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## Hot injection method ppt

Quantum dots of selenium (CdSe) (Q-dots) are prepared using an uncoordinating octaded solvent instead of the trioxysphine oxide (TOPO) coordination agent. Reaction processes were carried out at different temperatures of 240°, 260°, 280° and 300° C under nitrogen atmosphere. Prepared CdSe Q-dots that are very stable show even size distribution and adjustment of optical absorption and photoluminescence (PL). The temperature of growth significantly affected the size of the particles; spectral behaviour, energy band gap and PL intensity and full width at half maximum (FWHM). Three different methods were used to determine particle size, and the average particle size of cdse Q-dots is 3.2 - 4.3 nm, grown at different temperatures. In addition, the technique of exchanging ligands prepared stable and mono-scattered cdse dots soluuous in water. Thus, water soluble dots, which are sensitive to basic pH can be important for biological applications. Semiconductor nanocrystals (NC), i.e. quantum dots (Q dots) have attracted a lot of attention due to their unique optical properties dependent on size, namely absorption and photoluminescence (PL).1–51. D. L. Klein, R. Roth, A. K. L. Lim, A. P. Alivisatos and P. L. McEuen, Nature 389, 699 (1997). . I. L. Medintz, H. T. Uyeda, E. R. Goldman and H. Mattoussi, Nature Materials 4, 435 (2005) . H.J. Lee, J. H. Yum, H.C. Leventis, S.M. Zakeeruddin, S. A. Haque, P. Chen, S. Il. Seok, M. Gratzel and Md. K. Nazeeruddin, J. Phys. Chem.C 112, 11600 (2008). . M.S. Mehata, Appl. Mr. Phys. Mr. Lett. 100, 151908 (2012). . M.S. Mehata, Sci. Tail. 5, 12056 (2015). CdSe Q dots can be adjusted within the visible wavelength range of 475 - 670 nm and used for a wide range of possible applications, including light emitting diodes (LEDs), single-electron photovoltaic transistors, and fluorescent markings for biological imaging.1-91. D. L. Klein, R. Roth, A. K. L. Lim, A. P. Alivisatos and P. L. 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Semiconductor nanocrystals or Q dots such as CdX and PbX (X = S, Se and Te) and metal-chalkogenide-based are used to collect light energy.5,8,115. M.S. Mehata, Sci. Tail. 5, 12056 (2015). . J. H. Bang and P. V. Kamat, ACS Nano 3, 1467 (2009) . . A. Kongkanand, K. Tvrđy, K. Takechi, M. Kuno and P. V. Kamat, J. Am. Chem. Soc. 130, 4007 (2008). In semiconductor question points, excited electrons are injected into the semiconductor of large band spacing such as (ZnO or TiO2), and the holes are emptied by redox par.88. J. H. Bang and P. V. Kamat, ACS Nano 3, 1467 (2009). One of the most exciting and challenging questions is to produce mono-scattered CdSe Q-dots. There are different methods for growing CdSe Q-dots in which the most popular and effective route is the chemical method of hot injection.12-1512. J. R. Heine, J. Rodriguez-Viejo, M. G. Bawendi and K. F. Jensen, J. Cryst. Growth 195, 564 (1998). 98)0646-013. D. Crouch, S. Norager, P.O. Brien, J. H. Park, and N. Pickett, Phil. 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Smatralo se da je TOPO bitan zahtjev za proizvodnju visoko monoprostornih nanokristala II– VI, međutim, TOPO može proizvesti nepoznate onečišćujuće tvari.23–2723. D. Battaglia i X. G. Peng, Nano Lett. 2, 1027 (2002). . W. W. Yu i X. G. Peng, Angew Chem. Int. Ed. 41, 2368 (2000). 20020703)41:13&lt;2368::AID-ANIE2368&gt;3.0.CO;2-G25. C. R. Bullen i P. Mulvaney, Nano Lett. 4, 2303 (2004). . J. Jasieniak, C. Bullen, J.V. Embden and P. Mulvaney, J. Phys. Chem.B 109, 20665 (2005). . M. Sun i X. Yang, J. Phys. Chem.C 113, 8701 (2009). TOPO bi se mogao zamijeniti nekoordinativnim otkecenom otapala (ODE) i proizvodi mono-raspršene nanokristale CdSe.17–2617. C.B. Murray, D. J. Norris i M. G. Bawendi, J. Am. Chem. Soc. 115, 8706 (1993). . L. Manna, E.C. Scher i A. P. Alivisatos, J. Am. Chem. Soc. 122, 12700 (2000). 19. X. G. Peng, L. Manna, W. D. Yang, J. Wickham, E. Scher, A. Kadavanich i A. P. Alivisatos, Priroda 404, 59 (2000). . Q. Dai, D. Li, H. Chen, S. Kan, H. Li, S. Gao, Y. Hou, B. Liu i G. Zou, J. Phys. Chem.B 110, 16508 (2006). . L. H. Qu i X. G. Peng, J. Am. Chem. Soc. 124, 2049 (2002). . X. G. Peng, Chem. Eur. J. 8, 334 (2002). 20020118)8:2&lt;334::AID-CHEM334&gt;3.0.CO;2-T23. D. Battaglia i X. G. Peng, Nano Lett. 2, 1027 (2002). . W. W. Yu i X. G. Peng, Angew&lt;/334::AID-CHEM334&gt; &lt;/2368::AID-ANIE2368&gt; Int. Ed. 41, 2368 (2000). 20020703)41:13&lt;2368::AID-ANIE2368&gt;3.0.CO;2-G25. C.R. Bullen and P. Mulvaney, Nano Lett. 4, 2303 (2004). . J. Jasieniak, C. Bullen, J.V. Embden and P. Mulvaney, J. Phys. Chem.B 109, 20665 (2005). In addition to the ode non-compliant solvent, a supportive solvent trimethylpentil phosphic acid (TMPPA), hexadecail amine (HDA) and tributyl phosphine (TBP) were also used to control the reaction state. Thus, replacing TOPO makes synthesis cheaper, safer and greener than other solvents.17–2617. C.B Murray, D.J. Norris and M. G. Bawendi, J. Am. Chem. Soc. 115, 8706 (1993). . L. Manna, E.C. Scher and A. P. Alivisatos, J. Am. Chem. Soc. 122, 12700 (2000). 19. X. G. Peng, L. Manna, W. D. Yang, J. Wickham, E. Scher, A. Kadavanich and A. P. Alivisatos, Nature 404, 59 (2000). . Q. Dai, D. Li, H. Chen, S. Kan, H. Li, S. Gao, Y. Hou, B. Liu and G. Zou, J. Phys. Chem.B 110, 16508 (2006). . L.H. Qu, and X.G. Peng, J.Am. Chem. Soc. 124, 2049 (2002). . X.G. Peng, Chem. Eur. J. 8, 334 (2002). 20020118)8:2&lt;334::AID-CHEM334&gt;3.0.CO;2-T23. D. Battaglia and X.G. Peng, Nano Lett. 2, 1027 (2002). . W.W. Yu and X.G. Peng, Angew Chem. Int. Ed. 41, 2368 (2000). 20020703)41:13&lt;2368::AID-ANIE2368&gt;3.0.CO;2-G25. C.R. Bullen and P. Mulvaney, Nano Lett. 4, 2303 (2004). . J. Jasieniak, C. Bullen, J.V. Embden and P. Mulvaney, J. Phys. Chem.B 109, 20665 (2005). it is therefore desirable to make environmentally friendly Q-point dots less using TOPO. In this report, we have prepared CdSe Q-dots in a TOPO free environment at a different temperature of 240 to 300° C. Prepared CdSe Q-dots are homogeneous, stable and adjustable in size (3.2-4.3 nm) and show significant quantum yield (QY). Water-solubable QY Q-dots are higher at higher pH.All chemicals including kadmium oxide (CdO), selenium powder (Se) (99.9%), triocetylphosfin (TOP, 90%), oleic acid (OA, 90%), mercapto propionic acid (MPA, 95%) and 1-octaded (ODE, 90%) purchased from Aldrich and used without further purification. Synthesis was performed in a nitrogen (N2) environment after the described procedure earlier33. H.J. Lee, J. H. Yum, H.C. Leventis, S.M. Zakeeruddin, S. A. Haque, P. Chen, S. Il. Seok, M. Gratzel and Md. K. Nazeeruddin, J. Phys. Chem.C 112, 11600 (2008). with some modifications and without consideration of TOPO. To &lt;/2368::AID-ANIE2368&gt; &lt;/334::AID-CHEM334&gt; rigorously oxygenated and environmentally free water for synthesis, all substances are dried and degassed before use. Initially, the solutions of cd precursors and Sea were prepared separately. To prepare the precursors, 0.0205 gm Se powder, 0.12032 ml TOP and 2 ml of octadecene (ODE) are mixed in a glass bottle and put the mixer on a hot plate stir for about one hour. Similarly, a mixer of 0.102 gm CdO, 1.23 ml of oleic acid and 14 ml ode was taken for the CD precursor and then the mixture (CdO-OA-ODE) was transferred to three neck flask and continuously mixed with rotational heating to 180 ° C until the solution became impenehus. For the growth of CdSe Q-dots, the further temperature is increased up to 240 ° or 260 ° or 280 ° or 300 ° C, when the solution attends the required temperature, and then 2 ml of the seed solution is injected into three neck flask (which contained a Cd solution) with constant mixing. After injection the precursor temperature dropped about 10 ° C from the visited value. The color of the reaction solution changed immediately after the injection of the precursor Se, which indicated a rapid nucleus and growth. After about 60 seconds of injection of the precursor Se, the heating mantle was removed, and then 20 ml methanol was added to the reaction mixture and eventually centrifuged the resulting solution at 6500 rpm for 10 minutes. The process is repeated 2-3 times to obtain clean CdSe Q-dots, and then the resulting eluent is sprayed into the toluene for further investigation. Similarly, the reaction procedure was repeated to obtain CdSe dots at different growth temperatures of 260°, 280° and 300° C.The method of preparing cdse Q-dots involved injecting a cold solution of Se precursors into a hot liquid (CDO mixture - OA-ODE). Injection of the precursor leads to the immediate formation of CdSe nuclei. With the increase in the temperature of growth, the size of the resulting CdSe nanocrystals increases. The formation of new nuclei is prevented at each growth temperature by reducing the growth temperature by about 10° C. So it seems that below the temperature of 300 ° C the nucleation process is controlled and almost mono-dispersed CdSe Q-dots.In is produced to



use point issues for biological applications such as marking and recording, we need to prepare water-soluble Q-dots because almost all biological environments are aquatic. However, the cdse dots prepared above are not soluble in water due to the presence of ligands (OA/TOP). These hydrophobic ligales can be replaced by water-soluble two-functioning MPA molecules, following the method of ligand exchange or phase transformation,2828. W.C. W. Chan and S.M. Nie, Science 281, 2016 (1998). in which one end is connected to the dots of the question and the other to the other is hydrophilic and reactive (Scheme 1). In the process, oleic acid is limited J. Chen, J. L. Song, X. W. Sun, W. Q. Deng, C. Y. Jiang, and W. Lei, Appl. Mr. Phys. Mr. Lett. 94, 153115 (2009). solutions were dissolved in 2 ml chloroform to obtain a high concentration (the optical density of the cdse solution is ~2) and add the same volume of the water solution containing MPA in a strong mixing. The amount of MPA is adjusted to approximately 150% of total CD atoms. After 3 hours, the NC's were transferred to the water phase, but the solution was not optically clear to pH ~5 to 8, possibly due to inter-particle hydrogen binding of carboxylic ligand functions. In order to remove excess MPA from the supernatant solution, these particles are separated by centrifugation and decantation. At higher pH (&gt;8), successive particle reshopping into water provides a clear solution. Optical measurements in stable condition were performed in toluen and water (for water-soluged Q-dots) at ambient temperature. Absorption spectra have been reported by UV/VIS/NIR spectrometer, Lambda-750, Perkin-Elmer. Photoluminescence spectra (PL) collected horiba jobin Yvon Fluorolog-3 spectrofluorometer equipped with double grilles at excitation as well as on monochrome emissions (1200 grooves/mm) and Hamamatsu R928P photomultiplier tubes (PMT). The source of the excitation was a 450 Watt CW xenon lamp. An instrumental correction was applied on all PL spectra to correct the wavelength-dependent PMT response and variations in the intensity of the lamp output and the efficiency of the grille. The excitation and slit width of the emission was selected to be 1.0 nm and the same for all measurements, and the excitation wavelength was 450 nm. High-resolution electron microscopy (HRTEM) images on which the tecnai G2 20 electron microscope worked at a voltage acceleration of 200 KV. For HRTEM images, CdSe samples are prepared on 300-mesh carbon-coated Cu grilles, a very small amount of Q-dots are scattered in toluen (for a dilute solution) and then placed on carbon-coated Cu grilles and then dried patterns. An energy dispersive X-ray (EDX) spectrum sample and X-ray diffraction (XRD) recorded Hitachi-640 when accelerating voltages of 15 KV and BRUKER-D8, respectively. Figure 1 shows XRD samples of CdSe Q dots coated over a glass substrate grown at a temperature of 240° C. XRD patterns expand compared to those in bulk, thus indicating the tops and spread to the nanocrystallist nature of cdse Q dots. The tips obtained at (2θ) angles (25.6), (42.7) and (50.0) correspond to the reflection from (111), (220) and (311) crystal planes respectively, as determined by standard JCPDS data (Card No. 111) and (311). The XRD sample clearly showed a hexagonal phase with a wurtzite structure and also indicates preferential crystallite growth in a given direction (111) plane. The average crystal size (d) was calculated on the basis of the width of the top (111) of the aircraft using the Scherr formula: where the ηητ, β and θ X-ray wavelength, full width at half maximum (FWHM) and Bragg's reflection angle are. The average crystal size calculated from the equation (1) is 4.2 nm. Figure 2 shows HRTEM images and corresponding histograms of distribution of the statistical size of CdSe Q dots grown at different temperatures of 240°, 260° and 300° C. HRTEM's images confirm that the prepared dots of the question are almost spherical uniform in size and shape. The images show particle size change, particle size increases monotonously with an increase in growth temperature from 240° to 300°C, indicating that the reaction temperature has essential effects on CdSe Q-dots. The average particle size of cdse Q-dots is 3.2 - 4.3 nm grown at a temperature of 240 ° - 300 ° C. In Sl. are given a spectrum of energy dispersive X-rays (EDX) and a percentage of the amount of each identified cdse Q-dot element grown at different temperatures. S13030. See additional material on fig bed. S1. S1. and Table I. TABLE I. Percentage ingredients of each identified element in the CDSE Q-point sample. SamplesElements Weight %Atom %CdSe at 240° CCd66.3258.04Se33.6841.96CdSe at 300° CCd72.3665.20Se27. 6434.80Figure 3 shows the absorption spectrum of cdse Q-dots in a stable state dispersed in toluen grown at a temperature of 240 - 300 ° C. The lowest energy absorption belt has shifted towards a longer wavelength, that is, from 547 to 572 nm as the growth temperature increases from 240 to 300° C. The red shift in maximum absorption clearly indicates an increase in particle size. The energy band gap or optical energy band (e.g.) of cdse Q-dots grown at different temperatures was estimated from the absorption spectrum using the Tauc ratio, as given: α is the absorption coefficient, the hūest is photon energy, A is a constant and n assumes values 1/2, 2, 3/2 and 3 for permissible direct, indirect, prohibited direct and indirect transitions. For permissible direct transitions, the relationship can be given as: Parcels between (tññ)2 vs (ññññ) obtained at different temperatures are given on etc. 4. A straight line of parcels over a wide range of photonic energy indicates a direct type of transition. E.g. Q-points are determined by extrapolating linear areas on the energy wast. The estimated values of NPR for CDSE Q-points grown at temperatures of 240°, 260°, 280° and 300° C, respectively, are 2.81, 2.78, 2.74 and 2.60 eV respectively, and are significantly higher than group CdSe (Egbulk=1.74 eV),3131. W.E. Mahmoud and H.M. El-Mallah, J. Phys. D: Appl. Mr. Phys. 42, 35502 (2009). they are the band spacing between the highest occupied molecular orbital (HOMO) to the lowest uninhased molecular orbital (LUMO) CdSe Q-dots.Furthermore, particle size was also estimated from the optical spectrum using effective mass approximation.3232. S. V. Gaponenko, Optical Properties of Semiconductor Nanocrystals (Cambridge University Press, Cambridge, 1998). A powerful closing mode and shutting energy of the first excited electronic state (lowest energy absorption band) can be approximated Brus equation: Eg = Egbulk+ h2 π22 R21me\* 1mh\* - 1.8e24π€€o R,(4) where the optical transition energy of the CDSE Q-dot, Egbulk is an energy gap in bulk cdse and I\* and mh\* are an effective mass of conductor belt electrons and valence band holes CdSe, respectively. The value of me\* is 0.13 m0 and mh\* is 0.45m033,3433. E. Cohen and M.D. Sturge, Phys. Rev.B 25, 3828 (1982). . C. T. Giner, A. Debernardi, M. Cardona, E.M. Proupin and A. I. Ekimov, Phys. Rev.B 57, 4664 (1998). and m0 is the rest mass of electrons (9.1 × 10–31 kg). The estimated size of CdSe Q dots grown at temperatures of 240°, 260°, 280° and 300° C is 3.2, 3.4, 3.6 and 4.0 nm, respectively. The size of the question dots estimated by the simplified relationship Brus (4) is approximately the same as estimated by HRTEM. Figure 5 shows the PL spectrum of cdse Q-dots grown at temperatures of 240°, 260°, 280° and 300° C. Excitation wavelength was 450 nm. PL spectra show an apparent red shift, i.e. pl band maximum shifted from 565 to 580 nm as the growth temperature increases from 240° - 300° C. However, the FWHM PL monotonously decreases with an increase in the temperature of growth. As an example, FWHM is 33.7, 33.3, 31.5 and 29.9 nm at 240°, 260°, 280° and 300°C, respectively. At 300°C, the FWHM is very small (&lt;30 nm) indicating that the size distribution of the Q-dots obtained is almost homogeneous/narrow, which is comparable to one of the best results obtained using TOPO.3535. L. Qu, Z.A. Peng, and X.G. Peng, Nano Lett. 1, 333 (2001). pl QY CdSe Q-point (Φs) is evaluated using relationships; ΦS=ΦRISIR1– 10–AR1– 10–ASnS2nR2(5) where ΦR absolute QY of reference genus 6G (0.95), IR and IS are integrated fluorescence intensity reference (rodmin 6G) and sample (Q dots), i.e. AR and AS are adequate absorption, a nR and nS are a refraction solvent index (toluene for Q-point dots and methanol for rodmin 6G). The estimated QY for CdSe Q-dots is 0.16 ± 0.01 at a temperature of 240 ° C.In times synthesis, oleic acid is used as a means of closure, which always forms a shell on the surface of cdse. Oleic Activity increases as the growth temperature increases. High growth temperature leads to faster connection / dissociation of oleic acid dynamics on the surface. When oleic acid detaches from the particle surface, smaller particles become larger to reduce surface energy. Therefore, at a higher reaction temperature, a larger particle size and a higher growth rate are obtained. It was suggested that at a higher temperature, smaller particles decreased while larger ones grew due to Oswald's maturation, and the size distribution became more uniform.3636. D. I. Lubyshev, P. G. Borrero, E. Marega, E. Pettiprez, N. Lascala and P. Basmaji, Appl. Mr. Phys. Mr. Lett. 68, 205 (1996). as a result, the PL spectrum became narrow compared to obtained at a lower growth temperature. The displacement of Stoke (the difference between the lowest energy absorption and PL maximum) decreases from 20 to 8 nm as the growth temperature increases from 240° to 300° C, which takes into account the interaction of coulomb and the effects of dielectric mismatches.3737. A. Bagga, P.K. Chattopadhyay, and S. Ghosh, Phys. Rev.B 74, 035341 (2006). 6 and 7 show optical absorption and PL spectra of soluble Water-solue CdSe Q-dots measured at different pH with constant concentration. The size and PL maximum of the initial CdSe Q-dots used to prepare water-soluged Q-dots is about 3.5 nm and 553 nm respectively. The lowest absorption belt (HOMO-LUMO band gap) cdse Q-dot at different pH is almost the same, that is, it lies on the same wavelength region. A similar trend has been observed for PL spectra, no significant spectral shift is obtained at different pHs. However, pl intensity changes with different pH, especially from 8.5 to 11.5, and PL intensity is highest at pH 11.5, although pl does not exactly follow the Gaussian form, which may be due to the presence of ligands. At the lower and higher pH spectrum, the PL spectrum shows no significant changes. QY increases by increasing the pH of water (from 8.5 -11.5), as shown in Table II. In basic pH, the cdse Q-dot affinity is more favorable due to the deprotonation of the group of tioalkyl acid, which becomes negatively charged and seeks coordination between ligands and semiconductor surfaces which has led to improved dispersability of nano particles and QY.3838. A.M. Coto-Garcia, T.M. Fernandez-Arguells, J.M. Costa-Fernandez, A. Sanz-Medel, M. Valledor, J.C. Campo and F.J. Ferrero, J. Nanopart. Res. 15, 1330 (2013). II Quantum yield cdse Q-dots soluble in water at different pH.Sample/pHQQuantum yieldCdSe (pH) 8.50.03 ± 0.01CdSe (pH) 9.50.04 ± 9.50.040.01CdSe (pH) 10.60.06 ± 0.01CdSe (pH) 11.50.10 ± 0.01CdSe Q-dots are prepared with hot colloidal method in a TOPO free environment. The prepared CdSe Q-dots have a hexagonal phase with a wurtzite structure. With the increase in the temperature of the reaction system, i.e. the temperature of growth from 240° to 300° C: (i) the average particle size of cdse Q-dots increased from 3.2 to 4.3 nm, (ii) the maximum absorption shifted towards a longer wavelength region and therefore NPR decreased from 2.8 to 2.6 eV, (iii) Stoke's displacement decreased from 18 to 8 nm and (iv) the FWHM PL spectrum decreased. In addition, after the ligand exchange process, stable Water-solumable CdSe Q-dots are prepared, as shown by higher QY at higher pH. Thus, the study also reflects that prepared water-solule-soluing CDSE Q-dots, which are sensitive to basic pH may be suitable for biological applications. The authors would like to thank the Indian Atomic Energy Department (DAE-BRNS), the Committee for Research on Nuclear Sciences in India (Ref. 2012/37P/20/BRNS) for its financial support.1 D. L. Klein, R. Roth, A. K. L. Lim, A. P. Alivisatos and P. L. McEuen, Nature 389, 699 (1997). Google ScholarCrossref2. I. 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