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UNIVERSITI TEKNIKAL MALAYSIA MELAKA

Faculty of Manufacturing Engineering

EFFECTS OF GRAPHENE LOADING ON MECHANICAL PROPERTIES OF 3D PRINTING FILAMENT MADE FROM r-ABS/GNPs NANOCOMPOSITES

Mohd Faizal Bin Rusli

Master of Manufacturing Engineering (Quality System Engineering)

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EFFECTS OF GRAPHENE LOADING ON MECHANICAL PROPERTIES OF 3D PRINTING FILAMENT MADE FROM r-ABS/GNPs NANOCOMPOSITES

MOHD FAIZAL BIN RUSLI

**A dissertation submitted
in fulfillment of the requirements for the degree of Master of Manufacturing Engineering in Quality System Engineering**

Faculty of Manufacturing Engineering

UNIVERSITI TEKNIKAL MALAYSIA MELAKA

2020

DECLARATION

I declare that this dissertation entitled "Effects of Graphene Loading on Mechanical Properties of 3D Printing Filament Made from r-ABS/GNPs Nanocomposites" is the results of my own research except as cited in the references. The dissertation has not been accepted for any degree and is not concurrently submitted in candidature of any other degree.

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APPROVAL

I hereby declare that I have read this dissertation and in my opinion this dissertation is sufficient in terms of scope and quality as a partial fulfilment of Master of Manufacturing Engineering (Quality System Engineering).

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DEDICATION

"Dengan nama Allah yang Maha Pemurah lagi Maha Penyayang"

To my beloved wife Nurul Izza Binti Zulkifli, my lovely childrens, my parents and my family for giving me encouragement, moral support, cooperation and understandings.

Thank you so much and love you all forever.

ABSTRACT

Industry 4.0 offers the opportunity for manufacturers to optimize their operations quickly and efficiently by manipulating recent advanced technologies such as additive manufacturing using printing. An advance in the use of polymeric additive manufacturing has opened up wide possibilities for advanced based product manufacturing. With recent development of petrochemical industry, plastic products have made our society more convenient. However, the disposal of plastic based products has become a serious threat to the environment. This has contributed towards the environmental problem. Nanocomposite technology has been described as frontier of material science due to the employment of few amount of nano filler which significantly enhanced the resulted mechanical, thermal, physical and barrier properties. Therefore, this study is a preliminary effort to investigate the feasibility of creating recycled ABS/GNPs nanocomposites materials and determine their usability to be used as filament materials for 3D printer application. The r-ABS based nanocomposites were set at between of 0.00, 0.25, 0.75, 1.00, and 3.00 wt. % of GNPs nanomaterials addition. The samples properties of r-ABS/GNPs nanocomposites that were prepared by using extrusion and hot press machine were studied. The mechanical, morphological, thermal and physical characterization were determined by using Universal Testing Machine (UTM) machine, Scanning Electron Microscopy (SEM) machine and Differential Scanning Calorimetry (DSC). The addition of GNPs as nanofiller in r-ABS matrix was found not improving the resulted mechanical properties. Through SEM analysis, it was found that GNPs not compatible with r-ABS and create major phase separation between the r-ABS matrix and GNPs nanofiller. Thermal stability of composites indicates that the glass transition temperature was largely unchanged as a result of GNPs addition. Although the r-ABS/GNPs nanocomposites did not shows any increased of mechanical strength that were hypothesized to occur, large increased in material stiffness could still be contributed for 3D printed parts where these stiffness characteristic could served as reinforcing material to traditional ABS or other printed type materials. In overall by conducting this study, it can be said that the utilization of r-ABS as recycled polymer was not compatible for incorporation with GNPs nanofiller. For future improvement, it can be suggested that the GNPs nanofiller used could be surface treated or modified beforehand, prior of the mixing with polymer phases. Hence, there are still hopes for this research work in improving the properties of r-ABS by adding the nanofiller for 3D printing usage.

ABSTRAK

Industri 4,0 memberi peluang bagi pengeluar untuk mengoptimumkan operasi mereka dengan cepat dan cekap dengan memanipulasi teknologi canggih terkini seperti pembuatan bahan termoden menggunakan percetakan. Penggunaan bahan tambahan polimer dalam proses pembuatan telah membuka kebarangkalian yang luas untuk penghasilan produk termaju dan termoden. Dengan perkembangan dalam industri petrokimia, produk plastik telah membuatkan kehidupan masyarakat lebih mudah. Walau bagaimanapun, penjualan produk berasaskan plastik telah menjadi satu ancaman yang serius kepada alam sekitar. Ini telah menyumbang kepada masalah alam sekitar. Teknologi nanokomposit telah digambarkan sebagai penemuan yang amat hebat kerana dengan penambahan sejumlah kecil bahan nano, ia menyebabkan sifat-sifat mekanikal, haba dan fizikal dipertingkatkan dengan ketara. Oleh itu, kajian ini merupakan usaha awal untuk mengkaji kemungkinan untuk mewujudkan gabungan ABS yang dikitar semula dengan bahan nano(GNPs) dan menentukan kebolehgunaannya untuk digunakan sebagai bahan filamen untuk proses pencetakan 3D. Kombinasi r-ABS ditetapkan antara 0.00, 0.25, 0.75, 1.00, dan 3.00 wt. % daripada bahan nano GNPs. Sampel telah dikaji untuk penentuan sifat r-ABS/GNPs nanokomposit yang disediakan oleh mesin penyemperitan dan mesin tekanan panas. Sifat mekanikal, morfologi, haba dan ciri-ciri fizikal ditentukan dengan menggunakan mesin mikroskopi pengimbasan elektron (SEM), Mesin Universal Testing Machine (UTM) dan Calorimetri Pengimbasan Berbeza (DSC). Penambahan GNPs sebagai pengisi nano dalam r-ABS matriks tidak dapat meningkatkan sifat-sifat mekanikal. Melalui analisis SEM, didapati bahawa GNPs tidak boleh diikat dengan r-ABS dan wujud pemisahan fasa utama antara matriks r-ABS dan GNPs. Kestabilan termo haba komposit menunjukkan bahawa suhu peralihan kaca dalam analisa DSC sebahagian besarnya tidak berubah akibat penambahan GNPs. Walaupun r-ABS/GNPs nanokomposit tidak menunjukkan peningkatan dalam kekuatan mekanikal yang dijangka berlaku, peningkatan besar dalam kekakuan bahan masih boleh digunakan untuk pencetakan 3D serta boleh berfungsi sebagai bahan pengukuh untuk bahan asli ABS atau mencetak bahan-bahan lain. Secara keseluruhannya, penggunaan r-ABS sebagai polimer kitar semula tidak begitu sesuai digunakan bersama GNPs. Untuk penambahbaikan masa hadapan, kaedah perawatan permukaan ataupun pengubahsuaian pada GNPs perlu dilakukan sebelum percampuran bersama fasa polimer. Walaubagaimanapun, masih ada ruang untuk melihat peningkatan sifat r-ABS apabila dicampur dengan bahan nano untuk kegunaaan pencetakan 3D.

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LIST OF ABBREVIATIONS AND SYMBOLS

SEM	–	Scanning Electron Microscope
DSC	–	Differential Scanning Calorimeters
ASTM	–	American Standard Testing of Materials
EAB	–	Elongation at Break
g	–	Gram
g/l	–	Gram/Litre
^{o}C	–	Degree Celcius
Mpa	–	Mega Pascal
Gpa	–	Giga Pascal
µm	–	Micrometer
wt %	–	Weight percent

CHAPTER 1

INTRODUCTION

1.1 Introduction

Progress in an additive manufacturing has empowered engineers to decrease time to visualize and to study complex and costly assembly parts that involve in prototyping technique. With fast progression in 3D print technology, various unique and promising innovations could be actualized. Material selection has been very restricted in 3D printer advances, with direct metal laser sintering (DMLS), thermoset plastics, and UV curable materials ruling the market. DMLS based 3D-printing could in any case be restricted costly for some organizations and research foundations, and the normal plastics and UV curable materials utilized in printers today may not have the required quality for demanding applications. Therefore the integration between advances in polymer composites and 3D printing is a way forward that could possibly results in higher quality rapid prototyping for usage in numerous specialized applications. This additionally opens the entryway for tuning 3D print materials to explicit prerequisites for later use in exceptionally concentrated applications. 3D printing was a generally new innovation that has been initiated in the late 1980's by specialists in Japan. Aggressive innovations have seen the 3D printer based product quality, manufacturing speed, and its convenience has improved quickly while cost has been drastically reduced. This permits numerous organizations and research group to acquire 3D printers to be utilized in a wide range of rising applications. Most regular 3D printers that are accessible today have used thermoplastic filament to assemble models through layer by layer (LBL) technique. These printers used similar fundamental parts independent of printer quality. The most widely recognized materials presently utilized in 3D printing are Acrylonitrile Butadiene Styrene (ABS), Nylon and Polylactic Acid (PLA), Polycarbonate. These filament style printers works by utilizing software to break the designs down into separate layer slices, which are then printed by using design specifications set by the user. The control variables parameters are printing temperature, printing speed, layer height, number of perimeters, top and bottom infill pattern, number of solid top and bottom layers, infill percentage for non-solid layers, and infill pattern for interior layers (Washburn et al., 2015).

Many propelled 3D printers could print two materials at the same time by using two separate extruder heads. This takes into consideration the printer to make supports and overhang in the 3D printed part using the second material as a support material. This material can be soaked in a dissolvable solution that just responds with the support material and dissolved. A polymer nanocomposite is a composite material that uses nanoscale particulate support inside the polymer framework stage. The addition of graphene nanoplatelets (GNPs) to polymers and plastic is a strategy that has proven promising to improve the resulted properties of printed composite parts for good mechanical, electrical and thermal performance exhibited by graphene nanofiller (Miguel et al., 2019). Graphene is an incredibly strong material with an extra-ordinary tensile strength of 130000 MPa, that was over 70 times better than steel (Potts et al., 2011). It was composed of single layer of carbon atoms formed inside a honeycomb lattice (Akhaven et al., 2009). Graphene also provides higher conductivity, which measured up to 3500 S/m in stacked graphene oxide (Potts et al., 2011). Graphene based nanocomposites have been appeared to have the best properties of carbon nanotube based polymer composites.

This project is a collaborative project between the National Youth and Skill Institute (IKTBN) Sepang under Ministry of Youth and Sports (KBS), where as previously the scrap from injection molding process from the teaching and learning activities has been disposed without any recycling initiative. By using the ABS waste from injection molding process, it was hoping that the combination between r-ABS/GNPs could replace the utilization of virgin ABS as 3D printing filament for teaching and learning purpose, in order to reduce the overall cost of teaching materials. This recycling project is a way forward to convert waste into something that could benefited for technical teaching and learning activities at IKTBN Sepang.

1.2 Problem statement

There are abundant researches about polymer recycling has been extensively performed. However there are too little or no specific focused researches that specifically studied the combination effects between the r-ABS and GNPs from an injection molding (IM) waste or scrapped materials. Therefore this research has mainly focused to develop the best formulation of r-ABS and GNPs materials, to produce the 3D printing filament for substituting the existing expensive ABS type filament. By combining both r-ABS and GNPs as r-ABS/GNPs nanocomposites, the properties and performance could be significantly enhanced.

In Polymer Laboratory at National Youth and Skill Institute (IKTBN) Sepang and Pagoh, the students' needs to mastery in their mold making and process operation by using the IM process. Thermoplastic materials are the most commonly used materials to form IM based products. The problems had been noticed when there were massive of thermoplastic scraps and rejected parts that have been generated due to high volume utilization of IM process. Usually the wastes are being scrapped then disposed without any further recycling initiative as shown in Figure 1.0. Certainly this scenario could harmed the environment and helps to generate waste at the alarming rate. Hence the noble aims of this study are to recycle the wastes from IM process into parts that can be beneficial for others special usage related with teaching and learning activities.

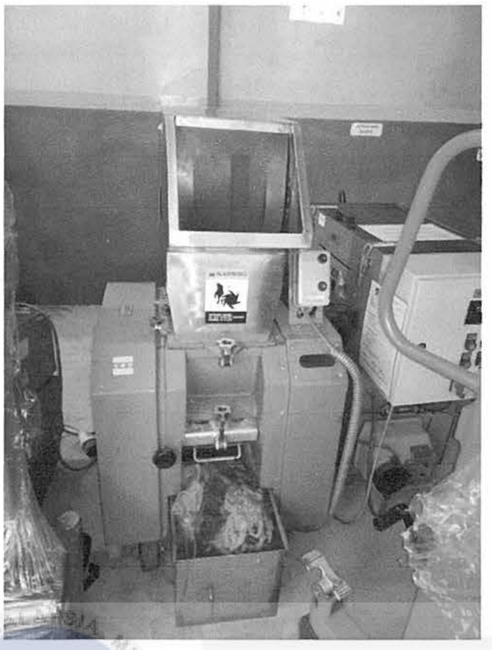

Figure 1.1: Waste materials from injection molding process

The optimum composition of r-ABS/GNPs nanocomposites could be found by combining various ratios of GNPs nanofiller loading and comparing their attributes by performing the mechanical tests and suggesting them as 3D printing filament. In our normal practice for machining activities during the teaching and learning session, virgin or pristine ABS material was used as filament to build their product prototypes. ABS based 3D printing filament as shown in Figure 1.2 is one of the important versatile feedstock used as 3D printing filament (Dul et al., 2016). However ABS was still expensive among other types of commercial polymer. In due to that, the innovative solution to utilize scrap materials of ABS from IM process has been performed in this research to test its feasibility in combining them with GNPs as nanofiller phase for r-ABS/GNPs nanocomposites.

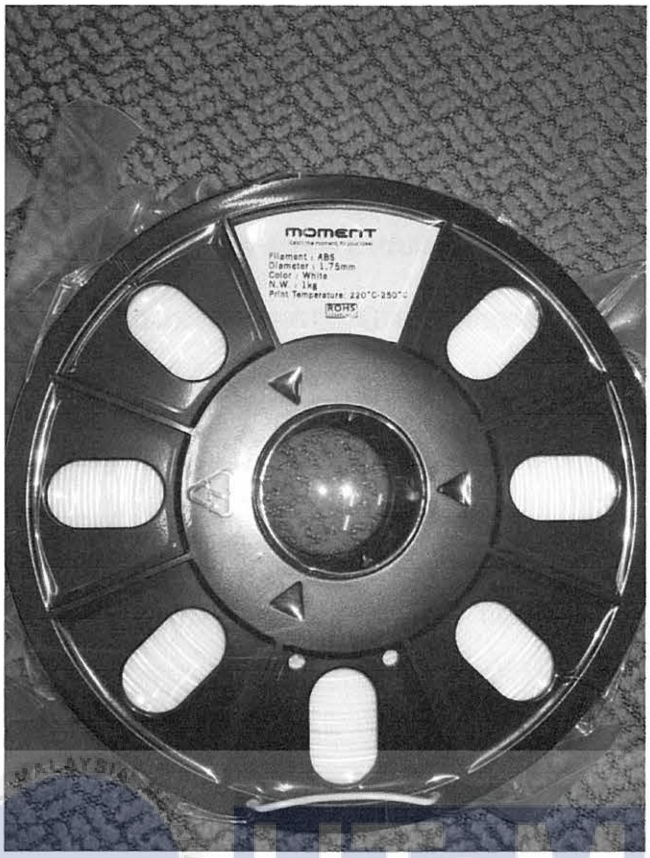

Figure 1.2 : ABS 3D printing filament

By considering all these matters, the motivation of performing this research has been clearly justified and essential for further exploration. This was because there are no previous similar studies have been conducted in the existing literature, specifically about utilization of r-ABS from IM waste materials and its integration with GNPs as nanofiller for r-ABS/GNPs nanocomposites development, specifically as 3D printing filament application, for fused deposition machining (FDM) purpose.

1.3 Objectives

The main objective of this study was to investigate the feasibility of developing new recycled ABS/GNPs nanocomposite advanced material and to determine their usability to be used as alternative filament materials for 3D printer application. Towards achieving this main objective, this study has sets out the specific objective as follows:

i. To evaluate the mechanical, thermal and physical properties of r-ABS/GNPs nanocomposites made from twin-screw extruder process and hot press machine.

ii. To correlate the performances of r-ABS/GNPs nanocomposites with its fracture morphologies observed under the Scanning Electron Microscope (SEM) observation.

iii. To test the performance of selected r-ABS/GNPs nanocomposites, r-ABS and ABS (control sample) as 3D printer filament by using the tensile test prior of printing process.

1.4 Scope of Study

The scope of this project is to formulate the best combination of composition between recycled ABS (noted as r-ABS) and graphene nanoplatelets (GNPs). The r-ABS based nanocomposites are filled with GNPs at between 0.25, 0.75, 1.00, and 3.00 wt. % of GNPs nanomaterials filler addition. The impact, tensile strength, modulus of elasticity, elongation at break and flexural strength of r-ABS/GNPs nanocomposites samples will be tested. The fractured morphology form the fractured samples will be observed through the SEM. Other than that, the thermal properties characterization also will be performed by using Differential Scanning Calorimetry (DSC). The density and water absorption of produced r-ABS/GNPs nanocomposites samples will be measured. The maximum result from the samples will be extruded into filament for 3D printing purposes.

To actualize the objectives of this study, the following research guideline have been derived:

a) Raw Materials: The recycled ABS and GNPs nanomaterials additive based nanocomposites will be set at between 0.25, 0.75, 1.00, and 3.00 wt. % of GNPs nanomaterials addition.

b) Preparation of specimens: The ABS scrap materials from injection molding process will be separately crushed by using the industrial grinder machine. Subsequently, it will be divided into five (5) different formulations. Later the hot press machine was utilized to convert it into specific dimension in accordance to their standard testing dimension. The following are the control samples and formulation recipes that has been utilized in this research study:

   i) r-ABS and 0.00 wt. % of GNPs nanomaterials
   ii) r-ABS and 0.25 wt. % of GNPs nanomaterials
   iii) r-ABS and 0.75 wt. % of GNPs nanomaterials
   iv) r-ABS and 1.00 wt. % of GNPs nanomaterials
   v) r-ABS and 3.00 wt. % of GNPs nanomaterials

c) Preparation of filament: The materials extruded into 3D printing filament. The extruded materials are:-

   i) r-ABS
   ii) r-ABS /0.75 wt. % of GNPs nanocomposites
   i) Testing techniques : Mechanical tensile properties testing is the tensile testing that is used to evaluate the force required to break a plastic sample specimen and the extent to which the specimen elongates or stretches to that breaking point. Tensile tests for plastics provide the information on ultimate tensile strength, Young modulus, flexural strength and elongation at break. The three-point loading that applying the centre loading technique, will be applied for the flexural testing of r-ABS/GNPs nanocomposites. Two span

at the bottom support the study, with span to depth ratio of 16: 1. For each r-ABS/GNPs nanocomposites sample formulation, specimens will be tested at room temperature and the average strength will be taken as a final results.

ii) Macroscopic analysis is performed by visual inspection of the fractured surfaces by using a Scanning Electron Microscopy (SEM). About 5.00 mm section of all the fractured fragments were subjected to SEM observation for microscopic analysis to verify the fracture morphological behavior.

iii) Physical testing for the specimen were tested by using ASTM D792 for density measurement. The density or specific gravity of a solid is a property conveniently measured to indicate the physical changes in a sample, to indicate degree of uniformity among different specimens, or to indicate the average density of a large item. The water absorption testing is used to test the ability and resistance towards water uptake. The conducted tests were confirmed in accordance to the ASTM and ISO standards.

iv) Differential Scanning Calorimetry (DSC) is a thermal analysis method used to calculate changes in material transfer heat flows. The DSC technique will be used to make qualitative statements about whether or not produced r-ABS/GNPs nanocomposites systems are well blended.

v) Performance analysis: The maximum result of nanocomposites then extruded into filament using extrusion machine. The filament then used in 3D printer and the results were compared with the performance of r-ABS filament. If the result from the samples much better, it can be suggested to replace pure ABS filament during teaching and learning for reducing cost strategy.

1.5 Dissertation Overview

This dissertation is separated into five chapters that explain the analytical and experimental research performed.

Chapter 1: Introduction to the study that presents research history, problem statement, objectives, scope and outline of the dissertation.

Chapter 2: Detailed literature review of important theory related to the 3D printing, ABS and GNPs. Previous studies on the issues and current studies of recycled ABS and GNPs nanocomposites are discussed as well.

Chapter 3: This chapter describes the methods used for overall research work, raw materials, specimen's characterization, samples preparation and procedure property testing and analysis.

Chapter 4: In this chapter, the results of the characterization, analysis of different engineering properties on r-ABS and GNPs nanocomposites are presented in detailed. The r-ABS and GNPs nanocomposite performance was determined through seven (7) test analysis.

Chapter 5: In this chapter, the conclusion of the results will be discussed.

1.6 Dissertation Motivation

The expenditure of manufacturing some products with ABS is prohibitively costly despite the fact that the material characteristics of the ABS are well suited for the produce of the products. Acrylonitrile butadiene styrene (ABS) is a standout amongst the most utilized materials in the FDM process. It has high potential to substitute the conventional polyesters and synthetic polymer in wide varieties of application. This bio-polymer is an alternative to compete with traditional plastic. The non-degradable plastics such as packing materials have created a disposal problem to the environment. Over the time, traditional plastic operation cost depends on oil prices that have been increased. Many researchers do modification on ABS to enhance the mechanical and thermal properties.

This project will derive new nanocomposites materials to be applied in 3D printing environment. It believed to greatly contribute to the national agenda in boosting the utilization of graphene by providing products at greener choices.