

THE EFFECT OF DIFFERENT CERAMIC FILLER LOADING ON THE
MECHANICAL PROPERTIES OF POLYMER MATRIX COMPOSITE

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This report is submitted in partial fulfillment of requirements for the award of Bachelor
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To My Husband and Parents

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ABSTRAK

Kajian ini akan menumpukan kepada kesan komposisi bahan pengisi seramik yang berbeza terhadap sifat mekanikal bahan komposit matrik polimer. Kajian ini menggunakan Polypropylene (PP) sebagai bahan polimer. Polypropylene digunakan sebagai bahan komposit polimer kerana kecekapannya dalam meningkatkan kualiti proses dan amat baik digunakan dalam pasaran. Kajian ini dibuat bertujuan untuk mengetahui sifat mekanikal komposit matrik polimer khususnya di bawah Ujian Ketegangan. Di dalam kajian ini, 4 jenis specimen akan digunakan iaitu 100%PP, 80%PP 20%Silica, 70%PP 30%Silica, and 60%PP 40%Silica di mana kesemuanya di dalam bentuk tulang anjing, di mana 3 specimen telah dibuat untuk setiap komposisi bahan, berikutan dengan ASTM D3039 yang dirujuk. Mesin *Extruder* dan *Hot Isostatic Press* telah digunakan untuk menghasilkan specimen. Daripada keputusan yang diperolehi, PP tulen menunjukkan nilai ketegangan yang paling tinggi iaitu 31.54 ± 0.64 MPa. Ini diikuti dengan keputusan 80%PP 20%Silica di mana 20.81 ± 5.49 MPa untuk nilai ketegangan, 70%PP 30%Silica di mana 15.34 ± 3.54 MPa untuk nilai ketegangan dan 60%PP 40%Silica di mana 12.30 ± 2.05 MPa untuk nilai ketegangan. Daripada keputusan yang telah dianalisa, dapat disimpulkan bahawa silica akan mengurangkan nilai ketegangan yang terdapat dalam komposit matrik polimer.

ABSTRACT

This research is focus on the effect of different ceramic filler loading on the mechanical properties of polymer matrix composites. The raw materials that is used are silica and polypropylene. This research is dedicated to the study of mechanical properties of polymer matrix composites, specifically under tensile loading. In this study, 4 types of composites filler loading which consist 100%PP, 80%PP 20%Silica, 70%PP 30%Silica, and 60%PP 40%Silica with the shape of dog-bone were fabricated 3 for each type, in accordance to Tensile Testing as per ASTM D3039. The Mixing via Extrusion and Hot Isostatic Pressing was used to produce the specimens. From the results obtained from the tensile testing, the plain PP has highest tensile strength which is 31.54 ± 0.64 MPa . This is followed by the result obtained for 80%PP 20%Silica with 20.81 ± 5.49 MPa for tensile strength, 70%PP 30%Silica with 15.34 ± 3.54 MPa for tensile strength and 60%PP 40%Silica with 12.30 ± 2.05 MPa for tensile strength. From all result that was analyzed it can be conclude that the effect of silica reduced the tensile strength of polymer matrix composite.

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LIST OF ABBREVIATIONS

SYMBOLS	DESCRIPTION
PP	Polypropylene
SiO ₂	Silica
SEM	Scanning Electron Microscope

CHAPTER I

INTRODUCTION

1.1 Introduction

We know that polymer materials have served mankind for decades. They are used in a wide range of industrial applications including packaging, transportation, construction, pharmacy and the food industry world wide. Elastomers are probably the most versatile and useful groups of polymers ever known to man. These materials are used to manufacture articles such as tires, isolation bearings, roofing sheets, seals, electrical cables and hovercraft skirts.

This research is about the effect of different ceramic filler loading on the mechanical properties of polymer matrix composite. Different ceramic filler loading is important factor that influence the extent of performance enhancement of the composites. In recent years, ceramic filled polymer composites have received increasing research interests of materials scientists because the filler/matrix interface in these composites might constitute a better interface bonding in composite which is increase material properties at low filler concentration. as compared to conventional micro-particulate composites.

1.2 Problem Statement

Previous research, many researchers uses micron size filler material in producing composite. To improve the addition between filler and matrix material, this research will carry out the different ceramic filler loading to choose the best ratio of polymer matrix composites. This is to improve mechanical properties in the composite system. Mechanical properties are very important to define the toughness, ductility and many more in the material that was produced.

1.3 Objective

The objectives of this research are:

- a) To fabricate polymer matrix composite with different ceramic filler loading (40wt%, 30wt%, 20wt %)
- b) To study the effect of different filler loading on the mechanical properties of polymer-matrix composites.

1.4 Scope

These researches are to fabricate ceramic filled polymer matrix composites via extrusion process. This research also to carry out mechanical testing via Tensile Test .

1.5 Planning and execution

This research activity that was carried out throughout the process is shown in the Gantt Chart in Figure 1.1.

Year	2007				2008			
Month-Activities	JUL	OGOS	SEPT	OKT	JAN	FEB	MAC	APR
Literature review	■	■	■					
Experimental activities: a) Design of experiment b) Fabrication of composites c) Sample preparation for testing d) Mechanical Testing : Tensile Test as per ASTM D3039					■	■	■	
Data collection							■	
Result & Analysis							■	
Report Writing for PSM 1 and PSM II		■	■	■		■	■	
Preparation for PSM Seminar 1 and Seminar PSM II				■			■	■
Submission of report & log book				■			■	

Figure 1.1: Gantt chart for execute the PSM

CHAPTER II

LITERATURE REVIEW

2.1 Introduction to filler

Particulate reinforced thermoplastic composites are designed to improve the properties and to lower the overall cost of engineering plastics. Particle filled polymer composites have become attraction because of their wide applications. Incorporating organic mineral fillers into plastics resin improves various physical properties of the materials such as mechanical strength, modulus and heat distortion temperature. In general the mechanical properties of particulate fillers polymer composites depend strongly the size, shape and distribution of filler particles in a matrix polymer and good adhesion at the interface surface Further more, it is worth that in the case of micrometer-sized particulates, high filler content (typically higher than 20% by volume) is generally required to bring the above-stated positive effects of the fillers into play. This would detrimentally affect some important properties of the matrix polymers such as process ability, appearance, density and ageing performance. Therefore a composite with improve performance and low particle concentration is highly desired (Wu et. al, 2001)

2.1.1 Types of fillers

1. Mica

Flakes or platelets represent a special class of reinforcing fillers for thermoplastics and thermoset. Mica is one type of filler. By virtue of its crystal habit, it may easily be cleaved into flakes by ordinary grinding methods. Such flakes when aligned in a thermoplastic matrix, may impart a relatively high degree of reinforcement in terms of strength and stiffness. There are most widely used engineering plastics such as in automobile, electrical, packaging, textiles and consumer applications because of their excellent mechanical properties. However, limitations in mechanical properties, the low distortion temperature, high water absorption of pure nylon has prevented applications to structural components.

2. Silica

Silica is the name given to a group of minerals composed of silicon and oxygen. Silica is found commonly in the crystalline state and rarely in an amorphous state. It is composed of one atom of silicon and two atoms of oxygen resulting in the chemical formula SiO_2 . Silica contributes to today's information technology revolution being used in the plastics of computer mouse and providing the raw material for silicon chips.

2.2 Matrix

2.2.1 Polypropylene (PP)

A polymer prepared catalytically from propylene having an isostatic replacement of a hydrogen atom by a methyl group on alternate carbon atoms in the main chain. Although largely un-reactive chemically the presence of the methyl group makes Polypropylene slightly more susceptible to attack by strong oxidizing agents. A major advantage is Polypropylene's higher temperature resistance; this makes PP particularly suitable for items such as trays, funnels, pails, bottles, carboys and instrument jars that have to be sterilized frequently for use in a clinical environment. Apart from that,

Polypropylene (PP) is a commodity plastic with wide applications that derived from its lightweight, good mechanical performance, economy in fabricating and acceptance to many filler. Polypropylene (PP) is a translucent material with excellent mechanical properties

2.2.2 Polypropylene (PP) resistance

PP has excellent resistance (no attack) to dilute and concentrated Acids, Alcohols, Bases and Mineral Oils. It also has good resistance (minor attack) to Aldehydes, Esters, Aliphatic, Hydrocarbons, Ketones and Vegetable Oils. Than PP has limited resistance (moderate attack and suitable for short term use only) to Aromatic and Halogenated Hydrocarbons and Oxidizing Agents.[1]

2.2.3 Polypropylene (PP) fabrication

PP has great chemical resistance makes Polypropylene a popular choice for plating and chemical tanks, as well as laboratory cabinetry and semi-conductor bench tops. This material machines well and is available in many profiles. Propylene is autoclave able for usage in biohazardous environments. Other uses include fittings and connectors, storage containers, sinks, and hoods. The best joining method is hot air or nitrogen welding. Can be mechanically joined with screws or rivets. Ultrasonic inserts work well.[1]

Table 1.1 : Polypropylene (PP) properties [1]

Maximum Temperature	135 ⁰ C
Minimum Temperature	0 ⁰ C
Melting Point	170 ⁰ C
Tensile Strength	4500psi
UV Resistance	Poor
Hardness	R95
Color	Translucent

2.3 Introduction to particle filled polymer

In recent years, inorganic particles filled polymer composites have received increasing research interests of materials scientists compared to conventional micro-particulate composites. It was happened because of the filler/matrix interface in these composites might constitute a much greater area and hence influence the composites' properties to a much greater extent at rather low filler concentration. Considering the versatility of production facilities and raw materials, dispersive mixing of ready-made particles and polymers is still one of the main manufacturing methods to make composites. [1]

Wu et. al,(2004) said that it is worth noting that the market available silica generally takes the form of agglomerates, which are hard to be broken apart during compounding due to the strong interaction among the silica, the limited shear force provided by the mixing device and the high melt viscosity of polymer melts. They also discussed that modification with coupling agents, which can only react with the exterior silica of the agglomerates as restricted by the larger molecules, is helpless to well disperse the silica. In some cases, as a result, the composites with the addition of nano-silica would exhibit properties worse than microcomposites. To bring the effect of the nano-silica into play, graft polymerization onto the particulates surface was developed by the authors as a pre-treatment technique [1]. The low molecular weights of the grafting monomers allow them to penetrate into the agglomerated silica and react with the particles both inside and outside the agglomerates.

Taking the advantage of this, the following benefits can be gained. The hydrophilic particles surfaces are converted into hydrophobic in favour of improving miscibility between the components. The loosened silica agglomerates are turned into compact nanocomposite structure consisting of the particles, the grafting polymers and the homopolymers generated in the course of graft polymerization; and the interfacial interaction between the filler particles and the surrounding matrix is enhanced through entanglement of the grafting polymers attached to the silica with the matrix molecules. Therefore, stress can be transferred to all the silica when the composites are subjected to applied force, while stiffening, reinforcing and toughening effects are observed at very low silica content. It is believed that a double percolation of effective stress volumes takes the responsibility for the overall enhancement of the composites [1].

Fumed nano-silica was employed as the predominant filler particles and the feasibility of graft pre-treatment of these silica for acquiring mechanical properties improvement has been investigated by using lab-scale and industrial scale compounding machines, respectively. Besides, surface morphologies of the silica before and after grafting were also characterized [1]. For conducting systematic researches, precipitated nano-silica, which is synthesized by a process different from the one for making fumed silica, is used in the present work. On the basis of this filler selection, some other important factors that have not yet been reported in the literature, like the effects of matrix ductility and particulate size on the composites mechanical performance, are studied hereinafter. To maintain the continuity of our work on this subject, Poly Propylene (PP) acts as the matrix polymer once more.

Precipitated silica is manufactured by a wet procedure by treating silicates with mineral acids to obtain fine hydrated silica particles in the course of precipitation. The reaction and drying conditions determine the porosity, surface area, surface chemistry and the degree of impurities in the precipitated silica. In general, precipitated silica is cheap and has a particle size higher than 10 μm [1]. However, they can be made as tiny as nano-scale under specific circumstances [1]. Fumed silica is manufactured by high-temperature hydrolysis of silicon tetrachloride in a flame. Silanol and siloxane groups are created on the silica surface, leading to hydrophilic nature of the particles. The use of fumed silica as fillers in thermoplastics has been well documented not only by our own works stated above but also by other groups [1]. It is thus expected that the applicability of nano-silica in thermoplastics modification would be further broadened if precipitated silica prove to be as useful as fumed ones.

2.4 Effects of matrix ductility and particles species

From Wu et. al,(2004) studied about nano- CaCO_3 /PP composites, they found the importance of the matrix toughness They said that a much more higher ductility as matrix, while the same particles did not result in a similar remarkable increase in impact resistance was observed in the composites with a PP copolymer possessing improvement in a PP homopolymer with lower ductility. So, they suggested that the polymer to be toughened by particles should possess at least certain toughness, which is different from the case when Elastomers acts as toughened

So, to find out whether silica/PP composites follow the same law, Wu et. al,(2004) was used three types of PP: EPS30R, T30S and PP700. They found that, nano-silica filled T30S has acquired significantly high static toughness. So, they said that it is clear that the matrix ductility or the capability of the matrix to plastically deform is key factor influencing the toughening effect of the particles. In the case of suitable matrix ductility like T30S, large scale of matrix polymer is successfully involved in plastic deformation as induced by the particles, leading to high impact toughness of the composites [1].

Wu et. al,(2004) has studied that precipitated silica is able to provide PP with stiffening, reinforcing and toughening effects at rather low filler concentration as fumed silica. They also discussed that ductility of the matrix PP determines the toughening effect of the particles. Only in the case of moderate matrix ductility, the composites can receive the highest extent of toughness increase. Besides, the size and surface area of the particles are also important influencing factors. The smaller particles lead to higher Young's modulus and impact strength of the composites, and reduce the sensitivity of the static toughness to the status of filler distribution.

2.5 The influence of surface modification on the structure and properties of a nanosilica filled thermoplastic elastomer

Aso et. al, (2007) said that to disperse the filler, melt mixing methods are usually preferred due to the versatility of the production facilities and raw materials. As the small filler particles should be adhered to the surface of the polymeric pellets and dosage is usually carried out in a continuous closed stage no nanodust liberation occurs. Further, melt mixing has the added advantage of being an environmentally friendly process due to the lack of solvents and moreover, it is carried out in widely available equipments. On the other hand, the low heat-resistance of the modifiers makes them prone to degradation under these conditions, and the presence of aggregates still occurs. The latter happens because the aggregates may be difficult to be broken up by the limited shear forces in the melt, which only act on the outer nanoparticles. [5]

They also discussed that the mechanical properties of the composites filled with nanoparticles are generally superior to those filled with micron-sized particles of

the same filler. Moreover, other properties such as surface smoothness and barrier properties usually improve. Mineral filler can act as a nucleating agent for crystallization, and can also reduce the mobility of the polymer chains close to it due to interactions with the surface of the fillers. The presence of either micro or nanofillers can also lead to compatibilization in polymer blends either through an increase in the viscosity of the matrix, or through their presence at the interface. Under external load the particle agglomerates may induce stress concentrations that increase with their size, although this effect is less important in the case of nanoparticles. Finally, in the case of nanocomposites, the dramatic increase in the interfacial area per volume content of filler should lead to a composite system, which should combine to some extent the advantages of its two phases. [5]

Aso et. al, (2007) also said that silica is one of the most commonly used nanofillers. It is very versatile, since it has been applied in electrical engineering, electronics and consumer goods. If dispersed to the scale of 10–50 nm, it can improve the heat distortion temperature, the modulus and the barrier properties of the matrix. Many research efforts have been devoted to the surface modification of silica leading to hydrophobic silica. The aim is to reduce the high surface energy and the particle interactions. Additionally, filler treatment will lead to a better dispersion due to the more probable interactions of the polymer with the modification than with the inorganic particle surface. [5]

Aso et. al, (2007) have studied about the effects of the filler content and the filler surface chemistry on the structure and mechanical properties of the nanocomposites. Then, from their experiment, they have concluded that the interactions between matrix and filler are weak. A possible adsorption of polymer molecules on an uncovered filler surface would lead to the development of a polymeric interface layer where the polymer presents lower mobility than the bulk matrix. [5] They also concluded that neither the filler modification nor the filler concentration influenced the crystalline morphology or the crystallization ability. This indicated that the decrease in the mobility of the matrix in the melt state with increasing amounts of filler did not hinder crystallization. However, the crystalline content of PET increased with both SiO_2 and mSiO_2 content, and epoxy-modified SiO_2 nucleated the crystallization of a propylene–ethylene copolymer. [5]

Aso et. al, (2007) have observed that the modulus clearly increased after both SiO₂ and mSiO₂ addition. This is because both moduli are measured at small strain values, and because the contribution of E'' compared to that of E' is very small in these basically elastic materials. They also have been seen that, the modulus values were slightly higher after SiO₂ addition than after mSiO₂ addition. This indicates that (as in the case of storage modulus) the silane modification of the SiO₂ had a slightly negative influence on the modulus. The dependence of composite modulus on the interfacial thickness was proved and the lower strengthening ability of surface modified SiO₂ was attributed to the existence of a flexible interlayer between the bulk polymer and the filler surface introduced by the silane treatment. The presence of an interfacial layer has to be taken into account mainly in nanosize particles. This is because their surface area and the correspondent volume of interphase region become very large, up to reach a volume comparable with that of the dispersed phase. The effects of this silane flexible interlayer are also observed as a decrease in the strengthening effect of the rigid unmodified particles and an increase in the ductility and impact energy compared with untreated silica. [5]

Widely dispersed polymer nanocomposites based in a thermoplastic elastomer (Hytrel) can be obtained by addition of SiO₂ nanoparticles. The main structural characteristics of the amorphous and crystalline phases of Hy did not change in presence of modified or unmodified SiO₂. Upon processing, the dispersion of mSiO₂ was very uniform; the typical clusters had only 2–3 particles and many individual particles were observed. In the nanocomposites with SiO₂ dispersion was also good, although slightly larger particle clusters and sporadic particle agglomeration were produced. The dispersion obtained is attributed to the energetic processing used and to the increase in the organophilic nature of the SiO₂ surface after silane modification. The former promotes better particle dispersion, while the latter hinders ulterior agglomeration. [5]

2.6 Tensile performance improvement of low particles filled poly propylene composites.

Wu et. al, (2002) said that low nanoparticles loaded polymer composites with improved mechanical performance can be prepared by conventional compounding technique in which the nanoparticles are pre-grafted by some polymers using