Supplementary Information

Surfactant-Driven Direct Synthesis of Hierarchical Hollow MgO Nanofiber-Nanoparticle Composite by Electrospinning

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Experimental

Chemicals: PVP (Polyvinyl pyrrolidone, M.W ~ 1,300,000, Sigma-Aldrich – USA), $Mg(ac)_2.4H_2O$ (>= 98% purity, M.W = 214.45 g/mol, Sigma Aldrich- Germany), DMF (N,N – Dimethylformamide, Merck, India) and anhydrous ethanol(Heyman,China)

Synthesis:

1. Effect on the fiber diameter and particle size on varying concentration of CTAB addition

The viscous solution for electrospinning was prepared by first adding 20wt% of PVP to 10ml of solvent (DMF) and stirred for 15 minutes to obtain a uniform clear solution. Later 16wt% of precursor ($Mg(ac)_2.4H_2O$) was added to the clear polymer solution while stirring. To see the effect of CTAB on the fiber diameter and particle size varying concentrations (0.1g, 0.25g, 0.5g and 0.75g) was added to the different polymer solutions prepared as above and stirred for 15hrs. After 15hrs of stirring the final solution obtained is turbid and for making it a clear solution, it is stirred at 35^0C for 5-10 minutes before keeping for electrospinning. The viscous precursor

solution is taken in a 20 ml syringe with 0.90 X 38mm needle attached to it. The voltage applied between the needle tip and the aluminium foil collector which was maintained at a distance of 17cm is 29kV (+25kV and -4kV). Humidity and temperature inside the electrospinning chamber was maintained at 20% and 25^0C. The solution was pumped into the needle tip inside the chamber at a flow rate of 1ml/hr. The as prepared $Mg(ac)_2.4H_2O$/PVP/CTAB composite fiber was then heat treated in air at 500^0C for 12hrs to obtain hollow MgO nanofibers with faceted MgO nanoparticles on the fiber surface.

2. ***Role of solvents in the surface modification of fibers***

To see the effect of solvent in the surface modification as well as faceting of surface modifying particles, two different viscous solutions for electrospinning was prepared. First solution was prepared by adding 20wt% of PVP to 10ml of solvent (DMF) and stirred for 15 minutes to obtain a uniform clear solution. Second solution was prepared by adding 20wt% of PVP in 10ml anhydrous ethanol and stirred for 15 minutes. Later 16wt% of precursor ($Mg(ac)_2.4H_2O$) was added to the clear polymer solutions and while stirring.0.75g CTAB was added to the above solution mixtures and stirred for 15hrs. After 15hrs of stirring the final solution obtained is turbid and for making it a clear solution, it is stirred at 35^0C for 5-10 minutes before keeping for electrospinning. The viscous precursor solution is taken in a 20 ml syringe with 0.90X38mm needle attached to it. The voltage applied between the needle tip and the aluminium foil collector which was maintained at a distance of 17cm is 29kV (+25kV and -4kV). Humidity and temperature inside the electrospinning chamber was maintained at 20% and 25^0C. The solution was pumped into the needle tip inside the chamber at a flow rate of 1ml/hr. The as prepared $Mg(ac)_2.4H_2O$/PVP/CTAB composite fiber was then heat treated in air at 500^0C for 12hrs to obtain hollow MgOnanofibers with faceted MgO nanoparticles on the fiber surface.

To see the effect of solvent in the surface modification and faceting of those particles the sintered fibers were characterized by TEM analysis. It showed that fibers prepared in DMF as well as an. ethanol both were surface modified with MgO. It is clear that the solvents have no role in faceting of the surface modifying particles or CTAB has same effect on both DMF and an. Ethanol solvents.

Characterization:

High resolution scanning Electron Microscopy was done using AURIGA- Cross Beam (FIB-SEM) and low magnification scanning Electron Microscopy was done using jeol (Jeol 6490 LA – 30 kV, , TEM - Transmission Electron Microscopy and EDX – Energy Dispersive X-ray Spectroscopy was performed using FEI-TECNAI F20 operating at200kV and viscosity measurements was done by DV-11 + Pro Viscometer (Brookfield).

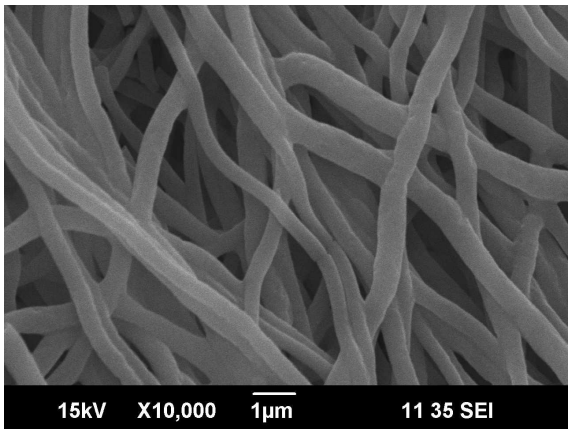

Figure S1. Showing the SEM images of MgOnanofiberswith out the addition of surfactant CTAB

Figure S2. FESEM images of 0.75g CTAB added sintered MgOnanofibers (a) & (b) showing fibers with non uniformly faceted nanoparticles projected on the fiber surfaces.

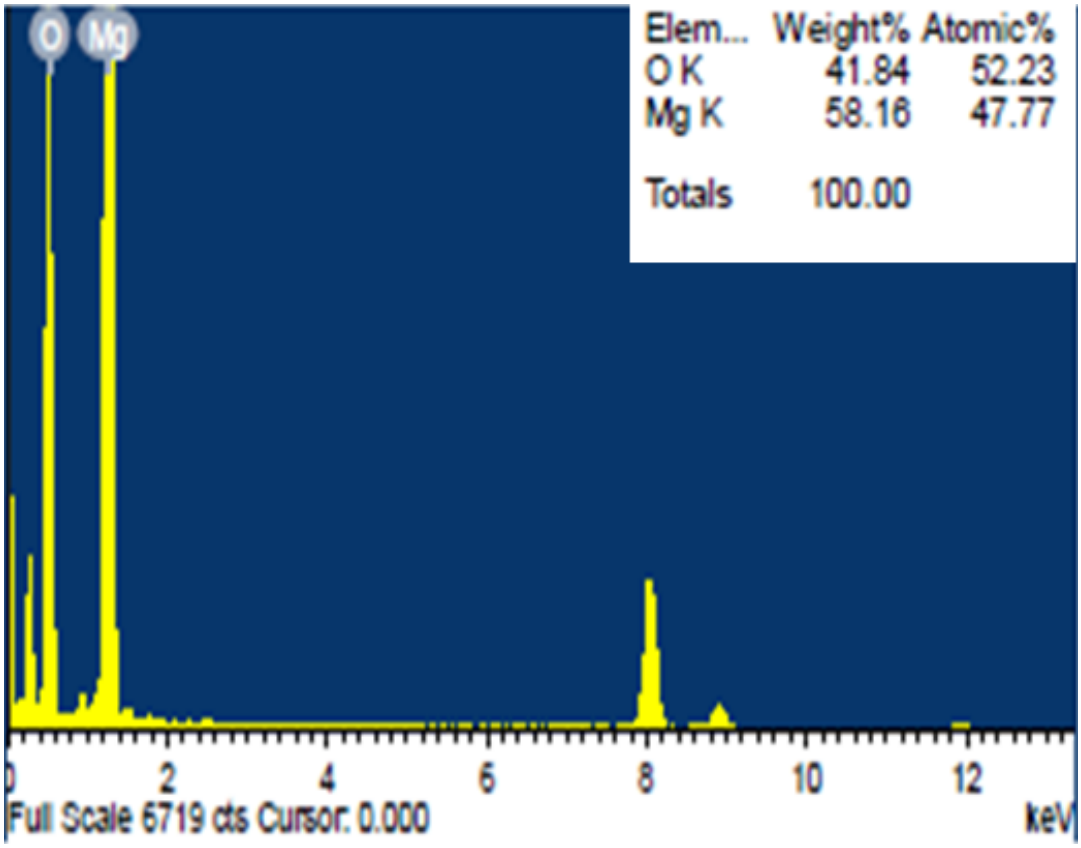

Figure 2. TEM-EDX of sintered MgOnanofiber-nanoparticle composite.

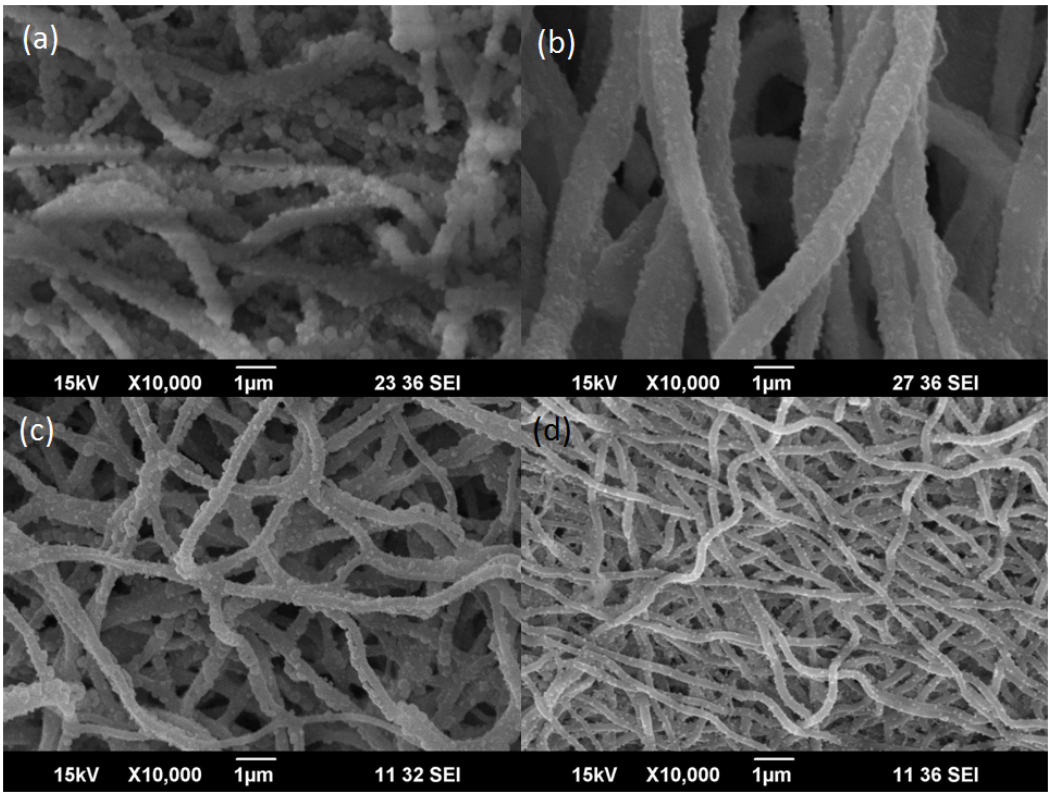

Figure S3.SEM images of sintered MgO fibers showing the gradualreduction in the diameter with increasing concentrations of CTAB addition. (a) 0.1g, (b) 0.25g, (c)0.5g and (d) 0.75g of CTAB.

S.No	Polymer : Precursor ratio	CTAB Conc. (g)	Viscosiy at 25^0C (cP)
1.	0.8	0.1	1470
2.	0.8	0.25	1830
3.	0.8	0.5	1320
4.	0.8	0.75	5400

Table 1. Viscosity values of the spinning solutions with different concentrations of CTAB addition.

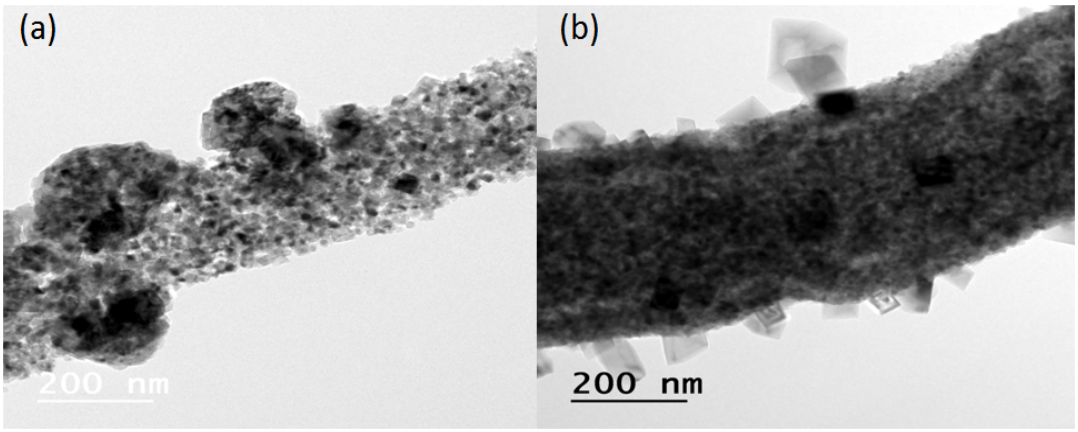

Figure S4. TEM images of (a) 0.1g and (b) 0.75g CTAB added sintered fibers showing clearly the size reduction and faceting of nanoparticles at higher concentrations

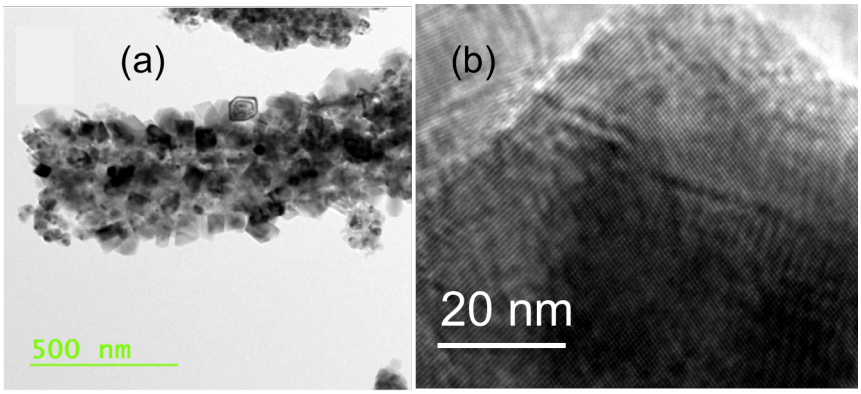

Figure S5. TEM images of sintered fibers (a) Low magnification TEM image of NF/NP composite prepared using ethanol as a solvent with 0.75 g CTAB. (b) High resolution TEM image of faceted MgO particle prepared with anhydrous ethanol solvent

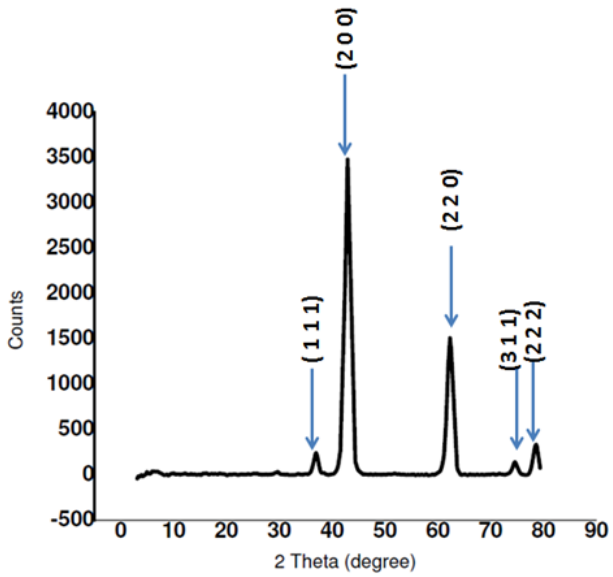

Figure S6. XRD profile of hollow MgOnanofiber/ faceted nanoparticle composite.