UNIVERSITI PUTRA MALAYSIA

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SYNTHESIS AND CHARACTERIZATION OF ZnO PARTICLES AND MWNTs-ZnO NANOCOMPOSITES

By

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SYNTHESIS AND CHARACTERIZATION OF ZnO PARTICLES AND MWNTs-ZnO NANOCOMPOSITES

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July 2012

Chair : Professor Mohd Zobir Bin Hussein, PhD

Faculty : Science

Multi-walled carbon nanotubes (MWNTs) were functionalized using various acid treatments to introduce functional groups, especially carboxylic acid groups on the surface of MWNTs. Different acid solutions namely nitric acid, citric acid, chromic acid and mixture of nitric and sulphuric acid were used for this purpose.

There are different parameters that had been used for the synthesis MWNTs-ZnO nanocomposites, including different acid treatments, the ratios between MWNTs and zinc precursor and surface adsorption of anionic and cationic surfactants. Each parameter was explored to investigate the effect on the physio-chemical properties of the MWNTs-ZnO nanocomposites prepared by simple, co-precipitation methods.

Through various acid treated of MWNTs, ZnO nanoparticles were selectively attached to the nanotubes and the presence of functional groups were further confirmed. MWNTs-ZnO nanocomposites that were prepared by pre-treatment of MWNTs with different acids gave dispersion of ZnO nanoparticles attached on the MWNTs but it was not formed uniformly on the surface of the MWNTs. Each of

MWNTs-ZnO nanocomposites prepared by pre-treatment of MWNTs with different acids gave different morphologies such as thin flake ZnO nanoparticles shown by FESEM study. On the other hand, different ratios between MWNTs and zinc precursors gave ZnO nanoparticles time to formed different shapes on the surfaces of MWNTs such flower-like shape.

By verifying the ratios of MWNTs and the precursors, the newly formed of ZnO nanoparticles morphologies such as flower-like ZnO nanoparticles attached on the MWNTs lead to new properties of MWNTs-ZnO nanocomposites. The ratio of 1:0.5 between MWNTs to Zn leads to better dispersion in the formation of ZnO on the surface of MWNTs. The increase in the ratios of MWNTs to ZnO leads the agglomeration of ZnO nanoparticles and increased the size of ZnO.

The morphologies of ZnO nanoparticles showed non-uniform, long finger-like irregular granular structure and rod-like structure when it was synthesized by co-precipitation method, with the addition of CTAB and SDS, respectively. The surface adsorption of Sodium dodecyl sulphate (SDS) and cetyltrimethylammonium bromide (CTAB) enable the decoration of MWNTs with a uniform size of zinc nanoparticles. MWNTs-ZnO nanocomposites prepared with addition of surfactants gives better distribution of ZnO nanoparticles formation on the surface of MWNTs and decreased their particle size to 15 nm.

The main problem in this research is the agglomeration of ZnO nanoparticles. They were agglomerated on the surface of MWNTs and makes the potential properties of the nanocomposites cannot fully discover. The inertness of MWNTs were also a challenge, but it has overcome with vary the type acid used.

The use of green beans as biotemplating agent showed that, ZnO nanoparticles can be formed with desired morphology and dimension. The result indicates that several surface morphology can be formed by adjusting the concentration of precursor solution. The average diameter of the particles was less than 1 μm. The green beans are thought to be responsible to control the size and morphology of the resulting ZnO nanoparticles by biotemplating process.

Abstrak tesis yang dikemukakan kepada Senat Universiti Putra Malaysia sebagai memenuhi keperluan ijazah untuk Master Sains

SINTESIS DAN PENCIRIAN ZnO PARTIKEL DAN MWNTs-ZnO NANOKOMPOSIT

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Karbon nanotiub (KNTs) dipelbagai fungsi, menggunakan pelbagai rawatan asid untuk memperkenalkan kumpulan berfungsi, terutamanya asid karbosilik pada permukaan KNTs. Pelbagai jenis asid digunakan iaitu asid nitrik, asid sitrik, asid kromik dan campuran asid nitrik dan sulfurik untuk tujuan ini.

Parameter yang berbeza telah digunakan dalam sintesis KNT bersama zink oksida, termasuklah asid yang berbeza, nisbah berat antara karbon nanotiub dan zink oksida nanopartikel (ZnO), dan serapan permukaan oleh kationik dan anionic surfaktan. Kesan setiap parameter ke atas sifat fizik-kimia KNT-ZnO yang dihasilkan menggunakn cara pemendakan telah dikaji.

Melalui rawatan pelbagai asid ke atas KNT, zink oksida secara terpilih malekat pada permukaan nanotiub dan lokasi kumpulan berfungsi telah disahkan. Rawatan pelbagai asid ke atas KNT-ZnO nanokomposit telah menghasilkan zink oksida nanopartikel yang melekat pada KNT secara tidak menyeluruh. Tetapi, setiap satu KNT-ZnO nanokomposit yang disediakan dengan cara ini menghasilkan kelainan pada morfologi seperti kepingan nipis zink oksida nanopartikel.

Dengan mempelbagaikan nisbah berat KNT dan zink ion, morfologi zink oksida yang baru terbentuk seperti zink oksida berbentuk bunga yang boleh menjadikan ia satu ciri baru untuk KNT-ZnO nanokomposit. Nisbah 1:0.5 antara KNT kepada zink ion memberi penyebaran yang lebih sekata dalam pembentukan ZnO pada permukaan KNT. Nisbah peningkatan zink ion dalam nisbah berat ini menyebabkan penggumpalan ZnO nanopartikel dan ini meningkatkan saiz ZnO.

Morfologi ZnO nanopartikel yang dipengaruhi surfaktan; SDS dan CTAB menunjukkan tidak keseragaman struktur berbentuk jari dan rod apabila disintesis dengan menggunakan kaedah pemendakan. Penggunaan surfaktan menyebabkan penyebaran pembentukan ZnO yang lebih sekata pada pemukaan KNT dan menyebabkan penurunan saiz ZnO sehingga 15 nm.

Masalah utama dalam kajian ini adalah pengumpulan ZnO nanopartikel. Ianya mengumpul di atas permukaan KNT dan menyebabkan potensi sifat nanokomposit tidak dapat ditunjukkan. Sifat lengai KNT juga satu cabaran, tapi ia dapat diatasi dengan menggunakan pelbagai jenis asid.

Penggunaan kacang hijau sebagai agen biotemplat manyebabkan ZnO nanopartikel dapat dibentuk dengan morfologi yang diingini dan dimensi yang spesifik. Hasil kajian menunjukkan pelbagai morfologi dapat dibentuk melalui kepelbagaian kepekatan cecair prekursor. Diameter purata pada setiap ZnO partikel yang dibentuk adalah kurang daripada 1 micrometer. Jadi, penggunaan kacang hijau sebagai agen biotemplat dapat mengawal saiz dan morfologi dalam sistesis ZnO partikel.

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I certify that a Thesis Examination Committee has met on the **13th July 2012** to conduct the final examination of Suzanita binti Latip on her thesis entitled Synthesis of ZnO particles and MWNTs-ZnO nanocomposites in accordance with the Universities and University Colleges Act 1971 and the Constitution of the Universiti Putra Malaysia [P.U. (A) 106] 15 March 1998. The committee recommends that the student be awarded the degree of Master of Science.

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DECLARATION

I declare that the thesis is my original work except for quotations and citation which have been dully acknowledged. I also declare that it has not been previously, and is not concurrently, submitted for any other degree at Universiti Putra Malaysia or at any other institution.

SUZANITA BINTI LATIP

Date: 13 July 2012

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