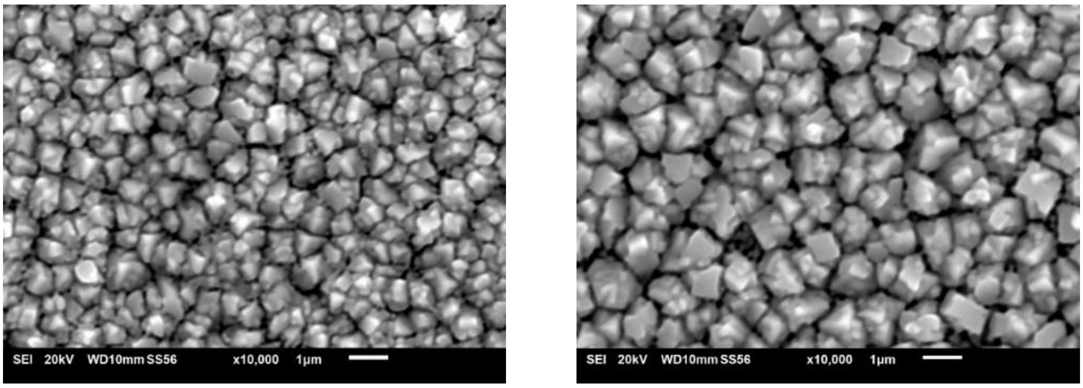
SEM analysis was used to study the surface morphology and grain sizes of $MAPbI_3$ thin films deposited using different solvents (DMF and a mixture of DMF and DMSO). Figure 7(a) and (b) show the SEM image of $MAPbI_3$ with magnifications of 10,000 for $MAPbI_3$ thin films prepared using DMF solvent and a mixture of DMF and DMSO solvent, respectively. The results show that both the samples exhibit similar cuboid crystals, as reported by H. Wei et al. [20]. In addition, the grains are relatively uniform in size with high compactness. Figure 8(a) and (b) show the analysis from image J on the perovskites layer's crystals size with DMF is smaller than the perovskites layer with a mixture of DMF and DMSO with an average of 0.560 um and 0.953 um, respectively.



(a)

(b)

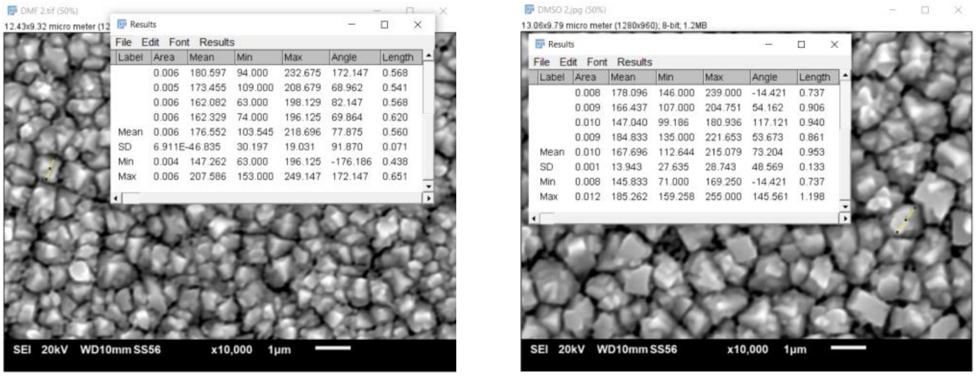
Figure 6. (a) UV-Vis absorption spectrum of the $MAPbI_3$ thin films after one day (b) UV-Vis absorption spectrum of the $MAPbI_3$ thin films after two days



(a)

(b)

Figure 7. Magnification 10.00kX of directly heated (a) $MAPbI_3$ thin films using DMF solvent, and (b) $MAPbI_3$ thin films using a mixture of DMF and DMSO solvent



Label	Area	Mean	Min	Max	Angle	Length
	0.006	180.597	94.000	232.675	172.147	0.568
	0.005	173.455	109.000	208.679	68.962	0.541
	0.006	162.082	63.000	198.129	82.147	0.568
	0.006	162.329	74.000	196.125	69.864	0.620
Mean	0.006	176.552	103.545	218.696	77.875	0.560
SD	6.911E-46.835		30.197	19.031	91.870	0.071
Min	0.004	147.262	63.000	196.125	-176.186	0.438
Max	0.006	207.586	153.000	249.147	172.147	0.651

Label	Area	Mean	Min	Max	Angle	Length
	0.008	178.096	146.000	239.000	-14.421	0.737
	0.009	166.437	107.000	204.751	54.162	0.906
	0.010	147.040	99.186	180.936	117.121	0.940
	0.009	184.833	135.000	221.653	53.673	0.861
Mean	0.010	167.696	112.644	215.079	73.204	0.953
SD	0.001	13.943	27.635	28.743	48.569	0.133
Min	0.008	145.833	71.000	169.250	-14.421	0.737
Max	0.012	185.262	159.258	255.000	145.561	1.198

(a) (b)

Figure 8. Crystal size of directly heated (a) $MAPbI_3$ thin films using DMF solvent, and (b) $MAPbI_3$ thin films using a mixture of DMF and DMSO solvent

4. EXPERIMENTAL RESULTS

4.1. The Degradation of $MAPbI_3$ Thin Films using a Mixture of Solvent

According to recent research, power conversion efficiency (PCE) for organometal halide perovskite ($MAPbI_3$) solar cells, often known as perovskite solar cells or PSCs, ranges from 3% to 21%. Nevertheless, it was noted that following production, efficiency values in PSCs significantly decline [25]. We refer to these progressive reductions in efficiency as aging, metastability, or instability. The sources of these reductions have been suggested to be interfacial interactions and stressors such as oxygen, water vapour, and UV radiation. Specific investigations through the studies on the decrease in efficiency illustrate that if multiple stress factors were present in the measurements at the same time, the stress factor would not be able to fully explain the outcome [26–31]. Therefore, it's unclear which threat has a higher correlation with degradation.

A significant observation is made regarding the degradation of $MAPbI_3$ thin films. Over a span of two weeks, these films undergo a noticeable transformation. Initially, they exhibit a smooth brownish-black appearance in Figure 9, which indicates the successful formation of $MAPbI_3$ perovskite through a two-step spin-coating process. However, this seemingly promising beginning takes a turnover time. After the first week, these films begin to show signs of degradation, gradually shifting to a yellowish hue, as depicted in Figure 10(a). The transformation becomes even more apparent after two weeks, when the $MAPbI_3$ perovskite has undergone complete degradation, giving way to the formation of PbI2 thin films, as shown in Figure 10(b). This sequence of events serves as a critical reference point for our discussion on the implications of perovskite degradation, particularly within the context of solar cell longevity. The distinct stages of degradation, marked by the change in colour and composition not only demonstrate the vulnerability of perovskite materials to environmental factors but also raise crucial questions about the practicality and durability of perovskite-based solar cells over extended periods.

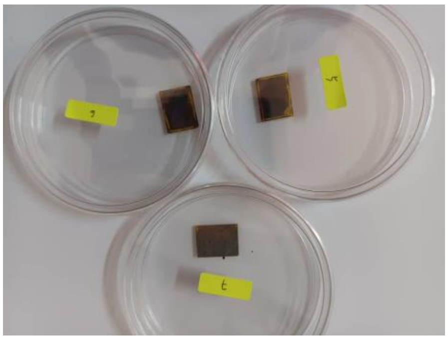
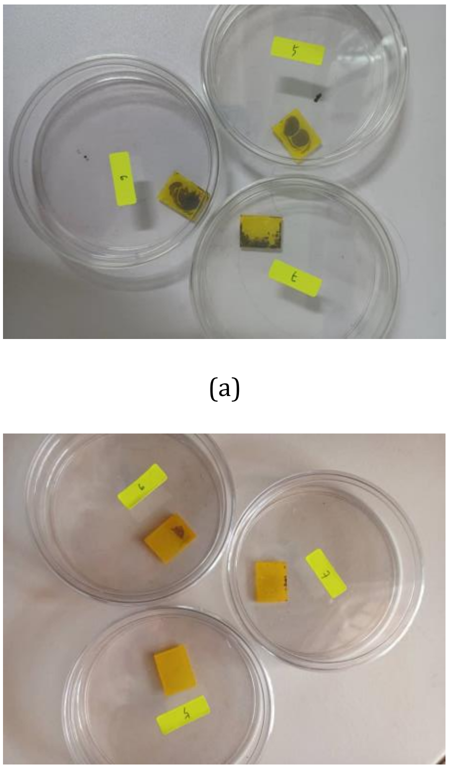


Figure 9. Sample $MAPbI_3$ after the deposition process



(a)

(b)

Figure 10. Sample $MAPbI_3$ thin films after (a) one week, and (b) two weeks

Future research is being conducted in numerous promising areas for preventing the degradation of perovskite materials in solar cells. These include the creation of more durable perovskite compositions through material modification and advanced encapsulation techniques intended to protect perovskite solar cells from external elements like moisture and oxygen. In order to improve perovskite stability, researchers are examining the impacts of alloying and doping as well as passivation layers as a potential extra barrier. An improved comprehension of degradation variables is made possible through environmental testing and in-situ monitoring. For practical application, long-term field testing and novel encapsulating materials are also being investigated.

5. CONCLUSION

In this work, the synthesis of $MAPbI_3$ perovskite was successfully coated on a glass substrate using a two-step spin coat method in an ambient condition. The $MAPbI_3$ thin films were investigated using XRD, UV-Vis spectroscopy, and SEM to see the structure properties, optical properties, and surface morphology. XRD analysis indicated that the $MAPbI_3$ thin films with DMF and DMSO solvent after one day showed the highest crystallization with principal peaks at 2θ of 14.08º (110), 28.37º (220), and 31.81º (310) which confirm the complete formation of $MAPbI_3$ perovskite film. UV-Vis showed that the layer of perovskite prepared with the mixture of DMF and DMSO as the solvent has a higher absorbance than the perovskite layer prepared with DMF. The SEM analysis shows that the samples exhibit similar cuboid crystals with relatively uniform grains in size with high compactness. Overall, it was found that the samples prepared with a mixture of DMF and DMSO had better results than using DMF only with high absorption and high crystallinity structure. In conclusion, $MAPbI_3$ perovskite was successfully synthesized in an ambient condition. However, the perovskite layer degrades rapidly in a short time and fully degrades after two weeks. Thus, the perovskite layer's stability remains an issue in the development of long-term stability PSCs.

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