UNIVERSITI PUTRA MALAYSIA

PREPARATION AND CHARACTERIZATION OF α-ALUMINA NANOPARTICLES AND α-ALUMINA-BASED POLYPROPYLENE NANOCOMPOSITES

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PREPARATION AND CHARACTERIZATION OF α-ALUMINA NANOPARTICLES AND α-ALUMINA-BASED POLYPROPYLENE NANOCOMPOSITES

By

FATEMEH MIRJALILI

Thesis Submitted to the School of Graduate Studies, University Putra Malaysia, and Fulfilment of the Requirements for the Degree of Doctor of Philosophy

July 2010

DEDICATION

Dedicated to:

My beloved family: my mother, my sister and my brother whose constant supports, unfading love, words of encouragement and patience has always been a great consolation for me to go through the challenging steps in my life.

Abstract of thesis presented to the Senate of Universiti of Putra Malaysia in fulfilment of the requirement for the degree of Doctor of Philosophy

PREPARATION AND CHARACTERIZATION OF α-ALUMINA NANOPARTICLES AND α-ALUMINA-BASED POLYPROPYLENE NANOCOMPOSITES

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July 2010

Chairman: Associate Professor Luqman Chuah Abdullah, PhD

Faculty: Engineering

In this study, nano α-alumina particles were synthesized by a sol–gel method. Sodium dodecylbenzen sulfonate (SDBS) and sodium bis-2-ethylhexyl sulfosuccinate (Na (AOT)) were implemented as the surfactant stabilizing agents. The prepared solution was, then, stirred at different times (24, 36, 48 and 60 hours) at 60ºC. SDBS application proved to present a better nanoparticle dispersion and finer particle size as compared to Na (AOT). The finest particle size ranging from 20-30 nm was obtained at 48 hour stirring time with SDBS surfactant being at 1200°C.

Similarly, the effect of temperature on production of α- nano alumina was put under investigation. The process included milling the mediums and uniformly mixing them with the hydrous alumina during grinding as well as utilizing ZnF_2 additives in order to reduce the transformation temperature and to modify the alumina particles shape. The non agglomerated nano plate like alpha alumina particles were observed to be formed at their finest size (15-20nm) at 950°C.

Later, self–produced nano α-alumina particles were utilized to develop the PP/ nano α-Al_2O_3 composite. This study aimed to investigate the mechanical, thermal and morphological properties of PP/ nano α-Al_2O_3 composite with and without addition of dispersant. Polypropylene matrix was filled and mixed with different weight percentages of nano α- alumina particles using a Haake internal mixer. Mixing was performed at 170ºC and 50 rpm for the rotor speed. Mechanical tests such as tensile, flexural and Izod impact tests were found to increase by the addition of content of nano α-Al_2O_3 as well as the dispersant. Maximum tensile strength (~16 %) and tensile modulus (~27%) can be attributed to the increase of nano α-Al_2O_3 loading from 1 to 4 wt % into PP matrix. Flexural analysis showed maximum values of 50.5 and 55.88 MPa for flexural strength and 1954 and 2818 MPa for flexural modulus for PP/ nano α-Al_2O_3 composite with and without dispersant ,respectively. Notched-Izod impact energy showed an increase up to ~ 43 J/m for the PP/4wt% nano α-Al_2O_3 composites. Higher concentration of nano α-Al_2O_3 loading resulted in a reduction of mechanical properties due to nano α-Al_2O_3 agglomeration. Dynamic mechanical analysis indicated that the storage and loss modulus improved with the addition of nano α-Al_2O_3 loading.

TGA and DSC analyses were conducted to recognize the thermal behavior of the composites produced. TGA results illustrated a drastic shift of the weight loss curve towards higher temperature, which led to reduction in the polypropylene heat release rate in both composites. DSC results showed that crystallinity of composites increased with the addition of the Al_2O_3 nanoparticles. Finally, FTIR study confirmed the existing of Al-O bond at 568 cm-1 resulted from the nano α-Al_2O_3 particles which were recently created.

Abstrak tesis yang dikemukakan kepada Senat Universiti Putra Malaysia untuk memenuhi keperluan untuk ijazah Doktor Falsafah.

PENYEDIAAN DAN PENCIRIAN PARTIKEL NANO

α-ALUMINA DAN KOMPOSIT POLIPROPELINA NANO

α-ALUMINA

Oleh

FATEMEH MIRJALILI

Julai 2010

Pengerusi: Profesor Madya Luqman Chuah Abdullah, PhD

Fakulti: Kejuteraan

Dalam kajian ini, partikel nano α-alumina telah disintesiskan dengan menggunakan kaedah sol-gel. Natrium dodesil benzene sulfonat (SDBS) dan natrium bis-2-etilheksil sulfosuksinat (Na (AOT)) telah digunakan sebagai agen penstabilan surfaktan. Larutan yang telah disediakan kemudian dikacau pada tempoh masa berbeza (24, 36, 48 dan 60 jam) pada suhu 60 °C. Penggunaan SDBS terbukti memberikan penyebaran partikel nano yang lebih baik dan saiz partikel yang lebih halus dibandingkan dengan Na (AOT). Saiz partikel yang paling halus adalah dalam

lingkungan 20 nm hingga 30 nm telah diperolehi pada 48 jam masa kacau dengan surfaktan SDBS pada suhu 1200°C.

Kesan suhu ke atas penghasilan partikel nano α- alumina juga telah dikaji. Proses tersebut melibatkan penggiling yang sederhana dan sekata dengan mencampurkannya dengan alumina hidrous semasa menggiling untuk menggunakan aditif ZnF_2 supaya dapat mengurangkan tranformasi suhu dan untuk mengubahsuai bentuk partikel alumina. Partikel nano alfa alumina yang tidak bergumpal dan berbentuk seperti plat telah diperhatikan berhasil pada saiz yang terhalus (15-20 nm) pada suhu 950°C.

Kemudian, partikel nano α-alumina telah digunakan untuk menghasilkan komposit PP/ nano α-Al_2O_3. Kajian ini bertujuan untuk menyiasat sifat-sifat mekanik, terma dan morfologi komposit PP/ nano α-Al_2O_3 dengan atau tanpa pertambahan agen penyerakan. Matrik polipropelina diisikan dan dicampurkan dengan peratusan berat berbeza partikel nano α- alumina menggunakan sebuah alat pengisar internal HAAKE. Pencampuran telah dilakukan pada suhu 170ºC dan 50 rpm untuk kelajuan pemutar. Ujian mekanikal seperti keregangan, lenturan dan hentaman Izod telah menunjukkan peningkatan dengan pertambahan pemuatan partikel nano α-Al_2O_3 dan juga bahan penyerakan. Kekuatan regangan maksimum (~16%) dan modulus regangan (~27%) adalah dicirikan kepada pertambahan pemuatan partikel nano α-Al_2O_3 daripada 1 hingga 4 wt % kedalam matrik PP. Analisis kelenturan menunjukkan nilai maksimum pada 50.5 dan 55.88 MPa untuk kekuatan lenturan dan 1954 dan 2818 MPa untuk modulus kelenturan untuk komposit PP/ nano α-Al_2O_3 dengan atau tanpa bahan penyerakan. Tenaga untuk ujian hentaman Izod menunjukkan peningkatan sehingga ~ 43 J/m untuk komposit PP/ 4wt% nano α-Al_2O_3. Kandungan tertinggi α-Al_2O_3 menunjukkan pengurangan dalam sifat-sifat mekanikal kerana pengumpalan nano α-Al_2O_3. Analisis mekanikal dinamik menunjukkan bahawa modulus storan dan modulus kehilangan meningkat dengan pertambahan pemuatan nano α-Al_2O_3.

Analisis TGA dan DSC dijalankan untuk mengenal sifat terma komposit yang dihasilkan. Keputusan TGA diilustrasikan anjakan mendadak dalam garis lengkungan kehilangan berat melawan suhu tertinggi, di mana menjuruskan kepada pengurangan dalam kadar pelepasan haba polipropelina dalam kedua-dua komposit. Keputusan DSC menunjukkan kehabluran komposit meningkat dengan pertambahan partikel nano Al_2O_3. Akhirnya, ujian FTIR mengesahkan kehadiran ikatan Al-O pada 568 cm^{-1} menunjukkan partikel nano telah terbentuk.

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my dream of studying. Their constant spiritual, financial and moral support was always a great consolation for me.

I certify that an Examination Committee has met on 15th July 2010 to conduct the final examination of Fatemeh Mirjalili on her Doctor of Philosophy thesis entitled "Production and characterization of nano α- alumina particles and α- alumina based polypropylene nanocomposites" in accordance with the Universities and University Colleges Act1971 and the Constitution of the Universiti Putra Malaysia[P.U.(A)106] 15 March1998. The Committee recommends that the student be awarded the Doctor of Philosophy.

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This thesis was submitted to the Senate of Universiti Putra Malaysia and has been accepted as fulfilment of the requirement for the degree of Doctor of Philosophy. The members of the Supervisory Committee were as follows:

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Date: 21 October, 2010

DECLARATION

I declare that the thesis is my original work except for quotation and citations, which have been duly acknowledged. I also declare that it has not been previously, and it is not concurrently, submitted for any other degree at University Putra Malaysia or other institutions.

FATEMEH MIRJALILI

Date: 15 July 2010

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