

**Study on Effect of Nanosilica Sand Addition on Physical and Mechanical
Properties of High Density Polyethylene (HDPE)**

by

Mohd Nor Azlan b Sudin

8177

Mechanical Engineering

Dissertation submitted in partial fulfilment of
the requirements for the
Bachelor of Engineering (Hons)
(Mechanical Engineering)

May 2010

Universiti Teknologi PETRONAS
Bandar Seri Iskandar
31750 Tronoh
Perak Darul Ridzuan

CERTIFICATION OF APPROVAL

**Study on Effect of Nanosilica Sand Addition on Physical and Mechanical
Properties of Thermoplastic**

by

Mohd Nor Azlan bin Sudin

A project dissertation submitted to the
Mechanical Engineering Programme
Universiti Teknologi PETRONAS
in partial fulfilment of the requirement for the
BACHELOR OF ENGINEERING (Hons)
(MECHANICAL ENGINEERING)

Approved by,

(A.P. Dr. Othman Mamat)

UNIVERSITI TEKNOLOGI PETRONAS
TRONOH, PERAK

May 2010

CERTIFICATION OF ORIGINALITY

This is to certify that I am responsible for the work submitted in this project, that the original work is my own except as specified in the references and acknowledgements, and that the original work contained herein have not been undertaken or done by unspecified sources or persons.

MOHD NOR AZLAN BIN SUDIN

ABSTRACT

Nanoparticles reinforced composite can be considered as a new technology and a good candidate material for research. Since this advanced composite are relatively new, studies need to be done to discover the properties changing in the composite. The aim of this project is to investigate the effect of nanosilica sand addition on physical and mechanical properties of thermoplastic. The thermoplastic that will be used in this research is TITANZEX HI 1100 high density polyethylene while the nanosilica is in the range of 0-100nm in size. The composite materials were manufactured using the compression molding process. Testing shows that addition of nanosilica provides improvement in mechanical properties of HDPE at low nanosilica percentage. The

optimum value of tensile strength, Young's Modulus and flexural strength is 11.23Mpa, 24.83MPa and 13.88MPa respectively when the content of SiO₂ is 15 wt%. Testing also shows that the density of the composite improves up to 1.35 g/cm³ at 20 wt% silica content. This research has opened a new horizon for thermoplastic polymer composite to be use as construction material.

ACKNOWLEDGEMENTS

First and foremost, thank god for His grace and mercy throughout the progress of this Final Year Project. I would like to acknowledge my family for always being there to provide financial and moral support. I would also like to take this exceptional opportunity to honorably thank to my Final Year Project supervisor, Associate Professor Dr. Othman Bin Mamat for giving me his personal time every week in the effort to further educate and guide me on the project undertaken. Not to be forgotten also to graduate assistant, Mr. Tahir Ahmed who have been with me through thick and thin in order to finish this project while contributing reading material which serves as reference. They also introduced me the aspect needed to do my FYP and the methods which are to be used to analyze the project so that the needed result can be obtained. Besides, I would also like to express my special thanks to the lecturers and others who have directly and indirectly contributed to the project by giving valuable information on how to conduct and improve the project. Without such assistance from these people, this Final Year Project might not be as successful as it is.

TABLE OF CONTENTS

CERTIFICATION OF APPROVAL	i
CERTIFICATION OF ORIGINALITY	ii
ABSTRACT	iii
ACKNOWLEDGEMENTS	iv
LIST OF TABLES	vii
LIST OF FIGURES	viii
CHAPTER 1:					
INTRODUCTION	1
1.1 Background of Study	1
1.2 Problem Statement	3
1.3 Objectives	3
1.4 Scope of Study	4
CHAPTER 2:					
LITERATURE REVIEW	5
2.1 Theory	7
2.1.1 HDPE Background	7
2.1.2 Thermoplastic Testing Standard	7

	2.1.3	Particulate Reinforce Composite	8
	2.1.4	Stress-Strain Diagram .	9
CHAPTER 3:	METHODOLOGY	10
	3.1	Materials	13
	3.2	Production of Nanosilica	13
	3.3	Composite Preparation	13
	3.4	Composite Characterization	16
CHAPTER 4:	RESULT AND DISCUSSION	18
	4.1	HDPE	18
	4.2	Sample Production	19
	4.2.1	Problems Faced	20
	4.3	Density of HDPE-SiO ₂ composites	21
	4.4	Mechanical Properties of composites	21
	4.4	Surface Fracture Observation	25
	4.5	Morphological Study	26
CHAPTER 5:	CONCLUSION & RECOMMENDATION		27
	5.1	Conclusion	27
	5.2	Recommendation	27
	5.2.1	Physical Properties	22
	5.2.2	Modifications of Nanosilica Sand	28
REFERENCES	29
APPENDICES	31

LIST OF TABLES

Table 2.1	Summary of Journals	5
Table 2.2	Properties of HDPE	7
Table 3.1	Gantt Chart & Project Milestone for FYP I	11
Table 3.2	Gantt Chart & Project Milestone for FYP II	12
Table 4.1	Weight Percentage of HDPE and Nanosilica	19
Table 4.2	Density of HDPE-SiO ₂ composites	21
Table 4.3	Mechanical Properties of HDPE-SiO ₂ composites	21

LIST OF FIGURES

Figure 2.1	A typical stress-strain diagram	9
Figure 3.1	Methodology Overview	10
Figure 3.2	Internal Mixer	14
Figure 3.3	Granulator	14
Figure 3.4	Dog-bone-shaped tensile bar	15
Figure 3.5	Carver Compression Molding Machine	15
Figure 3.6	Dog-bone-shape Mold	16
Figure 3.7	Universal Tensile Testing machine	16
Figure 3.8	Scanning Electron Micro-scope.	17
Figure 4.1	High Density Polyethylene (HDPE)	18
Figure 4.2	Broken Tensile Specimen of HDPE+SiO ₂ composites	20
Figure 4.3	Dog-bone-shape Mold	17
Figure 4.4	Young's Modulus of composite vs silica content.	22

Figure 4.5	Tensile Strength of composite vs silica content.	.	.	.	23
Figure 4.6	Flexural Strength of Composites	.	.	.	24
Figure 4.7	Tensile fractured surface	.	.	.	25
Figure 4.8	Tensile specimen fractured surface	.	.	.	26

CHAPTER 1

INTRODUCTION

This chapter is dedicated to introduction and explanation of the project topic, "Study on effect of Nanosilica Sand addition on Physical and Mechanical Properties of Thermoplastic". A background about this FYP project is given followed by statement of the problem to be addressed and lastly the objectives and scope of the work are pointed out.

1.1 Background Study

This FYP project will focus on the study of polymer properties when added with nano-particles material. Mineral fillers are added to polymers in commercial production primarily for the reason of cost reduction and stiffness improvement ^[1,2]. Polymer is a large molecule (macromolecule) composed of repeating structural units typically connected by covalent chemical bonds. Polymers refer to a large class of natural and synthetic materials with a variety of properties. One of the examples of polymer is polyethylene whose repeating unit is based on ethylene monomer. There are two types of polymer exist which are natural polymer and synthetic polymer.

Polymerization is the process of combining many small molecules known as monomers into a covalently bonded chain. One of polymerization process is laboratory synthesis. Laboratory synthetic methods are generally divided into two categories, step-growth polymerization and chain-growth polymerization ^[3]. The essential difference between the two is that in chain growth polymerization, monomers are added to the chain one at a time only ^[4], whereas in step-growth polymerization chains of monomers may combine with one another directly ^[5].

The microstructure of a polymer (sometimes called configuration) relates to the physical arrangement of monomer residues along the backbone of the chain. These are the elements of polymer structure that require the breaking of a covalent bond in order to change. Structure has a strong influence on the other

properties of a polymer. For example, two samples of natural rubber may exhibit different durability, even though their molecules comprise the same monomers.

One of the groups in polymers family is thermoplastic. Thermoplastic is a polymer that turns to a liquid when heated and freezes to a very glassy state when cooled sufficiently. Most thermoplastics are high-molecular-weight polymers whose chains associate through weak Van Der Waals forces, stronger dipole-dipole interactions and hydrogen bonding (nylon). Thermoplastic polymers differ from thermosetting polymers (Bakelite; vulcanized rubber) as they can, unlike thermosetting polymers, be remelted and remoulded. Many thermoplastic materials are addition polymers ^[7].

For this FYP project, nanoparticles in the shape of nanosilica will be added into the polymer to investigate the properties changes. The transition from microparticles to nanoparticles can lead to a number of changes in physical properties. The two major factors in this are the increase in the ratio of surface area to volume, and the size of the particle moving into the realm where quantum effects predominate.

The increase in the surface-area-to-volume ratio, which is gradual progression as the particle gets smaller, leads to an increasing dominance of the behavior of atoms on the surface of a particle over that of those in the interior of the particle. This affects both the properties of the particle in isolation and its interaction with other materials. The large surface area of nanoparticles also results in a lot of interaction between the intermixed materials in nanocomposite, leading to special properties such as increased strength and increased chemical resistance.

A composite, in the present context, is a multiphase material that is artificially made, as opposed to one that occurs or forms naturally ^[6]. In addition, the constituent phases must be chemically dissimilar and separated by a distinct interface. Thus, most metallic alloys and many ceramics do not fit this definition because their multiple phases are formed as a consequence of natural phenomena.

In designing composite materials, scientists and engineer have ingeniously combined various metals, ceramics and polymer to produce a new generation of extraordinary materials. Most composite have been created to improved combination of mechanical characteristics such as stiffness, toughness, and ambient and high-temperature strength.

1.2 Problem Statement

Polymers have been used in almost every product on earth this day which leads to increases of demand for a stronger and cheaper type of polymer. For this FYP, we need to study the effect on nanosilica sand addition on physical and mechanical properties of polyethylene in order to know whether the effect on nanosilica sand addition on properties such as tensile strength, elasticity and melting point are making the composite better or not. We also need to study the new microstructure of composite produced from those additions.

1.3 Objective

The main objective of this project is to study the effect of nanosilica sand addition on physical and mechanical properties of high density polyethylene (HDPE).

1.4 Scope of Study

In this investigation, nanosilica sand was used as reinforcement in HDPE matrix. The nanosilica sand that was used in this study was in size of 0-100nm. The composites were prepared using four different filler loadings which were 5 wt%, 10 wt%, 15 wt% and 20 wt%. The mechanical testing that was performed includes tensile and flexural testing. The effect of the filler loading on the mechanical properties was studied through tensile and flexural test that were conducted. The morphology of the composites fracture surface was examined in order to study and understand the fibre-matrix adhesion of the composites.

CHAPTER 2

LITERATURE REVIEW

Table 2.1 below shows the summary of findings from journal paper related to this project.

Table 2.1: Summary of the Journals

Journal	Objective	Methodology	Result
[11]	To investigate the effect of particle surface treatment, matrix ductility and particle species on mechanical performance of the silica nanoparticles/polypropylene (PP) composites.	<ol style="list-style-type: none"> 1. Pre-treatment of the nanoparticles through graft polymerization and the related analysis. 2. Two-roll mill and screw extruder was used to create samples 3. Tensile test and was conducted. 	<ol style="list-style-type: none"> a) Both treated and untreated nanosilica are able to stiffen the matrix. b) Precipitated nanosilica provides PP with reinforcing effect at low filler concentration. c) The smaller nanoparticles can lead to higher Young's modulus
[12]	To examine the applicability of compounding technique to prepare low nanoparticle	<ol style="list-style-type: none"> 1. Polypropylene was compounded with nanosilica using twin screw extruder. 2. Injection molding 	<ol style="list-style-type: none"> a) Addition of nanosilica into PP brings both reinforcing and toughening effect at filler content as low as 5

	loaded composite with improved mechanical performance.	machine create the sample needed. 3. Tensile test was conducted on the samples.	vol%. b) Modification of nanosilica help to provide the composite with balanced performance
[13]	To investigate the influence surface modification on the structure and properties of a nanosilica filled thermoplastic elastomer	1. Nanocomposites SiO ₂ and mSiO ₂ nanoparticles were prepared in a co-rotating twin screw extruder-mixer 2. The nanocomposite morphology and the dispersion of the silica were observed by scanning electron microscopy (SEM) 3. Tensile tests were carried out at a crosshead speed of 50 mm/min.	a) The addition of nanofillers into Hytrel brings a simultaneous improvement in modulus, elongation at break and creep resistance. b) Widely dispersed polymer nanocomposites based in a thermoplastic elastomer (Hytrel) can be obtained by addition of SiO ₂ nanoparticles
[10]	To highlights the potential of Carbon nanotubes as nanofillers in polymers and limitation and challenges face dealing with nanoparticles in generals.	1. Dispersion and interfacial adhesion were apply to CNTs 2. Mechanical properties of CNTs composite were investigated for volume content below 1%.	a) CNTs able to improve fracture mechanical performance of polymer composite at low filler content. b) Proper dispersion needed to obtain best possible properties.

2.1 Theory

2.1.1 HDPE Background

HDPE is a polyethylene thermoplastic made from petroleum. In terms of properties, HDPE has little branching, giving it stronger intermolecular forces and tensile strength than low-density polyethylene. It is also harder and more opaque and can withstand somewhat higher temperatures (120 °C/ 248 °F for short periods, 110 °C /230 °F continuously). HDPE, unlike polypropylene, cannot withstand normally-required autoclaving conditions. The lack of branching is ensured by an appropriate choice of catalyst and reaction conditions. HDPE contains the chemical elements carbon and hydrogen. Table 2.2 below shows the properties of HDPE.

Table 2.2: Properties of HDPE

Properties	Value
Density (g/cm ³)	0.96
Surface Hardness	SD68
Tensile Strength (MPa)	32
Flexural Modulus (GPa)	1.25
Strain at Yield (%)	15
Melting Temp. Range (°C)	220 - 310
Max. Operating Temp. (°C)	55

2.1.2 Thermoplastic Testing Standard

Testing of thermoplastics can take various forms such as;

- *Tensile tests* — ISO 527 -1/-2 and ASTM D 638 set out the standardized test methods. These standards are technically equivalent. However, they are not fully comparable because of the difference in testing speeds. The modulus determination requires a high accuracy of ± 1 micrometer for the dilatometer.

- *Flexural tests* — 3-points flexural tests are among the most common and classic methods for semi rigid and rigid plastics.
- *Pendulum impact tests* — impact tests are used to measure the behavior of materials at higher deformation speeds. Pendulum impact testers are used to determine the energy required to break a standardized specimen by measuring the height to which the pendulum hammer rises after impacting the test piece.

2.1.3 Particulate Reinforced Composites

Particle composites consist of particles of one material dispersed in a matrix of a second material. Particles may have any shape or size, but are generally spherical, ellipsoidal, polyhedral, or irregular in shape. They may be added to a liquid matrix that later solidifies; grown in place by a reaction such as age hardening; or they may be pressed together and then inter-diffused via a powder process. The particles may be treated to be made compatible with the matrix, or they may be incorporated without such treatment. Particles are most often used to extend the strength or other properties of inexpensive materials by the addition of other materials.

For this project, particle of nanosilica was mixed with HDPE to create the composite. Nanosilica is silica particle with particle size in the range of 1 – 250 nm^[8]. It has been claimed that the properties of materials increases as the decreases of its particles size. For the example, hardness or density of material will slightly increase as its particles size reduced. It is because, the fine particles known as nano particles will diffuse the gab or pore at the grain boundaries of material.

Introduction nanosilica into a HDPE will change the intermolecular interaction of the matrix. The extremely high surface area is one of the most attractive characteristic of nanosilica because it facilitates creating a great amount of interphase in a composite. As estimated by Reynaud et al^[9], an interphase 1 nm thick represents roughly 0.3% of the total volume of polymer in the case of microparticle filled-composites, whereas it can reach 30% of the total volume in the case of nanocomposites.

2.1.4 Stress-Strain Diagram

A tensile test is used to determine a variety of mechanical characteristics of material. The specimen is mounted in a test machine and gradually loaded in tension in increasing increments. The total elongation over the gauge length is measured at each increment of the load and this is continued until failure of the specimen takes place. The loads (stress) are observed and the changes in length (strain) are recorded and plotted in stress-strain diagram. Figure 2.1 shows a typical stress-strain diagram^[14].

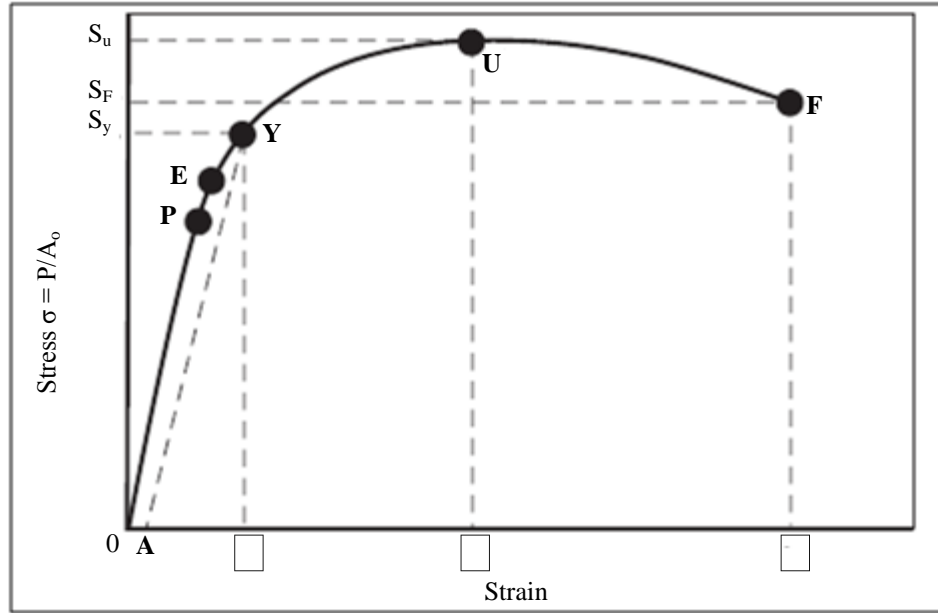


Figure 2.1: A typical stress-strain diagram ^[14]

Refers to Figure 2.1, point P is called the proportional limit where stress is proportional to strain. The slope of the line P is the modulus of elasticity. Point E is the elastic limit. At this point, if a part is loaded to a stress level below point E, no permanent deformation will be sustained. During the tension test, many materials reach a point where the strain begins to increase rapidly without a considerable increase in stress. This point is called the yield point which is represented by point Y. The ultimate or tensile strength is represented by point U. This is the maximum stress that can be withstood by a part. Point F is where the part ruptures. Rupture is the point at which specimen material breaks into two parts ^[14].

CHAPTER 3

METHODOLOGY

In order to achieve the objective of this project, proper planning for the methodology is very important. Figure 3.1 below shows the overview of the methodology while Table 3.1 and Table 3.2 shows the Gantt chart of specific project activities and milestone for FYP I and II.

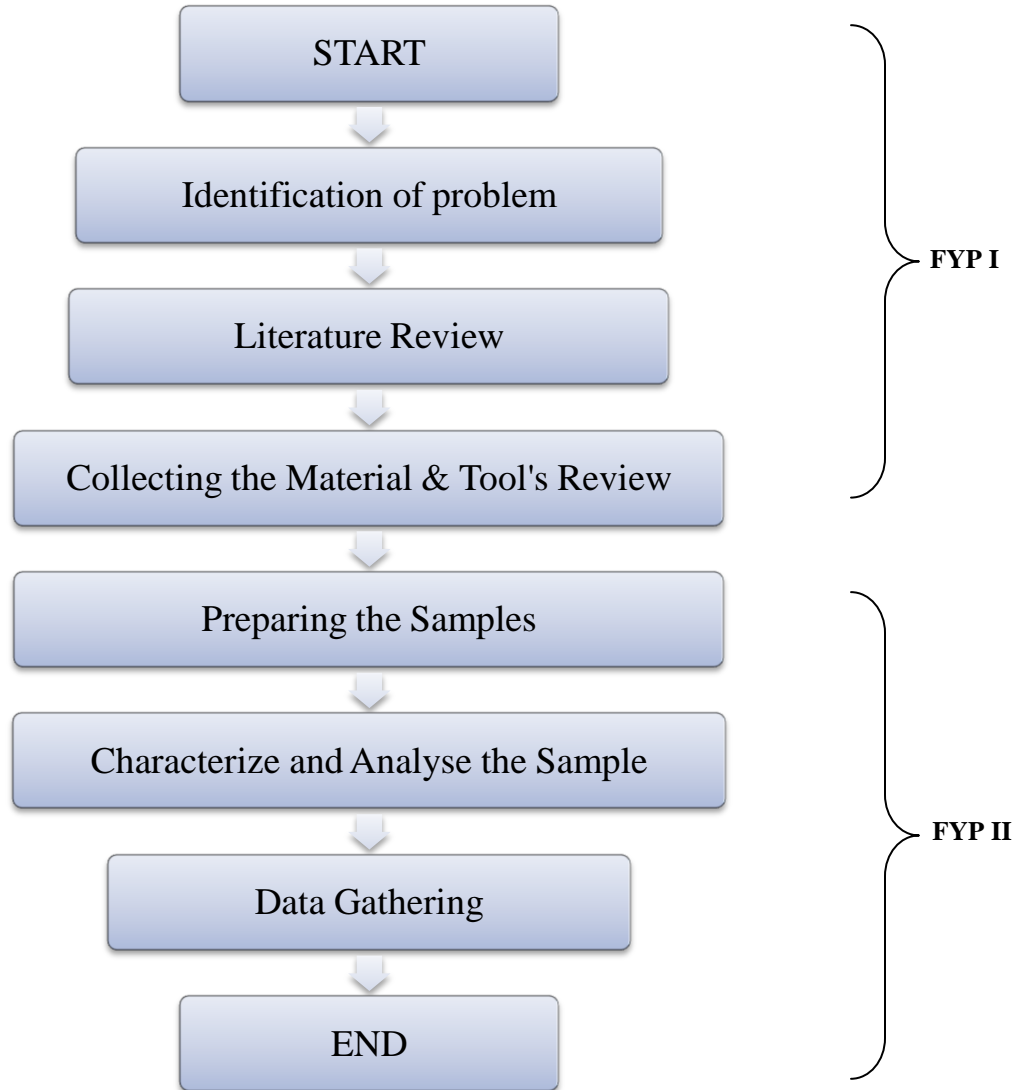


Figure 3.1: Methodology Overview

Specific project activities have been planned as summarize in the following Gantt chart.

Table 3.1: Gantt chart of Specific Project Activities and Milestone for FYP I

ACTIVITIES	1	2	3	4	5	6	7	8	9	Mid Semester Break					10	11	12	13	14
Prelim Research Work	Planned	Planned		Planned															
Research on Particulate Composite				Planned	Milestone														
Research on related journal				Planned	Milestone														
Research on HDPE System				Planned	Milestone	Milestone													
Research Work Phase 1				Planned	Planned	Planned	Planned	Planned	Planned					Planned	Planned	Planned	Planned		
Research on Background of Study				Planned	Planned	Planned	Milestone							Planned					
Research on silica Properties							Planned	Planned	Planned	Planned	Planned	Planned	Planned	Planned	Milestone				
Research on Nanosilica addition effects							Planned	Planned	Planned	Planned	Planned	Planned	Planned	Planned	Planned	Planned	Planned	Planned	Planned
Research on Suitable Methodology							Planned	Planned	Planned	Planned	Planned	Planned	Planned	Planned	Planned	Planned	Planned	Planned	Planned
Report Writing				Planned	Planned	Planned	Planned	Planned	Planned					Planned	Planned	Planned	Planned		
Prelim report				Planned	Milestone														
Progress report							Planned	Milestone											
Interim report														Planned	Planned	Planned	Milestone		
Seminar/Briefing		Planned		Planned											Planned				Planned
Presentation														Planned	Milestone				
Oral Presentation																		Planned	Planned
Preparation																		Planned	Milestone

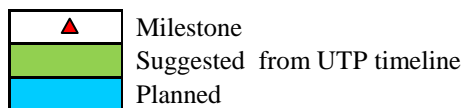





Table 3.2: Gantt chart of Specific Project Activities and Milestone for FYP II

ACTIVITIES	1	2	3	4	5	6	7	8	9	Break	10	11	12	13	14
------------	---	---	---	---	---	---	---	---	---	-------	----	----	----	----	----

Project Work Phase 1													
Confirming Methodology													
Conducting Experiment													
Conducting Physical Testing													
Result Analysis and Discussion													
Project Work Phase 2													
Conducting Experiment													
Mechanical Testing													
Result Analysis													
Conclusion													
Report Writing													
Progress report 1													
Progress report 2													
Disertation Final Report													
Final Report (Hardbound)													
Poster Exhibition													
Seminar/Briefing													
Oral Presentation													
Preparation													

-  Milestone
-  Suggested from UTP timeline
-  Planned

3.1 Materials

The thermoplastic that we are going to use is TITANZEX HI 1100 HDPE. Nanosilica particle with particle size in the range of 78 – 98 nm acted as the mineral filler.

3.2 Production of Nanosilica

The nanosilica sand was produced by material lab of UTP using the dry milling method. The size ranges of the nanosilica are 0-100nm.

3.3 Composite Preparation

Before mixing to HDPE polymer nano silica sand was heated at 200 °C to remove the possible absorbed water for 5 hours. The mixing of nanosilica sand with HDPE was carried by using internal mixer (Thermo Haake PolyLab System) (Figure 3.2) under Roller-Rotors R3000 with 50 rpm and 150 °C temperature for 10 minutes. After mixing the granular shape of the mixed particles were produced using granular machine in Figure 3.3. The resultant particles were moulded into dog-bone-shaped tensile bars (ASTM D638-97) (Figure 3.4) with a compression molding machine (Figure 3.5) at 160 °C. The mold use in this procedure was shown in Figure 3.6.



Figure 3.2: Internal mixer (Thermo Haake PolyLab System)



Figure 3.3: Granulator



Figure 3.4: Dog-bone-shaped tensile bar



Figure 3.5: Carver Compression Molding Machine

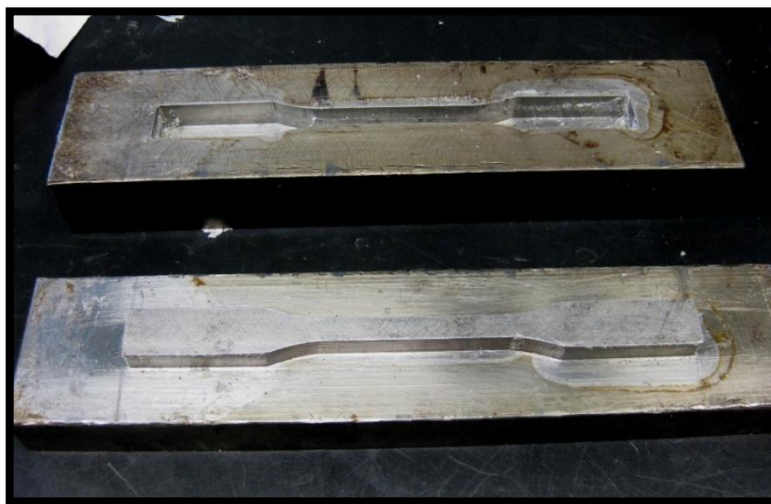


Figure 3.6: Dog-bone-shape Mold

3.4 Composite Characterization and Analysis

Room temperature tensile and flexural testing of the composites was conducted on a Universal Tensile Testing machine (5 KN) LLOYD Instruments, LR 5K (Figure 3.7) at crosshead speeds of 5 mm/min according to ASTM Standard D638-03 Tensile Test for plastic. The fractured surfaces of the samples were observed with a scanning electron micro-scope (SEM) at an accelerating voltage of 20 kV (Figure 3.8).



Figure 3.7: Universal Tensile Testing machine (5 KN) LLOYD Instruments, LR 5K



Figure 3.8: Scanning Electron Micro-scope

CHAPTER 4

RESULT AND DISCUSSION

This chapter will discuss about the material use in this project and also the process of preparing the experiment samples. There are also some elaborations about the process of using compression molding machine and also the problems face during the production of the samples.

4.1 HDPE

Polyethylene (PE) is the most widely used polyolefin in the world by volume. There are three types of PE, namely HDPE, Low Density Polyethylene (LDPE) and Linear Low Density Polyethylene (LLDPE). For this FYP, HDPE was use as the polymer. The type of HDPE is available at Material lab Block 17 is TITANZEX HI 1100 High Density Polyethylene. The material data sheet for this particular HDPE is enclosed together in **Appendix 1**.



Figure 4.1: High Density Polyethylene (HDPE)

4.2 Production of Samples

For each nanosilica weight percentage, three samples were produced. Table 4.1 below show the details of weight distribution in the mixing of nanosilica and HDPE. After a few testing run, we have decided to use 15g of HDPE for sample 1 in order to get the appropriate thickness of the specimen. Figure 4.2 shows broken tensile specimen produce by warm compression process. It is observed from the broken tensile specimen that each of the specimens is broken inside the gauge length which indicates proper mixing of the silica sand in HDPE.

Table 4.1: Weight Percentage of HDPE and Nanosilica

Sample No.	HDPE (wt %)	Nanosilica (wt %)
1	100	0
2	95	5
3	90	10
4	85	15
5	80	20

* Sample calculation for Sample no 3

$$95 \% \times 15 \text{ g} = 14.25 \text{ g} \rightarrow \text{HDPE}$$

$$5 \% \times 15 \text{ g} = 0.75 \text{ g} \rightarrow \text{Nanosilica}$$



Figure 4.2: Broken Tensile Specimen of HDPE+SiO₂ composites

4.2.1 Problems during Production Process

- 1) Thickness of the mold – the mold available is too thick and it takes more than 10 minutes to be heated once we put it on the clamp.
- 2) Condition of mold – the surface of the mold is not smooth which lead to imperfection of the sample surface.
- 3) Final product – unlike injection molding, compression molding process will create a lot of bubbles inside the sample due to not proper ventilation of mold and not proper cooling of mold.

4.3 Density Measurement of HDPE-SiO₂ composites

Table 4.2: Density of HDPE-SiO₂ composites

Composites	Density (g/cm ³)
Pure HDPE	0.96

5%Silica Sand+HDPE	1.13
10%Silica Sand+HDPE	1.25
15%Silica Sand+HDPE	1.30
20%Silica Sand+HDPE	1.35

From Table 4.2, the addition of nanosilica into HDPE has improved the density of the composite. The addition of reinforcement nanosilica particulate results in the increment of percent changes especially in weight. Therefore dramatically change in weight and slightly change in volume can cause the density of the composites increase with higher weight % of reinforcement.

4.4 Mechanical Properties of the HDPE Silica sand nanoparticles composites

Table 4.3: Mechanical Properties of HDPE-SiO₂ composites

Silica Sand+HDPE Composites	Tensile Strength (MPa)	Young's Modulus (MPa)	Flexural Strength (MPa)
Pure HDPE	9.025	18.9	8.70
5% Silica Sand+HDPE	10.42	21.84	10.22
10% Silica Sand+HDPE	11.22	24.32	12.57
15% Silica Sand+HDPE	11.60	24.83	13.88
20% Silica Sand+HDPE	8.93	18.60	10.22

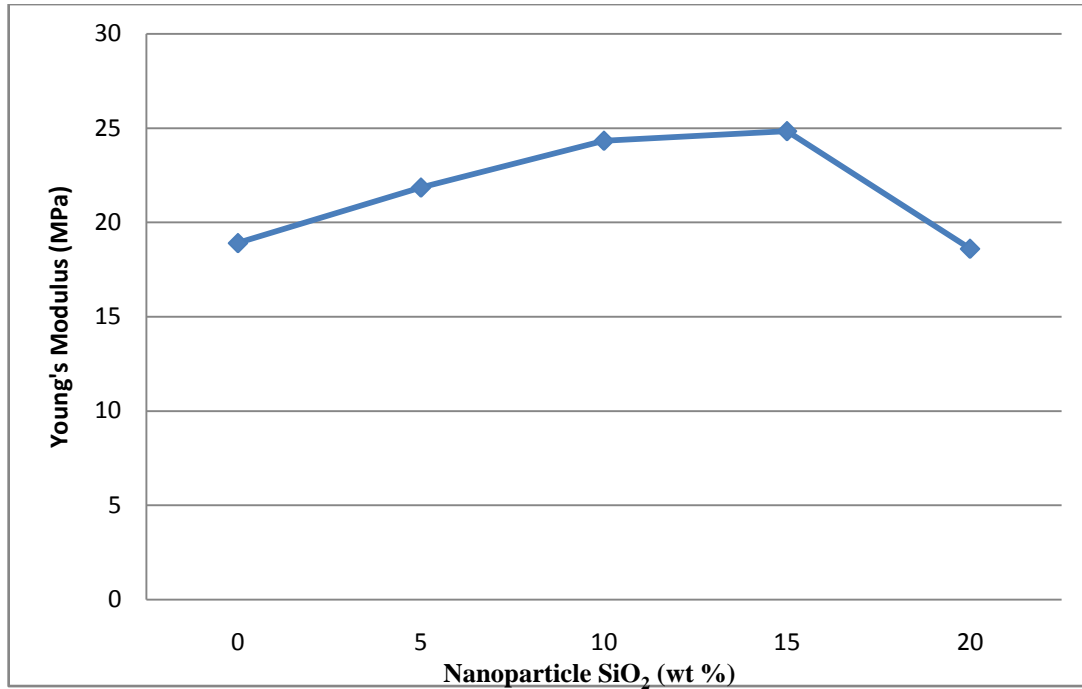


Figure 4.4 : Young's Modulus of nanosilica/HDPE composite with different silica content.

As for the details of the composites mechanical performance, it is seen that the addition of nanosilica sand with HDPE are able to stiffen the composites, as reflected by the proportional relationship between Young's modulus and the filler loading (Figure 4.4). The optimum value of tensile strength is 24.83 MPa when the content of SiO₂ is 15 wt%. This can be interpreted as the absence of soft interphase and the appearance of larger agglomerates of the nanoparticles in the matrix as a result of poor filler/matrix miscibility ^[12]. Under the same filler volume fraction ^[15], the latter effect would provide higher load carrying ability within small strain range. The above results factually reflect the contradiction between the stiffening effect of the rigid particles and the weakening effect of the soft interlayer ^[12].

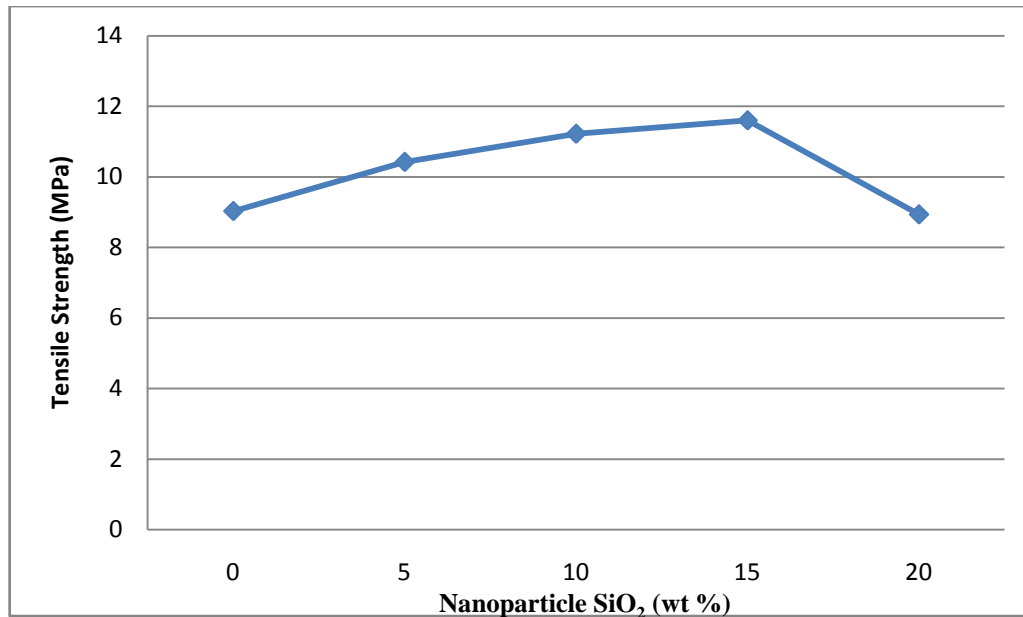


Figure 4.5 : Tensile Strength of nanosilica/HDPE composite with different silica content.

Besides stiffening effect, the addition of nanosilica sand can also provide HDPE with reinforcing effect at rather low nanosilica percentage (Figure 4.5). The optimum value of tensile strength is 11.6 MPa when the content of SiO₂ is 15 wt%. It is well known that the tensile strength of a particulate composite is usually reduced with filler content following a power law in the case of poor filler/matrix bonding ^[16, 17]. That means, the strength of the composite cannot be greater than that of the unfilled version because the filler particles do not bear any fraction of the external load. This contradicts the result shown in Figure 4.5. In fact, when bonding between fillers and matrix is strong enough, the tensile yield strength of a particulate composite can be higher than that of the matrix polymer ^[18, 19]. Although these models were developed based on the cases of microsized particulate composite, they are still valid for the explanation of the composites filled with nanoparticles ^[20].

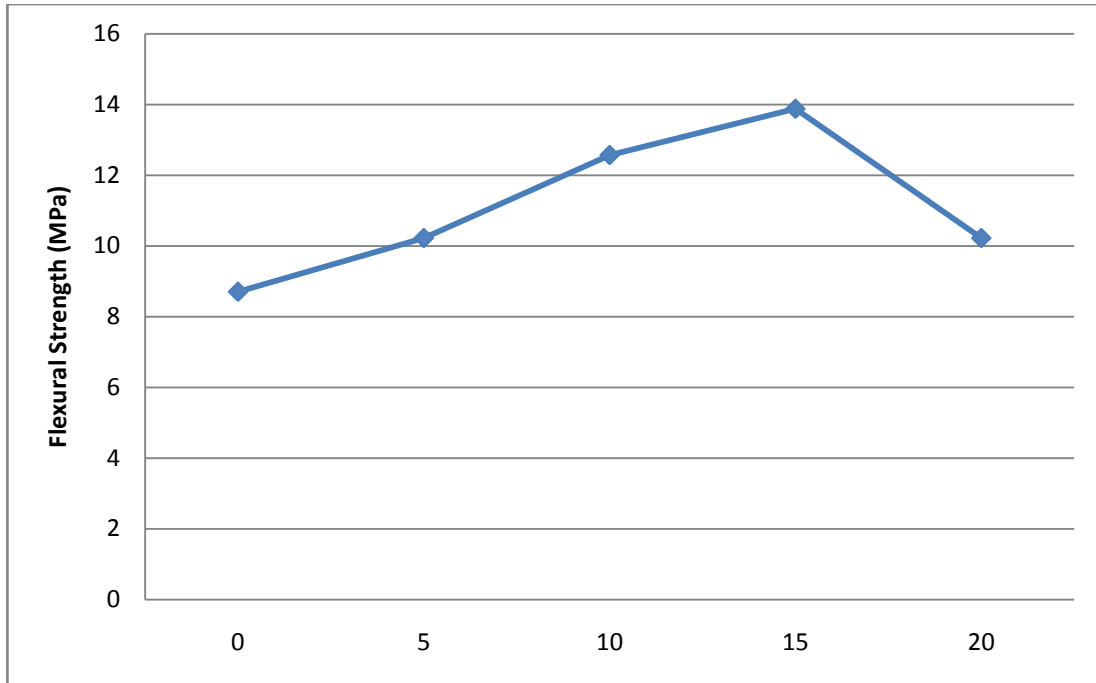


Figure 4.6: Flexural Strength of nanosilica/HDPE composite with different silica content.

Figure 4.6 shows the impact of nanosilica sand addition into HDPE on flexural strength of the composite. The value of flexural strength increase which provides clear evidence of the reinforcing effect that the filler exerts on the HDPE matrix. The optimum value for flexural strength is 13.88 MPa when the content of SiO_2 is 15 wt%.

4.5 Surface Fracture Observation

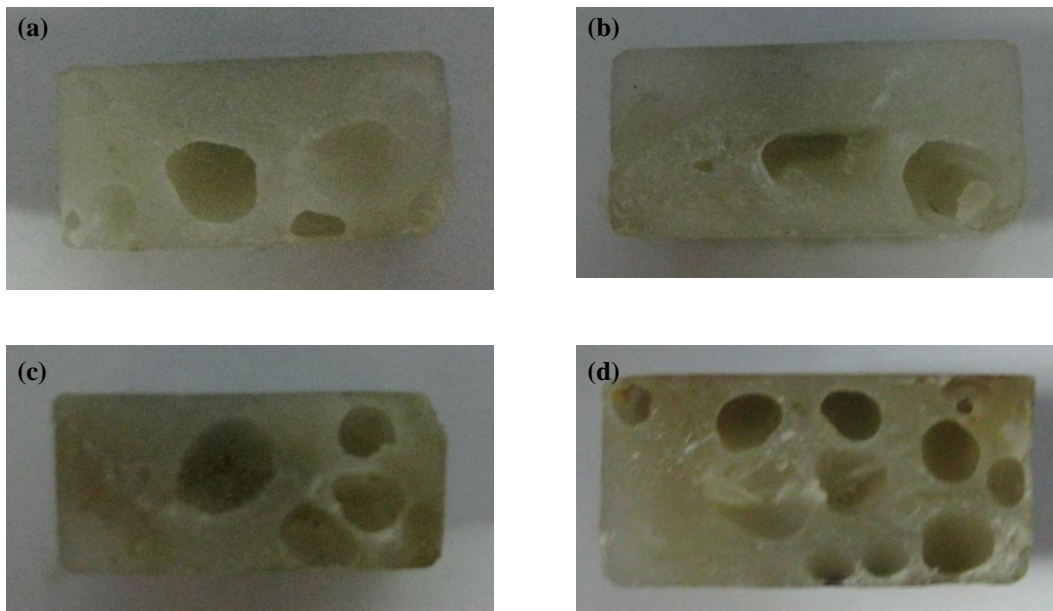


Figure 4.7: Tensile fractured surface of: (a) HDPE + 5 wt% of SiO₂, (b) HDPE + 10 wt% of SiO₂, (c) HDPE + 15 wt% of SiO₂, (d) HDPE + 20 wt% of SiO₂.

After the tensile test, the fracture surface of each sample was cut into small size. Figure 4.6 shows the fracture surface for four different weight percentages of nanosilica content. The appearances of pore spaces were observed in each of the sample fracture. This is an unavoidable circumstances due to production process which using warm compression method. The mold use in this method cannot be vacuum first which led to pore forming inside the sample. However, it was worth to noted that the sized of pore decreased as the nanosilica content increase. The numbers of pore also increase as the nanosilica content increase. This shows that nanosilica helps to fill the pore spaces thus decrease the pore size. All samples broke at pore area due to high stress concentration. Although this sample might not be perfect, it was still valid for this research since the only variable changed for this project was the method to produce the sample.

4.6 SEM Analysis

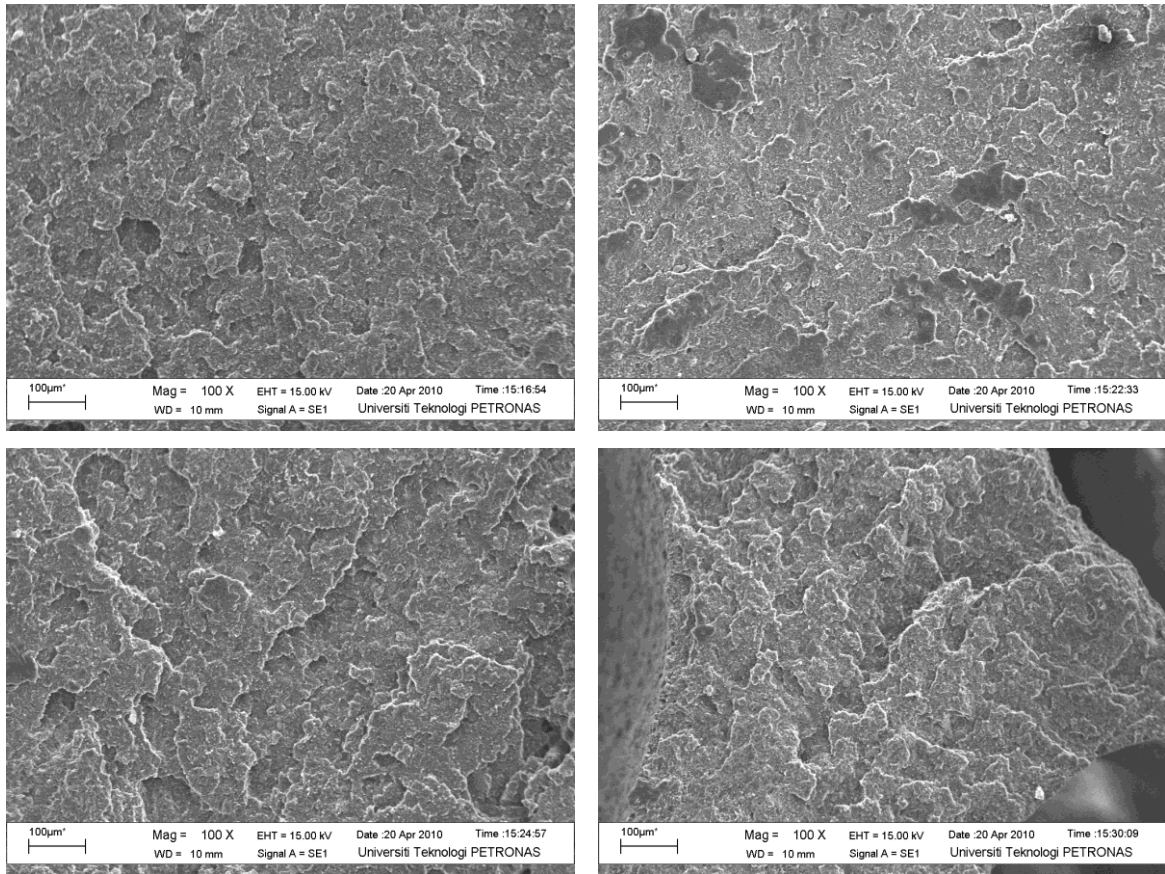


Figure 4.8: Tensile specimen fractured surface of: (a) HDPE + 5% wt SiO₂; (b) HDPE + 10% wt SiO₂; (c) HDPE + 15% wt SiO₂; (d) HDPE + 20% wt SiO₂.

To have clear image of the failure pattern of the composite under tension, SEM fractographs of the specimens with different filler contents are discussed hereinafter. Figure 4.7 shows the tensile fracture surface of HDPE/nanosilica composite generated at crosshead speed of 5 mm/min. All fracture surface has a relatively smooth fractured which indicating weak resistance to crack propagation. In general, an increased content of nanosilica sand would lead to larger agglomerates and hence greater probability of debonding due to the poor adhesion ^[12].

CHAPTER 5

CONCLUSION AND RECOMMENDATION

5.1 Conclusion

The result yield from the tensile testing shows that nanosilica sand addition are able to provide HDPE with reinforcing, stiffening and toughening effects at rather low nanosilica percentage. The optimum value of tensile strength, Young's Modulus and flexural strength is 11.23Mpa, 24.83 MPa and 13.88 MPa respectively when the content of SiO₂ is 15 wt%. Such a simultaneous improvement in modulus and strength is hard to be observed in conventional microsized particulate composites. SEM analysis reveals that All fracture surface has a relatively smooth fractured which indicating weak resistance to crack propagation. The addition of nanosilica sand in HDPE open a new horizon for research of thermoplastic polymers as constructions materials. There might be some room for improvement on the sample production process especially on the mixing techniques to produce a better sample thus lead to more accurate final result.

5.2 Recommendation

5.2.1 Physical Properties

In this research, only one physical property was studied which is the density of the composite. Perhaps in the future other researcher can continue this work by study more on physical properties such as surface hardness, melting temperature and maximum operating temperature.

5.2.2 Modifications of Nanosilica Sand

Research through web and journals shows that modification of nanosilica sand could provides the composite with extra performance in tensile modulus and strength. One of the techniques for modification was by grafting polymerization which helps to provide the composite with balanced performance. In addition, different species of the grafting monomers result in different interfacial interaction and different ultimate properties of the composite.

REFERENCES

- [1] Rothon RN. 1999. "*Mineral fillers in thermoplastics: filler manufacture and characterization.*" Adv Polym Sci
- [2] Pukanszky B. 1999. "*Particulate filled polypropylene composites.*" In: Karger-Kocsis J, editor. "*Polypropylene: an A-Z reference.*" Kluwer Academic
- [3] Sperling L. H., *Introduction to Physical Polymer Science*, Wiley & Sons, 2006, p10
- [4] Sperling L.H., *Introduction to Physical Polymer Science*, Wiley & Sons, 2006, p11
- [5] Sperling L. H., *Introduction to Physical Polymer Science*, Wiley & Sons, 2006, p15

- [6] William D. Callister 2006, *Materials Science And Engineering: An Introduction 7th Edition*, John Wiley & Sons
- [7] Wikipedia, The Free Encyclopedia. 7 September 2009, 03:37
 < <http://en.wikipedia.org/wiki/Thermoplastic> >
- [8] Michael J. Pitkethly, December 2004, “ *Nanomaterials – the driving force,*” QinetiQ Nanomaterials Ltd.
- [9] Reynaud E, Gauthier C, Perez J. 1999. “*Nanophases in polymers.*” Rev Metallurgie
- [10] Bodo Fiedler, Florian H. Gojny, Malte H.G. Wichman, Mathias C.M. Nolte, Karl Schulte, 17 December 2004, “ *Fundamental aspect of nano-reinforced composite,*” Elsevier Ltd.
- [11] Chun Lei Wu, Ming Qiu Zhang, Min Zhi Rong, Klaus friedrich, 9 November 2003, “*Silica nanoparticles filled polypropylene: effect of particle surface treatment, matrix ductility and particle species on mechanical performance of the composite,*” Elsevier Ltd.
- [12] Chun Lei Wu, Ming Qiu Zhang, Min Zhi Rong, Klaus Friedrich, 13 November 2001, “ *Tensile performance improvement of low nanoparticles filled-polypropylene composite,*” Elsevier Ltd.
- [13] O. Aso, J.I. Eguiaza bal, J. Naza bal, 19 July 2006, “The influence of surface modification on the structure and properties of a nanosilica filled thermoplastic elastomer,” Elsevier Ltd.
- [14] John Z. Lu. 2004. “Wood-Fiber/High-Density-Polyethylene Composites: Coupling Agent Performance” *Advance Polymer Technology*, Vol. 4, 143
- [15] Ahmed A, Jones FR. A review of particulate reinforcement theories for polymer composite. *J Mater Sci* 1990;25:4933-42
- [16] Nielsen LE. Simple theory of stress-strain properties of filled polymer. *J Appl Polym Sci* 1996;10:97-103.
- [17] Nicolais L, Narkis M. Stress-strain behavior of styrene-acrylonitrile/glass bead composites in the glassy region. *Polym Eng Sci* 1971;32:1394-9.
- [18] Jancar J, Dianselmo A, Dibenedetto AT. The Yield strength of particulate reinforced thermoplastic composite. *Polym Eng Sci* 1992;32:1394-9.
- [19] Fekete E, Molnar SZ, Kim GM, Michler GH, Pukanszky B. Aggregation, fracture initiation, and strength of PP/CaCO₃ composite. *J Macromol Sci Phys* 1999;B38:885-99.

- [20] Rong MZ, Zhang MQ, Zheng YX, Zeng HM, Friedrich K. Improvement of tensile properties of nano-SiO₂/PP composite in relation to percolation mechanism. *Polymer* 2001;42:3301-4