



UNIVERSITI PUTRA MALAYSIA

**PREPARATION AND CHARACTERIZATION OF
POLY(HYDROXYBUTYRATE-CO-HYDROXYVALERATE)/CLAY
NANOCOMPOSITES**

MOHD FIRDAUS BIN MOHD ANUAR

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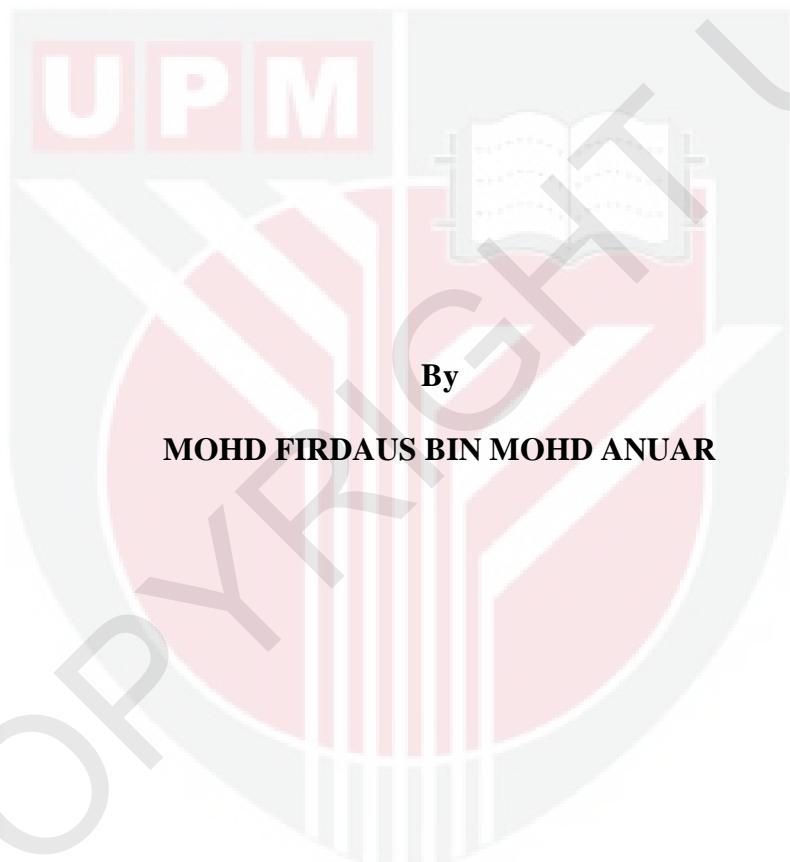
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POLY(HYDROXYBUTYRATE-CO-HYDROXYVALERATE)/CLAY
NANOCOMPOSITES**



MOHD FIRDAUS BIN MOHD ANUAR

**Thesis Submitted to the School of Graduate Studies, Universiti Putra
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Science**

August 2012

Abstract of Thesis Presented To The Senate Of Universiti Putra Malaysia In
Fulfillment of The Requirement For The Degree of Master of Science

**PREPARATION AND CHARACTERIZATION OF
POLY(HYDROXYBUTYRATE-CO-HYDROXYVALERATE)/CLAY
NANOCOMPOSITES**

By

MOHD FIRDAUS BIN MOHD ANUAR

August 2012

Chairman : Nor Azowa Ibrahim, PhD

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In this study, natural clay (Na-MMT) and synthetic layered double hydroxide (LDH) were modified to produce organo-modified clay. LDH was prepared via co-precipitation method by adding dropwise a solution of NaOH (1M) into a solution of $Mg(NO_3)_2 \cdot 6H_2O$ and $Al(NO_3)_3 \cdot 9H_2O$. Both Na-MMT and LDH were modified via ion exchange technique using octadecylamine (ODA) and sodium stearate respectively to perform organo-modified clays. The modification was carried out by stirring the clay in an aqueous solution of a mixture of Na-MMT and ODA, and LDH and sodium stearate. The modified clays were then used in preparation of poly(hydroxybutyrate-co-hydroxyvalerate), PHBV nanocomposites. The intercalation of the modifier in the clays layer were characterized by X-ray Diffraction (XRD) Analysis and Fourier Transform Infrared (FTIR) Spectroscopy.

PHBV/clay nanocomposites were prepared via solution casting technique. The clays were added into chloroform solution of PHBV based on a fixed amount of clay loading. The amount of clays added were 0.25, 0.5, and 1.0 wt% for all type of clays. For further dispersion of clay, the mixture of PHBV/clay solution was sonicated for 30 min. The viscous solutions were casted in a glass petri dish and dried in the solvent atmosphere to obtain the nanocomposite films.

The effect of clay loading on PHBV/clay nanocomposites were characterized by means of mechanical, thermal and morphology properties. The addition of natural and modified clays was found not to increase the tensile strength property of the nanocomposites. However, the elongation at break property was found to increase for all nanocomposites. At 0.5 wt% of clay loading of ODA-MMT, the maximum elongation at break was achieved. This result is in accordance with Scanning Electron Microscopy (SEM) analysis which shows the stretching images at 0.5 wt% of clay loading for all nanocomposites. The thermal stability of PHBV/clay nanocomposites was found to increase with the increase of clay loading. Transmission Electron Microscopy (TEM) images showed that exfoliated and intercalated type of nanocomposites was successfully prepared with the incorporation of 1.0 wt% of modified clays loading.

Abstrak Tesis Yang Dikemukakan Kepada Senat Universiti Putra Malaysia Bagi
Memenuhi Keperluan Untuk Ijazah Master Sains

**PENYEDIAAN DAN PENCIRIAN POLI(HIDROKSIBUTIRAT-
BERSAMA-HIDROKSIVALERAT)/TANAH LIAT NANOKOMPOSIT**

Oleh

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Dalam kajian ini, tanah liat semula jadi (Na-MMT) dan sintetik hidroksida berlapis dua (LDH) telah diubahsuai untuk menghasilkan tanah liat organo-terubahsuai. LDH telah disediakan melalui kaedah pemendakan bersama dengan menambah titisan larutan NaOH ($1M$) ke dalam larutan $Mg(NO_3)_2 \cdot 6H_2O$ dan $Al(NO_3)_3 \cdot 9H_2O$. Kedua-dua Na-MMT dan LDH telah diubahsuai melalui teknik pertukaran ion dengan menggunakan oktadesilamina (ODA) dan natrium stearat untuk menyediakan tanah liat organo-terubahsuai.

Pengubahsuaian telah dijalankan dengan mengacau tanah liat dalam larutan akueus campuran Na-MMT dan ODA, dan LDH dan natrium stearat. Tanah liat yang telah diubahsuai digunakan dalam penyediaan poli(hidroksibutirat-bersama-hidroksivalerat), PHBV nanokomposit. Interkalasi pengubahsuaian dalam lapisan tanah liat telah dicirikan oleh analisis pembezaan sinar-X (XRD) dan spektroskopi infra-merah fourier (FTIR). Nanokomposit PHBV/tanah liat telah disediakan melalui teknik pemutus larutan. Tanah liat telah dicampurkan ke dalam larutan kloroform PHBV berdasarkan jumlah muatan tanah liat yang telah ditetapkan.

Jumlah tanah liat yang ditambah ialah 0.25, 0.5, dan 1.0% berat bagi semua jenis tanah liat. Untuk penyebaran lanjut daripada tanah liat, campuran larutan PHBV / tanah liat telah disonikasi selama 30 minit.

Larutan yang likat telah diisi ke dalam piring Petri kaca dan dikeringkan untuk mendapatkan filem-filem nanokomposit. Kesan muatan tanah liat pada nanokomposit PHBV/tanah liat telah dicirikan melalui sifat-sifat mekanik, terma dan morfologi. Selain itu, tanah liat semulajadi dan terubahsuai didapati tidak meningkatkan kekuatan tegangan nanokomposit. Pemanjangan ketika putus didapati meningkat untuk semua nanokomposit. Pada kadar 0.5% berat muatan tanah liat ODA-MMT, pemanjangan maksimum telah dicapai ketika putus. Keputusan ini adalah selaras dengan analisis Pengimbas Mikroskopi Elektron (SEM) yang menunjukkan imej regangan pada kadar 0.5% berat muatan tanah liat untuk semua nanokomposit. Kestabilan haba nanokomposit PHBV/tanah liat didapati meningkat dengan setiap penambahan muatan tanah liat. Imej Mikroskopi Transmisi Elektron (TEM) menunjukkan bahawa nanokomposit eksfoliasi dan interkalasi telah berjaya dihasilkan dengan penambahan 1.0% berat tanah liat terubahsuai.

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I certify that an Examination Committee has met on to conduct the final examination of Mohd Firdaus Bin Mohd Anuar on his Master of Science thesis entitled "Preparation and Characterization of Poly(Hydroxybutyrate-co-hydroxyvalerate)/Clay Nanocomposites" in accordance with Universiti Pertanian Malaysia (Higher Degree) Act 1980 and Universiti Pertanian Malaysia (Higher Degree) Regulations 1981. The Committee recommends that the student be awarded the Master of Science.

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DECLARATION

I declare that this thesis is my original work except for quotations, citations and illustrations which have been duly 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 institutes.

MOHD FIRDAUS B MOHD ANUAR

Date: 13th August 2012



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