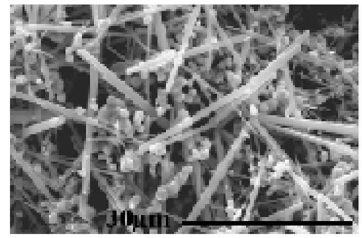
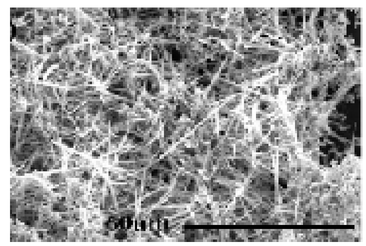
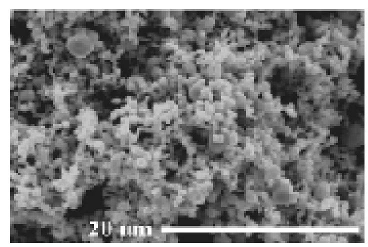
Microwave Assisted Shape-Controlled Bulk Synthesis of Ag and Fe Nanoparticles in Poly(ethylene glycol) Solutions

Ag and Fe nanoparticles, which have gained attention as catalysts, electronic nanodevices, and magnetic contrasting agents, were prepared using microwave (MW) irradiation via the "polyol process" – a more ecofriendly approach in material synthesis, by Nadagouda and Varma at the Environmental Protection Agency.[1] In many cases, microwave irradiation allows for a rapid and efficient uniform heating which leads to the shape and size controlled synthesis of many types of nanomaterials. Their work is highlighted in Scheme 1. Varying amounts of $AgNO_3$ or $Fe(NO_3)_3$• XH_2O along with 300 Da poly(ethylene glycol) (PEG) were heated in a CEM Discover® dedicated microwave synthesizer, as shown in Figure 1, at 100 ^{o}C for 1 h. After only two minutes, Ag nanomaterial precipitated out of solution (further heating for 1 h was required for maximum yield, generally around 90%); no precipitation was observed in the solution heated in an oil bath at the same temperature for 1 h.

Figure 1. CEM Discover® Microwave

The factor that determined the formation of nanorods versus nanoparticles was the concentration of Ag. In samples where the concentration of Ag was small, nanoparticle formation was favored, whereas at high concentrations, the formation of nanoparticles and nanorods were favored. Eventually, solely nanorod formation was possible by tailoring the reaction mixture. SEM images of nanoparticles (bottom), nanorods (middle), and a combination of rods and particles (top) are shown in scheme 1. This same technique was also applied to synthesis iron nanorods. In conclusion, microwave heating provided uniform, inside-out heating through the strongly microwave absorbing solvent to give nanomaterials with a high degree of crystallinity that was not obtained through conventional heating.

Scheme 1. Selected Examples[2]:

Reactant	Conditions	$PEG:AgNO_3$	Product	
$AgNO_3$	300 Da PEG → MW, 100 °C, 1h	1:7 2:6 3:5	Nanorods and nanoparticles	
$AgNO_3$	300 Da PEG → MW, 100 °C, 1h	4:4 5:3	Nanorods	
$AgNO_3$	300 Da PEG → MW, 100 °C, 1h	6:2 7:1 7.5:0.5	Nanoparticles	

References

1) Nadagouda, M. N.; Varma, R. S. *Gryst. Growth Des*. **2008**, *8*, 291 – 295.
2) Reprinted with permission from Nadagouda, M. N.; Varma, R. S. *Gryst. Growth Des*. **2008**, *8*, 291 – 295. Copyright 2008 American Chemical Society. **DOI**: 10.1021/cg070473i