

UNIVERSITI PUTRA MALAYSIA

GAMMA RADIATION INDUCED SYNTHESIS AND OPTICAL CHARACTERIZATION OF COBALT AND NICKEL NANOPARTICLES STABILIZED IN POLYMER

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Thesis Submitted to the School of Graduate Studies, Universiti Putra Malaysia, in Fulfilment of the Requirement for the Degree of Master of Science

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DEDICATION

To my beloved parents, Haji Ilias Bin Haji Yunus and Hajah Ramlah Binti Haji Mat Sani, my siblings:

Suraya Hanis Binti Ilias
Suliana Hanim Binti Ilias
Sharul Rizan Bin Ilias
Sharul Nizam Bin Ilias
Last but not least to my grandmother,
brothers and sister in law, nephews, nieces and all my family.
Thank you very much.

Abstract of the thesis presented to the Senate of Universiti Putra Malaysia in fulfilment of requirement for the Master of Science

GAMMA RADIATION INDUCED SYNTHESIS AND OPTIC

CHARACTERIZATION OF COBALT AND NICKEL NANOPARTICLES STABILIZED IN POLYMER

By

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March 2011

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Faculty : Science

An attempt was made to synthesize Cobalt (Co) and Nickel (Ni) nanoparticles by radiation induction method with polyvinyl alcohol (PVA) and polyvinyl pyrrolidone (PVP) used as capping agents. Metal precursors CoCl₂ and NiSO₄ at various concentrations together with deionized water, polymer capping agent, and ascorbic acid anti-oxidant were stirred homogenously for 6 hours and the solutions were irradiated with ⁶⁰Co gamma rays at various doses up to 50 kGy. Free electrons and hydrated electrons generated by γ radiation interaction with water interacts with cobalt ions Co⁺ and nickel ions Ni²⁺ reduced them into Co and Ni zero valent atoms. After aggregation of atoms, metal nanoparticles are formed. The irradiated solutions containing PVA/Co nanoparticles, PVP/Co nanoparticles, PVA/Ni nanoparticles, and PVP/Ni nanoparticles were characterized for their size, shape, optical and electronic structures. The average particle sizes were measured by photon cross correlation spectroscopy which shows that the particle size was influenced by the precursor concentration and absorbed dose. The transmission electron micrographs were used to determine the particle structural morphology. The average particle diameter decreases with an increase of dose and a

decrease of precursor concentration. The effect of PVA and PVP on the particle sizes is not very significant for the formation of Co and Ni nanoparticles.

The UV-Visible spectrophotometer measurements revealed the formation of metal nanoparticles by exhibiting the absorption peak λ_{max} at about 520 and 400 nm for Co and Ni nanoparticles respectively. The absorption energy is referred as the conduction band energy determined from the absorption peak λ_{max} by $E_{CB} = hc/\lambda_{max}$. For a given precursor concentration the absorption peak λ_{max} blue shifts redundant with the increase of dose, indicating the conduction band of Co and Ni nanoparticles increases as the dose increases. The conduction band for Co nanoparticles in PVA increased from 2.416 eV at 10 kGy to 2.440 eV at 50 kGy for 1.0% wt CoCl₂ and from 2.398 eV at 10 kGy to 2.421 eV at 50 kGy for 3.0% wt CoCl₂. For Ni nanoparticles in PVA, the conduction band increased from 3.160 eV at 10 kGy to 3.184 eV at 50 kGy for 1.0% wt NiSO₄ and from 3.120 eV at 10 kGy to 3.152 eV at 50 Gy for 3.0% wt NiSO₄. The conduction band has a large energy value at the smaller particle sizes with an increase of dose. An increase of the conduction band with a decrease in particle size confirmed the phenomenon of quantum confinement effect for Co and Ni nanoparticles.

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

SINTESIS KAEDAH SINAR GAMMA DAN PENCIRIAN OPTIK NANOZARAH KOBALT DAN NIKEL DISTABILKAN OLEH POLIMER

Oleh

SUHAILA HANI BINTI ILIAS

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Percubaan dalam menghasilkan nanozarah Kobalt (Co) dan Nikel (Ni) melalui kaedah radiasi dengan polyvinyl alcohol (PVA) dan polyvinyl pyrrolidone sebagai agen pemerangkap. Garam CoCl₂ dan NiSO₄ dalam pelbagai kepekatan bersama dengan air penyahionan, ejen penukupan polimer dan bahan anti pengoksidaan iaitu asid askorbik telah dilarutkan selama 6 jam dan larutan dikenakan radiasi sinar gamma 60Co pada pelbagai dos sehingga mencapai 50 kGy. Elektron bebas dan elektron terhidrat dihasilkan mengunakan pengaruh radiasi γ interaksi air dengan ion Kobalt Co⁺ dan ion Nikel Ni²⁺ dikurangkan menjadi Co dan Ni atom sifar valen. Selepas pengumpulan, logam nanozarah akan terbentuk. Larutan yang telah diradiasi mengandungi nanozarah PVA/Co, nanozarah PVP/Co, nanozarah PVA/Ni dan nanozarah PVP/Ni telah dikategorikan mengikut saiz, bentuk, sifat optik dan struktur elektronik. Purata saiz zarah diukur melalui spektroskopi foton silangan perkaitan yang menunjukkan saiz zarah dipengaruhi oleh kepelbagaian kepekatan dan dos yang diserap. Mikroskop transmisi elektron telah digunakan untuk menentukan struktur morfologi zarah. Diameter saiz zarah menurun dengan kenaikan dos dan menurun dengan kepekatan pendahuluan. Kesan penggunaan PVA dan PVP ke atas saiz zarah tidak begitu penting bagi pembentukan nanozarah Co dan Ni.

Cahaya nampak-lembayung ultra mendedahkan pembentukan garam nanozarah melalui pempameran puncak penyerapan λ_{max} pada 520 dan 400 nm bagi nanozarah Co dan Ni. Penyerapan tenaga merujuk kepada jalur tenaga kekonduksian ditentukan daripada puncak penyerapan λ_{max} melalui $E_{CB} = hc/\lambda_{max}$. Bagi kepekatan pendahuluan, puncak penyerapan λ_{max} menunjukkan peningkatan dos, menandakan bahawa jalur kekonduksian untuk nanozarah Co dan Ni berlebihan dengan kenaikan dos. Jalur kekonduksian bagi nanozarah Co didalam PVA meningkat daripada 2.416 eV pada 10 kGy kepada 2.440 eV pada 50 kGy untuk 1.0% wt CoCl₂ dan daripada 2.398 eV pada 10 kGy kepada 2.421 eV pada 50 kGy untuk 3.0% wt CoCl₂. Untuk nanozarah Ni didalam PVA, jalur kekonduksian meningkat daripada 3.160 eV pada 10 kGy kepada 3.184 eV pada 50 kGy untuk 1.0% wt NiSO₄ dan daripada 3.120 eV pada 10 kGy kepada 3.152 eV pada 50 kGy untuk 3.0% wt NiSO₄. Jalur kekonduksian mempunyai nilai tenaga yang besar pada saiz zarah yang lebih kecil dengan kenaikan dos. Peningkatan jalur kekonduksian dan penurunan saiz zarah disahkan dengan fenomenon kesan pengurungan kuantum bagi nanozarah Co dan Ni.

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I certify that an Thesis Examination Committee has met on 1 March, 2011 to conduct the final examination of Suhaila Hani Binti Ilias on her thesis entitled "Gamma Radiation Induced Synthesis and Characterization of Cobalt and Nickel Nanoparticles Stabilized in Polymer" in accordance with Universities and University College 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 Master of Science.

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DECLARATION

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

SUHAILA HANI BINTI ILIAS

Date: 1 March 2011



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