Electronic Supplementary Material (ESI) for RSC Advances.
This journal is © The Royal Society of Chemistry 2016

Supporting information

Selective growth of fullerene octahedron and flower-like particles by liquid-liquid interfacial precipitation method for super-hydrophobic applications

Thamodaran Partheeban and Marappan Sathish*
Functional Materials Division,
CSIR-Central Electrochemical Research Institute,
Karaikudi – 630003, INDIA.
E. mail: marappan.sathish@gmail.com; msathish@cecri.res.in

Experimental section

Fullerene micro-crystals were synthesized by mixing of fullerene saturated anisole solution with different volume of isopropyl alcohol (IPA) using LLIP method. In a typical precipitation, C_{60} saturated anisole solution was prepared by adding excess amount (150 mg) of C_{60} powder (99.5% pure, MTR Ltd) in 25 ml of anisole (AR grade, Merck). The mixture was ultrasonicated for 30 min followed by filtration to remove undissolved excess of C_{60} powder. Then, 1 ml of C_{60} saturated solution of anisole was taken in two 10 ml glass bottle and kept in ice water bath maintained 5 °C. Similarly, IPA was taken in glass beaker and stored in ice water bath 5 °C and calculated volume of IPA was added to C_{60} saturated solution of anisole gently and slowly through the glass bottle wall. For all the preparation, the 1 ml of C_{60} saturated solution was kept as constant and the volume of IPA is varied from 1 to 5 ml. The resulted solution was ultrasonicated for 30 sec and kept in an incubator at 5 °C. The formation of fullerene micro particles was observed within 1h at interfaces

Characterisation

The formation of fullerene microcrystals was initially confirmed using optical microscopy (Leica microsystems with magnification ranges from 50x to 1000x). The phase formation and crystalline nature of the C_{60} microcrystals were examined by powder X-ray diffraction (XRD) technique (X'Pert PRO, PANAlytical) using Cu Kα radiation (λ=1.5418 Å) in the 2θ ranges from

10 to 80° at 0.02° step with a count time of 0.2 s at each step. The morphology and the particle size of the C_{60} microcrystals were analyzed by Field-emission scanning electron microscope (FE-SEM, Carl Zeiss Supra 55VP) and high resolution transmission electron microscope (HR-TEM, Tecnai G2 TF20) working at an accelerating voltage of 5-30 KV and 200 KV, respectively. Water contact angle measurements was carried out using goniometer (OCA 35 Data Physics).

Water contact angle measurements

Super-hydrophobic properties of synthesized flower and octahedron like fullerene particles are measured in terms of water contact angle measurements. Thin film (20 µm) of fullerene microcrystals was fabricated on ITO substrate by dispersing 20 mg of fullerene microcrystals in 200 µl of hexane. Then, the solvent in the dispersion coated on ITO plate was slowly evaporated under room temperature.

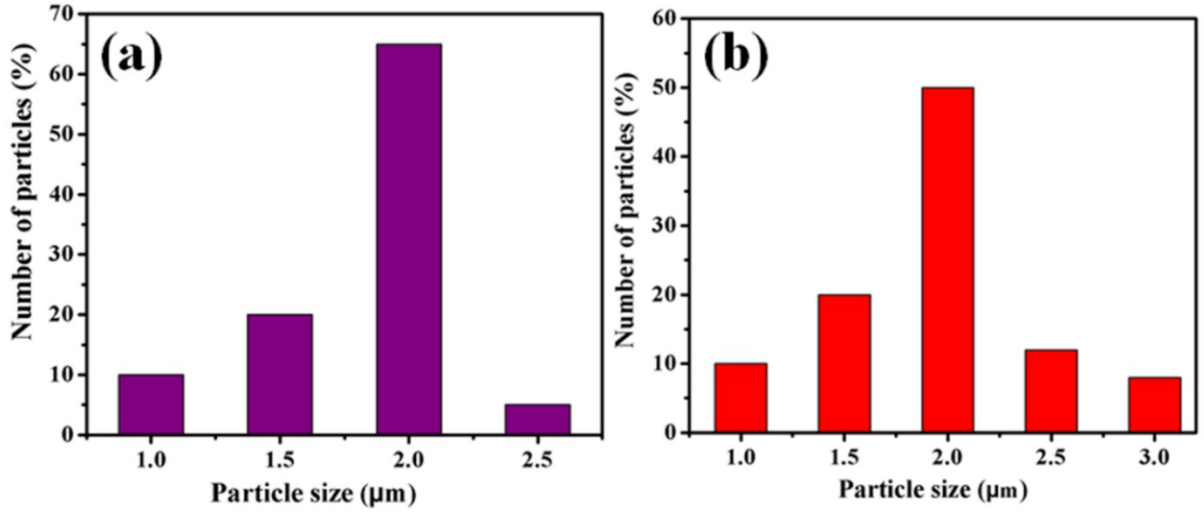

Figure S1. Size distribution of (a) flower-like and (b) octahedron-like fullerene micro-particles

X-ray photo electron spectroscopy is a powerful analytic tool to conform the presence of various elements and their oxidation states. The survey spectrum of fullerene flower-like crystals display strong line corresponding to carbon and a weak line corresponding to surface bound oxygen. The broad C1s peak can be deconvoluted into two peaks at 283.6 and 284.9 eV corresponding to C=C and C-C bonding, respectively and it is in good correspondence with the reported binding energy values[1].

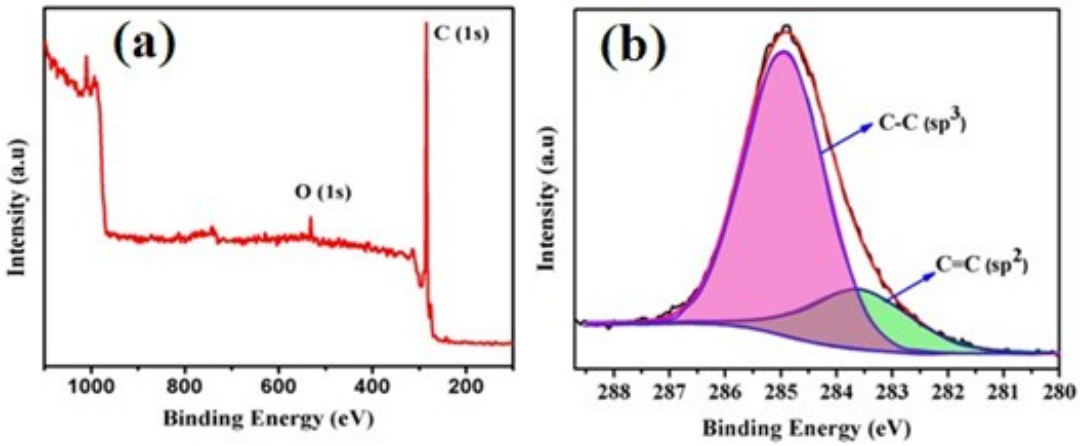

Figure S2. XPS (a) survey spectrum of flower-like fullerene crystals and (b) deconvoluted C 1s peak.

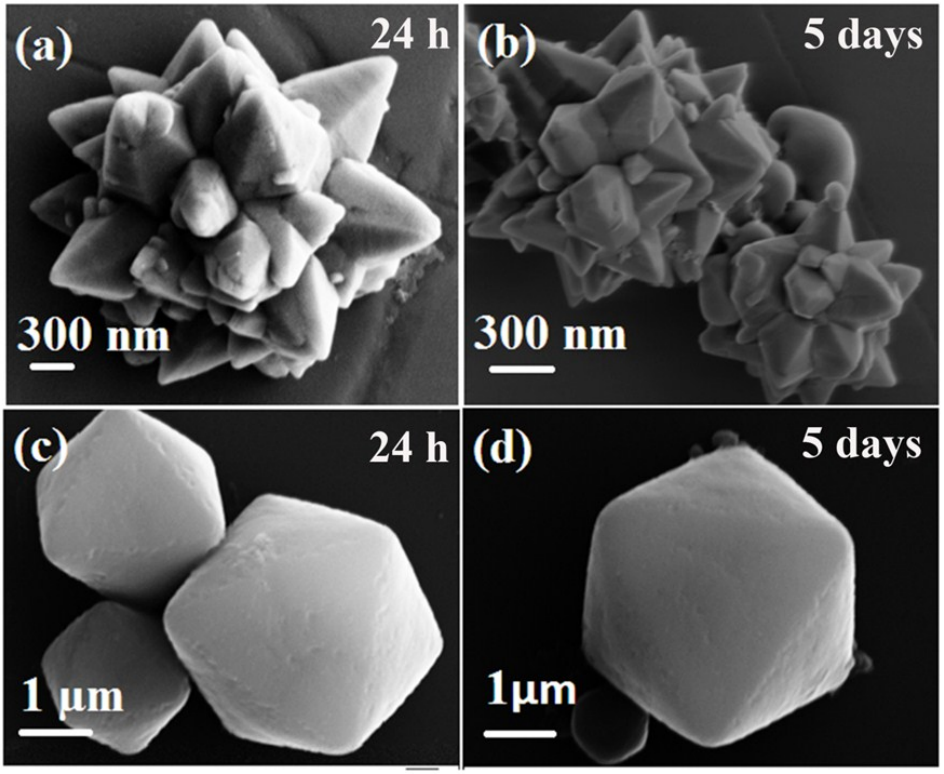

Figure S3. FE-SEM images of flower-like and octahedron-like fullerene micro-particles after 24 h (a & c) and 5 days (b & d).

Reference

1. R. G. Shrestha, L. K. Shrestha, A. H. Khan, G. S. Kumar, S. Acharya and K. Ariga, *ACS Appl. Mater. Interfaces*, 2014, **6**, 15597.