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THE DEVELOPMENT OF POLYPROPYLENE COMPOSITES REINFORCED MICRO AND NANOALUMINA FILLER FROM INDUSTRIAL WASTE

BY

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A dissertation submitted in fulfilment of the requirement for the degree of Master of Science (Materials Engineering)

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ABSTRACT

Excessive source of bauxite ore has led to the relentless production of primary Aluminium (Al) globally. Environmental hazards caused by the Al manufacturing activities and high cost procedure of its scheduled waste safe disposal pose serious problems and demands sustainable solutions for the Al industries. Malaysia has abundant deposit of Al dross resulted from Al smelting process and easily available thermoplastic polypropylene (PP) from the oil industry. Effective use of local Al dross waste recycling to alumina (Al_2O_3) as filler in PP composite system is an interesting economic prospect, since Malaysia currently imports its Al_2O_3 requirements. The aim of this work is to synthesize micro and nano α-Al_2O_3 from Al dross using thermal decomposition and wet milling method; followed by analyzing the effect of micro and nano α-Al_2O_3 content on mechanical, morphological, wear and thermal properties as well as addition of maleic anhydride grafted polypropylene (MAPP) compatibilizer in PP composites. PP composites (without and with MAPP) based on various formulations (1, 3, 5 and 7 weight percentage (wt.%)) for both micro and nanosized α-Al_2O_3 particles were prepared through different processing method. The processes involved were compounding method using twin screw extruder machine followed by hot pressing. The mechanical properties of micro and nanocomposites are studied through tensile and impact tests. Mechanical properties showed improvement in tensile strength and modulus with increases micro and nano α-Al_2O_3 content from 1 to 5 wt.%. An optimum tensile strength was obtained by PP composite with MAPP filled 5 wt.% nano α-Al_2O_3 particles. These findings were supported by field emmision scanning electron microscopy (FESEM) where in the MAPP addition particularly, better dispersion of 5 wt.% nanocomposite was observed. Impact strength values however decreases with increases of α-Al_2O_3 loading from 3 to 7 wt.% which indicates improved stiffness of both micro and nanocomposite. The highest wear rate attained by 7 wt.% nanocomposite was significant as increased in nano α-Al_2O_3 content along with MAPP addition resulted in better wear resistance. Similarly, α-Al_2O_3 particles increment up to 7 wt.% increases the decomposition and melting temperature (T_m) of nanocomposite with MAPP compared to microcomposite. Thus, micro and nanoalumina particles produced from Al dross is an appealing alternative in utilizing the industrial waste efficiently besides enhancing the composites' mechanical, wear and thermal properties.

خلاصة البحث

الاستخدام المفرط لمصادر خام البوكسيت أدى إلى زيادة مطَّردة في أنتاج الألومنيوم الأولي على الصعيد العالمي. إن المخاطر البيئية الناجمة عن الأنشطة الصناعية التحويلية للألومنيوم وإجراءات التكلفة العالية للتخلص الآمن تسبب مشاكل خطيرة وتتطلب حلولا مستدامة لصناعات الألومنيوم. ماليزيا لديها مخزون كبيرة من خبث الألومنيوم والناتجة من عملية الصهر والمتوفرة بسهولة من البلاستيك الحراري البولي بروبلين من صناعة النفط. الاستخدام الفعّال لإعادة تدوير نفايات خبث الألومنيوم المحلي وتحويلها الى أكسيد الألومينيوم كمواد حشو في مركبات البولي بروبلين يعتبر بمثابة فرصة اقتصادية مثيرة للاهتمام، حيث ان ماليزيا تستورد حاليا متطلباتها من أكسيد الألومينيوم. الهدف من هذا العمل هو تجميع الدقائق الصغيرة والمتناهية الصغر من أكسيد الألومينيوم من خبث الألومنيوم باستخدام التحلل الحراري وطريقة الطحن الرطبة؛ يليه تأثير محتوى جزئيات أكسيد الألومينيوم الصغيرة والمتناهية الصغر على الخواص الميكانيكية، البنيوية، القدرة والخواص الحرارية الى جانب إضافة أنهيدريد المالئيك المطعمة البولي بروبلين بناءً على صيغ مختلفة (1,3,5 و7 نسبة وزنية لكل من الجزيئات الصغيرة والمتناهية الصغر من أكسيد الألومينيوم حيث حضرت من خلال طرق معالجة مختلفة). العمليات التي استخدمت تتضمن طرق معقدة باستخدام آلة المفك المزدوج السحب يليه الضغط الساخن. يتم دراسة الخواص الميكانيكية الجزيئات الصغيرة والمتناهية الصغر من خلال اختبارات الشد والضغط. وأظهرت الخواص الميكانيكية تحسن في قوة الشد ومعامل المرونة مع الزيادات في الجزيئات الصغيرة والمتناهية الصغر من أكسيد الألومينيوم من 1 الى 5 % نسبة وزنية. تم الحصول على قوة الشد المثلى عن طريق مركبات البولي بروبلين مع أنهيدريد المالئيك المطعمة البولي بروبلين تملأ مع 5 % من جزيئات أكسيد الألومينيوم. ودعمت هذه النتائج عن طريق مجال المجهر الإلكتروني حيث في إضافة بشكل خاص البولي بروبلين مع أنهيدريد المالئيك لوحظ الانتشار الأفضل بنسبة 5%من النسبة الوزنية. لكن قوة الضغط تناقصت مع زيادة جزيئات أكسيد الألومينيوم مع زيادة القوة من 3- 7 % من الوزن مما يدل على تحسين صلابة الجزيئات الصغيرة والمتناهية الصغر. وكان أعلى معدل التآكل الذي حققته هو 7% بالوزن بمركب متناهي الصغر أكبر عن زيادة في نانو محتوى الأمونيا مع أنهيدريد المالئيك المطعمة البولي بروبلين أدى إلى تحسين مقاومة التآكل. وبالمثل زيادة جزيئات أكسيد الألومينيوم الصغيرة والمتناهية الصغر الى 7 % يزيد من تحلل وذوبان درجة الحرارة من الجزيئات الصغيرة مع أنهيدريد المالئيك المطعمة البولي بروبلين بالمقارنة مع الجزيئات المتناهية في الصغر. وبالتالي فإن التنمية في الجزيئات الصغيرة والمتناهية الصغر من خبث الألومنيوم المحلي يعج بديلاً جذاباً في الاستفادة من النفايات الصناعية بكفاءة إلى جانب تعزيز الخواص الميكانيكية والتآكل والخصائص الحرارية.

APPROVAL PAGE

I certify that I have supervised and read this study and that in my opinion it conforms to acceptable standards of scholarly presentation and is fully adequate, in scope and quality, as a dissertation for the degree of Master of Science (Materials Engineering).

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DECLARATION

I hereby declare that this dissertation is the result of my own investigations, except where otherwise stated. I also declare that it has not been previously or concurrently submitted as a whole for any other degrees at IIUM or other institutions.

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LIST OF ABBREVIATIONS

%	Percent
°C	Degree celcius
°C/min	Degree per minute
°F	Degree fahrenheit
1D	One dimension
2D	Two dimension
3D	Three dimension
α-Al_2O_3	Alpha-alumina
µl	Micro liter
µm	Micro meter
AACH	Ammonium aluminium carbonate hydroxide
Al	Aluminium
Al_2O_3	Alumina
$Al_2(SO_4)_3$	Aluminium sulphate
AlO_2Na	Sodium aluminate
AlO(OH)	Aluminium oxy-hydroxide
$Al(OH)_3$/ATH	Aluminium hydroxide/Aluminium trihydrate
$Al(OH)_4$	Hydroxoaluminates
ASTM	American society for testing and materials
C	Carbon
$CaCO_3$	Calcium carbonate
CaO	Calcium oxide
CH_2	Methylene
CH_3	Methyl
cm	Centimeter
cm^{-1}	Wavenumber
Cu	Copper
DSC	Differential Scanning Calorimetry
e.g	(example gratia) as for example
et al.	(*et alia*): and others
etc.	(*et cetera*): and so forth
FESEM	Field Emission Scanning Electron Microscopy
FTIR	Fourier Transform Infrared Spectroscopy
g	Gram
g/cm^3	Gram per centimeter cubic
g/L	Gram per liter
g/m^3	Gram per meter cubic
g/mL	Gram per mililiter
GCC	Gulf cooperation council
G-E	Glass fabric reinforced epoxy composites
GPa	Giga-Pascal

GPC	Gel permeation chromatography
h	Hour
H	Hydrogen
H_2O	Water
HDPE	High density polyethylene
He-Ne	Helium-Neon
HPP	Homopolymer polypropylene
IAI	International Aluminium Institution
i.e.	(*id est*): that is
ICP	Impact (block) copolymer
ICSD	Inorganic crystal structure database
IR	Infrared
J	Joule
J/g	Joule per gram
kJ/m^2	Kilo joule per meter square
kg	Kilogram
kg/cm^2	Kilogram per centimeter square
kg/mm^2	Kilogram per milimeter square
kN	Kilo-Newton
kV	Kilo-Volt
LDPE	Low density polyethylene
MA	Maleic anhydride
MAPP	Maleic anhydride grafted polypropylene
MFI	Melt flow index
min	Minute
mm	Milimeter
mm/min	Milimeter per minute
MPa	Mega-Pascal
MPa·m½	Mega-Pascal half meter square
MSc.	Master of science
NaOH	Sodium hydroxide
nm	Nanometer
O_2	Oxygen
OH	Hydroxide
PE	Polyethylene
PEEK	Polyetheretherketone
PMMA	Polymethyl methacrylate
PP	Polypropylene
PP/α-Al_2O_3	Polypropylene/alpha-alumina
PP-g-MA	Polypropylene-grafted-maleic anhydride
PP/SiO_2	Polypropylene/silicon oxide
PSA	Particle Size Analyzer
PTFE	Polytetrafluoroethylene
PVC	Polyvinyl chloride
RCP	Random copolymers
rpm	Revolution per minute
SEM	Scanning Electron Microscopy
SiO_2	Silicon oxide

SLS	Sodium lauryl sulphate
T_c	Crystallization temperature
TEM	Transmission Electron Microscopy
T_g	Glass transition temperature
TGA	Thermogravimetric Analysis
TG/DTA	Thermogravimetry Differential Thermal Analysis
T_m	Melting temperature
tpm	Tonnes per million
UTS	Ultimate tensile stress
UV	Ultraviolet
w/o	Without
W/m°K	Watt per meter kelvin/Thermal conductivity
wt.%	Weight percentage
XRD	X-Ray Diffraction
ZnO	Zinc oxide
ZnO-PP	Zinc oxide-Polypropylene

LIST OF SYMBOLS

°	Degree
α	Alpha
β	Beta
χ	Chi
δ	Delta
η	Eta
γ	Gamma
κ	Kappa
λ	Lamda/Wavelength
ρ	Rho
θ	Theta
<	Less-than
>	More-than
~	Approximately
±	Plus or minus

CHAPTER ONE

INTRODUCTION

1.1 BACKGROUND

Alumina, with Al_2O_3 chemical formula exists in a white powder. It is also known as a polymorphic crystalline aluminium oxide and commonly produced from bauxite ores. Alumina polymorphs become a very significant part of studies among researchers nowadays even though its properties are already known extensively. This is because different types of alumina polymorphs phases have different properties and therefore it is widely used in various industrial applications namely composites, ceramics, abrasive materials, biomaterials, catalyst and etc.

Basically, Al_2O_3 is one of the main constituents of aluminium (Al) dross extraction through Al refining processes (Adeosun et al., 2012). From Al refining process, the primary and secondary production of Al produces a residual waste known as Al dross which in latter became an industrial waste product. This Al dross waste can be recycled and it is produced almost five million tonnes every year as reported from the worldwide Al industry (International Aluminium Institute, 2014).

Alpha-alumina (α-Al_2O_3), which is one of the alumina's polymorphs exhibits the strongest and stiffest of the oxide ceramics (Wilfred et al., 2009). It is thermodynamically the stable phase and occurs naturally as corundum or sapphire. Alpha-alumina is frequently used in the industry as wear resistance coatings due to its hardness and high thermal stability. Other than that, α-Al_2O_3 is commonly synthesized by thermal decomposition method where high temperature is needed to produce alpha phase of alumina (Adkins et al., 1966). α-Al_2O_3 is produced at temperature higher than

1200 °C and the particles are microcrystal with the same crystalline structure as corundum (Souza-Santos et al., 2000).

Nanosized materials have become one of the most advanced materials in the market nowadays which give a very high impact toward the industries. The increase of surface area to volume ratio in nanomaterials has essentially increased its ability in influencing matter into impossible scales. The outcomes is benefited in the production of materials and products with new properties, contributing to solutions to the environmental problems, improving the innovation of existing technologies and also help in optimization of primary conditions for practical application (Lines, 2008).

Via thermal decomposition method, Al dross can be transformed into α-Al_2O_3 along with changes in its phases. Thus, in this research, α-Al_2O_3 will be used as micro and nanofiller in polypropylene (PP) composites. Coupling agent of maleic anhydride grafted polypropylene (MAPP) will be added to improve the surface adhesion between filler-matrix interface and consequently the properties for both of the composites. The influence of α-Al_2O_3 particle sizes, loading and the surface modification with MAPP coupling agent in the aspects of the composites' mechanical, morphological, wear resistance and thermal properties will be studied.

1.2 PROBLEM STATEMENT AND ITS SIGNIFICANCE

The production of primary Al globally has increased over the years due to the highest demand of Al in numerous industrial sectors. The abundance of Al dross, which is by-product of Al from the smelting process is classified as a schedule waste in Malaysia because it could pose environmental hazards when this waste is disposed on landfills and ground water (Meor et al., 2011). These environmental hazards have led to lots of problems including the occurrence of health issue such as cancer disease and bronchitis when these particles emitted in atmosphere (Berkum & McLachlan, 1986; Goyer, 1991; Shore & Wyatt, 1983 and Arieff et al., 1979). The safe disposal of this schedule waste is also a costly process that charged RM 2,000 fee per tonned. The Al manufacturing companies were burdened by this safe disposal of Al dross waste since its storage, transportation and disposal activities must be carried out only by licensed contractors (Meor et al., 2012). Consequently, the high disposal fee had caused an indiscriminate disposal of Al dross waste in secluded areas of palm oil plantation at Segamat, Johor (The Star, 2006). The incidence was marked as a major environmental disaster and authorities were urged to take strict actions on the violators ever since.

Meanwhile, special interests generated in the world of polymer engineering these days are the application of fillers in thermoplastic. Different kinds of fillers have been introduced in order to increase the number of filler selection in the market. Recycling alternative of Al dross waste into a value-added material like filler based polymer composite is one of such interests since it has received numerous attentions in materials science for both ecological and technological perspectives. As a new class of filler in polymer based composite, its improvement on the environmental effect is significant even at low filler loading because the effort itself has helped preserving a sustainable environment by saving natural resources through the use of secondary resources.

Extensive research work has been done for the fabrication of composite with different particle size of Al_2O_3 filler (Mirjalili et al., 2014; Orellana et al., 2014 and Kakde & Paul, 2014). Comparing micron and nanosized Al_2O_3 particles, nanoalumina exhibits many advantages. For example, PP/Al_2O_3 nanocomposite shows better mechanical strength and high in thermal stability. The addition of nanoalumina particles also improved the wear resistance in ABS polymer composite. However, the major drawback in nanocomposite is the cost-effectiveness of its synthesis and uniform dispersion of nanofiller against agglomeration throughout the polymer substrate.

Thermal decomposition is one of the cheap and convenient ways for the most stable α phase alumina to be produced from Al dross due to its simplicity as potential application at industrial scale (Darezereshki et al., 2011). Through this phase transformation, a smaller particle size of α alumina can be formed. There are several methods to synthesize nanoparticles. Among the various methods available, mechanical grinding which includes dry and wet milling technique is a promising method for the large-scale production of nanoparticles. In fact, an aqueous environment provided by the wet milling technique had been proven to be better than the dry milling in producing nanosize powders (Damm & Peukert, 2011).

Therefore, this study reports on a novel usage of Al dross waste, synthesized into micro and nano α-Al_2O_3 as reinforcement in PP based composite by thermal decomposition and wet milling method. In wet milling process, Sodium Lauryl Sulphate (SLS) dispersant is used to disperse and reduce the aggregation effect of nano α-Al_2O_3 filler. In the case of PP composites, the effect of micro and nano α-Al_2O_3 content on mechanical, morphological, wear resistance and thermal properties will be investigated. Other than that, the effectiveness of MAPP compatibilizer addition into micro and nanocomposite concerning its filler-matrix compatibility compared to the unmodified samples will be analyzed. The implementation of this alternative has many gains not just to eliminate hazardous waste, reduce pollution on landfills, conserves the natural resources and energy but also targets on developing product with good mechanical, morphological, wear resistance and thermal properties.

1.3 RESEARCH OBJECTIVES

The main objective of this research is to study the capability of Al_2O_3 from industrial waste as potential micro and nanofiller for PP composites. Therefore, in order to achieve this main objective, some specific objectives are addressed as follows:

i. To synthesize micro and nano α-Al_2O_3 particles from Al dross.
ii. To evaluate and compare the effect of micro and nano α-Al_2O_3 filler content in PP composite with respect to mechanical, morphological and thermal properties.
iii. To investigate the influence of MAPP compatibilizer on the properties of fabricated PP composites filled micro and nano α-Al_2O_3 particles.
iv. To determine the wear resistance of PP/α-Al_2O_3 micro and nanocomposites.

1.4 SCOPE OF RESEARCH

Al dross from industrial waste was one of the main materials involved in developing PP composites reinforced micro and nanoalumina filler. For this reason, different processes were applied to produce both micro and nano α-Al_2O_3 particles. Thermal decomposition method was used in synthesizing α phase Al_2O_3 from Al dross. The process included breaking the compound into simpler compound or element when calcined at 1300 °C for 3 h. In the synthesis of nano α-Al_2O_3 particles, wet milling method was employed. Then, PP/α-Al_2O_3 micro and nanocomposites (without and with MAPP) were fabricated through extrusion process followed by hot pressing.

In this study, 1, 3, 5, and 7 wt.% compositions of micro and nano α-Al_2O_3 filler were used which is based on de Araújo Silva et al. (2013) studies. Their work, which emphasized on the used of microscale filler in fabricating PP composite was done to investigate the effect of commercial Al_2O_3 with different particle sizes (80 and 6 μm), with and without silane surface treatment through increment in mechanical and thermal properties. On the other hand, this research concentrated on synthesizing micro and nano α-Al_2O_3 from Al dross waste as potential filler in PP composites. Previous study showed that Al_2O_3 was viable filler with gains in the mechanical and thermal properties of PP composite. The reduction of Al_2O_3 particles (6 μm) in PP matrix showed the best tensile result with significant increases in toughness while increase of the Al_2O_3 content (low loading) with silane treatment caused improvement in material's thermal stability. Again, previous study did not include wear characterization, internal bonding, crystalline and morphological behavior to observe the dispersion of filler.

Hence, the research scope of this work was to scrutinized the effect of micro and nano α-Al_2O_3 content as feasible reinforcement both in MAPP modified and unmodified PP composites toward better mechanical, morphological, wear and thermal properties. Besides, characterizations on the crystallinity behavior of the Al dross and alumina particles as well as the composites' filler-matrix interaction are necessary in order to synchronize with the mechanical and thermal properties. Finally, the fabricated PP/α-Al_2O_3 micro and nanocomposites (without and with MAPP) and neat PP were compared.

1.5 THESIS ORGANIZATION

This dissertation is divided into five chapters. The introduction and the background of the research done is briefly discussed in chapter one. The basic information regarding the presence of Al_2O_3 (constituent of Al dross waste) generated from the Al production processes, utilization as filler in composites followed by its growing development in nanocomposites is simply explained. Whereas the problems and the current issues that drive the force in diverting the Al dross waste to be recycled as filler based composite is stated in the problem statement and its significance. The targets in analyzing the capability of Al_2O_3 from industrial waste as potential micro and nanofiller for PP composites are specified in the research objectives followed by the research scope. Chapter two reviewed the types of Al dross, its production from mineral resources and recovery process, transition sequences, production of nanosized Al_2O_3 and implementation of Al_2O_3 as potential filler in composites established by the previous researches. The following chapter outlined the experimental setup and methodology including characterization and evaluation procedures throughout the research. Chapter four conferred the outcomes by giving inclusive clarification and comparison of the results obtained from the experimental analysis. The overall research work with additions of possible recommendations on the future work based on the present findings is concluded in the final chapter.