2021

Optimizing the Purification Process for Microwave Synthesized Lead Halide Perovskites

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INTRODUCTION

Lead halide perovskites (LHPs) have become a promising optoelectronic material due to their bright photoluminescence, low manufacturing cost, and application in solar cells and light emitting diodes (LEDs). Their quantum yield (QY) is a metric used to quantify the efficiency by which they absorb and emit light, defined as the ratio of number of photons absorbed to those emitted. Currently there are several commonly used routes for synthesizing LHPs, including the hot injection (HI) and room temperature supersaturated recrystallization (SR) methods. Each technique presents its own challenges in the form of high operating temperatures, low stability of resulting LHPs, or lack of precise control over the synthesis process.

Developments have been made toward a new synthesis technique, using a microwave chamber to produce highly stable and luminescent perovskites. This 'microwave synthesis' allows for the precise control of operating parameters while being simpler than traditional methods since it is a single pot technique. In order to fully optimize a Microwave Synthesis for LHPs, work must be done to improve the purification procedure. Purification is a necessary step in the synthesis of LHPs as it allows for better analysis of photoluminescent properties and can even improve overall QY. Herein, we attempt to further optimize perovskites produced via Microwave Synthesis by analyzing their different purification techniques to determine which produces the most luminescent QDs.

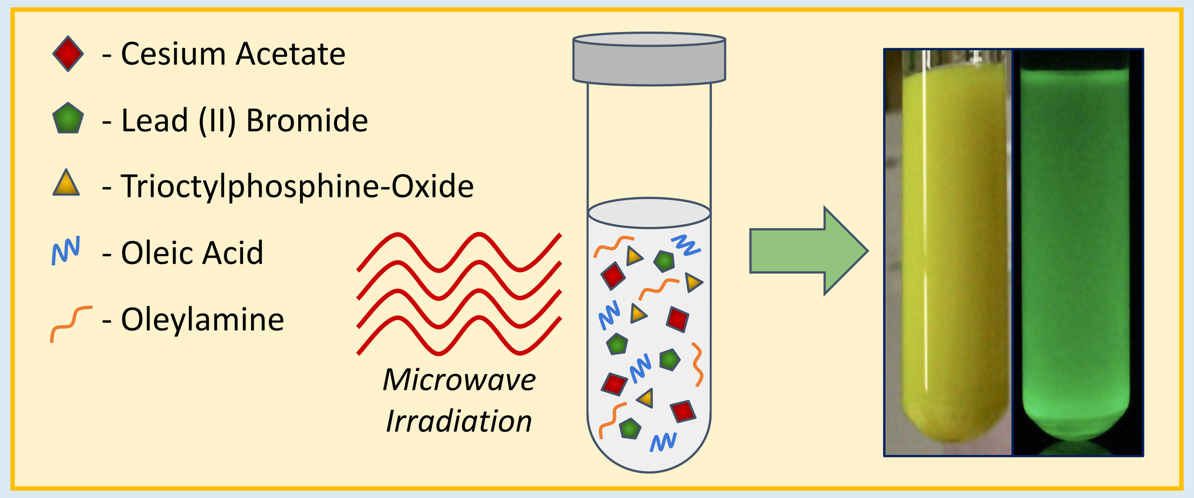
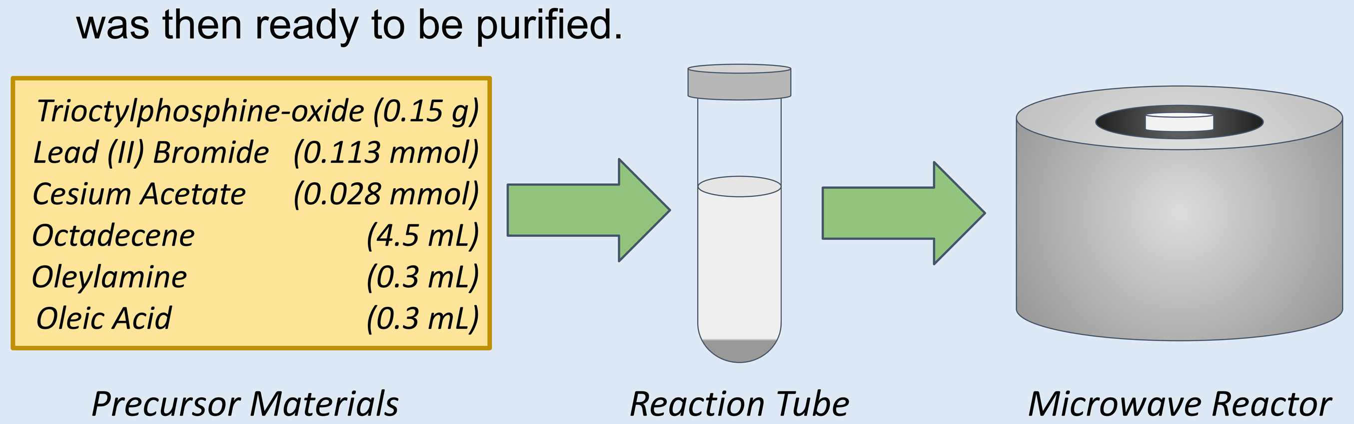


Figure 1. overview of synthesis procedure with pictures of resulting perovskites

METHODS

➔ **Microwave Synthesis of Lead Halide Perovskites**

A typical experiment involved combining all the precursor materials into a 10 mL reaction tube, then placing the tube into a microwave reactor at 160°C for 5 minutes. The resulting solution was then ready to be purified.



➔ **Purification of Lead Halide Perovskites**

Purification was done by centrifuging a certain ratio of perovskite solution to antisolvent solution, then drying under argon gas and redispersing in the desired solvent (toluene).

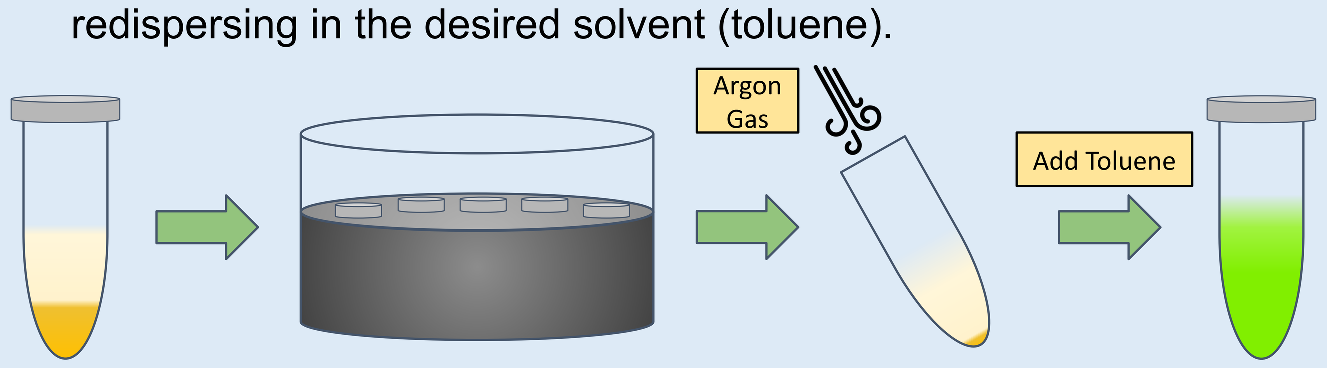


Figure 2. Microwave synthesis procedure. Perovskites with anti-solvents are placed in centrifuge. After drying, they are then redispersed.

Microwave-Synthesized lead halide perovskites result in the **highest quantum yield** when cleaned with **Acetone**

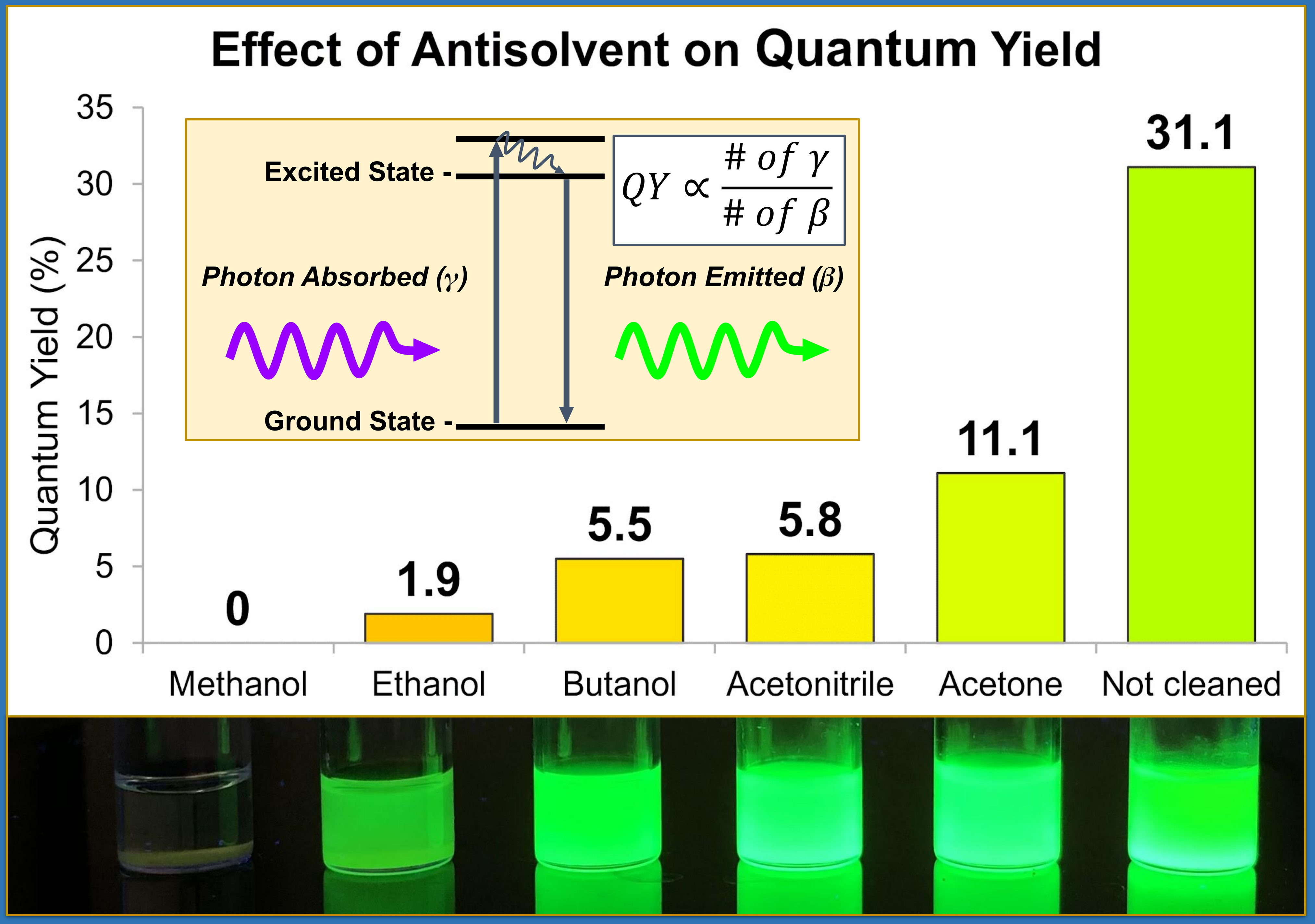


Figure 3: Comparison of resulting quantum yields from cleaning with different antisolvents. Acetone resulted in the highest yield. Methanol was incompatible resulting in a 0% yield.

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RESULTS

Acetonitrile was predicted to be the best cleaning agent, based on literature precedence.[1] However, Acetone consistently produced the highest yield in the as-prepared LHPs (Fig. 3). Moreover, methanol was entirely incompatible as a cleaning agent. Upon making this determination, several samples with varying ratios of sample to antisolvent were cleaned using acetone to determine which resulted in the best yield.

Figure 4. Normalized Photoluminescence for Perovskites Cleaned With Acetone

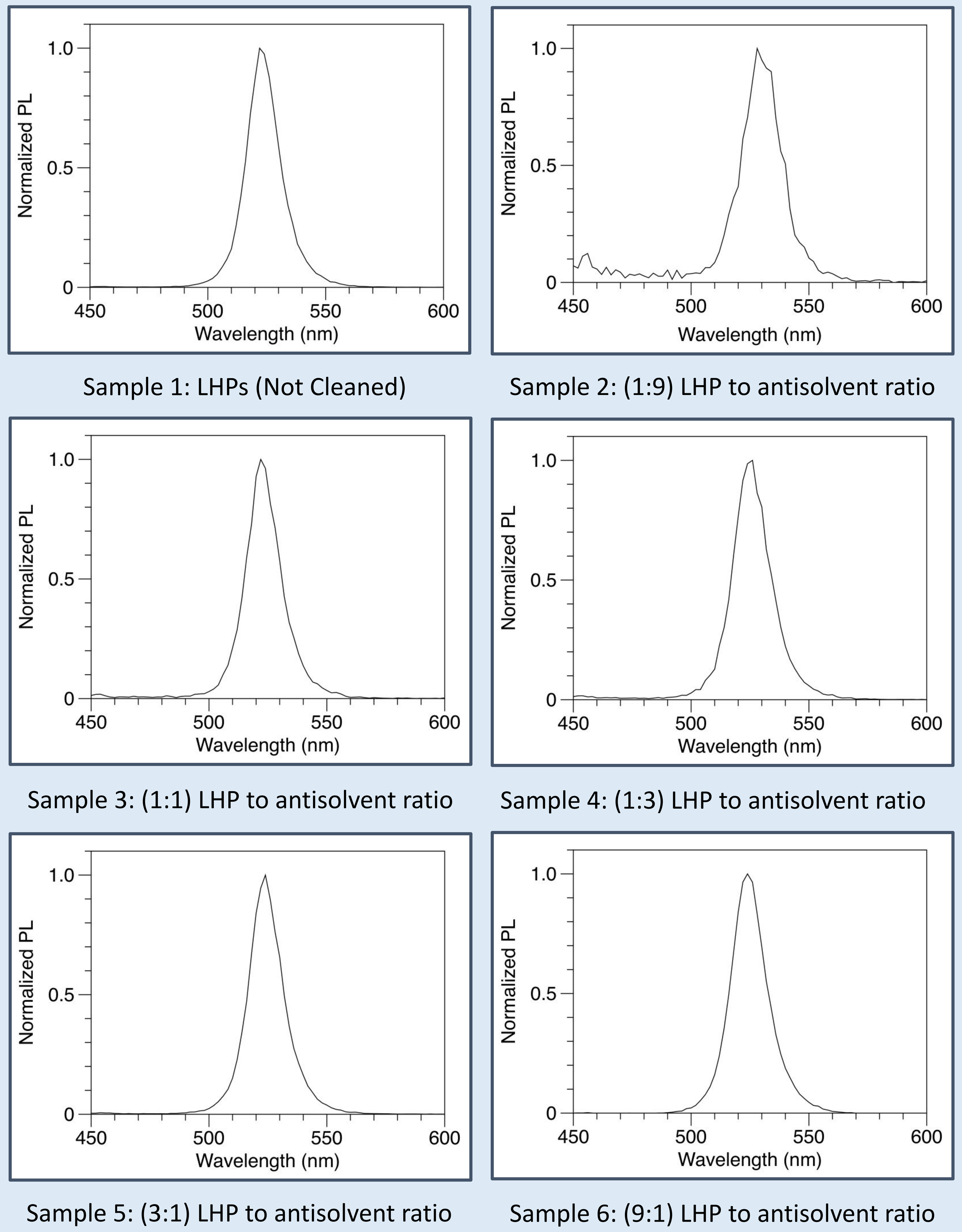

Table 1. Comparing Quantum Yields of LHPs Cleaned with Varying Ratios of Acetone

Sample	Ratio of Solution : Antisolvent	Quantum Yield (%)
Not Cleaned	1 mL : 0 mL	29.3
Sample 2	0.1 mL : 0.9 mL	1.8
Sample 3	0.5 mL : 0.5 mL	8.7
Sample 4	0.25 mL : 0.75 mL	14.4
Sample 5	0.75 mL : 0.25 mL	24.8
Sample 6	0.9 mL : 0.1 mL	**30.8**

CONCLUSION

Microwave Synthesized $CsPbBr_3$ LHPs result in the highest quantum yield when cleaned with acetone at a 9:1 ratio (Figure 4, Table 1). Perovskites produced this way managed to achieve yields higher than their non-cleaned counterparts. Further work must be done to test the yield resulting from varying the ratio of solution to antisolvent with different antisolvents.