

Quantum Dots

1. Han, H.; Francesco, G. D.; Maye, M. M. Size Control and Photophysical Properties of Quantum Dots Prepared via a Novel Tunable Hydrothermal Route. *J. Phys. Chem. C* **2010**, *114*, 19270 – 19277 DOI: [10.1021/jp107702b](https://doi.org/10.1021/jp107702b)
 - CdSe core shell dots were synthesized in a Discover microwave at 60 – 180 °C for only two minutes
 - Cd(ClO₄)₂, Me₂NCSenH₂, Zn(ClO₄)₂, MeCSNH₂ were used as core and shell sources with water as the solvent
 - Higher temperatures produced bigger dots with a higher quantum yield. λ_{abs} from 490 – 580 nm, depending on [Cd]:[Se] ratio
 - Quantum yield drastically improved from shelling
2. Song, A; Ai, X.; Topuria, T.; Rice, P. m.; Alharbi, F. H.; Bagabas, A.; Bahattab, M.; Bass, j. D.; Kim, H-C.; Scott, J. C.; Miller, R. D. Microwave-assisted Synthesis of Monodispersed CdTe nanocrystals. *Chem. Commun.* **2010**, *46*, 4971 – 4973 DOI: [10.1039/c0cc00576b](https://doi.org/10.1039/c0cc00576b)
 - Scientist from IBM Almaden Research Center and The National Nanotechnology Center and the Petrochemicals Research Institute in Riyadh Saudi Arabia prepared monodispersed CdTe quantum dots
 - CdO and TeTBP (tributylphosphine) were combined in the presence of tetradecylphosphonic acid in octadecene.
 - The contents were heated in a CEM Discover[®] microwave with 300 W fixed power to temperatures ranging from 180-280 °C for 1-90 min
 - The high quality crystals were monodispersed with a size distribution of less than 5%
 - Tunable emission spectra (516 nm to 650 nm) by varying reaction time and temperature
 - Highly reproducible microwave assisted method for preparing CdTe nanocrystals
3. Li, W.; Li, D.; Zhang, W.; Hu, Y.; He, Y.; Fu, X. Microwave Synthesis of Zn_xCd_{1-x}S Nanorods and Their Photocatalytic Activity under Visible Light. *J. Phys. Chem. C* **2010**, *114*, 2154 – 2159 DOI: [10.1021/jp9066247](https://doi.org/10.1021/jp9066247)
 - Professor Danzhen Li at the Research Institute of Photocatalysis, State Key Laboratory Breeding Base of Photocatalysis, Fuzhou University
 - TEM showed MW method gave a more homogeneous nanorod compared to conventional heating
 - Precursors heated to 140 – 160 °C for only 10 min
 - Nanorods were monodispersed with diameters of 10 nm
 - MW sample had higher surface area and faster conversion of methy orange degradation
4. Schumacher, W.; Nagy, A.; Waldman, W. J.; Dutta, R. K. Direct Synthesis of Aqueous CdSe/ZnS-Based Quantum Dots using Microwave Irradiation. *J. Phys. Chem. C* **2009**, *113*, 12132 – 12139. DOI: [10.1021/jp901003r](https://doi.org/10.1021/jp901003r)
 - Professor Prabir Dutta, Department of Chemistry and Pathology, The Ohio State University
 - One-pot synthesis of CdSe/ZnS core/shell quantum dots using readily available and cheap starting material in water
 - CdSe nanocrystals from NaHse and Cd-MPA (3-mercaptopropionic acid) were heated in a MARS digestion vessel with Zn(NH₃)₄²⁺ solutions at 140 – 170 °C for 45 – 120 min to give 5 nm particles
 - MW synthesized QDs at 20 nM were readily detected within the macrophages after 20 min of incubation
5. Washington, A. L.; Strouse, G. F. Microwave Synthetic Route for Highly Emissive TOP/TOP-S Passivated CdS Quantum Dots. *Chem. Mater.* **2009**, *21*, 3586 – 3592. DOI: [10.1021/cm900624z](https://doi.org/10.1021/cm900624z)
 - Professor Geoffrey Strouse, Department of Chemistry and Biochemistry, Florida State University
 - Through selective microwave absorption, they demonstrate the ability to activate TOPS as an efficient sulfur donor, allowing the rapid (18 m) growth of highly emissive (PLQY=33%), Zn blended CdS quantum dots (QDs) passivated by TOP/TOPS in the 4-6 nm size regime (5% size dispersity)
 - The CdS QDs exhibited sharp absorption features and band edge photoluminescence even for the largest CdS sample
 - The use of MW chemistry for QD formation allows a highly reproducible synthetic protocol that is fully adaptable to industrial applications
6. Washington, A. L.; Strouse, G. F. Selective Microwave Absorption by Trioctyl Phosphine Selenide: Does it Play a Role in Producing Multiple Sized Quantum Dots in a Single Reaction? *Chem. Mater.* **2009**, *21*, 2770 – 2776. DOI: [10.1021/cm900305j](https://doi.org/10.1021/cm900305j)
 - Professor Geoffrey Strouse, Department of Chemistry and Biochemistry, Florida State University
 - Demonstrate unprecedented control over nucleation, growth, and Ostwald ripening in the formation of CdSe quantum dots (QDs), the quintessential quantum dot
 - The selectivity of the MW reactions is demonstrated by the ability to generate MW multiple, different sized QDs in the same reaction, where each QD component exhibits 6-7% size dispersity
 - The number of QDs in solution translates to color saturation (intensity), and the size of the QD translated to color index and is completely controlled by temperature and concentration in the MW reaction.

- The ability to repetitively generate nucleation and growth events in which a specific color index with defined color saturation is isolated from a single reaction offers potential for preparing mixed QD compositions for applications in optical barcoding, white light emitting diodes (LEDs), and photovoltaics (PVs).

7. He, Y. et al. Microwave Synthesis of Water-Dispersed CdTe/CdS/ZnS Core-Shell-Shell Quantum Dots with Excellent Photostability and Biocompatibility. *Adv. Mater.* **2008**, 20, 3416 – 3421. DOI: [10.1002/adma.200701166](https://doi.org/10.1002/adma.200701166)
- Professor Wei Huang and Lian-Hui Wang at the Institute of Advanced Materials, Nanjing University of Posts and Telecommunication and Laboratory of Advanced Materials, Fudan University
 - Synthesized CdTe/CdS/ZnS (this is a QD with a CdTe center, encased in a layer of CdS, followed by an outer layer of ZnS) QDs using MW irradiation
 - CdTe MW, 100 °C, 1 min
 - CdTe/CdS MW, 100 °C, 5 min
 - CdTe/CdS/ZnS, MW, 60 °C, 5 min. Final size of 3.4 – 4.5 nm.
 - Quantum Yield increased drastically from CdTe (30%) to CdTe/CdS/ZnS (80%)
 - This was the first example of water dispersed QDs that are made in water -which was assisted by MW irradiation
8. Roy, M. D.; Herzing, A. A.; Lacerda, S. H.; Becker, M. L. Emission-Tunable Microwave Synthesis of Highly Luminescent Water Soluble CdSe/ZnS Quantum Dots. *Chem. Commun.* **2008**, 2106 – 2108. DOI: [10.1039/b800060c](https://doi.org/10.1039/b800060c)
- Dr. Matthew Becker Polymers Division and Surface and Microanalysis Science Division, National Institute of Standards and Technology
 - Water soluble CdSe/ZnS nanoparticles with emission maxima from 511 nm to 596 nm and quantum efficiencies ranging from 11% to 28% were synthesized using MW irradiation
 - Particle size controlled by heating times. Reactions were run at lower temperatures (145 – 150 °C) under ambient atmosphere with shorter reaction times when compared to conventional reactions
 - This approach offered a great deal of control of particle size and particles were very monodispersed
9. Lovingood, D. D.; Strouse, G. F. Microwave Induced In-Situ Active Ion Etching of Growing InP nanocrystals. *Nano Lett.* **2008**, 8, 3394 – 3397. DOI: [10.1021/nl802075j](https://doi.org/10.1021/nl802075j)
- Professor Geoffrey Strouse, Florida State University, Department of Chemistry
 - Used MW irradiation and fluorinated ionic liquids to etch InP nanoparticles. Conventional procedure required HF. This technique eliminates the need for HF, making it much safer and more practical
 - 280 °C, 300 W, times of 70 s to 20 min
10. Washington, A. L.; Strouse, G. F. Microwave Synthesis of CdSe and CdTe Nanocrystals in Nonabsorbing Alkanes. *J. Am. Chem. Soc.* **2008**, 130, 8916 – 8922. DOI: [10.1021/ja711115r](https://doi.org/10.1021/ja711115r)
- Professor Geoffrey Strouse, Florida State University, Department of Chemistry
 - Synthesis of CdSe and CdTe quantum dots
 - Were found to be temperature, time, and power dependent in the presence of an Ionic Liquid and minohexadecane (strong MW absorbing solvents)
 - Temperature and time dependent in non MW absorbing solvent
 - By varying the reaction temperature, time, power, or a combination of both, Strouse and co-workers were successful in controlling particles size and quantum yields
 - Typical diameters were 2.5 – 5 nm, depending on power, temperature, and time
 - Typical Temperatures were 180 – 280 °C, time ranging from 30 – 600 sec, power 67 – 400 W
 - Higher temperatures and longer reaction times lead to larger particles with lower quantum yields
 - The higher the power the faster the ramp to temperature-which resulted in a higher quality QD.
 - Cooling at the end of this reaction played a crucial role in QD size. Slow cooling from the reaction temperature to ambient conditions resulted in larger size dispersity
11. Gerbec, J. A.; Magana, D.; Washington, A.; Strouse G. F. Microwave-Enhanced Reaction Rates for Nanoparticle Synthesis. *J. Am. Chem. Soc.* **2005**, 127, 15791 – 15800. DOI: [10.1021/ja052463g](https://doi.org/10.1021/ja052463g)
- See above reference
12. Firth, A. V.; Tao, Y.; Wang, D.; Ding, J.; Bensebaa, F. Microwave Assisted Synthesis of CdSe Nanocrystals for the Straightforward Integration into Composite Photovoltaic Devices. *J. Mater. Chem.* **2005**, 15, 4367 – 4372 DOI: [10.1039/b505891k](https://doi.org/10.1039/b505891k)
- Dr. Andrea Firth, National Research Council, Ottawa Canada
 - A preparative route for a nanocrystalline CdSe/polymer nanocomposite was developed with microwave irradiation.
 - Microwave assisted synthesis is used to initiate the reaction of relatively safe, inexpensive and air stable precursors.
 - Particles size may be varied from 20 Å to 60 Å in diameter. The CdSe nanocrystals show near band-edge photoemission, are crystalline

and may be incorporated into a poly(9,9-dioctylfluorene-2,7-diylalt-9-octylcarbazole-3,6-diyl) copolymer.
- Polymer/CdSe composite can be directly cast, without complicated work-up, for the preparation of photovoltaic devices.

Inorganic Nanomaterial

13. Yu, M.; Funke, H. H.; Noble, R. D.; Falconer, J. L. H₂ Separation Using Defect-Free, Inorganic Composite Membranes. *J. Am. Chem. Soc.* **2011**, *133*, 1748 – 1750. DOI: [10.1021/ja108681n](https://doi.org/10.1021/ja108681n)
- Professor Miao Yu, Department of Chemical and Biological Engineering, University of Colorado
 - Using a CEM MARS with Teflon vessels, SAPO-34 zeolite seed crystals were prepared for membrane synthesis
 - Molecular layer deposition (MLD) was used to deposit a thin, porous layer of alumina upon the zeolite support
 - Composite membranes showed much higher ability to separate H₂ from N₂ and CO₂ than conventional SAPO-34 membranes
14. Wang, H.-E.; Zheng, L.-X.; Liu, C.-P.; Liu, Y.-K.; Luan, C.-Y.; Cheng, H.; Li, Y. Y.; Martinu, L.; Zapien, J. A.; Bello, I. Rapid Microwave Synthesis of Porous TiO₂ Spheres and Their Applications in Dye-Sensitized Solar Cells. *J. Phys. Chem. C*, **2011**, *115*, 10419 – 10425. DOI: [10.1021/jp2011588](https://doi.org/10.1021/jp2011588)
- Professors Juan Antonio Zapien and Igor Bello, Department of Physics and Materials Science and Center of Super-Diamond and Advanced Films (COSDAF), City University of Hong Kong,
 - Porous TiO₂ nanospheres constructed in only 10 minutes by microwave synthesis in a CEM Discover
 - Products displayed good morphology and better electrochemical efficiency than commercially available nanopowders
15. Vernon, J. P.; Fang, Y.; Cai, Y.; Sandhage, K. H. Morphology-Preserving Conversion of a 3D Bioorganic Template into a Nanocrystalline Multicomponent Oxide Compound. *Angew. Chem. Int. Ed.* **2010**, *49*, 7765 – 7768. DOI: [10.1002/anie.201003170](https://doi.org/10.1002/anie.201003170)
- Professor Kenneth H. Sandhage, School of Materials Science and Engineering, Georgia Institute of Technology
 - A layer-by-layer surface sol-gel process was used to convert the structure of Morpho helenor butterfly wings to BiTiO₃ replicas
 - CEM MARS used for the final step, microwave hydrothermal conversion of anatase TiO₂ to BaTiO₃
 - The general process can be applied to microscale nanostructured bioorganic or synthetic organic templates
16. Mehta, R. J.; Karthik, C.; Jiang, W.; Singh, B.; Shi, Y.; Siegel, R. W.; Borca-Tasciuc, T.; Ramanath, G. High Electrical Conductivity Antimony Selenide Nanocrystals and Assemblies. *Nano Lett.* **2010**, *10*, 4417 – 4422. DOI: [10.1021/nl1020848](https://doi.org/10.1021/nl1020848)
- Professor Ganpati Ramanath, Rensselaer Polytechnic Institute, Department of Materials Science and Engineering.
 - One-dimensional nanocrystals of sulfurized antimony selenide were synthesized in a rapid and scalable manner
 - 10⁴-10¹⁰ times higher electrical conductivity than non-nanostructured bulk or thin film forms
 - Control of microwave heating times showed changes in wire morphology
17. Wang, H. Q.; Nann, T. Monodisperse Upconverting Nanocrystals by MW Assisted Synthesis. *ACS Nano* **2009**, *3*, 3804 – 3808. DOI: [10.1021/nn9012093](https://doi.org/10.1021/nn9012093)
- Professor Thomas Nann, School of Chemistry, University of East Anglia, Norwich
 - Nanocrystals consisting of different ratios of Na, Er, Y, and Li were synthesized
 - A microwave assisted synthesis approach allows for the synthesis of such monodisperse and luminescent upconverting nanocrystals within 5 min in a closed reaction vessel (even though the same reactants and solvents as with classical conductive heating reactions were used)
 - Microwave-assisted synthesis resulted in differently sized and shaped particles and provided superior reaction control. The nucleation and growth mechanism follows a La Mer scheme and can be controlled extremely accurately
- Addition/Correction DOI: [10.1021/nn100270n](https://doi.org/10.1021/nn100270n)
18. Baruwati, B.; Polshettiwar, V.; Varma, R. S. Glutathione Promoted Expeditious Green Synthesis of Silver Nanoparticles in Water using Microwaves. *Green Chem.* **2009**, *11*, 926 – 930. DOI: [10.1039/b902184a](https://doi.org/10.1039/b902184a)
- Varma at the EPA developed rapid, green synthesis of Ag, Pt, Pd, and Cu nanoparticles in Glutathione (benign antioxidant)
 - Reaction time of 30 – 60 sec at 50 W for Ag, and 45 – 60 sec at 75 W for Pt, Pd, and Cu
 - Completed at very low temperatures: 40 – 60 °C. Conventional temperatures > 150 °C
 - Monodispersed particles of 5 – 10 nm

19. Bolink, H. J.; Cappelli, L.; Coronado, E.; Grätzel, M.; Nazeeruddin, M. K. Efficient and Stable Solid-State Light-Emitting Electrochemical Cell Using Tris(4,7-diphenyl-1,10-phenanthroline)ruthenium(II) Hexafluorophosphate. *J. Am. Chem. Soc.* **2006**, *128*, 46 – 47. DOI: [10.1021/ja0565065](https://doi.org/10.1021/ja0565065)
- Professor Henk Bolink, Institute of Molecular Science at the University of Valencia
 - The title compound was prepared in a CEM Discover in only 5 minutes with a yield of 89% as compared to conventional reactions which can take days and generate <5% desired product
 - Ru(dpp)₃²⁺ incorporated into electrochemical cells demonstrating a high power efficiency of 1.9 Lum/W and luminous brightness of 390 cd/m²
20. Bilecka, I.; Elser, P.; Niederberger, M. Kinetic and Thermodynamic Aspects in the Microwave-Assisted Synthesis of ZnO Nanoparticles in Benzyl Alcohol. *ACS Nano* **2009**, *3*, 467 – 477. DOI: [10.1021/nn800842b](https://doi.org/10.1021/nn800842b)
- Professor Markus Niederberger- Laboratory for Multifunctional Materials, Department of Materials. ETH Zurich
 - Comparison of the microwave mediated route with conventional heating showed that microwave irradiation greatly accelerates nanoparticle formation by:
 - facilitating the dissolution of the precursor in the solvent
 - increasing the rate constants for the esterification reaction by 1 order of magnitude, resulting in faster production of monomer and consequently in an earlier nucleation event
 - increasing the rate constants *k* growth for the crystal growth from 3.9 nm/min (conventional heating) to 15.4 nm/min (microwave heating)
21. Quan, Z.; Yang, P.; Li, C.; Yand, J.; Yang, D.; Jin, Y.; Lian, H.; Li, H.; Lin, J. Shape and Phase Controlled Synthesis of KMgF Colloidal Nanocrystals via Microwave Irradiation. *J. Phys. Chem. C* **2009**, *113*, 4018 – 4025. DOI: [10.1021/jp810714k](https://doi.org/10.1021/jp810714k)
- Professor Jun Lin at the State Key Laboratory of Rare Earth Resource Utilization, Changchun Institute of Applied Chemistry, Academy of Sciences in collaboration with Graduate University of the Chinese Academy of Sciences
 - One step route to colloidal KMgF₃ nanocrystals via thermolysis using MW irradiation.
 - Shape of nanocrystals readily controlled resulting in the well defined near-spherical nanoparticles and nano-plates of cubic-phased KMgF₃ and nanorods of tetragonal-phased MgF₂
 - Substrates heated in MW at 100 °C for 10 min under inert atmosphere, then 290 °C for 30 min.
 - Morphology and size were controlled by changing the heating temperature, time, and by altering the ratio of surfactants
22. Hu, X.; Gong, J.; Zhang, L.; Yu, J. C. Continuous Size Tuning of Monodisperse ZnO Colloidal Nanocrystal Clusters by a Microwave-Polyol Process and Their Application for Humidity Sensing. *Adv. Mater.* **2008**, *20*, 4848 – 4850. DOI: [10.1002/adma.200801433](https://doi.org/10.1002/adma.200801433)
- Dr. Xianluo Hu and Professor Jimmy Yu. Key Laboratory of Pesticide and Chemical Biology of Ministry of Education, College of Chemistry Central China Normal University with the Department of Chemistry, The Chinese University of Hong Kong
 - Reaction is run under open vessel conditions - the NP size is directly controlled by the amount of Zn Acetate stock solution added
 - Open vessel allows Zn solution to be added during the reaction-this was crucial for monodispersed nanoparticle formation
 - Small ZnO nuclei are generated by the rapid MW induced hydrolysis of Zn²⁺ and dehydration of resulting Zn complexes at 180 deg C. This is achieved by the polar, and thus strong microwave absorbing, characteristic of Zn and ZnO, creating a super hot surface speeding up nanocrystal growth
23. Nadagouda, M. N.; Varma, R. S. Microwave Assisted Shape Controlled Bulk Synthesis of Ag and Fe Nanorods in PEG Solutions. *Cryst. Growth Des.* **2008**, *8*, 291 – 295. DOI: [10.1021/cg070473i](https://doi.org/10.1021/cg070473i)
- Raj Varma at EPA synthesized Ag and Fe nanorods and nanoparticles using MW.
 - Also synthesized Ag supported Pt nanocubes
 - 100 °C for 1 h. Same conditions with conventional heating yielded no product at all
 - Morphology controlled by ratio of solvent (PEG) to metal precursor
24. Hu, B.; Wang, S.-B.; Wang, K.; Zhang, M.; Yu, S.-Y. Microwave-Assisted Rapid Facile “Green” Synthesis of Uniform Silver Nanoparticles: Self-Assembly into Multilayered Films and Their Optical Properties. *J. Phys. Chem. C* **2008**, *112*, 11169 – 11174. DOI: [10.1021/jp801267j](https://doi.org/10.1021/jp801267j)
- Professor Yu, Division of Nanomaterials and Chemistry and Hefei National Laboratory for Physical Sciences at Microscale, the School of Chemistry and Materials, University of Science and Technology of China
 - Synthesized uniform Ag nanoparticles using water, soluble starch (dextrin) as a protecting agent, and basic amino acids L-lysine and L-arginine as mild, renewable, and nontoxic reducing agents.
 - The reaction was heated using microwave irradiation to 150 deg C and held for 10 s. The authors noted that higher power settings lead smaller nanoparticles. This could be due to faster ramp times, therefore decreasing the total crystal growth time, or that the higher energy could promote the generation of more nuclei
 - Using MW irradiation shortened the reaction time by 2-3 orders of magnitude when compared to conventional hydrothermal methods.
 - Recovered nanoparticles were more uniform in dimension than their conventional partners

25. Suib, S. L. et al. Systematic Control of Particle Size in Rapid Open Vessel Microwave Synthesis of K-OMS-2 Nanofibers. *J. Phys. Chem. C* **2008**, 112, 6786 – 6793. DOI: [10.1021/jp800672m](https://doi.org/10.1021/jp800672m)

- Professor Steven Suib, Department of Chemistry and Department of Chemical, Materials and Biomolecular Engineering. University of Connecticut
- Typical reaction conditions using an oil bath require anywhere from hours to days, multiple procedures under hydrothermal and refluxing conditions, and generally have no direct control of nanoparticle size and surface area-which play a crucial role in determining the catalytic properties of the material
- Developed a systematic approach to control particle size by using MW irradiation and varying the amount of co-solvent (DMSO) from 0–50% v/v in water
- Microwave irradiation formed nanofibers, ranging from 4 – 12.2 nm. These results could not be duplicated using an oil bath. No ordered nanoparticles were recovered after 90 min of reflux using conventional heating methods and poorly ordered manganese oxide with particle diameter of around 100 nm formed at room temperature. Well-formed needle-like fibers were only formed with the addition of microwave irradiation. The needle diameter was varied by changing the ratio of solvent to co-solvent

26. Zhu, j-F.; Zhu, Y-J.; Ma, M-G.; Yan, L-X.; G, L. Simultaneous and Rapid Microwave Synthesis of Polyacrylamide – Metal Sulfide (Ag₂S, Cu₂S, HgS) Nanocomposites. *J. Phys. Chem. C* **2007**, 111, 3920 –3926 DOI: [10.1021/jp0677851](https://doi.org/10.1021/jp0677851)

- Professor Ying-Ji Zhu at the State Key Laboratory of high Performance Ceramics and superfine Microstructures and Shanghai Institute of Ceramics, Chinese Academy of Sciences.
- Fast, microwave assisted synthesis of polyacrylamide metal sulfides using metal salt, sulfur powered, and acrylamide monomer
- Heated in ethylene glycol, which acted as solvent and reducing agent. Created low-cost preparation of polymeric metal sulfide nanoparticles without additional need for initiator or surfactant
- Reaction run in open vessel format, samples heated to 125 °C or 190 °C for 15 – 60 min.
- Took over 2 h to complete in oil bath
- Overall, variable heating times, temperatures resulted in monodispersed, size control synthesis of metal sulfide nanoparticles

Magnetic Metal Materials

27. Yi, D. K.; Lee, S. S.; Ying, J. Y. Synthesis and Applications of Magnetic Nanocomposite Catalysts. *Chem. Mater.* **2006**, 18, 2459 – 2461. DOI: [10.1021/cm052885p](https://doi.org/10.1021/cm052885p)

- Dr. Jackie Y. Ying, Institute of Bioengineering and Nanotechnology, Singapore
- Synthesis of Pd coated SiO₂/Fe₂O₃ nanoparticles
- Microwave irradiation allowed synthesis of more uniform and finer palladium particles than conventional methods
- Particles showed dramatically increased catalytic ability over commercial Pd/C

28. Liang, Y.-C.; Hwang, K. C.; Lo, S.-C. Solid-State Microwave-Arcing-Induced Formation and Surface Functionalization of Core/Shell Metal/Carbon Nanoparticles. *Small*, **2008**, 4, 405 – 409. DOI: [10.1002/sml.200700808](https://doi.org/10.1002/sml.200700808)

- Professor Kuo Chu Hwang, Department of Chemistry, National Tsing Hua University
- Silicon pieces and ferrocene powder were arced to form Fe/C nanoparticles which could then be surface functionalized
- Reactions were performed in the solid-state, and without the formation of carbon nanotubes
- Similar nanoparticles could be prepared using Ni or Co metallocenes

29. Amstad, E.; Gillich, T.; Bilecka, I.; Textor, M.; Reimhult E. Ultrastable Iron Oxide Nanoparticle Colloidal Suspensions using Dispersants with Catechol-Derived Anchor Groups. *Nano Lett.* **2009**, 9, 4042 – 4048. DOI: [10.1021/nl902212q](https://doi.org/10.1021/nl902212q)

- Professor Erick Reimhult at the Laboratory of Surface Science and Technology and Laboratory for Multifunctional Materials, ETH Zurich
- Fe(ac)₂ in BnOH were heated for 3 min at 180 °C
- Monodispersed 6 nm Fe₃O₄
- Dispersants later added to stabilize iron oxide in water

30. Taylor, K. M.; Rieter, W. J.; Lin, W. Manganese – Based Nanoscale Metal – Organic Frameworks for Magnetic Resonance Imaging. *J. Am. Chem. Soc.* **2008**, 130, 14358 – 14359. DOI: [10.1021/ja803777x](https://doi.org/10.1021/ja803777x)

- Professor Weblin Lin, Department of Chemistry, University of North Carolina at Chapel Hill
- There was a remarkable difference in the synthesis of Manganese Based Metal Organic Frameworks when microwave irradiation was utilized. Spiral nanorods (50 – 100 nm x 1000-2000 nm) were obtained under room temperature conditions while three dimensional nanocubes (50 – 300 nm) were synthesized via microwave irradiation

- MW conditions: 120 °C for 10 min, 800 W
- Nanocubes could not be duplicated under conventional conditions

31. Wang W. W.; Zhu, Y. J.; Ruan, M. L. Microwave – Assisted Synthesis and Magnetic Property of Magnetite and Hematite Nanoparticles. *J. Nanopart. Res.* **2007**, 9, 419 – 426. DOI: [10.1007/s11051-005-9051-8](https://doi.org/10.1007/s11051-005-9051-8)
- Professor Ying-Jie Zhu, State Key Laboratory of High Performance Ceramics and Superfine Microstructure and the Chinese Academy of Sciences and Analysis and Testing Center for Inorganic Materials Shanghai Institute of Ceramics
 - MW irradiation led to α -Fe₂O₃ ellipsoid nanocrystals (50 x 100 nm), while conventional heating, under the same conditions, lead to irregular nanorods and particles
 - Conditions: 100 °C, 10 min
32. Sreeja, V.; Joy, P.A. Microwave-Hydrothermal Synthesis of γ -Fe₂O₃ Nanoparticles and their Magnetic Properties. *Mater. Res. Bull.* **2007**, 42, 1570 – 1576. DOI: [10.1016/j.materresbull.2006.11.014](https://doi.org/10.1016/j.materresbull.2006.11.014)
- Professor P.A. Joy, Physical and Materials Chemistry Division, National Chemical Laboratory.
 - It is shown that microwave–hydrothermal method offered a convenient, fast and single step process for the synthesis of nanoparticles of γ -Fe₂O₃
 - Nanoparticles of γ -Fe₂O₃ could be synthesized at 150 °C in short time duration of 25 min by the microwave hydrothermal method
33. Nguyen, H. L.; Howard, I. E. M.; Giblin, S. R.; Tanner, B. K.; Terry, I.; Hughes, A. K.; Ross, i. M.; Serres, A.; Burckstummer, H.; Evans, J. S. O. Synthesis of Monodispersed fcc and fct FePt/FePd Nanoparticles by Microwave Irradiation. *J. Mater. Chem.* **2005**, 15, 5136 – 5143. DOI: [10.1039/b511850f](https://doi.org/10.1039/b511850f)
- Professor John Evans, Department of Chemistry, University Science Laboratories, University of Durham, in collaboration with Department of Physics and Department of Electronic and Electrical Engineering
 - Monodispersed superparamagnetic fcc FePt and fct FePd nanoparticles were synthesized in short reaction times, 6 – 90 min at high temperatures (150 – 282 °C).
 - For maximum power, several of the reactions were run in open vessel format under inert atmosphere at 282 °C
34. Katsuki, H.; Komarneni, S. Microwave-Hydrothermal Synthesis of Monodispersed Nanophase α -Fe₂O₃. *J. Am. Ceram. Soc.* **2001**, 84, 2313 – 2317. DOI: [10.1111/j.1151-2916.2001.tb01007.x](https://doi.org/10.1111/j.1151-2916.2001.tb01007.x)
- Dr. Hiroaki Katsuki and Professor Sridhar Komarneni at Saga Ceramics Research Laboratory and Materials Research Laboratory and Department of Agronomy, The Pennsylvania State University
 - “Red, spherical α -Fe₂O₃ particles, 50–100 nm in diameter, were formed with β -FeOOH crystals after 13 h via the C-H reaction. α -Fe₂O₃, 100–180 nm in diameter, was preferentially formed after 24 h of treatment... With the M-H reaction at 100°C, monodispersed α -Fe₂O₃ particles, 31 nm in diameter, were formed in 2 h without the formation of β -FeOOH.”

Organic Nanostructures

35. Silva, P.; Vieira, F.; Gomes, A. C.; Ananias, D.; Fernandes, J. A.; Bruno, S. M.; Soares, R.; Valente, A. A.; Rocha, J.; Paz, F. A. A. Thermal Transformation of a Layered Multi-Functional Network into a Metal-Organic Framework based on a Polymeric Organic Linker. *J. Am. Chem. Soc.* **2011**, Just accepted DOI: [10.1021/ja205243w](https://doi.org/10.1021/ja205243w)
- Professor Filipe A. Almeida Paz, Department of Chemistry, CICECO, University of Aveiro
 - Layered [La(H₃nmp)] was synthesized using both dynamic hydrothermal methods and microwave synthesis
 - Conventional reaction required 2 days at 165 °C while microwave reactions completed in only 1 minute at 140 °C
 - Further heating above 300 °C converted the layered solid to a three-dimensional metal organic framework (MOF)
 - [La(H₃nmp)] MOF was shown to have excellent selectivity and reactivity as a heterogeneous catalyst in epoxide ring opening reactions
36. Zhuang, G.-L.; Chen, W.-X.; Zhao, H.-X.; Kong, X.-J.; Long, L.-S.; Huang, R.-B.; Zheng, L.-S. Two Three-Dimensional 2p–3d–4f Heterometallic Frameworks Featuring a Ln₆Cu₂₄Na₁₂ Cluster as a Node. *Inorg. Chem.* **2011**, 50, 3843 – 3845. DOI: [10.1021/ic200077r](https://doi.org/10.1021/ic200077r)
- Professors Xiang-Jian Kong and La-Sheng Long, State Key Laboratory of Physical Chemistry of Solid Surface and Department of Chemistry, College of Chemistry and Chemical Engineering, Xiamen University
 - Microwave heating used to synthesize two heterometallic metal organic frameworks (MOFs) containing either Gd or Dy
 - Nodes of MOF feature rare copper-sodium-lanthanide nanocluster
 - Testing of magnetic and electrical properties performed demonstrate ferrimagnetism and proton conductivity respectively

37. Rifai, S.; Breen, C. A.; Solis, D. J.; Swager, T. M. Facile in Situ Silver Nanoparticle Formation in Insulating Porous Polymer Matrices. *Chem. Mater.* **2006**, *18*, 21 – 25. DOI: [10.1021/cm0511419](https://doi.org/10.1021/cm0511419)
- Professor Timothy M. Swager, Department of Chemistry, Massachusetts Institute of Technology
 - Silver/poly(aryl ether) (PAE) nanoparticles were synthesized using varied microwave pulses
 - Resultant structures showed good uniformity and could be synthesized in as quickly as 5 minutes
 - Ag/PAE particles were then incorporated into an insulating polymer matrix resulting in a “relatively uniform polymer-ion thin film”
38. Wang, X.; Xia, T.; Ntim, S. A.; Ji, Z.; Meng, H.; Zhang, H.; Castranova, V.; Mitra, S.; Nel, A. E. Quantitative Techniques for Assessing and Controlling the Dispersion and Biological Effects of Multiwalled Carbon Nanotubes in Mammalian Tissue Culture Cells. *ACS Nano* **2010**, *4*, 7241 – 7252. DOI: [10.1021/nn102112b](https://doi.org/10.1021/nn102112b)
- Professor Andre E. Nel, Division of NanoMedicine, Department of Medicine, and California NanoSystems Institute, UCLA
 - CEM MARS used for purification and functionalization of multiwalled carbon nanotubes (MWCNT)
 - Modified nanotubes used to assay dispersion of MWCNT in tissue culture media
 - It was determined that hydrophobicity is the major factor determining agglomeration
39. Dössel, L.; Gherghel, L.; Feng, X.; Müllen, K. Graphene Nanoribbons by Chemists: Nanometer-Sized, Soluble, and Defect-Free. *Angew. Chem. Int. Ed.* **2011**, *50*, 2540 – 2543. DOI: [10.1002/anie.201006593](https://doi.org/10.1002/anie.201006593)
- Professor Klaus Müllen, Max-Planck-Institut für Polymerforschung
 - Researchers detail a bottom-up organic synthesis of defect-free graphene nanoribbons
 - Microwave irradiation was used to construct polymer precursors which were efficiently converted to graphene nanoribbons by an intramolecular Scholl reaction
40. Centrone, A.; Yang, Y.; Speakman, S.; Bromberg, L.; Rutledge, G. C.; Hatton, T. A. Growth of Metal – Organic Frameworks on Polymer Surfaces. *J. Am. Chem. Soc.* **2010**, *132*, 15687 – 15691 DOI: [10.1021/ja106381x](https://doi.org/10.1021/ja106381x)
- Scientist at MIT synthesized metal organic frameworks (MOFs) directly on polyacrylonitrile using microwave irradiation
 - Growth of MOF was studied by varying microwave irradiation time
 - Reagents were heated in DI water to 200 °C for 5 s to 30 min.
 - MOF agglomerates formed on the polymer after just 5 s of MW irradiation. The MOFs were only found on the polymer and not in the bulk solution
 - Provides rapid, reproducible method to coat polymer fibers with MOFs
41. Lhoste, J.; Rocquefelte, X.; Adil, K.; Dessapt, R.; Jobic, S.; Leblanc, M.; Maisonneuve, V.; Bujoli-Doeuff, M. A New Organic–Inorganic Hybrid Oxyfluorotitanate [Hgua]₂(Ti₅O₅F₁₂) as a Transparent UV Filter. *Inorg. Chem.* **2011**, *50*, 5671 – 5678. DOI: [10.1021/ic200407h](https://doi.org/10.1021/ic200407h)
- Prof. V. Maisonneuve and Dr. M. Bujoli-Doeuff, Faculté des Sciences et Techniques, Université du Maine and IMN, France
 - A unique UV absorber with the structure [Hgua]₂(Ti₅O₅F₁₂) was synthesized using microwave hydrothermal synthesis
 - Heating materials in a CEM MARS with Teflon vessels to 190 °C for 1 hour generated the desired product efficiently
 - Investigations of the atomic, optical, and electrical properties show that this hybrid may have unique applications for UV shielding
42. Gizdavic-Nikolaidis, M. R.; Stanisavljev, D. R.; Easteal, A. J.; Zujovic, Z. D. Microwave-Assisted Synthesis of Functionalized Polyaniline Nanostructures with Advanced Antioxidant Properties. *J. Phys. Chem. C* **2010**, *114*, 18790 – 18796. DOI: [10.1021/jp106213m](https://doi.org/10.1021/jp106213m)
- Utilized microwave irradiation to synthesize copolymers of aniline and 2-aminobenzoic acid or 20aminosulfonic acid
 - When compared to conventionally synthesized copolymers, MW yields were 2.5 – 3.0 times higher
 - MW samples were also 2.1 – 2.4 times better radical scavenger efficacy
 - MW times were between 5 and 10 min
43. Centrone, A.; Harada, T.; Speakman, S.; Hatton, T. A. Facile Synthesis of Vanadium Metal – Organic Frameworks and their Magnetic Properties. *Small* **2010**, *15*, 1598 – 1602 DOI: [10.1002/smll.201000773](https://doi.org/10.1002/smll.201000773)
- Scientist at MIT presented a microwave synthesis of MIL-47 and 6 new vanadium MOF's
 - Precursors were heated to 200 °C for only 10 min
 - Represents a very fast and reproducible method for MOF's

44. Economopoulos, S. P.; Pagona, G.; Yudasaka, M.; Iijima, S.; Tagmatarchis, N. Solvent-free Microwave-Assisted Bingel Reaction in Carbon Nanohorns. *J. Mater. Chem.* **2009**, *19*, 7326 – 7331. DOI: [10.1039/b910947a](https://doi.org/10.1039/b910947a)
- Dr. Nikos Tagmatarchis at the Theoretical and Physical Chemistry Institute- National Hellenic Research Foundation (Athens, Greece) and in collaboration with the Nanotube Research Center, National Institute of Advanced Industrial Science and Technology(Higashi, Japan) and NEC Corporation(Ibaraki, Japan)
 - Functionalized carbon nano-horns with fluorescent linkers through Bingel reaction without solvent
 - MW irradiation provided a high degree of functionalization
 - Pulse method: 5 – 45 second of MW irradiation was used. Total reaction time of 5 min with a max temp of 120 – 140 °C
 - Conventional synthesis: 60 °C for 20 h
45. Fujita, D.; Takahashi, A.; Sato, S.; Fujita, M. Self-Assembly of Pt(II) Spherical Complexes via Temporary Labilization of the Metal-Ligand Association in 2,2,2-Trifluoroethanol. *J. Am. Chem. Soc.* **2011**, ASAP DOI: [10.1021/ja2059236](https://doi.org/10.1021/ja2059236)
- Professor Makoto Fujita, Department of Applied Chemistry, School of Engineering, The University of Tokyo, and CREST, Japan Science and Technology Agency
 - Researchers used solvent conditions and metal ion selection to control geometry and self-assembly of spherical complexes
 - Previous syntheses of complexes required 4h or more of reaction time under conventional conditions; microwave reactions complete in 30 minutes or less
46. Wang, Y.; Su, F.; Lee, J. Y.; Zhao, X. S. Crystalline Carbon Hollow Spheres, Crystalline Carbon-SnO₂ Hollow Spheres, and Crystalline SnO₂ Hollow Spheres: Synthesis and Performance in Reversible Li-Ion Storage. *Chem. Mater.* **2006**, *18*, 1347 – 1353. DOI: [10.1021/cm052219o](https://doi.org/10.1021/cm052219o)
- Professor Jim Yang. Lee, Department of Chemical and Biomolecular Engineering, National University of Singapore
 - SnO₂ nanoparticles prepared with microwave heating showed uniform dispersion and no agglomeration on carbon hollow sphere surface as compared to conventional heating methods
 - Microwave synthesis completed in only 3 minutes; conventional heating required 3 hours
 - Electrochemical testing of different products demonstrated potential applications of tin composite nanostructures in Li-ion storage
47. Campbell, N. L.; Clowes, R.; Ritchie, L. K.; Cooper, A. I. Rapid Microwave Synthesis and Purification of Porous Covalent Organic Frameworks. *Chem. Mater.* **2009**, *21*, 204 – 206. DOI: [10.1021/cm802981m](https://doi.org/10.1021/cm802981m)
- Professor Andrew Cooper at University of Liverpool – Dept of Chemistry and materials discovery
 - MW irradiation offered a convenient and rapid synthesis of covalent organic frameworks.
 - 200 times faster than conventional methods
 - Reactions were run in closed vessel and open vessel format at 100 °C, 200W
 - Reaction times as low as 20 min
 - COFs had surface area equal to or greater than conventional made COF's and synthesized 200 times faster
48. Economopoulos, S. P.; Rotas, G.; Miyata, Y.; Shinohara, H.; Tagmatarchis, N. Exfoliation and Chemical Modification Using Microwave Irradiation Affording Highly Functionalized Graphene. *ACS Nano* **2010**, *4*, 7499 – 7507. DOI: [10.1021/nn101735e](https://doi.org/10.1021/nn101735e)
- Dr. Nikos Tagmatarchis, Theoretical and Physical Chemistry Institute, National Hellenic Research Foundation
 - Used MW to multi-functionalize graphene
 - Series of electrochemical, spectroscopic, gravimetric, and thermal experiments performed to fully characterize new material
49. Brunetti, F. G.; Herrero, M. A.; Munoz, J. M.; Diaz-Ortiz, A.; Alfonsi, J.; Meneghetti, M.; Prot, M.; Vazquez, E. Microwave-Induced Multiple Functionalization of Carbon Nanotubes. *J. Am. Chem. Soc.* **2008**, *130*, 8094 – 8100. DOI: [10.1021/ja801971k](https://doi.org/10.1021/ja801971k)
- Professor Maurizio Prato and Professor Ester Vazquez. Universita degli Studi di Trieste and Universidad de Castilla-La Mancha
 - Used MW to multi-functionalize CNTs
 - Conventional conditions generally require high temperatures and/or pressures, long reaction times, and organic solvents or mineral acids
 - MW reduced amount of organic waste, and reduced the reaction time from 5 days to 2.5 hours.
 - Typical temperatures for [3+2] cycloaddition step was 160 °C for 90 min (100 W), and no solvent, the reaction was done under neat conditions. Arene addition step: 80 °C, 90 min (100 W) and in water
50. Brunetti, F. G.; Herrero, M. A.; Munoz, J. M.; Giordani, S.; Diaz-Ortiz, A.; Filppone, S.; Ruaro, G.; Meneghetti, M.; Prot, M.; Vazquez, E. Reversible Microwave Assisted Cycloaddition of Aziridines to Carbon Nanotubes. *J. Am. Chem. Soc.* **2007**, *129*, 14580 – 14581. DOI: [10.1021/ja077927k](https://doi.org/10.1021/ja077927k)
- See previous reference