One-Pot Synthesis of High-Quality Zinc-Blende CdS Nanocrystals

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Measurement of the number of stable nuclei in the synthesis of CdS nanocrystals

In the nanocrystal nucleation stage, two types of nuclei are often formed: (a) unstable nuclei and (b) stable nuclei. While unstable nuclei will dissolve with particle growth, the stable ones will grow to form nanocrystals. Therefore, the number of stable nuclei (N_{sn}) is equal to the number of nanocrystals (N_n) in the growth stage, and so the concentration of stable nuclei (C_{sn}) is equal to the concentration of nanocrystals (C_n). In other words, the concentration of stable nuclei can be obtained by measuring the concentration of nanocrystals in the growth stage.

$$N_{sn} = N_n \quad (1)$$

$$C_{sn} = C_n \quad (2)$$

Finally, we can obtain the number of stable nuclei by Eq. 3.

$$N_{sn} = C_{sn} * V_0 = C_n * V_0 \quad (3)$$

Where, V_0 is the volume of solution during the nucleation stage

In this paper, UV-Vis spectrometry is used to measure the concentration of nanocrystals in the synthesis (Eq. 4).

$$C_n = A/(\varepsilon * l) \quad (4)$$

Where, ε is the absorption coefficient at the first absorption peak of the nanocrystals, l is pathlength, and A is absorbance.

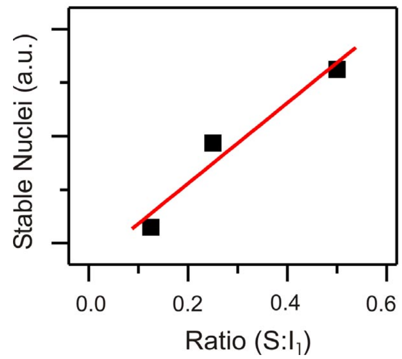
$$C_{n1} / C_{n2} = [(A_1/(\varepsilon_1 * l)]/[(A_2/(\varepsilon_2 * l)] \quad (5)$$

$$C_{n1}/C_{n2} = A_1/A_2 \quad (6)$$

$$N_{sn1}/N_{sn2} = A_1/A_2 \quad (7)$$

The absorption coefficient (ε) depends on the size of the nanocrystals, and same-sized CdS particles have a nearly identical absorption coefficient regardless of their surface passivation.[1] Therefore, when measuring same-sized particles in different reactions, Eq. 5 becomes Eq. 6. Then the combination of Eqs. 6 and 3 yields Eq. 7. So, the ratio between the numbers of stable nuclei in two different reactions equals that of the optical density of the aliquots. In other words, Eq. 7 allows the comparison among the numbers of nuclei in different reactions without knowing the absorption coefficients of the nanocrystals.

Six syntheses were carried out to check the effect of I_2 on the number of stable nuclei. The amount of chemicals except I_2 was kept the same as in the typical experiment (main text). The ratios of S: I_2 were 1:1/32, 1:1/16, 1:1/8, 1:1/4, 1:3/8, and 1:1/2. In each synthesis at least three different-sized particles were chosen to evaluate the number of stable nuclei. Then, the number of stable nuclei was normalized with that found in the synthesis when the ratio of S:I_2 was 1:1/32. The relative number of nuclei as a function of the ratio of S: I_2 is shown in Figure 2A in main text (solid boxes). In addition, three syntheses were carried out to check the effect of I_1 on the number of stable nuclei. The relative number of nuclei as a function of the ratio of S:I_1 is shown in Supporting Figure 1 (solid boxes).



Supporting Figure 1. The number of stable nuclei in the synthesis with different S:I_1 ratios. The number of stable nuclei increases with the amount of initiator I_1.

Sample preparation for X-ray diffraction (XRD).

Powder X-ray diffraction patterns were measured on a Philips APD 3720 X-ray diffractometer with Cu Kα radiation. Approximately 8 mg of CdS nanocrystals were dispersed in a minimum volume of toluene. The nanocrystal solution was deposited onto low-scattering quartz plates, and the solvent was evaporated under mild vacuum.

Reference:

1. Yu, W. W.; Qu, L.; Guo, W.; Peng, X. *Chem. Mater*. **2003**, *15*, 2854.