Supplementary Information

Impact of Microwave Irradiation on Cyclo-octeno-1, 2, 3-selenadizole: Formation of Selenium Nanoparticles and its Polymorphs

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Reproducibility testing of Se nano experiments.

The experiments were repeated several times on domestic microwave oven and results were similar as presented in the paper. The experiments were also conducted on laboratory standard MW oven (Description- CEM model no. 908010, (USA)) for authenticity at similar MW power, at 100 ^{0}C and it is observed that reaction time get reduced as compared to domestic MW oven. The results obtained by using laboratory standard MW oven were compared with conventional MW oven which are presented as supplementary information. During reproducibility test it is observed that there is only a slight variation in UV-Visible spectra of black Se. Which could be considered as a natural outcome as this is the most stable form of selenium and due to clustering its dispersion will get adversely affected thereby affecting the absorption wavelength. Clustering of nanoparticles may vary experiment to experiment even in normal thermal chemical reactions related to semiconductor or metal nanoparticles. Following experiments were conducted to test reproducibility of data;

Synthesis of red Se nanoparticles by using laboratory standard MW oven:

Cyclo-octeno-1,2,3-Selenadiazole taken (6.9 mmol) in a beaker added oleic acid (4 ml) and diphenyl ether 6 ml), This reaction mixture was transferred in to MW glass tube (15 ml) subjected to microwave irradiation on 180 watt and at 100^{0}C temperature under closed condition. Reaction continued for 6 minutes. For work up hexane and methanol added for precipitation of nanoparticles. Se nanoparticles were separated by centrifugation with repeated washing with methanol and dried in hot air oven at about 50 ^{0}C. Red powder is obtained, which was used for characterization.

Synthesis of black Se nanoparticles by using laboratory standard MW oven: Black selenium was obtained by increasing reaction time by 10 min. using same procedure as above. The work up was carried out as per the protocol described above.

Table 1 Analytical data of Se nanoparticles synthesized using domestic and laboratory standard MW oven.

Compounds	Reaction Time (min.)	$\lambda_{(abs)}$ (nm)	$\lambda_{(em)}$ (nm)	Raman (cm^{-1})	XRD 2θ value for (100) plane	% Yield
[1*]Red Se NPs (R I)	12-15	400	430 ($\lambda_{(ex)}$ @ 350 nm)	234	23.93	45
[2*]Red Se NPs (R II)	6	396	430 ($\lambda_{(ex)}$ @ 350 nm)	234	23.63	51
[1*]Black Se NPs (B I)	20-25	490	520 ($\lambda_{(ex)}$ @ 400 nm)	236	23.47	60
[2*]Black Se NPs (B II)	15	440	530 ($\lambda_{(ex)}$ @ 400 nm)	236	23.53	62

[1*] Experiments conducted using domestic MW oven.
[2*] Experiments conducted using laboratory standard MW oven.

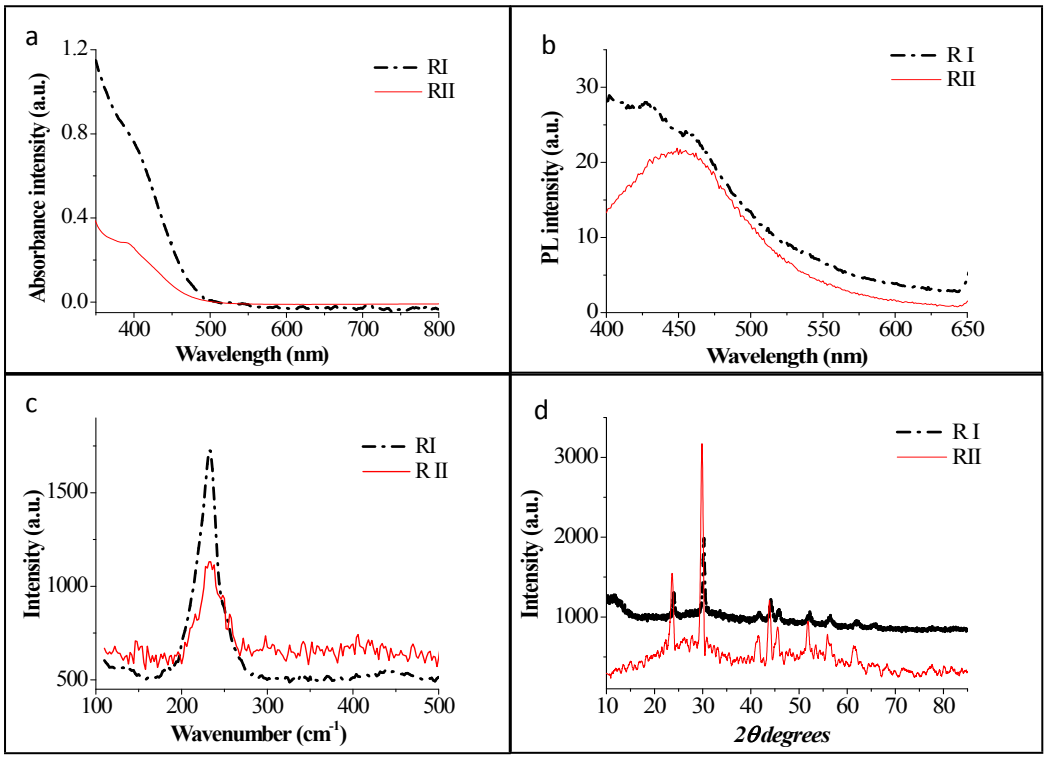

Fig. 1. UV–Visible spectra (a), PL spectra (b) , Raman (C) and XRD (d) Spectra of red Se nanoparticles synthesized using domestic and laboratory standard MW oven.

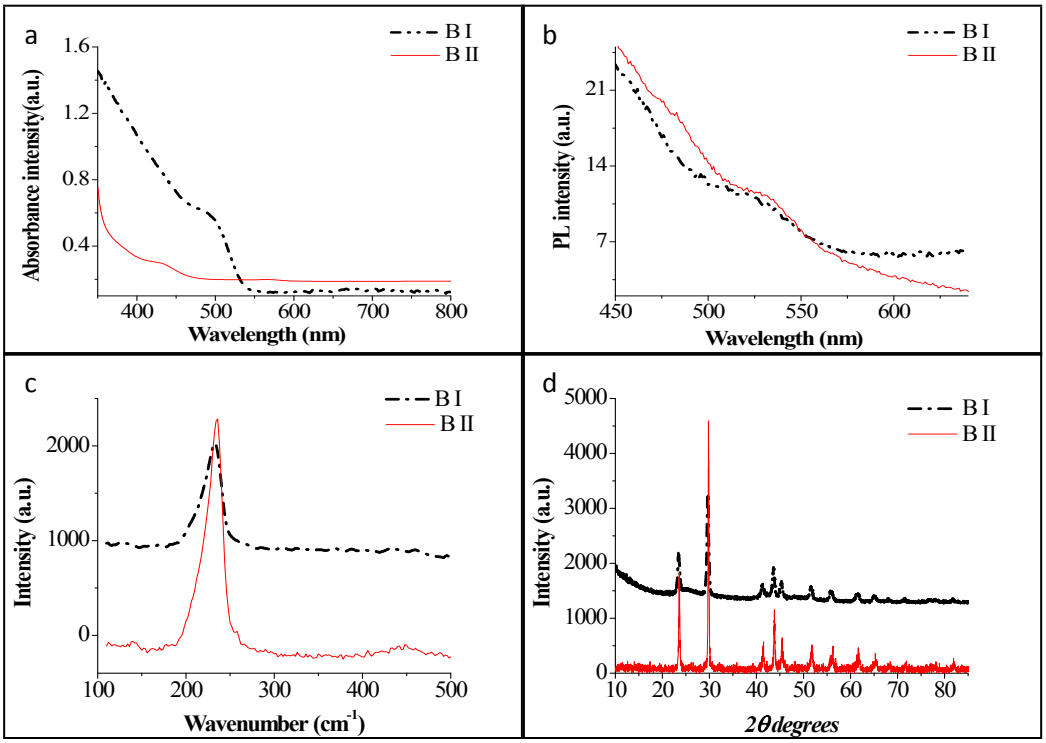

Fig. 2. UV–Visible spectra (a) ,PL (b) spectra, Raman (C) and XRD (d) Spectra of black Se nanoparticles synthesized using domestic and laboratory standard MW oven.