Supplementary Information :

High-Throughput Workflow for the Synthesis of CdSe Nanocrystals Using a Sonochemical Materials Acceleration Platform

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1 Sonochemical Materials Acceleration Platform

1.1 Design Goals

The design of a custom autonomous platform was deemed necessary to significantly scale the volume of conducted experiments. This platform, called the sonication station, provides a repeatable means of sonicating batches of samples according to a user specification.

The platform largely derives from an existing open source CNC platform, Jubilee, which emerged as an ideal choice as a starting point from which to design the sonication station. This is largely due to the stock platform's automatic tool-changing capabilities and means of customization through the design of custom tools and bed plates. The project itself also features an active Discord community that could provide further troubleshooting assistance.

1.2 Jubilee Platform Modifications

The stock Jubilee platform was modified with three key changes: (1) a custom bed plate for handling SLAS microplates; (2) two custom tools, a sonicator probe and a camera for registration; and (3) a software package written in Python to provide a simplified script interface to the platform.

A custom bed was designed to accommodate six SLAS-style microplates. Each location features a preload element such that microplates can be installed and removed in a spatially repeatable manner. Consequently, scripts that operate on the same type of labware may be run repeatedly without any realignments. The bed's construction consists of both laser-cut and off-the-shelf components, and it is released as open source with a bill-of-materials (BOM) and set of assembly instructions in the project repository.

The Jubilee platform's documentation includes a mechanical design pattern standard by which tools designed to this standard can be readily mounted into the carriage. The sonicator probe tool is a Q125 sonicator with a 3D printed housing designed to this standard. It enables the platform to sonicate various well locations on the bed plate (x/y axes) with the tip submerged to a specified depth (z axis). The camera tool consists of a downward-facing imaging sensor (OV5647) and lens also designed to this standard. It serves as a visual assist, enabling the user to register the location of the labware relative to the platform by identifying features on the bed plate with the camera's video feed.

The Python package encapsulates platform-specific details and provides a simplified script-like interface to the Sonication Station. It also enables scripts to double as a reproducible record of the actions performed on various samples in one or more microplates.

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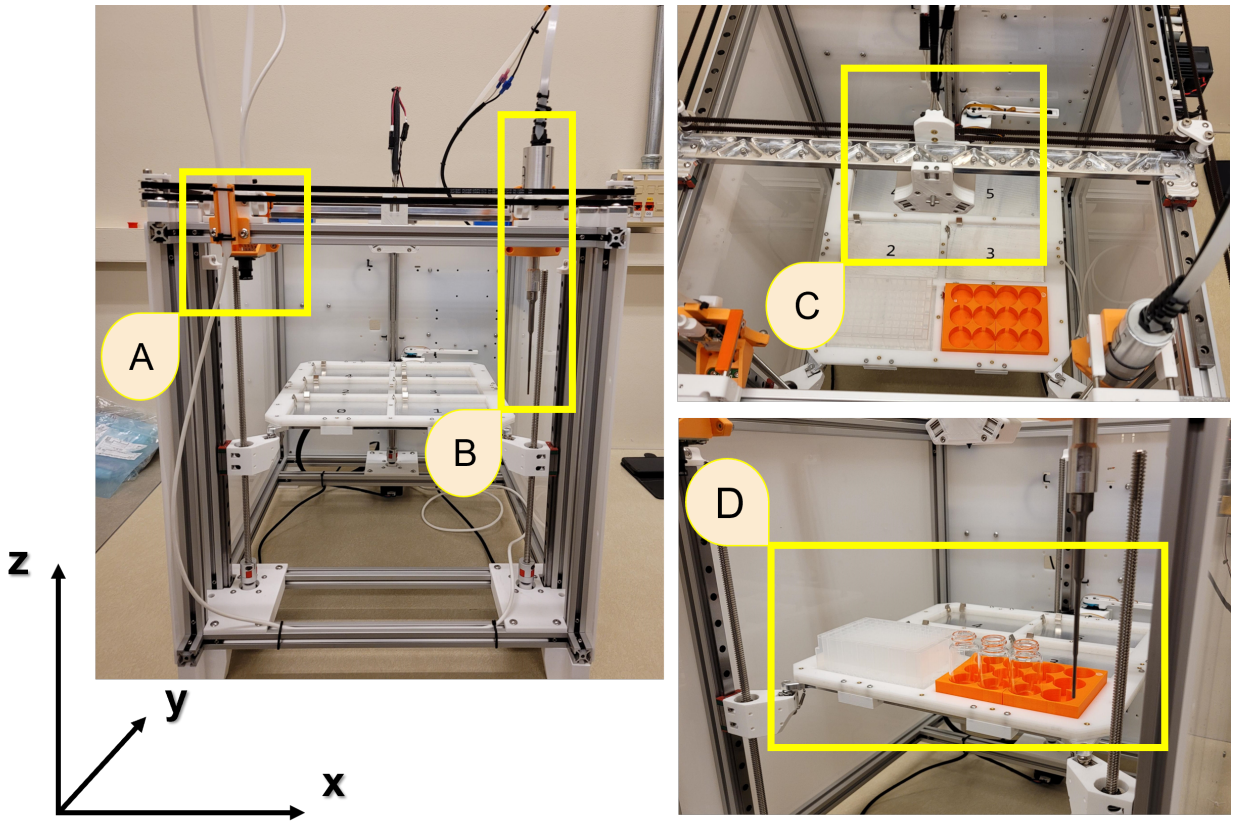

Figure S1: Sonication Station components representation. A) Camera tool. This is used to perform a calibration step for any labware implemented on the platform. B) Sonication horn tool used to process samples. C) Toolchanger component. The t-shaped metal component is used to lock and secure the selected tool. The Toolchanger arm moves in the x,y plane. D) Deck with 6 total slots which can house SLAS standard size labware

1.3 Sonication Station Workflow

The following details a typical workflow on the sonication station.

1. Sample specification : The user first aggregates a batch of samples on up to six distinct labware and determines the sonication specifications (plunge depth, time, power, etc.) for each sample. These specifications are entered into a python script.

2. Sample registration: The user then loads the corresponding labware (e.g., 96 deep-well wellplate) with the prepared samples into the sonication station. With the camera tool selected, the user can easily calibrate the wellplate and annotates the centroids of the corner wells on each plate. With the camera displaying a crosshair overlay onto a live video feed, the user navigates to the (1) bottom left, (2) bottom right, and (3) upper right centroids of the three extreme corner on the selected labware. Internally, the software interpolates these corners to create a grid pattern of sonication points specific to this machine. The labware calibration is tied to a specific deck slot and can be reused without further modifications.

3. Autocleaning setup: It is possible to include an autocleaning protocol to be execute between the processing of each sample. This eliminates possible cross contamination among samples. The user has full control on how this protocol looks like and how it is executed. For example,

it is possible to control how many cleaning baths to use, how long and with what power to clean the sonication probe tip in each bath and where these cleaning labware are placed (e.g., all on the same labware, or specific wells of an the sample labware).

4. Script execution: With the samples loaded and registered and the protocol fully defined, the user can finally autonomously execute the script. These can be saved and reused for future experimental campaigns, as well as shared with the community for reproducibility of the implemented workflow.

1.4 Key Results

In total, the stock platform, combined with the modifications detailed above, can produce a viable laboratory instrument for under $ 5000 (USD). The full set of Jubilee modifications to design a sonication station are documented at `https://github.com/machineagency/sonication_station`.

A sonication station can sonicate samples batched on up to 6 SLAS-compatible labware plates at a time. It can be run unsupervised autonomously via script, and samples can be collected for downstream processing upon completion.

Scripts are machine-instance agnostic. Note that specifications are annotated by well plate index, not spatial location, such that the script may be run on a duplicate machine to yield identical results. In such a scenario, only the sample registration step would need to be run. This design choice affords the practical scaling up of sonication stations, any of which could be used on-demand to run a set of samples.

2 Bulk Temperature Gradient During Sonochemical Synthesis

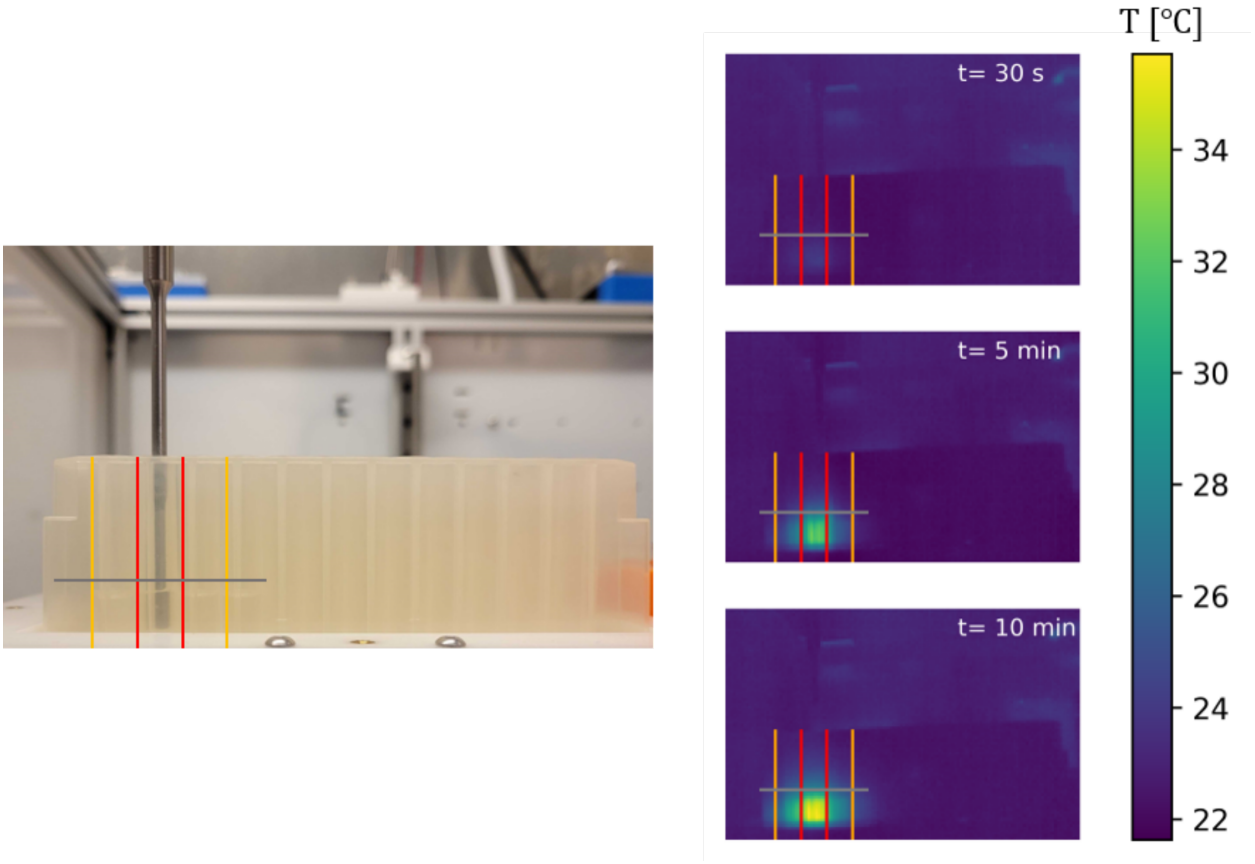

Figure S2: Bulk temperature evolution of a sample during sonochemical processing. Red lines delimit the sample well walls, whereas the orange lines indicate the walls of the two contiguous wells; the grey line represents the height of the liquid level inside each well (0.5mL each). The bulk temperature of the samples was monitored using an IR camera. The temperature is encoded in the pixel values of the images on the right panel. The maximum bulk temperature of the sample being sonicated was recorded to be approximately 35 ℃ , whereas the neighboring wells only experience a maximum temperature of about 28 ℃ due to conduction.

3 Pre-Sonication

3.1 UV-Vis Spectra

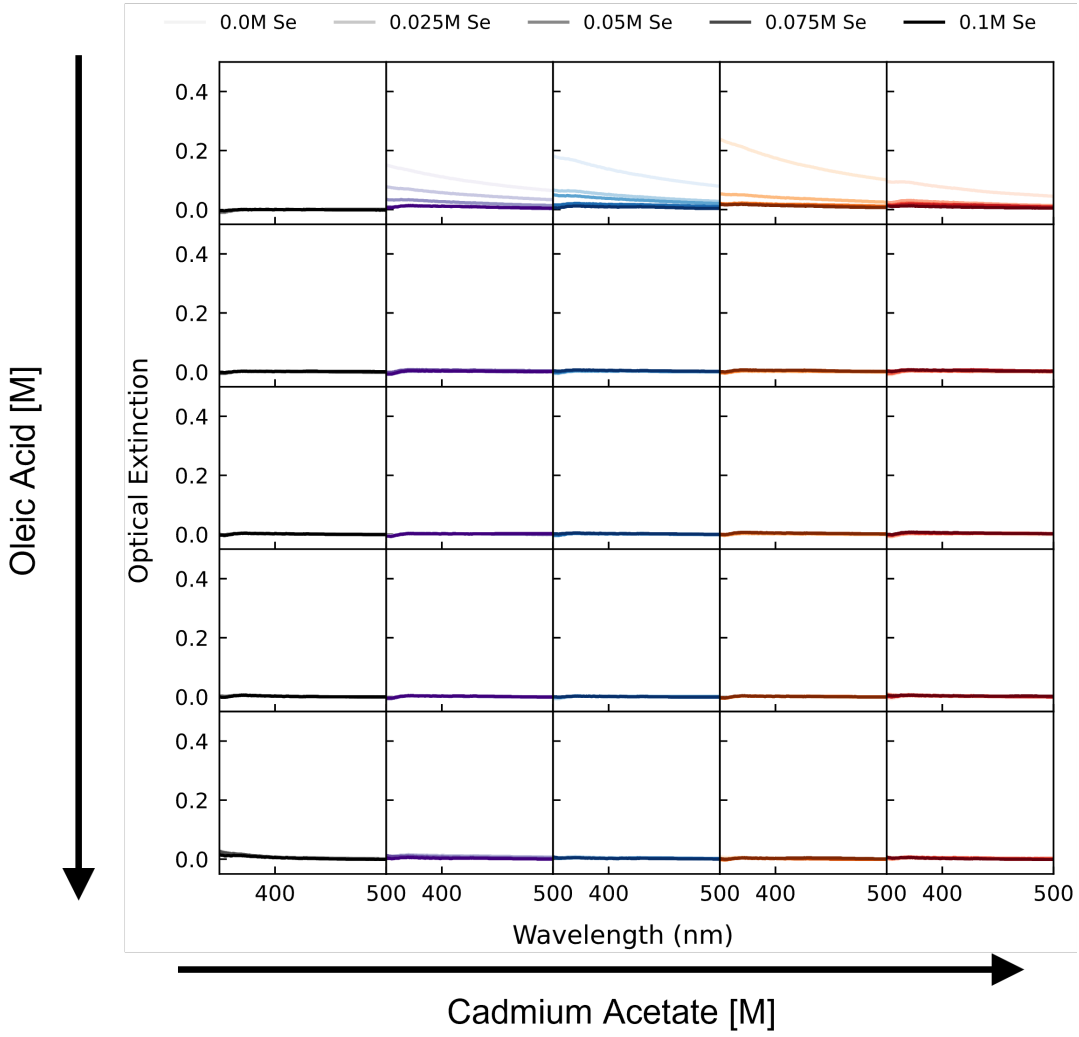

Figure S3: UV-Vis absorption spectra of the CdSe QDs synthesized at varying precursors and oleic acid conditions, prior to sonochemical processing. The concentration of oleylamine is 0 M. The color of the spectra corresponds to different selenium conditions (see legend), whereas the concentration of cadmium acetate increases moving from left to right (0 M, 0.025 M, 0.05 M, 0.075 M, and 0.1 M, respectively). Additionally, moving from top to bottom, the concentration of oleic acid increases from 0M to 0.1M .

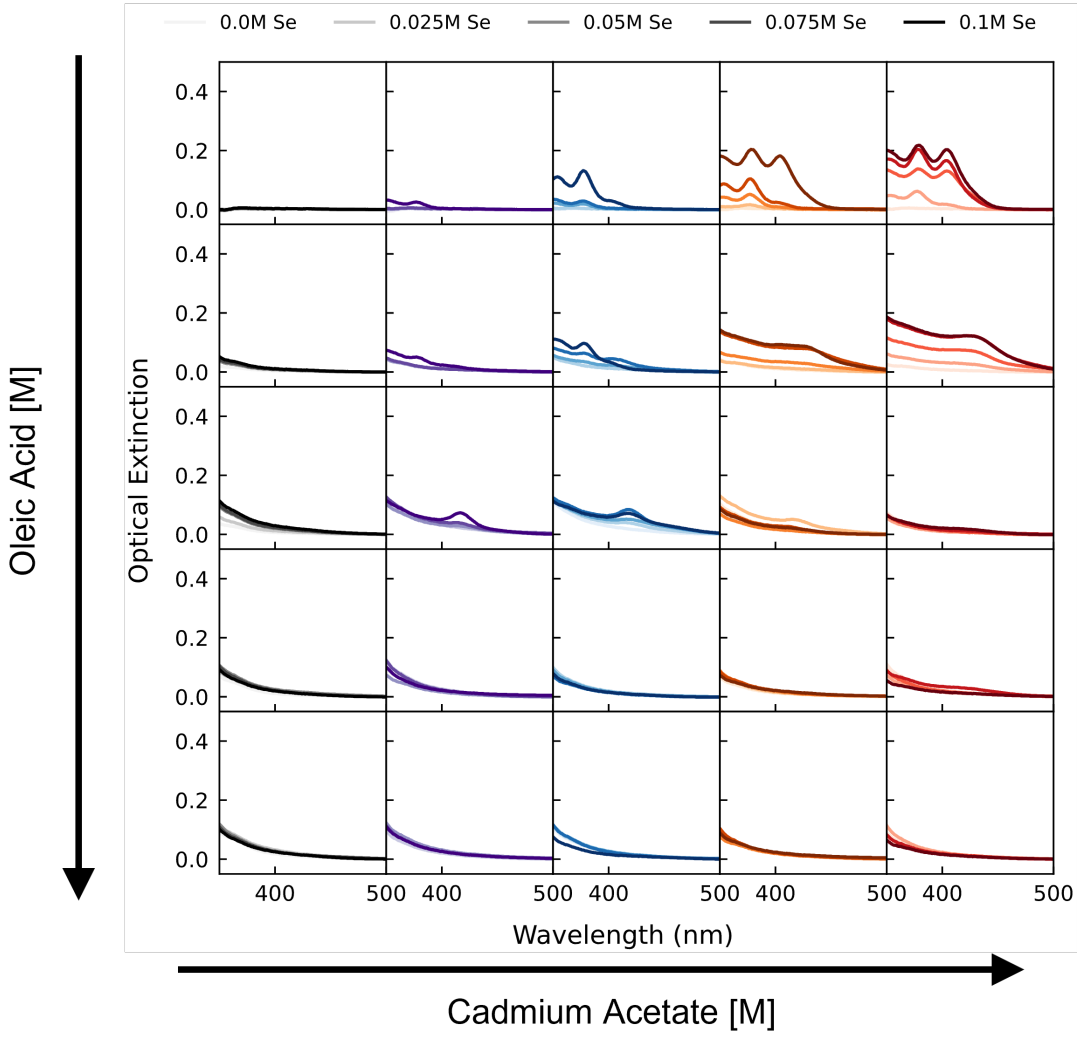

Figure S4: UV-Vis absorption spectra of the CdSe QDs synthesized at varying precursors and oleic acid conditions, prior to sonochemical processing. The concentration of oleylamine is 0.125 M. The color of the spectra corresponds to different selenium conditions (see legend), whereas the concentration of cadmium acetate increases moving from left to right (0 M, 0.025 M, 0.05 M, 0.075 M, and 0.1 M, respectively). Additionally, moving from top to bottom, the concentration of oleic acid increases from 0M to 0.1M.

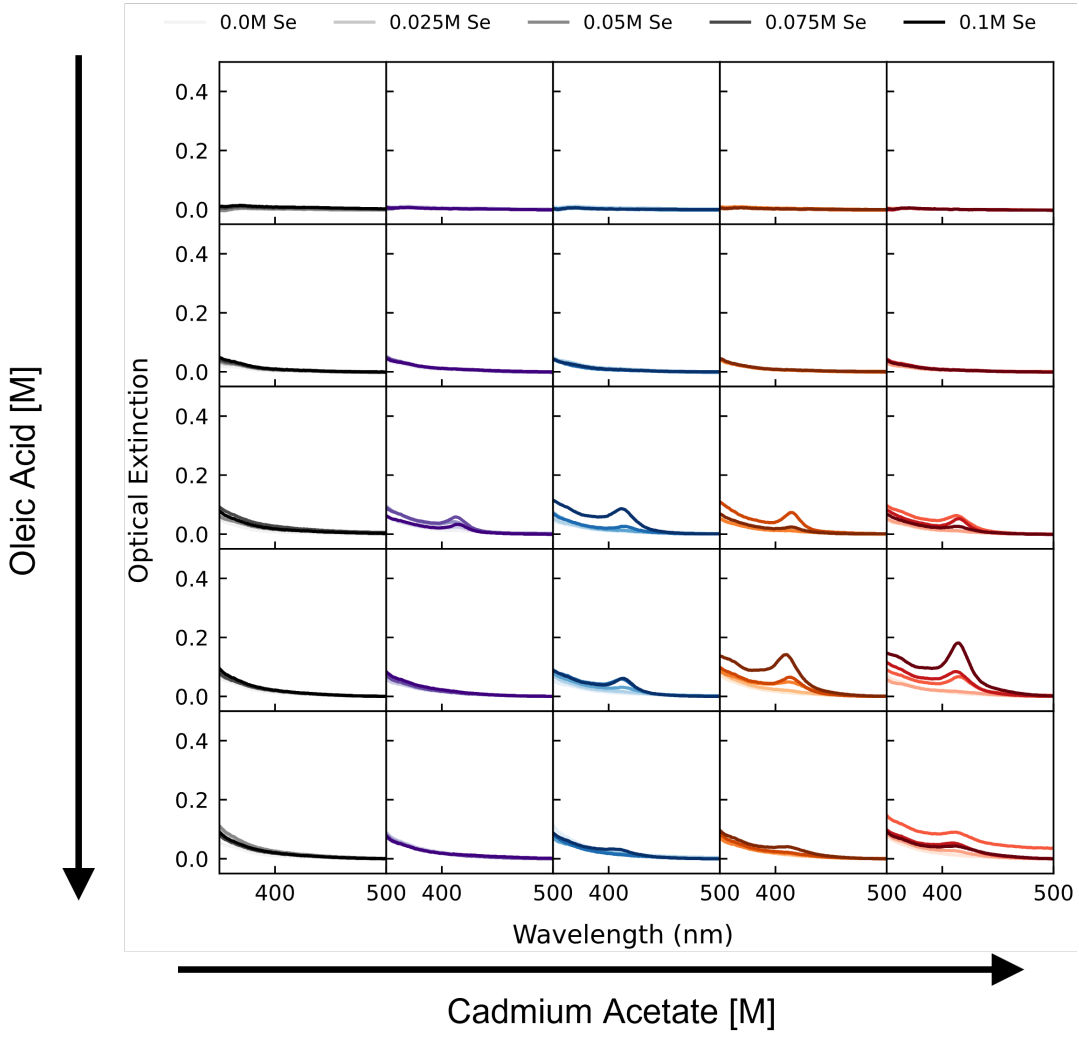

Figure S5: UV-Vis absorption spectra of the CdSe QDs synthesized at varying precursors and oleic acid conditions, prior to sonochemical processing. The concentration of oleylamine is 0.25 M. The color of the spectra corresponds to different selenium conditions (see legend), whereas the concentration of cadmium acetate increases moving from left to right (0 M, 0.025 M, 0.05 M, 0.075 M, and 0.1 M, respectively). Additionally, moving from top to bottom, the concentration of oleic acid increases from 0M to 0.1M.

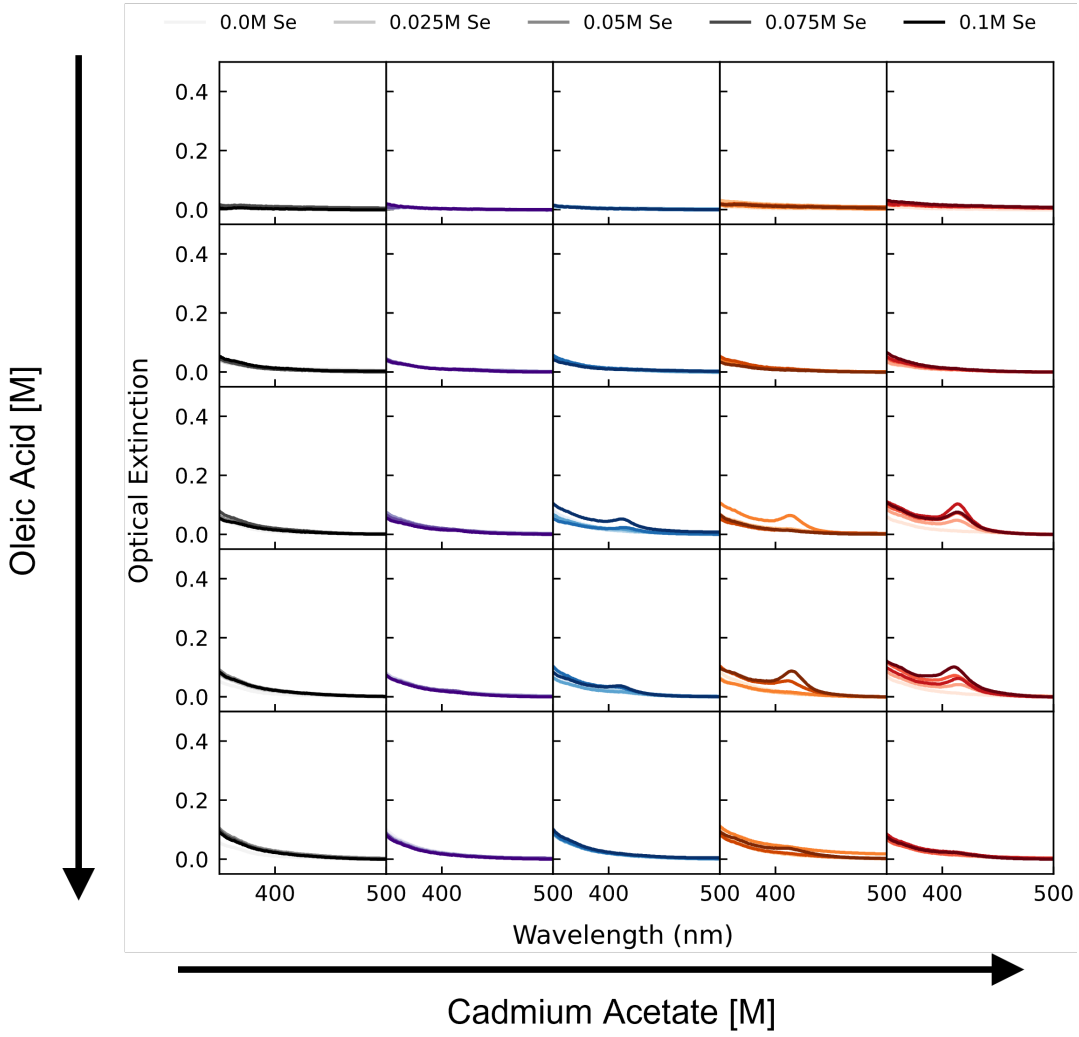

Figure S6: UV-Vis absorption spectra of the CdSe QDs synthesized at varying precursors and oleic acid conditions, prior to sonochemical processing. The concentration of oleylamine is 0.125 M. The color of the spectra corresponds to different selenium conditions (see legend), whereas the concentration of cadmium acetate increases moving from left to right (0 M, 0.025 M, 0.05 M, 0.075 M, and 0.1 M, respectively). Additionally, moving from top to bottom, the concentration of oleic acid increases from 0M to 0.1M.

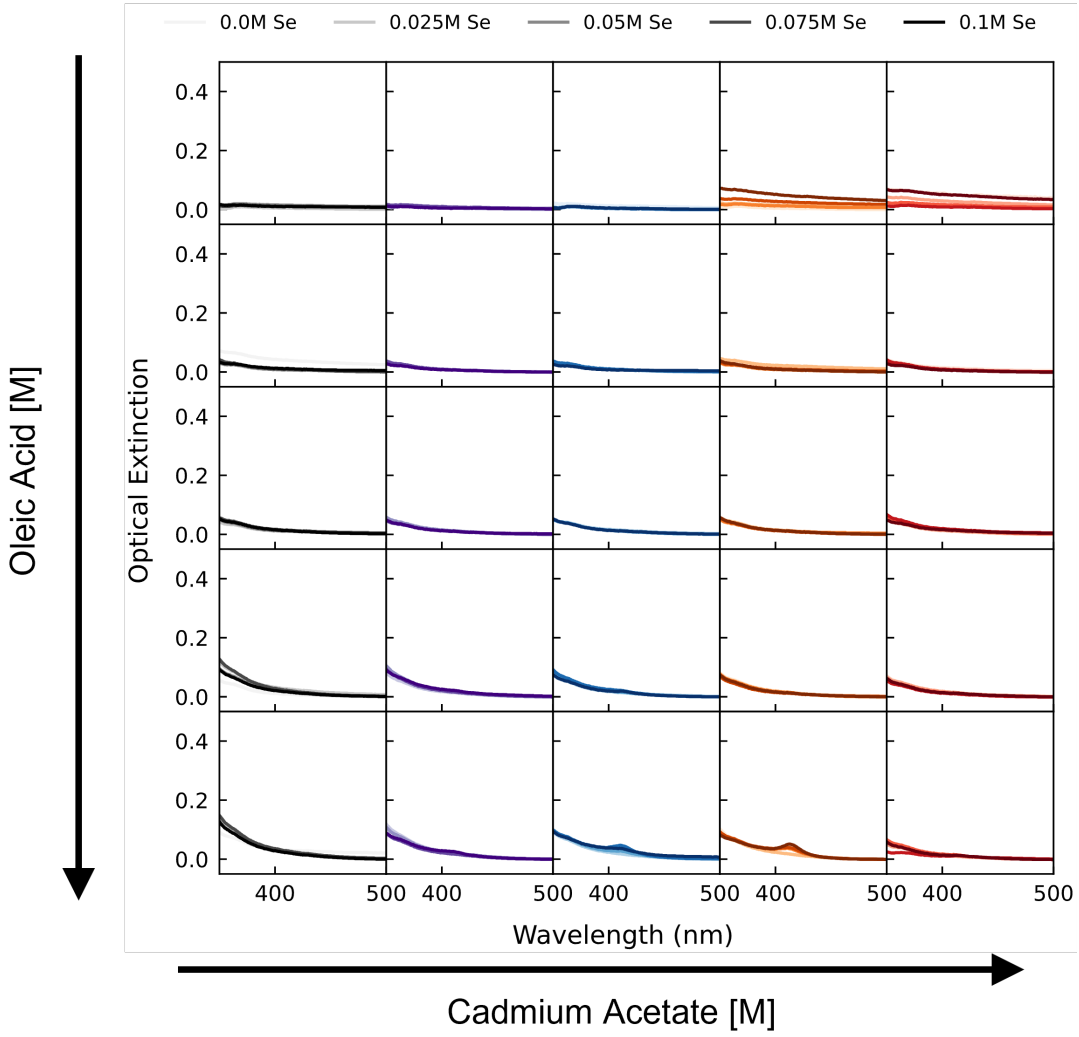

Figure S7: UV-Vis absorption spectra of the CdSe QDs synthesized at varying precursors and oleic acid conditions, prior to sonochemical processing. The concentration of oleylamine is 0.125 M. The color of the spectra corresponds to different selenium conditions (see legend), whereas the concentration of cadmium acetate increases moving from left to right (0 M, 0.025 M, 0.05 M, 0.075 M, and 0.1 M, respectively). Additionally, moving from top to bottom, the concentration of oleic acid increases from 0M to 0.1M.

3.2 Photoluminescence Spectra

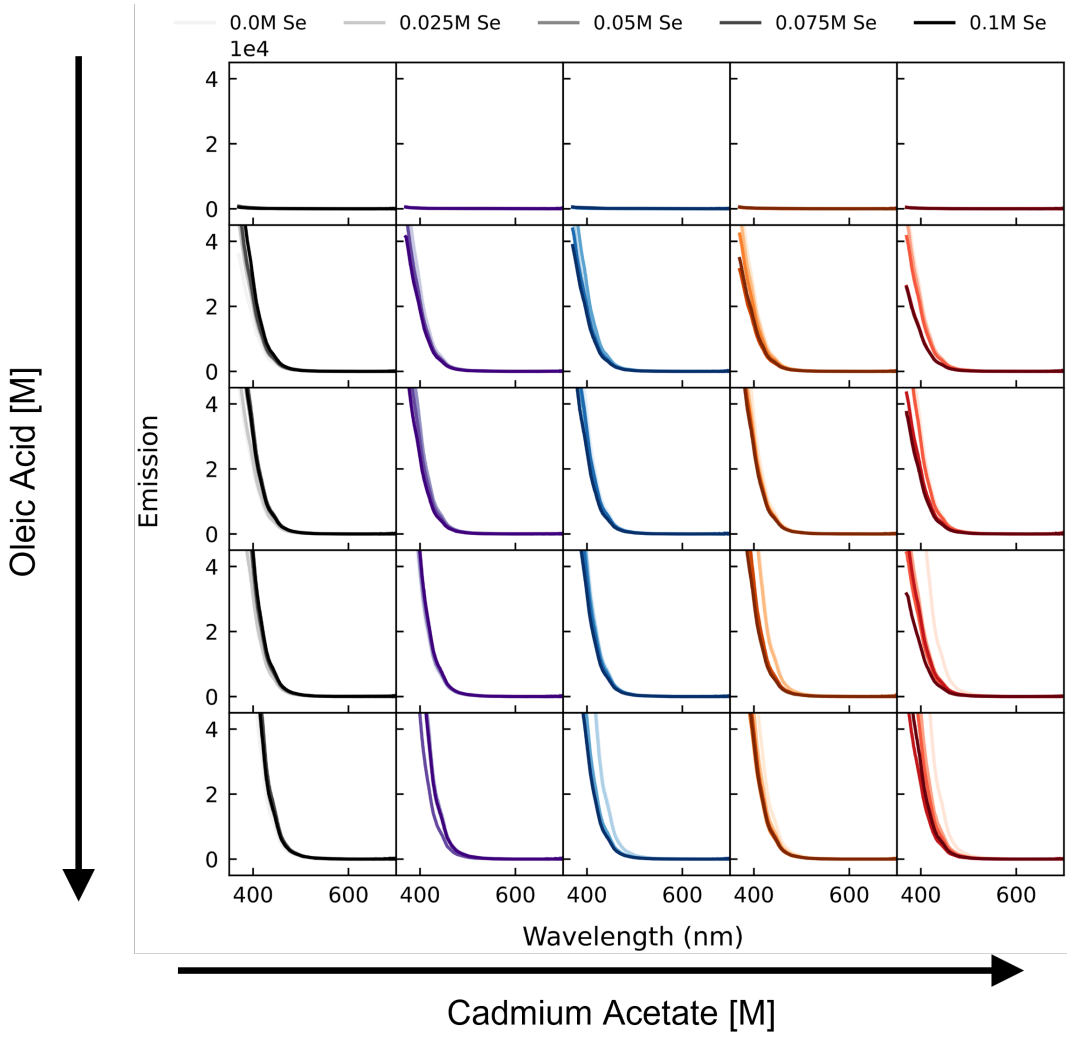

Figure S8: Photoluminescence emission spectra of the CdSe QDs synthesized at varying precursors and oleic acid conditions, before sonochemical processing. The concentration of oleylamine is 0 M. The color of the spectra corresponds to different selenium conditions (see legend), whereas the concentration of cadmium acetate increases moving from left to right (0 M, 0.025 M, 0.05 M, 0.075 M, and 0.1 M, respectively). Additionally, moving from top to bottom, the concentration of oleic acid increases from 0M to 0.1M.

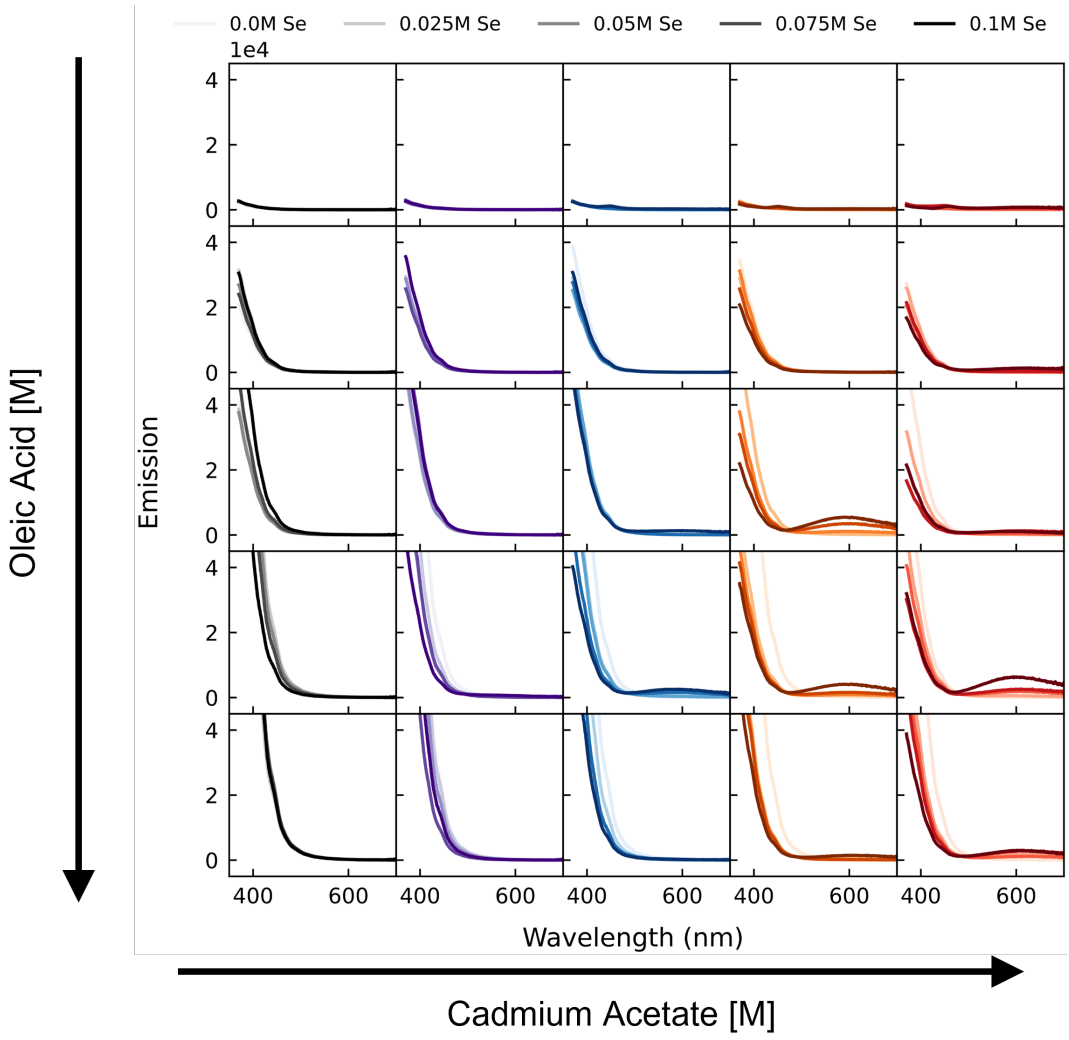

Figure S9: Photoluminescence emission spectra of the CdSe QDs synthesized at varying precursors and oleic acid conditions, before sonochemical processing. The concentration of oleylamine is 0.125 M. The color of the spectra corresponds to different selenium conditions (see legend), whereas the concentration of cadmium acetate increases moving from left to right (0 M, 0.025 M, 0.05 M, 0.075 M, and 0.1 M, respectively). Additionally, moving from top to bottom, the concentration of oleic acid increases from 0M to 0.1M.

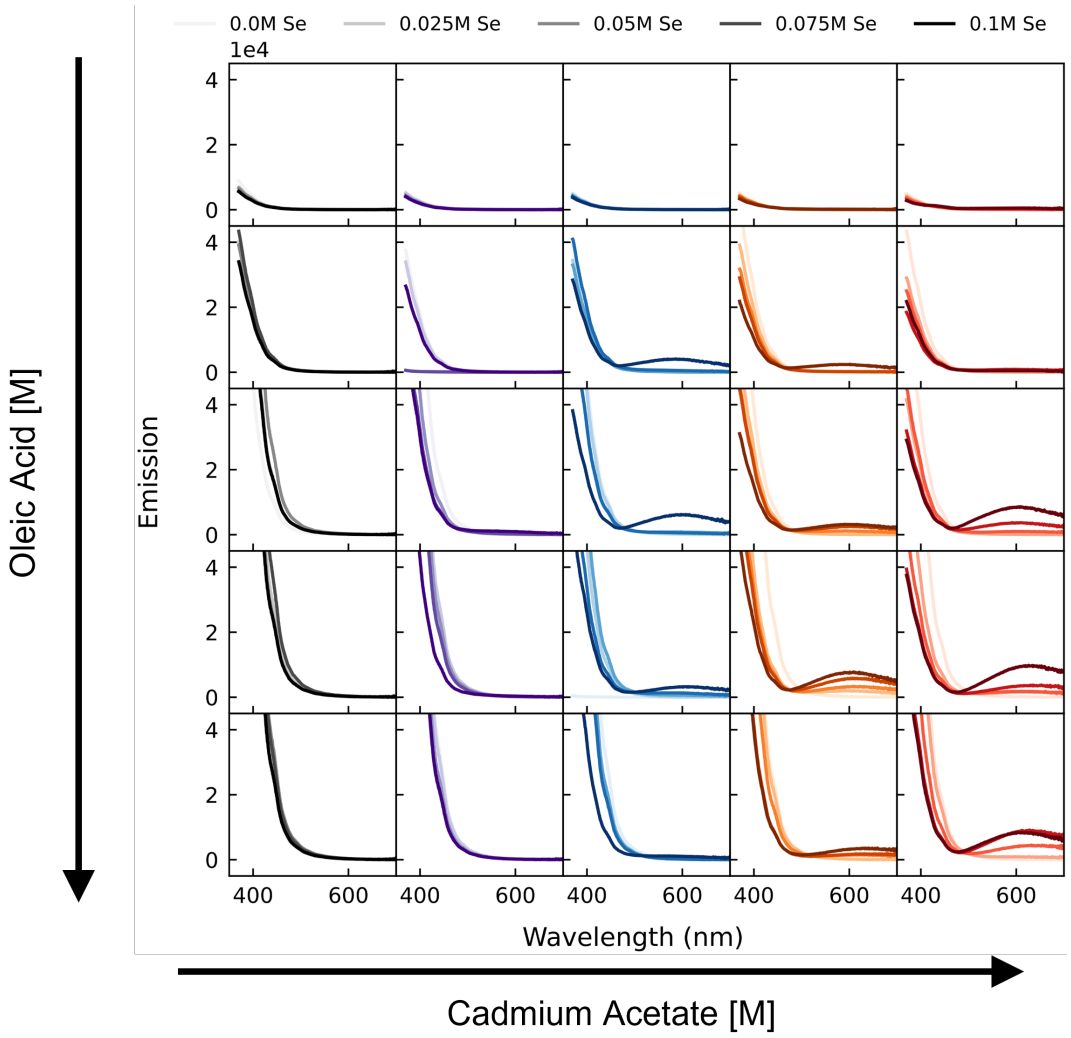

Figure S10: Photoluminescence emission spectra of the CdSe QDs synthesized at varying precursors and oleic acid conditions, before sonochemical processing. The concentration of oleylamine is 0.25 M. The color of the spectra corresponds to different selenium conditions (see legend), whereas the concentration of cadmium acetate increases moving from left to right (0 M, 0.025 M, 0.05 M, 0.075 M, and 0.1 M, respectively). Additionally, moving from top to bottom, the concentration of oleic acid increases from 0M to 0.1M.

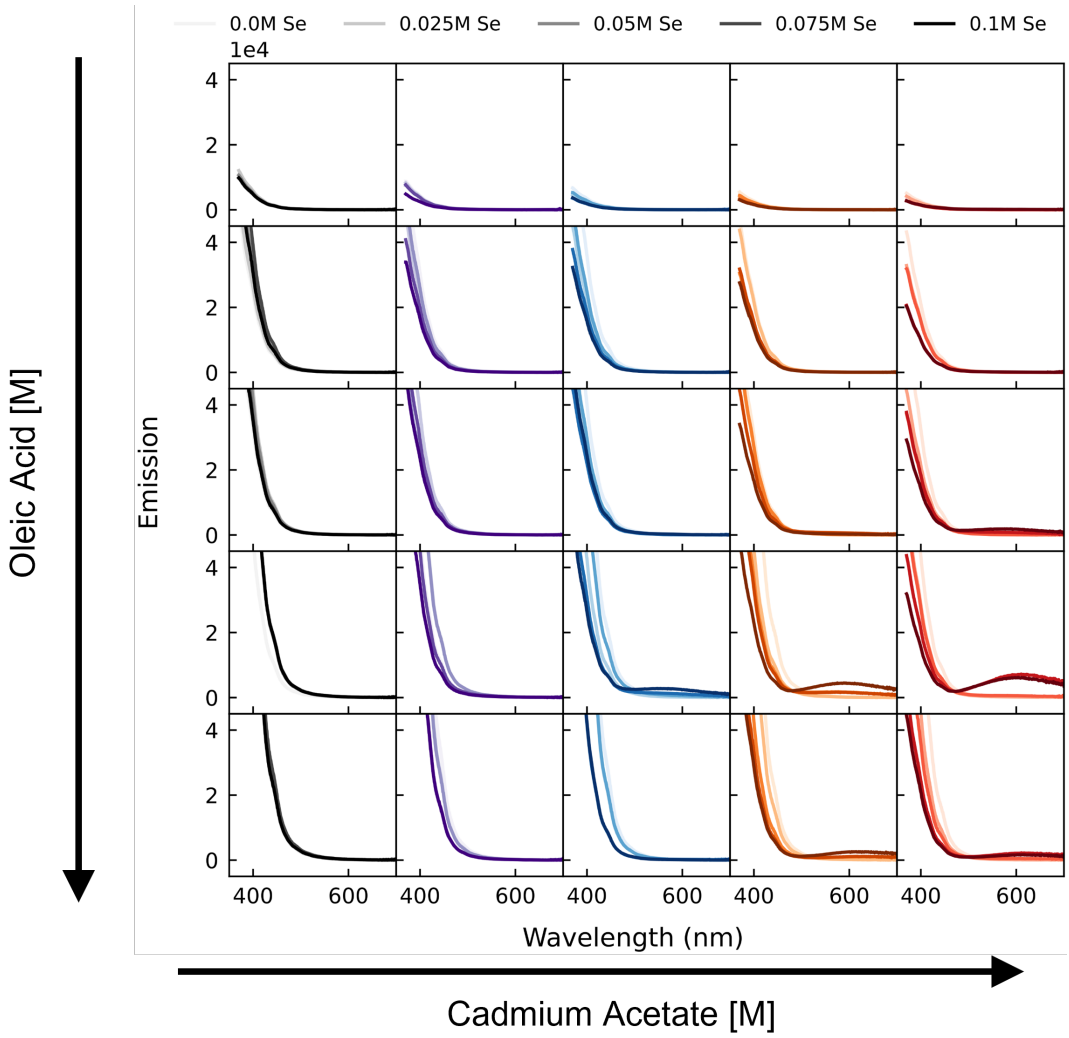

Figure S11: Photoluminescence emission spectra of the CdSe QDs synthesized at varying precursors and oleic acid conditions, before sonochemical processing. The concentration of oleylamine is 0.125 M. The color of the spectra corresponds to different selenium conditions (see legend), whereas the concentration of cadmium acetate increases moving from left to right (0 M, 0.025 M, 0.05 M, 0.075 M, and 0.1 M, respectively). Additionally, moving from top to bottom, the concentration of oleic acid increases from 0M to 0.1M.

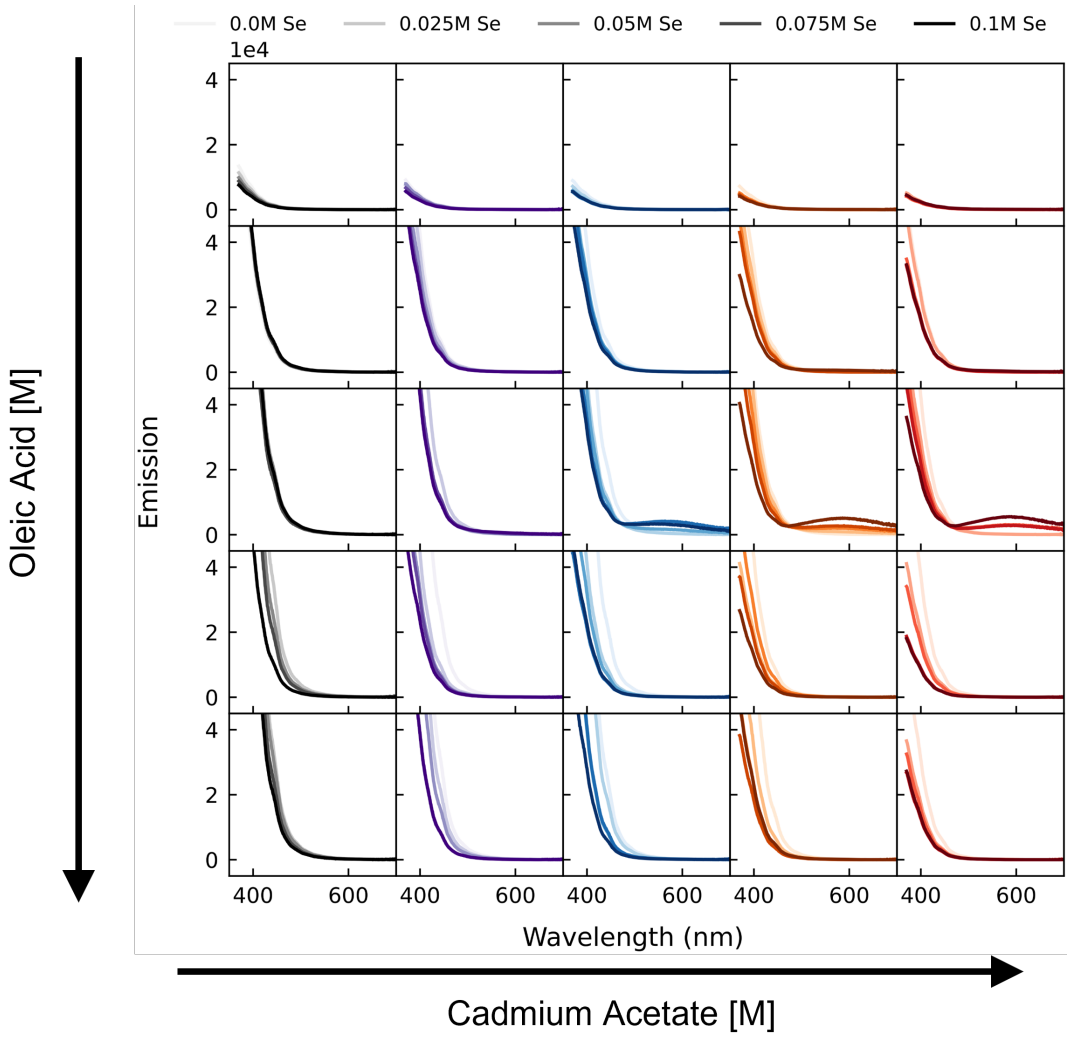

Figure S12: Photoluminescence emission spectra of the CdSe QDs synthesized at varying precursors and oleic acid conditions, before sonochemical processing. The concentration of oleylamine is 0.125 M. The color of the spectra corresponds to different selenium conditions (see legend), whereas the concentration of cadmium acetate increases moving from left to right (0 M, 0.025 M, 0.05 M, 0.075 M, and 0.1 M, respectively). Additionally, moving from top to bottom, the concentration of oleic acid increases from 0M to 0.1M.

4 Post-Sonication

4.1 UV-Vis Spectra

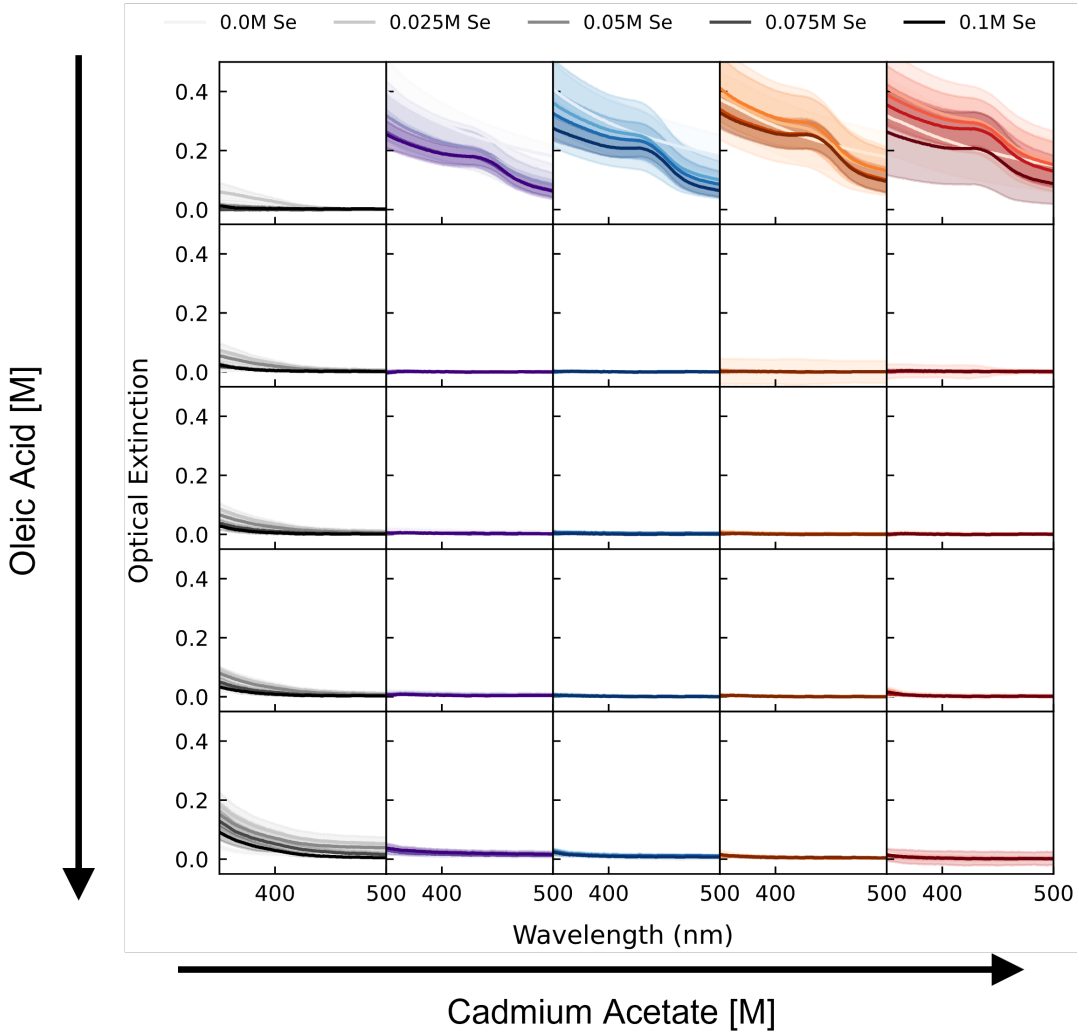

Figure S13: UV-Vis absorption spectra of the CdSe QDs synthesized at varying precursors and oleic acid conditions. The concentration of oleylamine is 0 M. The color of the spectra corresponds to different selenium conditions (see legend), whereas the concentration of cadmium acetate increases moving from left to right (0 M, 0.025 M, 0.05 M, 0.075 M, and 0.1 M, respectively). Additionally, moving from top to bottom, the concentration of oleic acid increases from 0M to 0.1M. Since the data was collected in triplicates, the shaded area of the corresponding color shows the variation of response across the different batches tested.

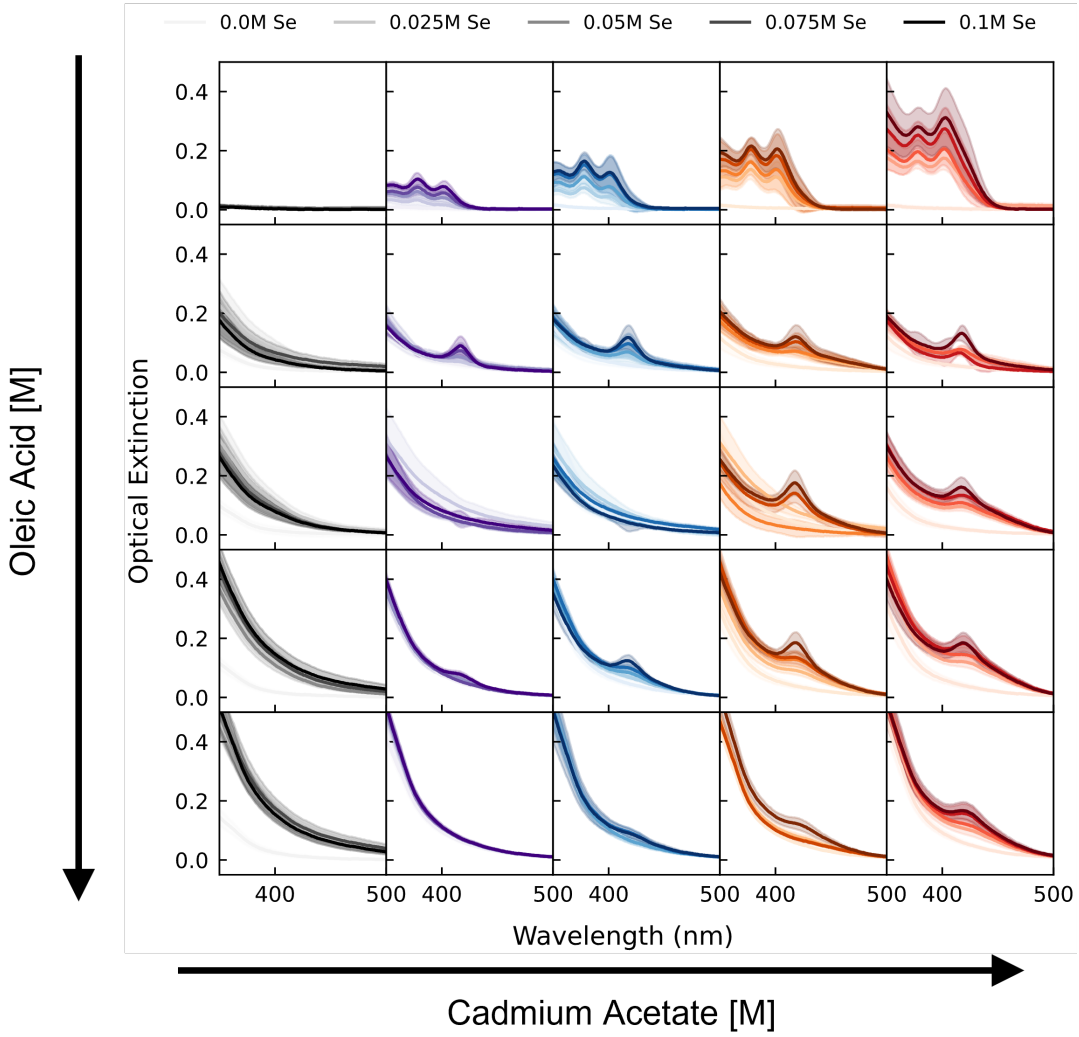

Figure S14: UV-Vis absorption spectra of the CdSe QDs synthesized at varying precursors and oleic acid conditions. The concentration of oleylamine is 0.125 M. The color of the spectra corresponds to different selenium conditions (see legend), whereas the concentration of cadmium acetate increases moving from left to right (0 M, 0.025 M, 0.05 M, 0.075 M, and 0.1 M, respectively). Additionally, moving from top to bottom, the concentration of oleic acid increases from 0M to 0.1M. Since the data was collected in triplicates, the shaded area of the corresponding color shows the variation of response across the different batches tested.

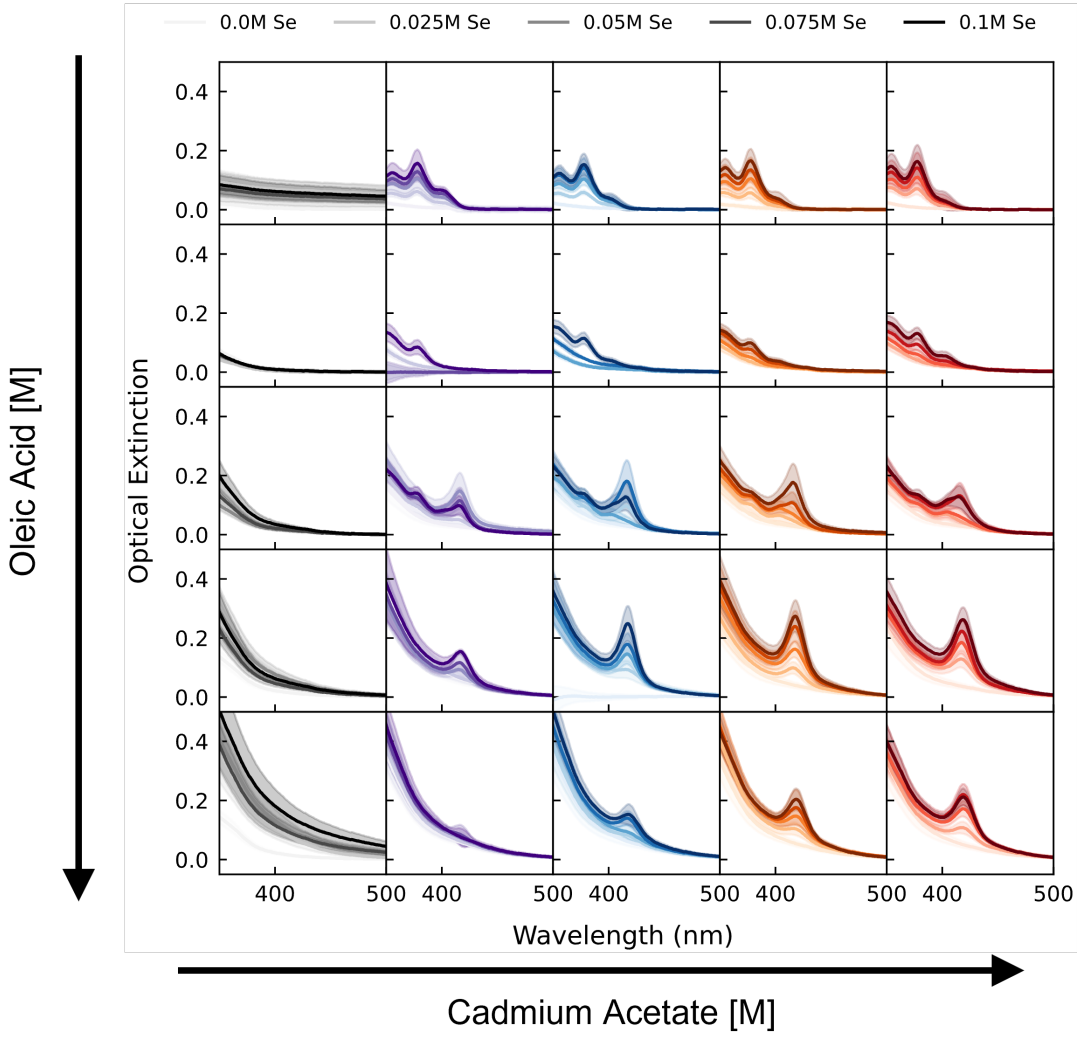

Figure S15: UV-Vis absorption spectra of the CdSe QDs synthesized at varying precursors and oleic acid conditions. The concentration of oleylamine is 0.25 M. The color of the spectra corresponds to different selenium conditions (see legend), whereas the concentration of cadmium acetate increases moving from left to right (0 M, 0.025 M, 0.05 M, 0.075 M, and 0.1 M, respectively). Additionally, moving from top to bottom, the concentration of oleic acid increases from 0M to 0.1M. Since the data was collected in triplicates, the shaded area of the corresponding color shows the variation of response across the different batches tested.

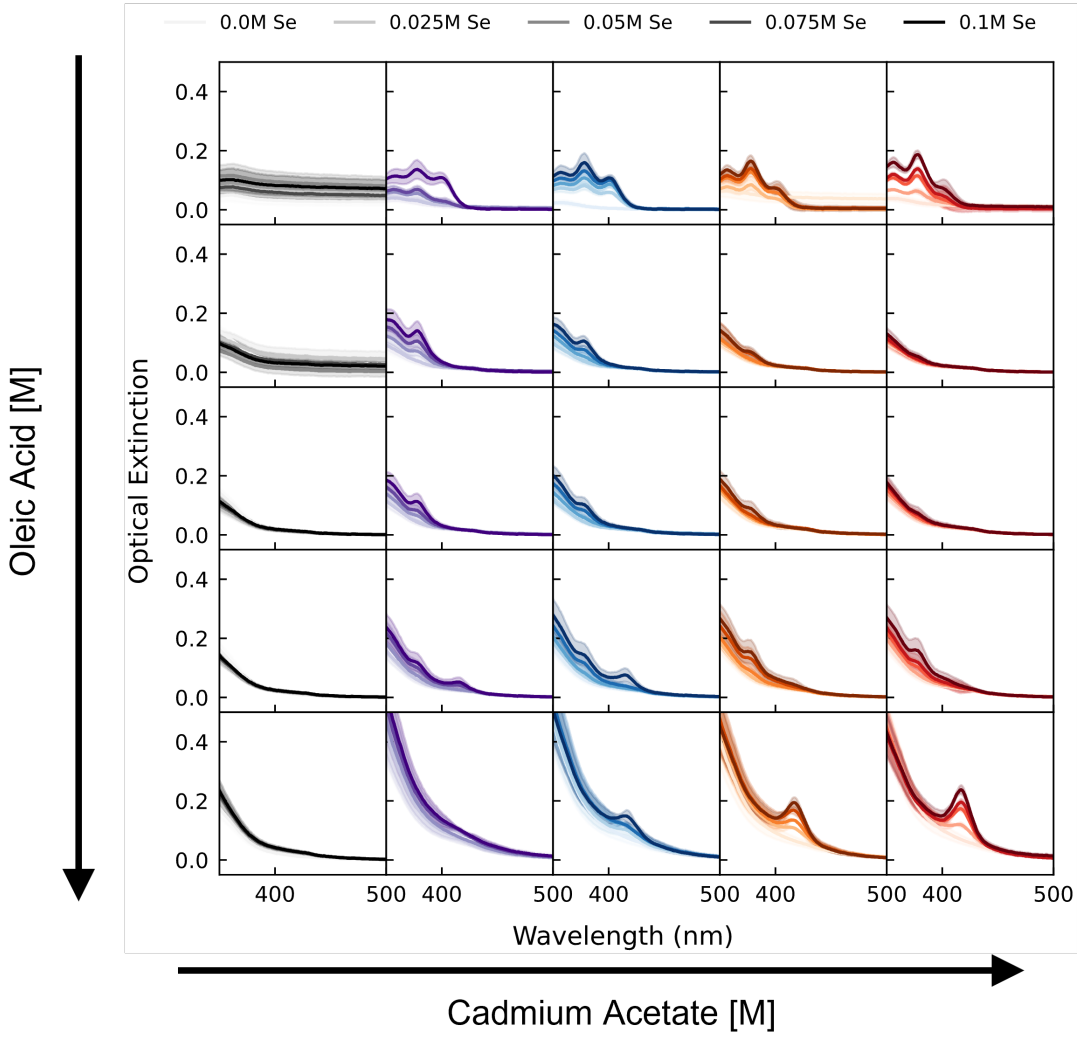

Figure S16: UV-Vis absorption spectra of the CdSe QDs synthesized at varying precursors and oleic acid conditions. The concentration of oleylamine is 0.125 M. The color of the spectra corresponds to different selenium conditions (see legend), whereas the concentration of cadmium acetate increases moving from left to right (0 M, 0.025 M, 0.05 M, 0.075 M, and 0.1 M, respectively). Additionally, moving from top to bottom, the concentration of oleic acid increases from 0M to 0.1M. Since the data was collected in triplicates, the shaded area of the corresponding color shows the variation of response across the different batches tested.

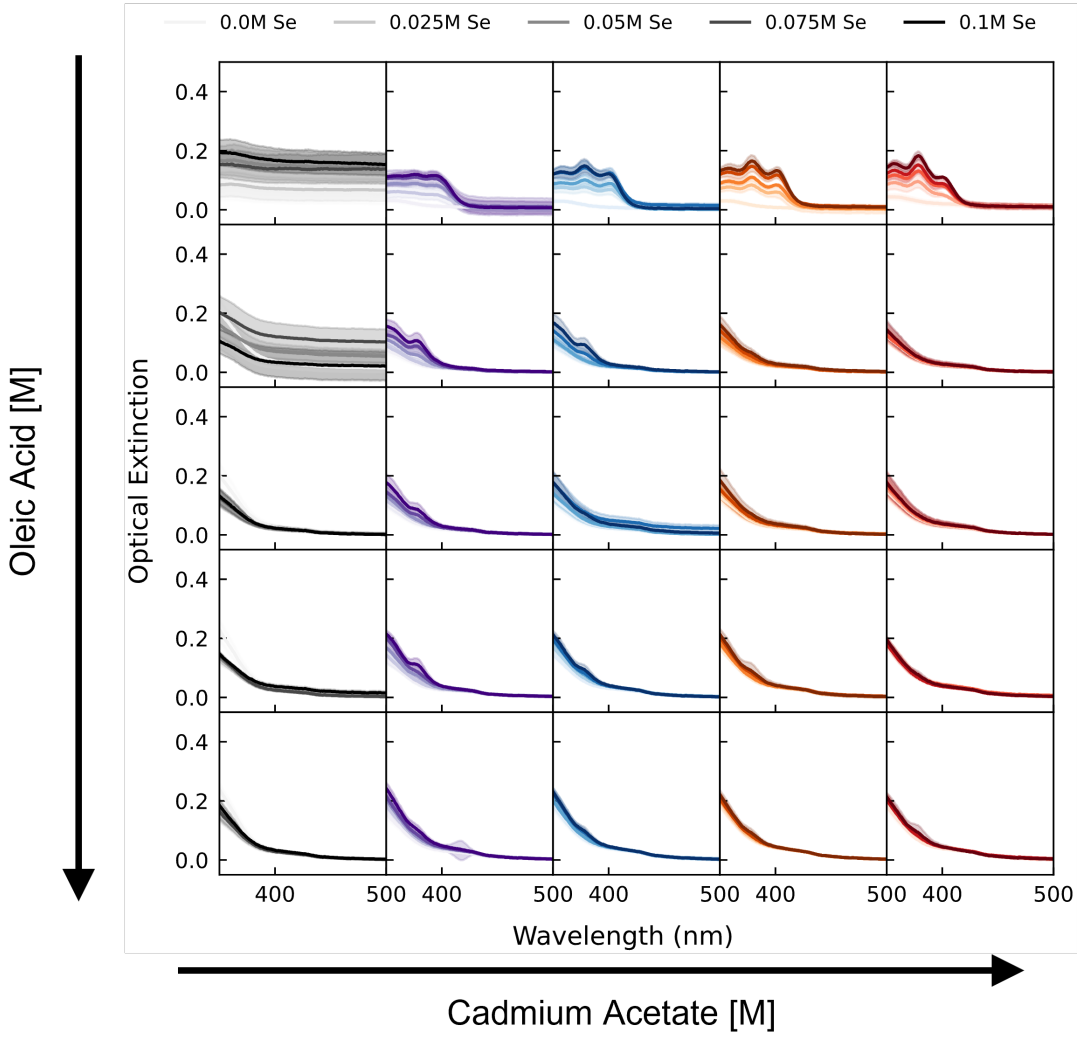

Figure S17: UV-Vis absorption spectra of the CdSe QDs synthesized at varying precursors and oleic acid conditions. The concentration of oleylamine is 0.125 M. The color of the spectra corresponds to different selenium conditions (see legend), whereas the concentration of cadmium acetate increases moving from left to right (0 M, 0.025 M, 0.05 M, 0.075 M, and 0.1 M, respectively). Additionally, moving from top to bottom, the concentration of oleic acid increases from 0M to 0.1M. Since the data was collected in triplicates, the shaded area of the corresponding color shows the variation of response across the different batches tested.

4.2 Photoluminescence Spectra

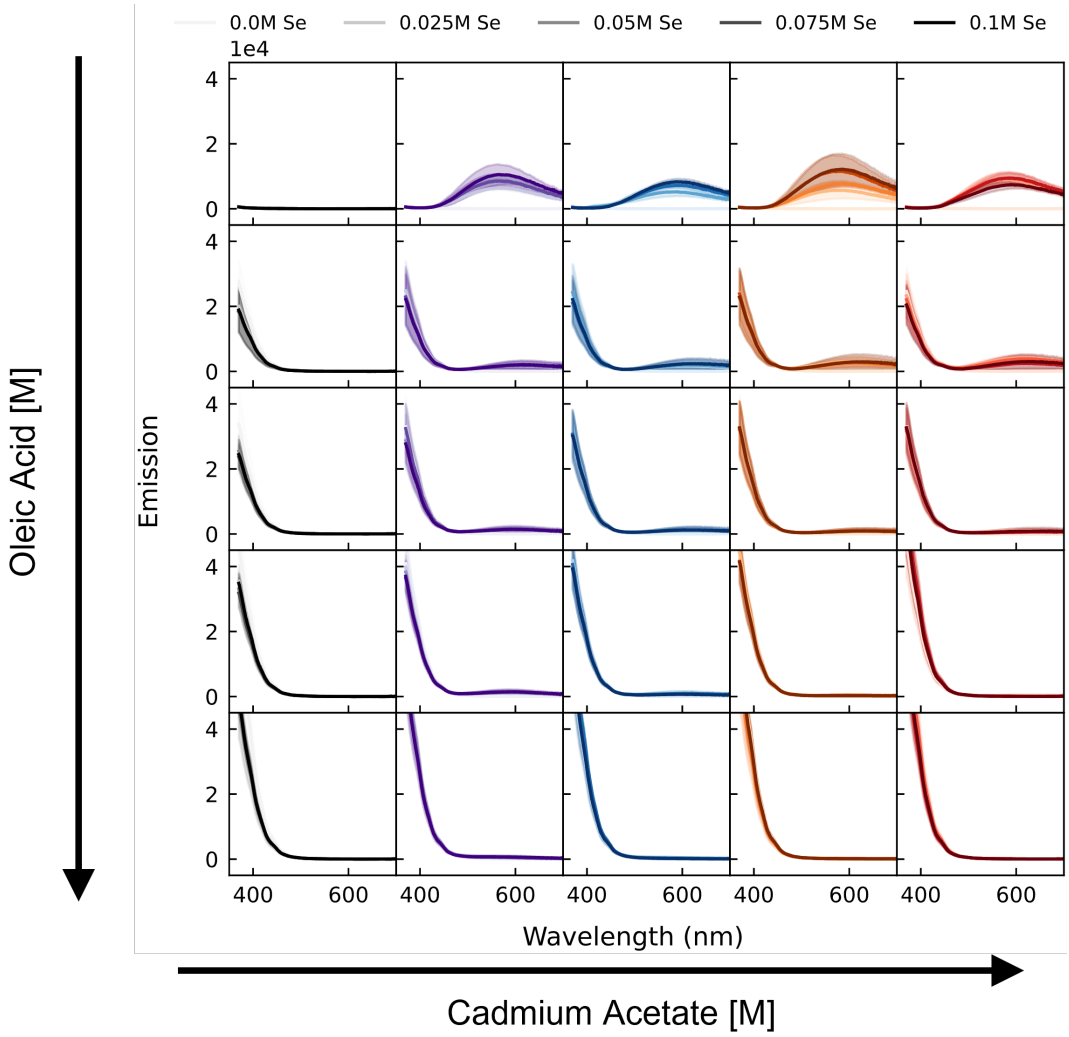

Figure S18: Photoluminescence emission spectra of the CdSe QDs synthesized at varying precursors and oleic acid conditions. The concentration of oleylamine is 0 M. The color of the spectra corresponds to different selenium conditions (see legend), whereas the concentration of cadmium acetate increases moving from left to right (0 M, 0.025 M, 0.05 M, 0.075 M, and 0.1 M, respectively). Additionally, moving from top to bottom, the concentration of oleic acid increases from 0M to 0.1M. Since the data was collected in triplicates, the shaded area of the corresponding color shows the variation of response across the different batches tested.

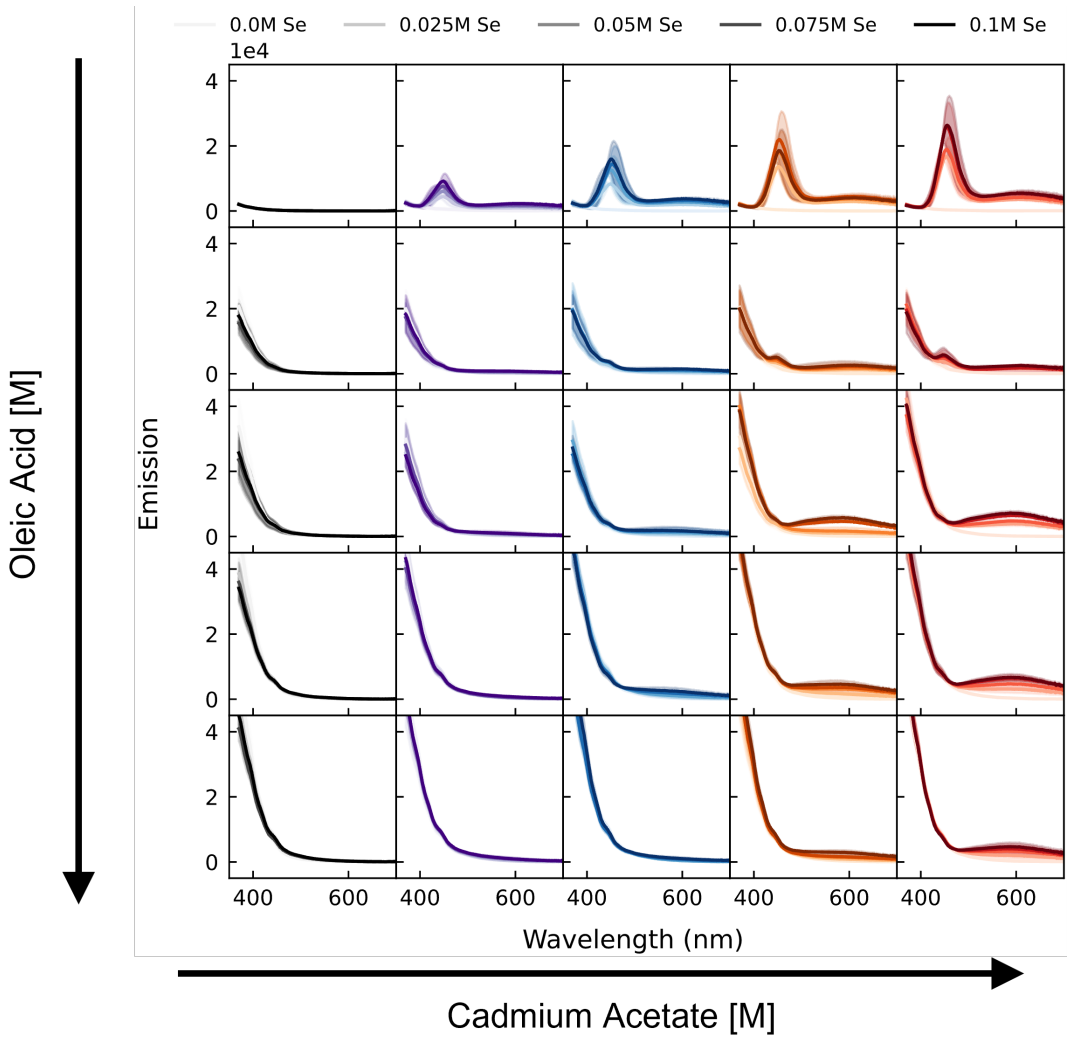

Figure S19: Photoluminescence emission spectra of the CdSe QDs synthesized at varying precursors and oleic acid conditions. The concentration of oleylamine is 0.125 M. The color of the spectra corresponds to different selenium conditions (see legend), whereas the concentration of cadmium acetate increases moving from left to right (0 M, 0.025 M, 0.05 M, 0.075 M, and 0.1 M, respectively). Additionally, moving from top to bottom, the concentration of oleic acid increases from 0M to 0.1M. Since the data was collected in triplicates, the shaded area of the corresponding color shows the variation of response across the different batches tested.

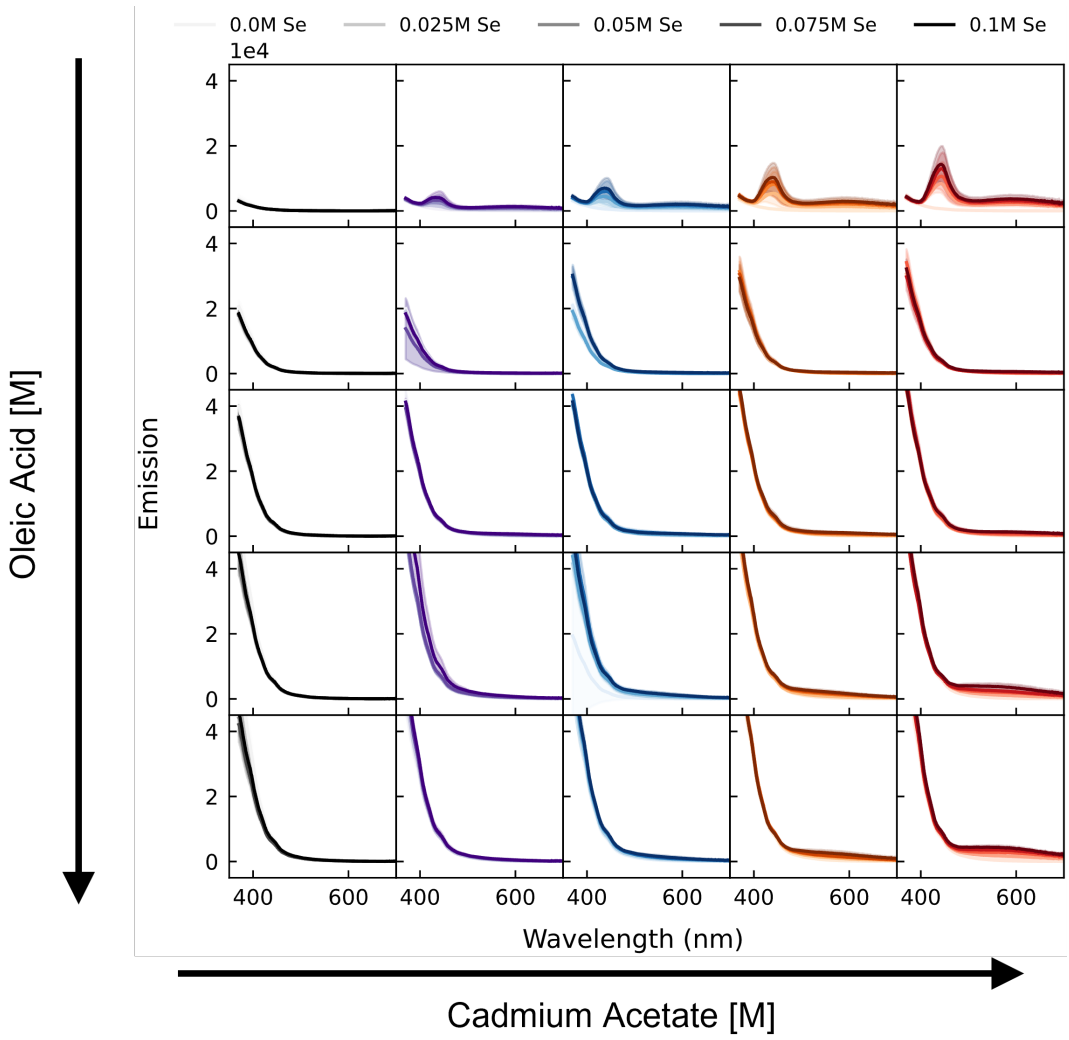

Figure S20: Photoluminescence emission spectra of the CdSe QDs synthesized at varying precursors and oleic acid conditions. The concentration of oleylamine is 0.25 M. The color of the spectra corresponds to different selenium conditions (see legend), whereas the concentration of cadmium acetate increases moving from left to right (0 M, 0.025 M, 0.05 M, 0.075 M, and 0.1 M, respectively). Additionally, moving from top to bottom, the concentration of oleic acid increases from 0M to 0.1M. Since the data was collected in triplicates, the shaded area of the corresponding color shows the variation of response across the different batches tested.

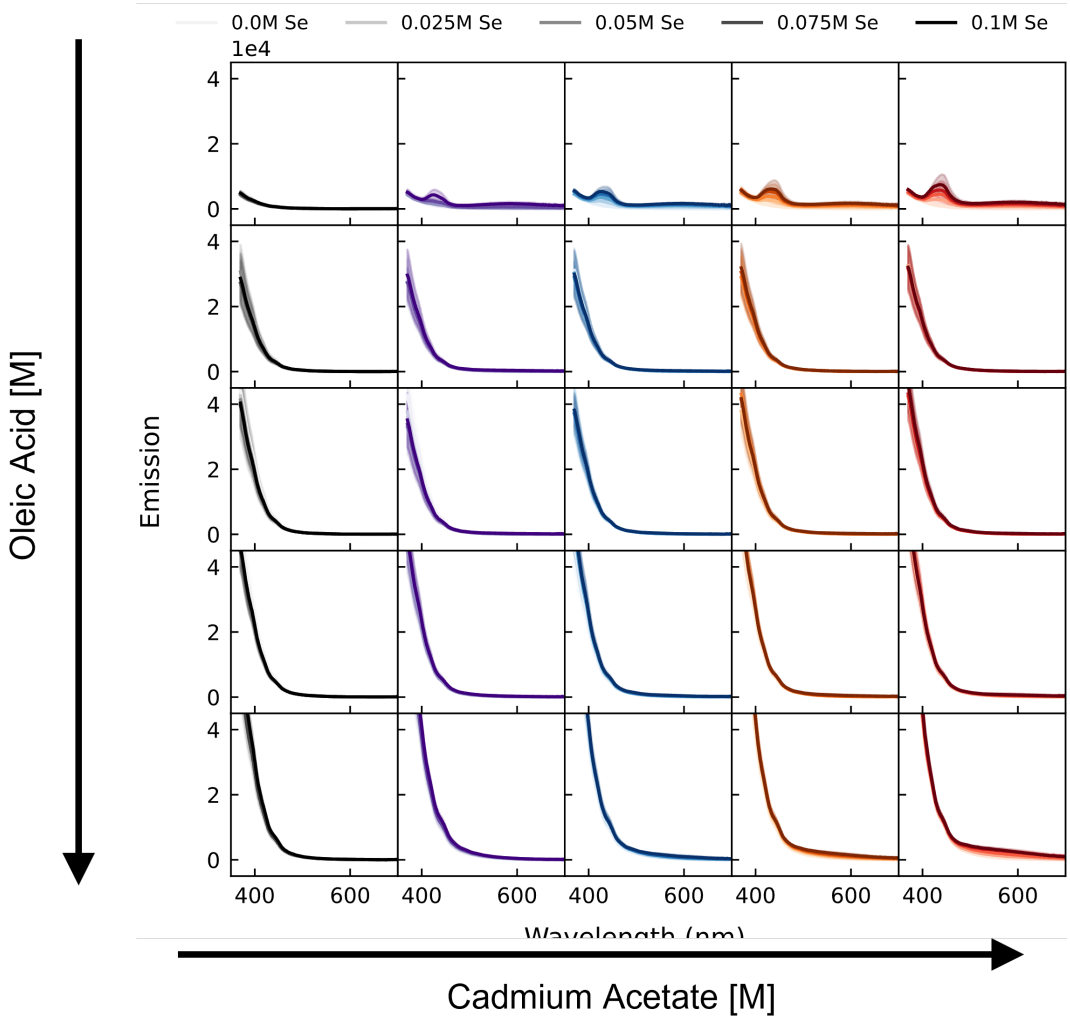

Figure S21: Photoluminescence emission spectra of the CdSe QDs synthesized at varying precursors and oleic acid conditions. The concentration of oleylamine is 0.125 M. The color of the spectra corresponds to different selenium conditions (see legend), whereas the concentration of cadmium acetate increases moving from left to right (0 M, 0.025 M, 0.05 M, 0.075 M, and 0.1 M, respectively). Additionally, moving from top to bottom, the concentration of oleic acid increases from 0M to 0.1M. Since the data was collected in triplicates, the shaded area of the corresponding color shows the variation of response across the different batches tested.

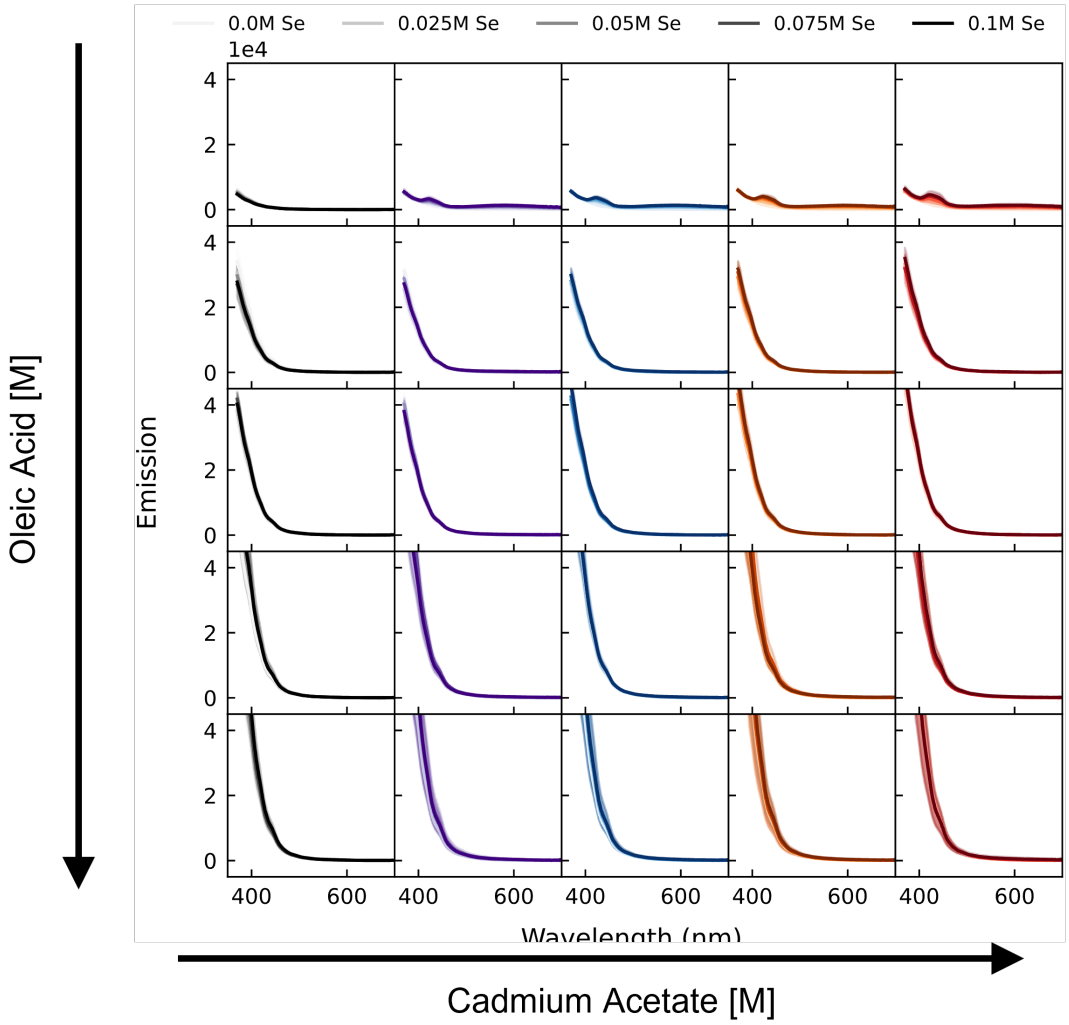

Figure S22: Photoluminescence emission spectra of the CdSe QDs synthesized at varying precursors and oleic acid conditions. The concentration of oleylamine is 0.125 M. The color of the spectra corresponds to different selenium conditions (see legend), whereas the concentration of cadmium acetate increases moving from left to right (0 M, 0.025 M, 0.05 M, 0.075 M, and 0.1 M, respectively). Additionally, moving from top to bottom, the concentration of oleic acid increases from 0M to 0.1M. Since the data was collected in triplicates, the shaded area of the corresponding color shows the variation of response across the different batches tested.

5 Small angle X-Ray Scattering

Table 1: Sample composition for the subset of conditions characterized using Small Angle X-Ray Scattering.

Sample Name	**Sample Composition [M]**			
	OlAc	OlAm	CdAc	Se
CdSe 1	0	0	0.1	0.1
CdSe 2	0	0.125	0.1	0.1
CdSe 3	0.25	0.125	0.1	0.1
CdSe 4	0.5	0.125	0.1	0.1
CdSe 5	0	0.25	0.1	0.1
CdSe 6	0.25	0.25	0.1	0.1
CdSe 7	0.5	0.25	0.1	0.1
CdSe 8	0	0.375	0.1	0.1
CdSe 9	0.5	0.375	0.1	0.1
CdSe 10	0	0.5	0.1	0.1

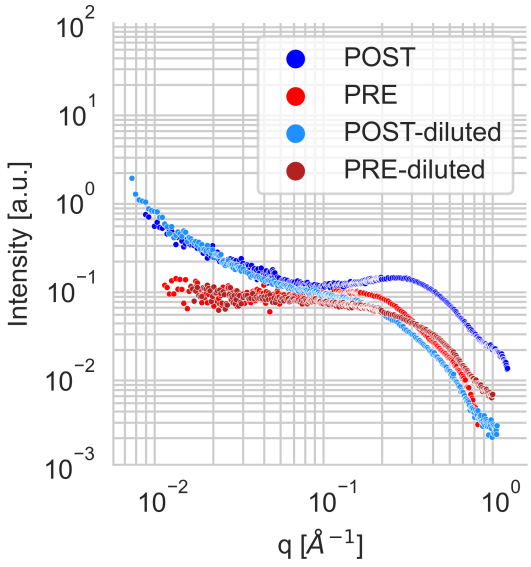

Figure S23: SAXS profiles of CdSe QDs sonochemically synthesized with oleic acid at 0.25M, oleylamine at 0.125M and metal precursors at 0.1 M each. SAXS profiles were collected for samples before and after sonochemical processing. Furthermore, to gain better insight on the particle size distribution, the samples were also diluted 4-fold in n-decane, see legend.

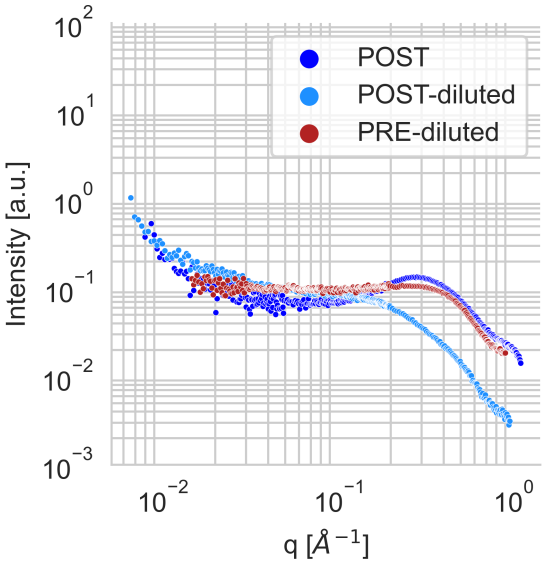

Figure S24: SAXS profiles of CdSe QDs sonochemically synthesized with oleic acid at 0.5M, oleylamine at 0.125M and metal precursors at 0.1 M each. SAXS profiles were collected for samples before and after sonochemical processing. Furthermore, to gain better insight on the particle size distribution, the samples were also diluted 4-fold in n-decane, see legend.

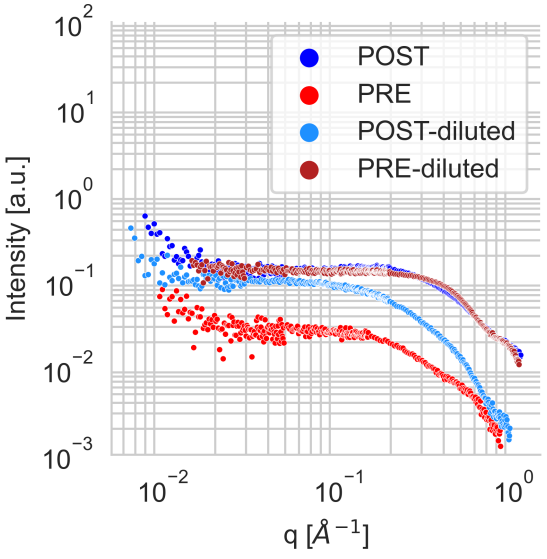

Figure S25: SAXS profiles of CdSe QDs sonochemically synthesized with oleic acid at 0M, oleylamine at 0.25M and metal precursors at 0.1 M each. SAXS profiles were collected for samples before and after sonochemical processing. Furthermore, to gain better insight on the particle size distribution, the samples were also diluted 4-fold in n-decane, see legend.

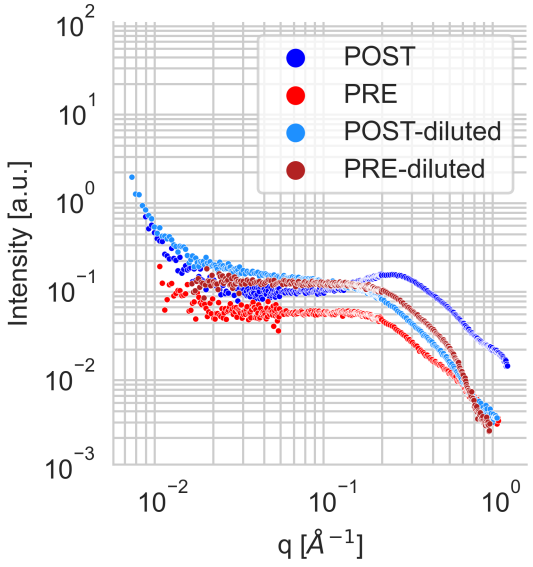

Figure S26: SAXS profiles of CdSe QDs sonochemically synthesized with oleic acid at 0.25M, oleylamine at 0.25M and metal precursors at 0.1 M each. SAXS profiles were collected for samples before and after sonochemical processing. Furthermore, to gain better insight on the particle size distribution, the samples were also diluted 4-fold in n-decane, see legend.

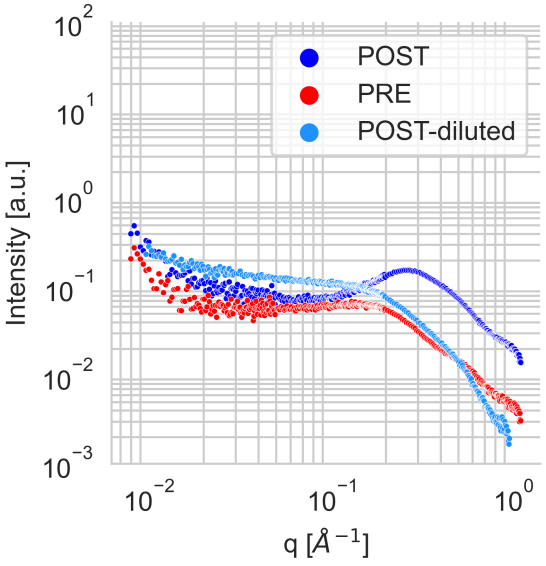

Figure S27: SAXS profiles of CdSe QDs sonochemically synthesized with oleic acid at 0.5M, oleylamine at 0.25M and metal precursors at 0.1 M each. SAXS profiles were collected for samples before and after sonochemical processing. Furthermore, to gain better insight on the particle size distribution, the samples were also diluted 4-fold in n-decane, see legend.

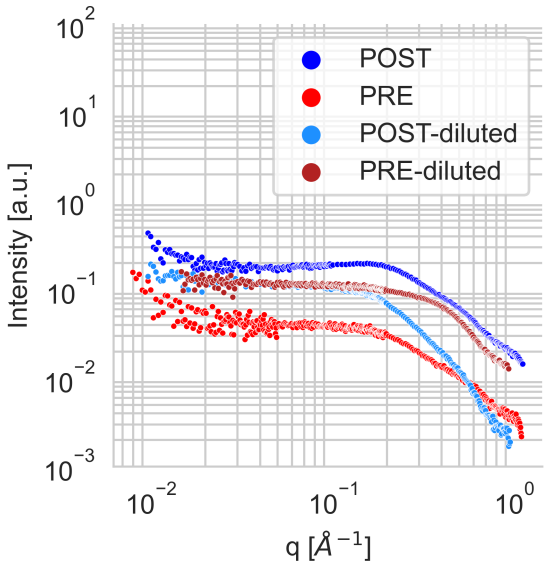

Figure S28: SAXS profiles of CdSe QDs sonochemically synthesized with oleic acid at 0M, oleylamine at 0.375M and metal precursors at 0.1 M each. SAXS profiles were collected for samples before and after sonochemical processing. Furthermore, to gain better insight on the particle size distribution, the samples were also diluted 4-fold in n-decane, see legend.

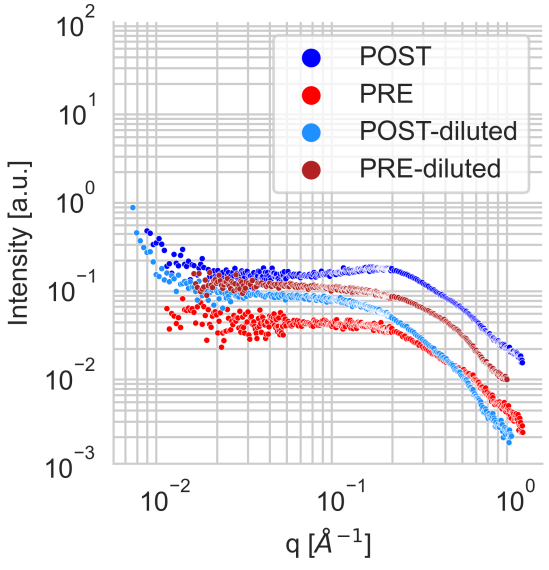

Figure S29: SAXS profiles of CdSe QDs sonochemically synthesized with oleic acid at 0M, oleylamine at 0.5M and metal precursors at 0.1 M each. SAXS profiles were collected for samples before and after sonochemical processing. Furthermore, to gain better insight on the particle size distribution, the samples were also diluted 4-fold in n-decane, see legend.

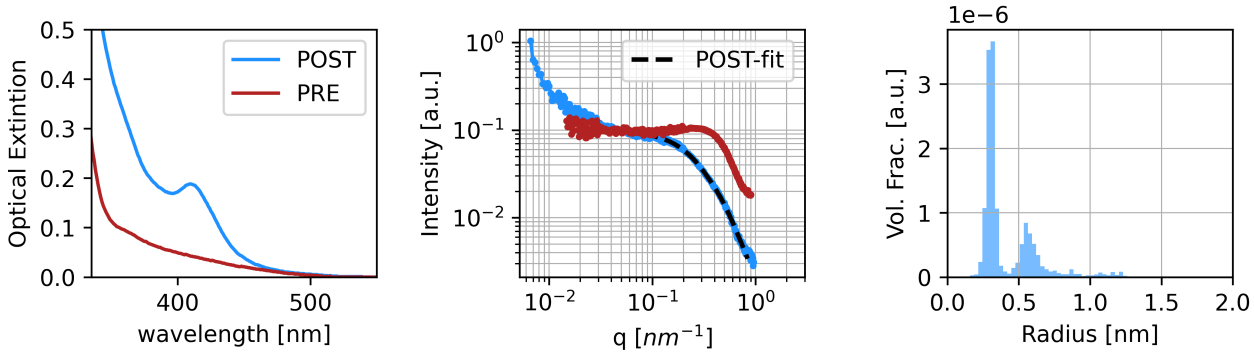

Figure S30: Summary of results for the characterization of samples CdSe 4 (0.5 M OlAc, 0.125 M OlAm and 0.1M for both CdAc and Se). In all the plots displayed, the red colored dataset represents the sample in its pre-sonicated state, whereas the blue dataset depicts the samples in its post-sonications state. Left) Uv-Vis spectra for the CdSe4 samples diluted in Octadecene (1:10). Center) Reduced SAXS profiles. Both curves represents the samples diluted 25% in n-decane for better data resolution. Right) Histogram of particle sized obtained from fitting the SAXS data (center) using the McSAS software. No fitting was successful for CdSe4 in its pre-sonicated state due to the presence of a structure factor. This indicates a high-concentration of particle which are interacting with each other.

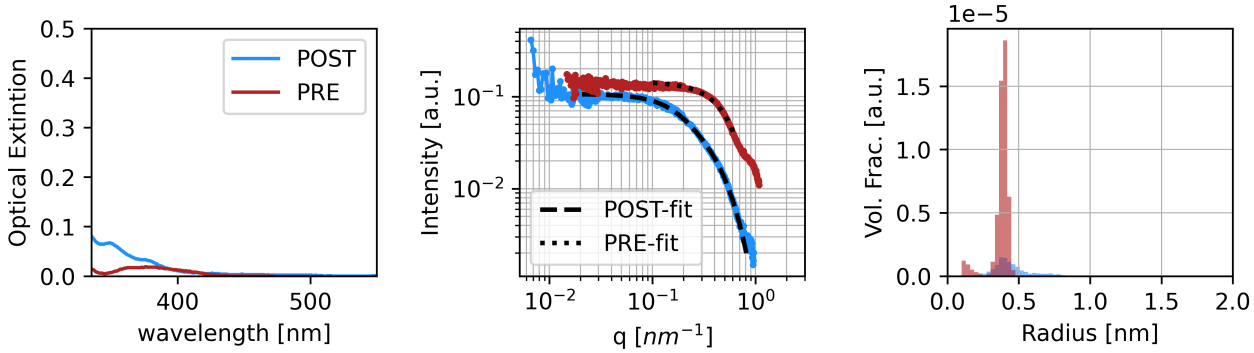

Figure S31: Summary of results for the characterization of samples CdSe 5 (0 M OlAc, 0.25 M OlAm and 0.1M for both CdAc and Se). In all the plots displayed, the red colored dataset represents the sample in its pre-sonicated state, whereas the blue dataset depicts the samples in its post-sonications state. Left) Uv-Vis spectra for the CdSe 5 samples diluted in Octadecene (1:10). Center) Reduced SAXS profiles. Both curves represents the samples diluted 25% in n-decane for better data resolution. Right) Histogram of particle sized obtained from fitting the SAXS data (center) using the McSAS software.

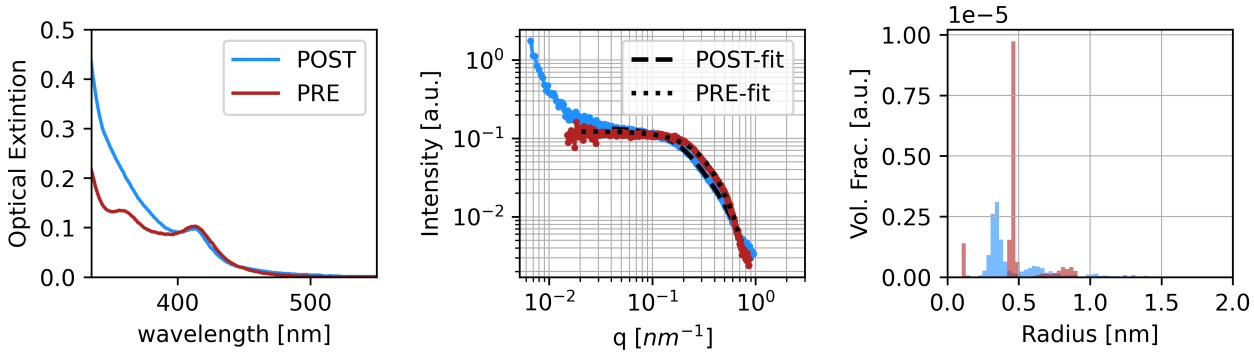

Figure S32: Summary of results for the characterization of samples CdSe 6 (0.25 M OlAc, 0.25 M OlAm and 0.1M for both CdAc and Se). In all the plots displayed, the red colored dataset represents the sample in its pre-sonicated state, whereas the blue dataset depicts the samples in its post-sonications state. Left) Uv-Vis spectra for the CdSe 6 samples diluted in Octadecene (1:10). Center) Reduced SAXS profiles. Both curves represents the samples diluted 25% in n-decane for better data resolution. Right) Histogram of particle sized obtained from fitting the SAXS data (center) using the McSAS software.

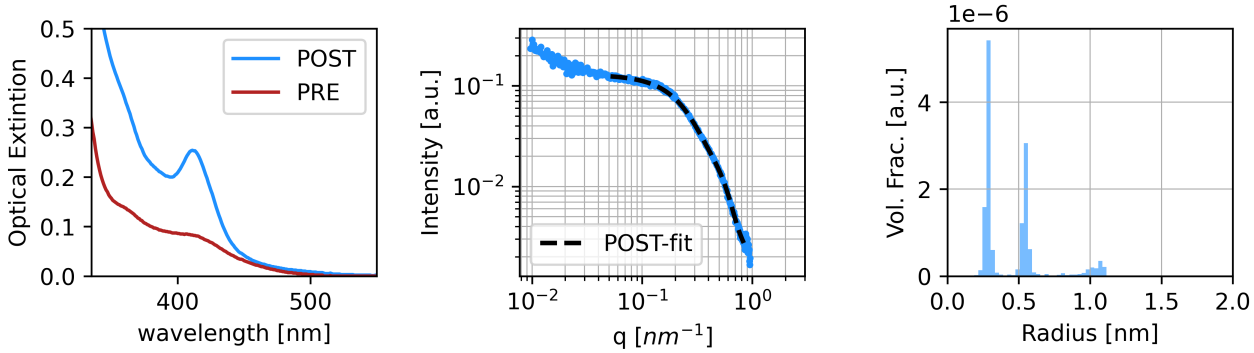

Figure S33: Summary of results for the characterization of samples CdSe 7 (0.5 M OlAc, 0.25 M OlAm and 0.1M for both CdAc and Se). In all the plots displayed, the red colored dataset represents the sample in its pre-sonicated state, whereas the blue dataset depicts the samples in its post-sonications state. Left) Uv-Vis spectra for the CdSe 7 samples diluted in Octadecene (1:10). Center) SAXS spectra of CdSe7 samples. Both curves represents the samples diluted 25% in n-decane for better data resolution. Right) Histogram of particle sized obtained from fitting the SAXS data (center) using the McSAS software. No data for the pre-sonication state is displayed for the SAXS profile and fitting as the sample showed intensities matching the buffer one, indicating an overly-diluted samples.

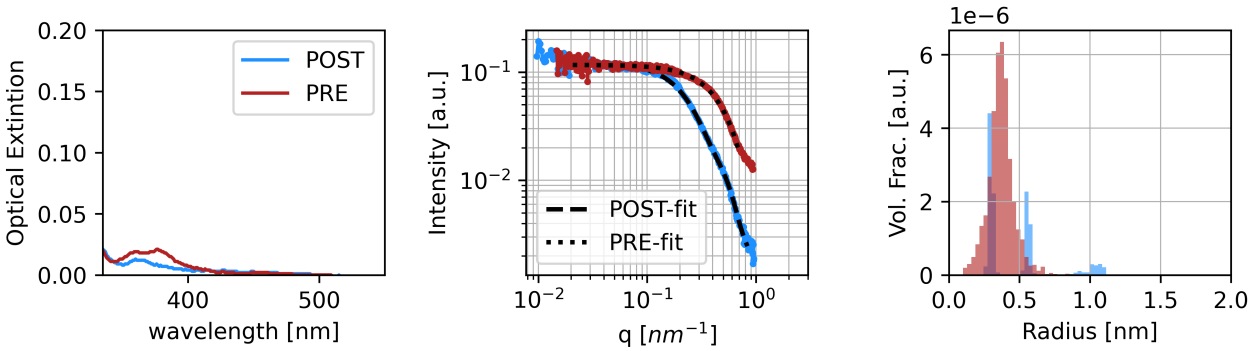

Figure S34: Summary of results for the characterization of samples CdSe 8 (0 M OlAc, 0.375 M OlAm and 0.1M for both CdAc and Se). In all the plots displayed, the red colored dataset represents the sample in its pre-sonicated state, whereas the blue dataset depicts the samples in its post-sonications state. Left) Uv-Vis spectra for the CdSe 8 samples diluted in Octadecene (1:10). Center) SAXS spectra of CdSe7 samples. Both curves represents the samples diluted 25% in n-decane for better data resolution. Right) Histogram of particle sized obtained from fitting the SAXS data (center) using the McSAS software.

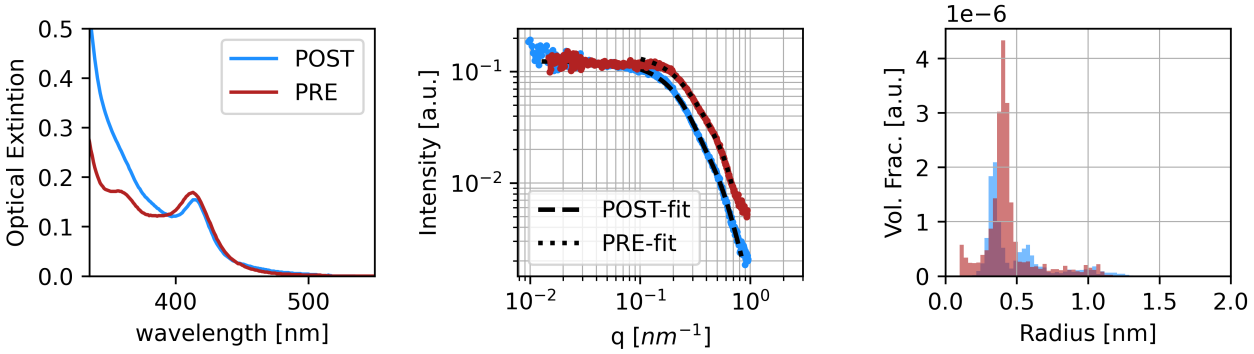

Figure S35: Summary of results for the characterization of samples CdSe 9 (0.5 M OlAc, 0.375 M OlAm and 0.1M for both CdAc and Se). In all the plots displayed, the red colored dataset represents the sample in its pre-sonicated state, whereas the blue dataset depicts the samples in its post-sonications state. Left) Uv-Vis spectra for the CdSe 9 samples diluted in Octadecene (1:10). Center) SAXS spectra of CdSe7 samples. Both curves represents the samples diluted 25% in n-decane for better data resolution. Right) Histogram of particle sized obtained from fitting the SAXS data (center) using the McSAS software.

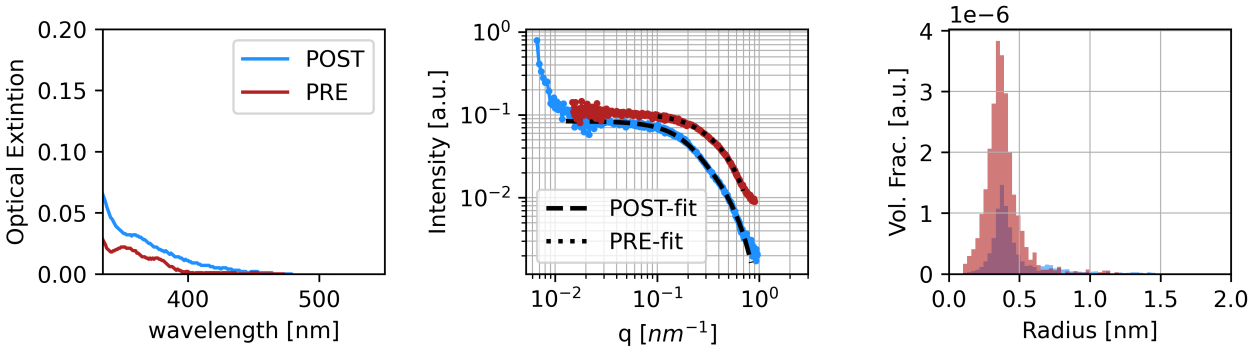

Figure S36: Summary of results for the characterization of samples CdSe 10 (0 M OlAc, 0.5 M OlAm and 0.1M for both CdAc and Se). In all the plots displayed, the red colored dataset represents the sample in its pre-sonicated state, whereas the blue dataset depicts the samples in its post-sonications state. Left) Uv-Vis spectra for the CdSe 10 samples diluted in Octadecene (1:10). Center) SAXS spectra of CdSe7 samples. Both curves represents the samples diluted 25% in n-decane for better data resolution. Right) Histogram of particle sized obtained from fitting the SAXS data (center) using the McSAS software.