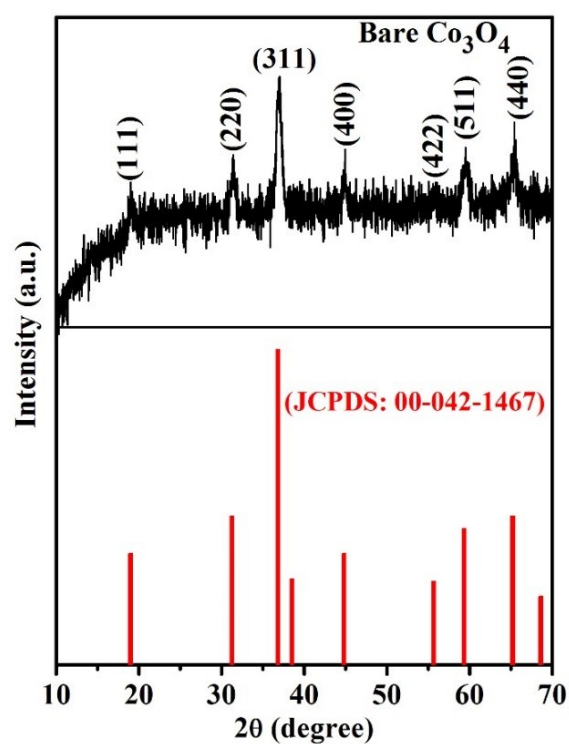


## Supporting Information

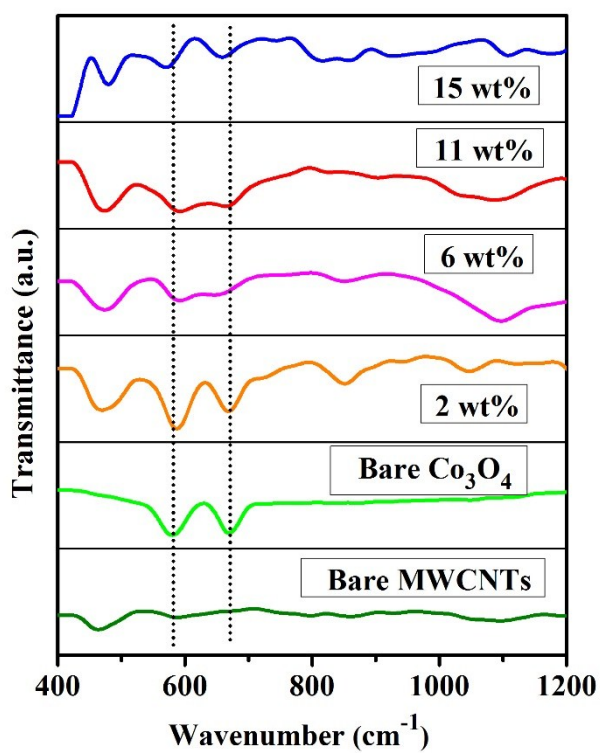
### **Anchoring of ultrafine Co<sub>3</sub>O<sub>4</sub> nanoparticles on MWCNTs using supercritical fluid processing and its performance evaluation towards electrocatalytic oxygen reduction reaction**

**Pitchai Thangasamy<sup>a</sup>, Karuppiah Selvakumar<sup>b</sup>, Marappan Sathish\*<sup>a</sup>, Sakkarapalayam Murugesan Senthil Kumar\*<sup>b</sup> and Rangasamy Thangamuthu<sup>b</sup>**

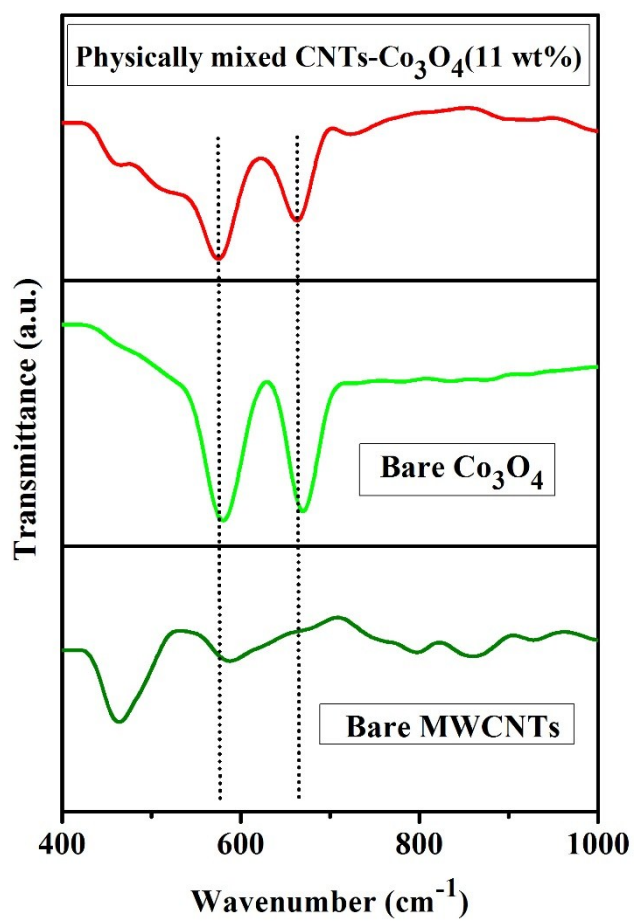
<sup>a</sup>Functional Materials Division, <sup>b</sup>Electrochemical Materials Science Division,  
CSIR-Central Electrochemical Research Institute, Karaikudi, Tamil Nadu, 630 003, India.  
E-mail address: [msathish@cecri.res.in](mailto:msathish@cecri.res.in); [senthilkumarsm@cecri.res.in](mailto:senthilkumarsm@cecri.res.in)



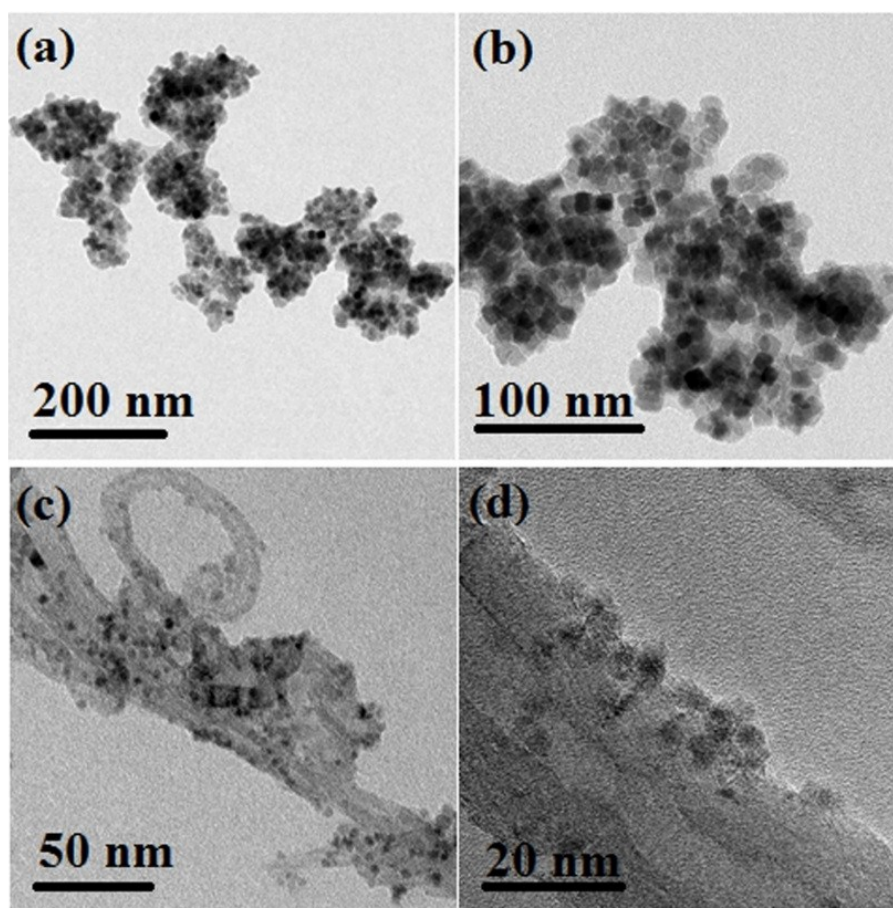
**Fig. S1** XRD pattern of bare  $\text{Co}_3\text{O}_4$  nanoparticles with standard reference pattern of cubic phase of  $\text{Co}_3\text{O}_4$  (JCPDS card No: 00-042-1467).



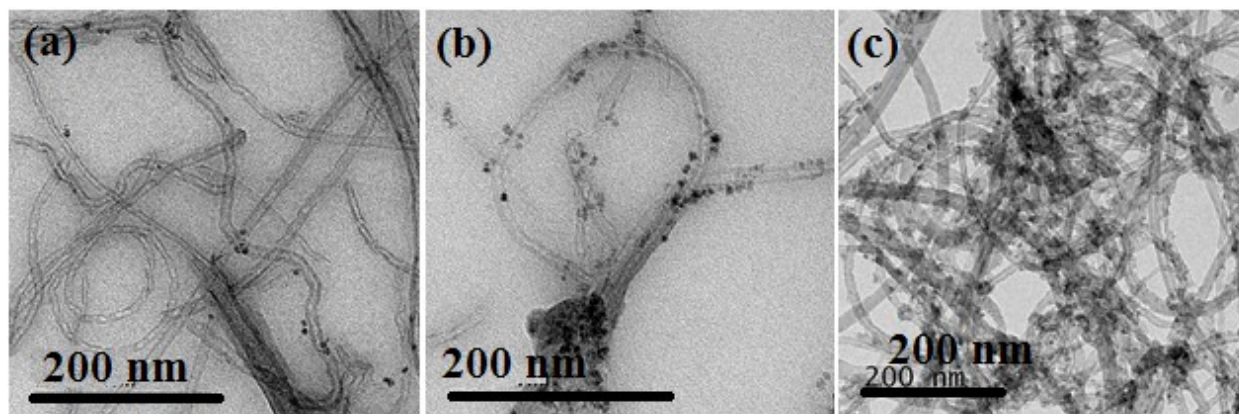
**Fig. S2** FT-IR spectra of bare MWCNTs,  $\text{Co}_3\text{O}_4$  and MWCNTs- $\text{Co}_3\text{O}_4$  with different weight loadings.



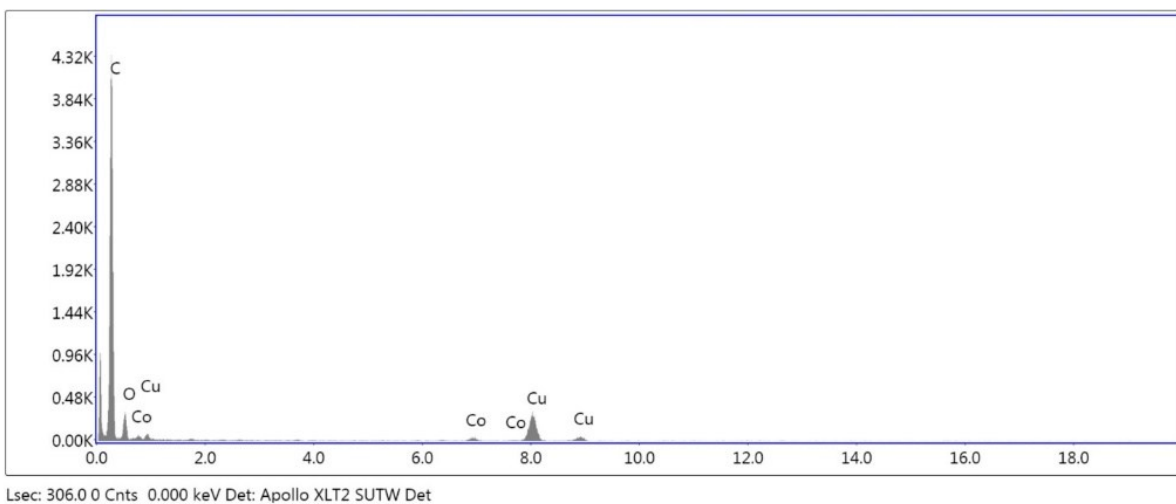
**Fig. S3** FT-IR spectra of bare MWCNTs, Co<sub>3</sub>O<sub>4</sub> and Physically mixed MWCNTs-Co<sub>3</sub>O<sub>4</sub> (11 wt%) composite.



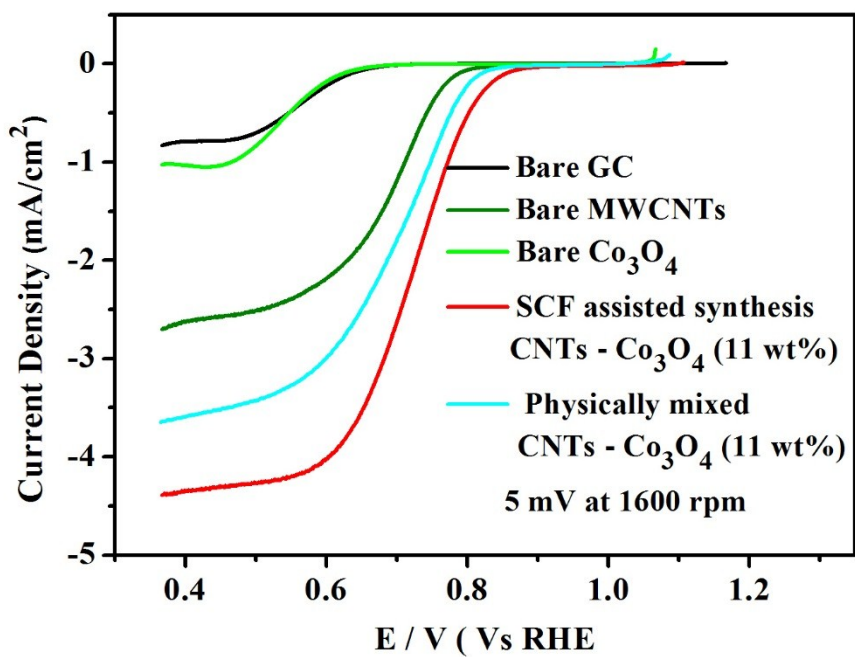
**Fig. S4** HR-TEM images of (a, b) bare  $\text{Co}_3\text{O}_4$  and (c, d) MWCNTs/ $\text{Co}_3\text{O}_4$  (11 wt%).



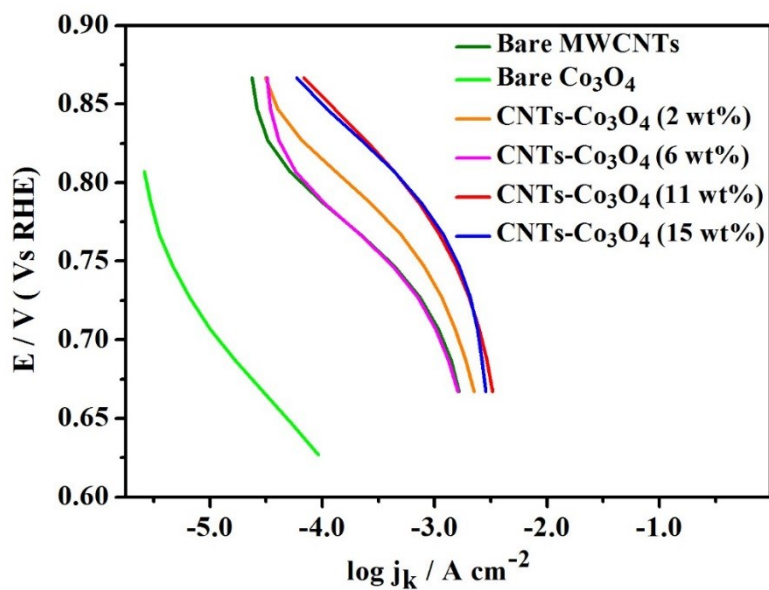
**Fig. S5** HR-TEM images of (a) MWCNTs/ $\text{Co}_3\text{O}_4$  (2 wt%), (b) MWCNTs/ $\text{Co}_3\text{O}_4$  (6 wt%) and (c) MWCNTs/ $\text{Co}_3\text{O}_4$  (15 wt%).



**Fig. S6** TEM-EDX analysis of MWCNTs/  $\text{Co}_3\text{O}_4$  (11 wt%).



**Fig. S7** Rotating ring disc electrode response recorded at  $5 \text{ mV s}^{-1}$  scan rate with a rotation rate 1600 rpm in  $\text{O}_2$  saturated 0.1 M KOH solution.



**Fig. S8** Tafel plots for various catalysts studied in this work as mentioned in the figure.

**Table ST1:** Electrode kinetic parameters, like, Tafel slope, n-value and % of peroxide produced for various CNTs-Co<sub>3</sub>O<sub>4</sub> catalysts studied in this work.

| Sample  | n-value in ORR (KL plot method) | n-value in ORR (RRDE method) | % of H <sub>2</sub> O <sub>2</sub> | Tafel slope / mV decade <sup>-1</sup> |
|---|---------------------------------|------------------------------|------------------------------------|---------------------------------------|
| MWCNTs-Co <sub>3</sub> O <sub>4</sub> (0wt%)  | 2.00                            | 2.66                         | 17                                 | 82                                    |
| MWCNTs-Co <sub>3</sub> O <sub>4</sub> (2wt%)  | 2.20                            | 2.25                         | 71                                 | 68                                    |
| MWCNTs-Co <sub>3</sub> O <sub>4</sub> (6wt%)  | 2.45                            | 2.99                         | 72                                 | 68                                    |
| MWCNTs-Co <sub>3</sub> O <sub>4</sub> (11wt%) | 3.80                            | 3.52                         | 31                                 | 77                                    |
| MWCNTs-Co <sub>3</sub> O <sub>4</sub> (15wt%) | 3.50                            | 3.57                         | 27                                 | 69                                    |