Study on Effects of Nanosilica Sand Addition on Physical and Mechanical Properties of Polypropylene (PP)

by

Dzul Khairee Bin Yasaa

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

SEPTEMBER 2011

Universiti Teknologi PETRONAS
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CERTIFICATION OF APPROVAL

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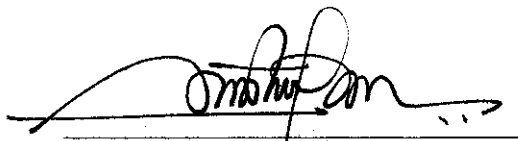
Approved by,

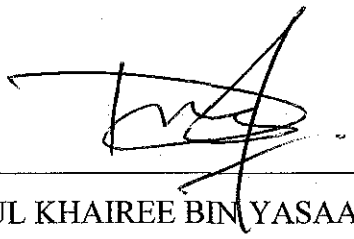


(AP Dr. Othman Bin Mamat)

UNIVERSITI TEKNOLOGI PETRONAS
TRONOH, PERAK
SEPTEMBER 2011

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.



DZUL KHAIREE BIN YASAA

ABSTRACT

Problem that arises for attention is polymer degradation issue. Polymer degradation is a change in the properties - tensile strength, colour, shape - of a polymer or polymer based product under the influence of one or more environmental factors. These changes are usually undesirable, such as changes during use, cracking and depolymerisation of products or, more rarely, desirable, as in biodegradation or deliberately lowering the molecular weight of a polymer for recycling. It was found that the addition of nanoparticles in polypropylene will increase the performance of mechanical properties of the composites. The improvement mechanical and physical properties of PP will improve all plastic products in industry such as bottle, dustbin, pipe, jug and chair. Objective of this project is to study on effects of nanosilica sand addition on physical and mechanical properties of polypropylene (PP). One of the physical properties that need to be studied is density and the mechanical properties are tensile strength, Young's modulus and flexural strength. To examinate the applicability of the study, the silica sand nanoparticles with average particle size of less than 100nm was produced by several stages of ball mill and heating combinations. PP matrix composites with 5, 10, 15 and 20 wt% of silica sand nanoparticles were developed. The silica sand will be grounded to nanoparticles by using ball mill with zirconium ball as grinding media. The mixing of nanosilica sand with PP was carried using internal mixer. After mixing the granular shape of the mixed particles were produced using granular machine. The resultant particles were moulded into dog-bone-shaped tensile bars (ASTM D638-97) with an injection molding machine. Tensile and densities properties of the composites were measured. Based on the result obtained, there was an increasing trend in Young modulus. The tensile strength decreased. An increasing trend of density also was obtained with the increasing content of silica sand nanoparticles in PP. However an improvement in flexural strength was observed. From experimental values, PP matrix with 20% addition of nanosilica could be proposed as the optimum mixture ratio. In this project, although pure PP recorded the highest tensile strength value, common experimental trend in polymer nanocomposites can be observed from 5% to 20% particle addition. The effects of silica sand nanoparticles in PP increased its density and flexural strength but slightly decreased its tensile strength.

ACKNOWLEDGEMENT

First of all, the author would like to express utmost gratitude and appreciation to Allah for His guidance and blessings throughout the entire course of Final Year Project. Alhamdulillah, all praises to Him that the author have been able to complete this project on time.

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Finally many thanks to the author's family and fellow colleagues for their ideas and motivation throughout the completion of this study. They inspired the authors deeply to complete the project. Their inspirations were a tremendous gift to the author. Hopefully that the outcome of this report will bring beneficial output to others as well. Thank you very much everyone.

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1 INTRODUCTION

1.1 Project Background

1.1.1 Polypropylene (PP)

Polypropylene is an economical material that offers a combination of outstanding physical, chemical, mechanical, thermal and electrical properties not found in any other thermoplastic. Compared to low or high density polyethylene, it has a lower impact strength, but superior working temperature and tensile strength.

Polypropylene possesses excellent resistance to organic solvents, degreasing agents and electrolytic attack. It has lower impact strength, but its working temperatures and tensile strength are superior to low or high density polyethylene. It is light in weight, resistant to staining, and has a low moisture absorption rate. This is a tough, heat-resistant, semi-rigid material, ideal for the transfer of hot liquids or gases. It is recommended for vacuum systems and where higher heats and pressures are encountered. It has excellent resistance to acids and alkalies, but poor aromatic, aliphatic and chlorinated solvent resistance.[9]

Table 1 shows the mechanical and physical properties for polypropylene.

Table 1: Mechanical and physical properties for polypropylene [8]

No	Properties	Values	ASTM
1	Elastic Modulus (MPa)	7590 - 10350	D638
2	Flexural Modulus (MPa)	6555 - 6900	D790
3	Tensile Strength (MPa)	58 - 104	D638
4	Compressive Strength (MPa) at yield or break	61 - 68	D695
5	Flexural Strength (MPa) at yield or break	72 - 15	D790
6	Elongation at break (%)	2 - 4	D638
7	Hardness	102 - 111	D638
8	Specific Gravity (g/cm^3)	1.22 - 1.23	D792

1.1.2 Nanosilica

Nanosilica generally is high dispersed and amorphous where they are produced by high-temperature hydrolysis of silicon tetrachloride in an oxyhydrogen gas flame. It is a white, fluffy powder consisting of spherically shaped primary particles, of which ranged between 7 to 40 nm. Primary particles are free from pores. The specific surface areas range between 50 and 380m^2/g. Nanosilica is nearly insoluble in water and acids, but dissolve in strong alkaline media to form silicates. [19]

1.1.3 Silica nanoparticles filled Polypropylene (PP)

Mineral fillers are added to polymers in most commercial production for the reasons of stiffness and toughness improvement. Some researchers showed the improvement of toughness can be increased by adding nanoparticles in polymers. It is worth noting 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.[2]

Nanosilica filled polypropylene composites are hard to be broken apart during compounding due to the strong interaction among the nanoparticles, the limited shear force provided by the mixing device and the high melt viscosity of polymer melts. This will affect some important properties of the matrix polymers such as appearance, density and process performance. Other than that, this also affects the mechanical properties of the polymers such as the improvement of tensile strength and toughness. Therefore, a composite with improved performance and low particle concentration is highly required. With regard to this, the newly developed nano composites would be competitive candidates.

1.1.4 Injection molding

Injection molding is a manufacturing process for producing parts from both thermoplastic and thermosetting plastic materials. Material is fed into a heated barrel, mixed, and forced into a mold cavity where it cools and hardens to the configuration of the mold cavity. After a product is designed, usually by an industrial, mechanical designer or an engineer, molds are made by a mold maker (or toolmaker) from metal, usually either or aluminum, and precision-machined to form the features of the desired part. Injection molding is widely used for manufacturing a variety of parts, from the smallest component to entire body panels of cars.

Process overview

- Utilizes a ram or screw-type plunger to force molten plastic material into a mold cavity
- Produces a solid or open-ended shape which has conformed to the contour of the mold
- Uses thermoplastic or thermoset materials
- Produces a parting line, sprue, and gate marks
- Ejector pin marks are usually present

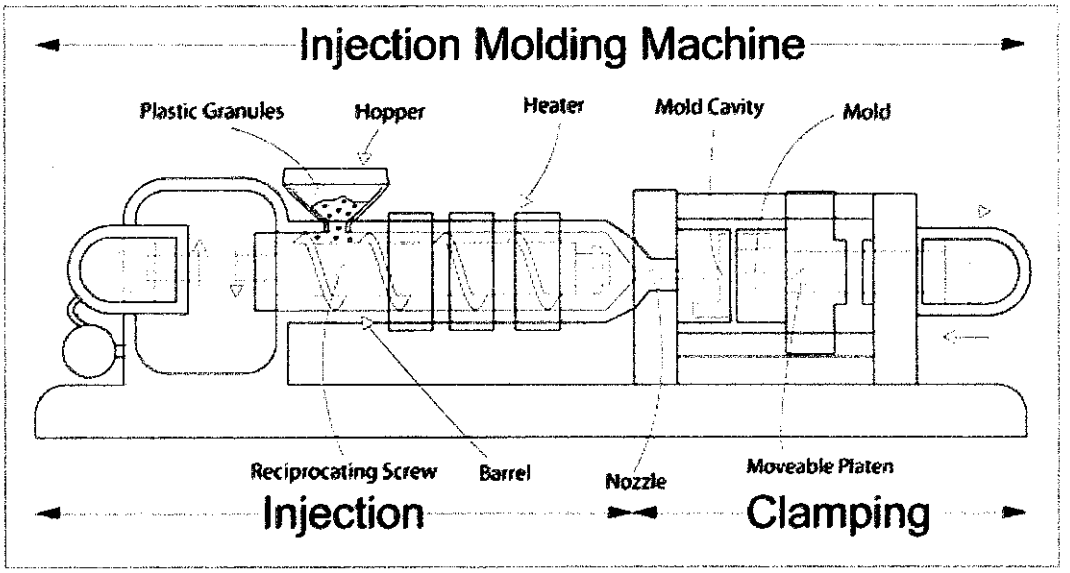

Figure 1: Injection molding drawing [7]

Figure 1 shows the main components of injection molding machine. Injection molding is used to create many things such as wire spools, packaging, bottle caps, automotive dashboards, pocket combs, and most other plastic products available today. Injection molding is the most common method of part manufacturing. It is ideal for producing high volumes of the same object. Some advantages of injection molding are high production rates, repeatable high tolerances, the ability to use a wide range of materials, low labor cost, minimal scrap losses, and little need to finish parts after molding. Some disadvantages of this process are expensive equipment investment, potentially high running costs, and the need to design moldable parts[7]

1.1.5 Scanning Electron Microscope (SEM)

The Scanning Electron Microscope (SEM) is a microscope that uses electrons rather than light to form an image. There are many advantages to using the SEM instead of a light microscope.

The SEM has a large depth of field, which allows a large amount of the sample to be in focus at one time. The SEM also produces images of high resolution, which means that closely spaced features can be examined at a high magnification. Preparation of the samples is relatively easy since most SEMs only require the sample to be conductive. The combination of higher magnification, larger depth of focus, greater resolution, and ease of sample observation makes the SEM one of the most heavily used instruments in research areas today.[5]

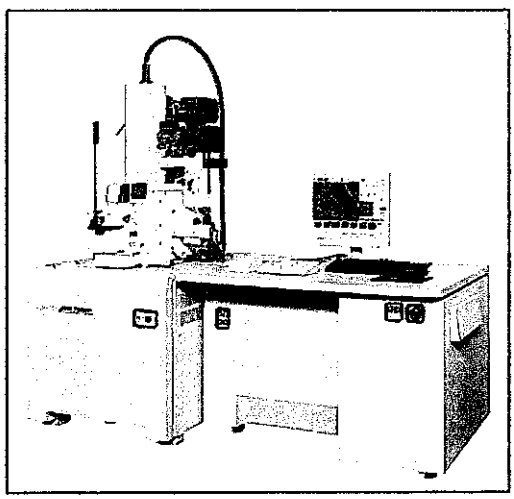

Figure 2: Scanning Electron Microscope [13]

1.1.6 Tensile Test

A tensile test, also known as a tension test, tests a material's strength. It's a mechanical test where a pulling force is applied to a material from both sides until the sample changes its shape or breaks. It's is a common and important test that provides a variety of information about the material being tested, including the elongation, yield point, tensile strength, and ultimate strength of the material. Tensile tests are commonly performed on substances such as metals, plastics, wood, and ceramics.

The tensile strength of a sample of material describes how it reacts when tension is applied to it. By measuring the changes in the material as tension is applied, engineers can determine a variety of things about the material, which is helpful in determining whether the material is a suitable choice for the application they have in mind. In addition to whether a material changes in shape, a tensile test will also show a material's "ultimate strength." The ultimate strength refers to the maximum tensile load that the material can stand. A tension test also uncovers the material's "yield point," which is the amount of tension that causes the sample to break or fail.[14] Figure 3 shows the shape of ductile specimen at various stage of testing.

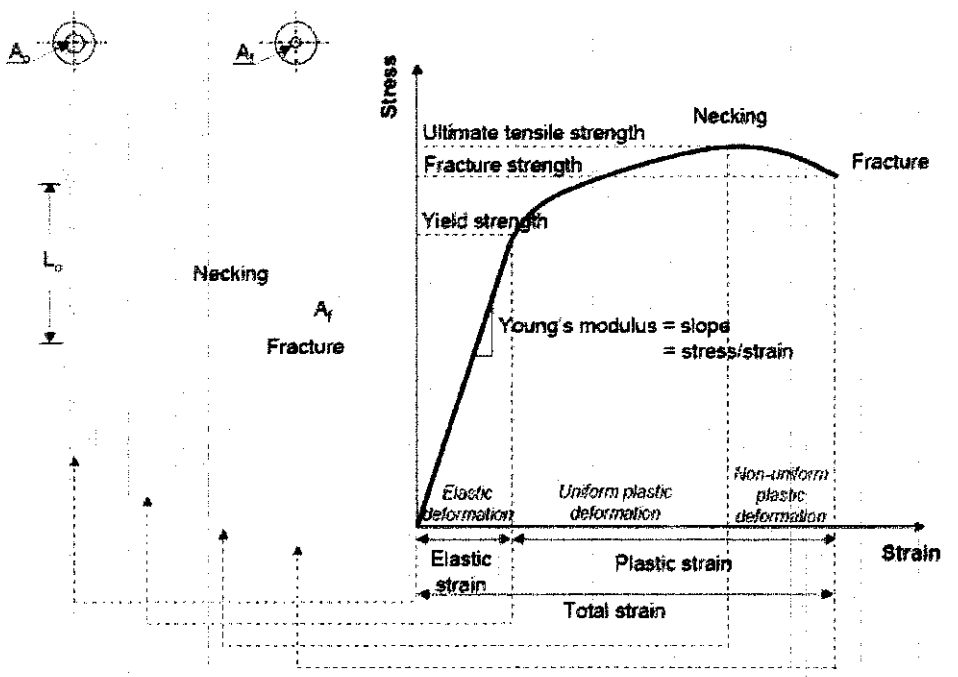

Figure 3: Shape of ductile specimen at various stages of testing[15]

1.1.7 Flexural Test

This mechanical testing method measures the behavior of materials subjected to simple bending loads. Like tensile modulus, flexural modulus (stiffness) is calculated from the slope of the bending load vs. deflection curve.

Flexural testing involves the bending of a material, rather than pushing or pulling, to determine the relationship between bending stress and deflection. Flexural testing is commonly used on brittle materials such as ceramics, stone, masonry and glasses. It can also be used to examine the behavior of materials which are intended to bend during their useful life, such as wire insulation and other elastomeric products. [17]

Figure 4 below shows the test geometry for ASTM D790. Specimen of 1/8" x 1/2" x 5" is placed on two supports and a load is applied at the center. The load at yield is the sample material's flexural strength. These tests also give the procedure to measure a material's flexural modulus (the ratio of stress to strain in flexural deformation). [18]

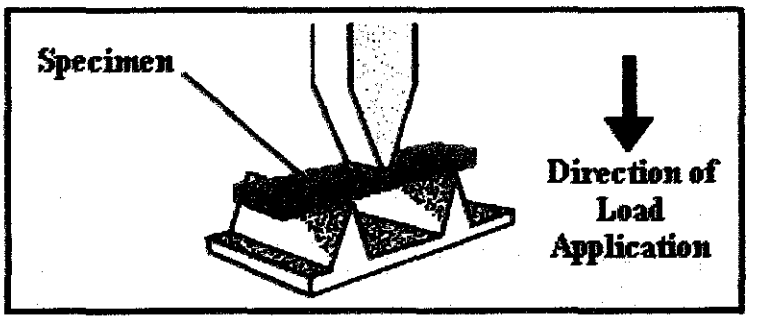

Figure 4: 3 Point Bending Test [18]

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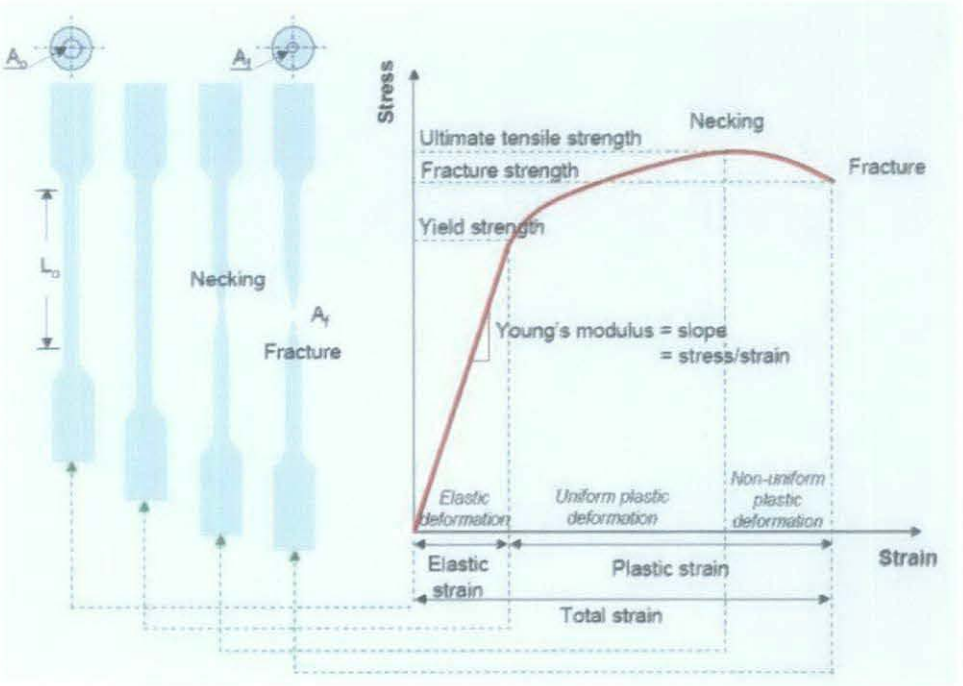

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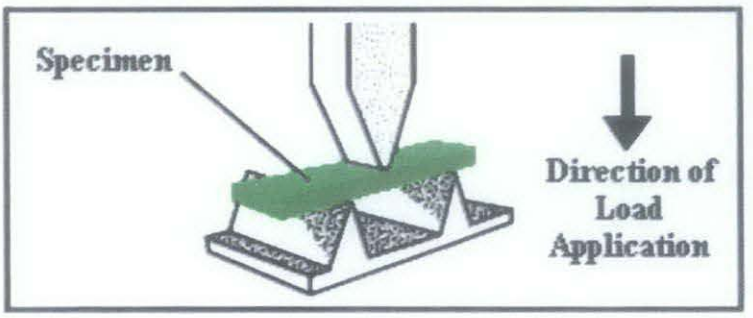


Figure 4: 3 Point Bending Test [18]

1.2 Problem Statement

A variety of window films (also known as sample support) for X-ray Fluorescence (XRF) are available to fill a variety of needs. Decisions on the type of film to use are made based on factors such as cost, x-ray transmission, chemical resistance, reproducibility, and ease of use. Although Mylar (Figure 5) has worse transmission characteristics than most other film, it is often the first choice material to make the XRF film because it is low in cost and has a high tensile strength so it gives very reproducible results. Polypropylene is the next most popular film used in XRF. It is the next least expensive film after Mylar and is resistant to most acids. It also has better x-ray transmission than Mylar. It usually comes in a 6.3 μm thickness and is useful for analyzing elements from Al up in the periodic table. Polypropylene's weakness or problem is that it has poor tensile strength and stretches readily, meaning it has poorer reproducibility than Mylar.[20]

Another problem that arises for attention is polymer degradation issue. Polymer degradation is a change in the properties - tensile strength, colour, shape - of a polymer or polymer based product under the influence of one or more environmental factors such as heat, light or chemicals such as acids, alkalis and some salts. These changes are usually undesirable, such as changes during use, cracking and depolymerisation of products or, more rarely, desirable, as in biodegradation or deliberately lowering the molecular weight of a polymer for recycling. The changes in properties are often termed "ageing". Therefore, polymer degradation is one of major concern in polymer industries which needs continuous attention and improvement.[21]

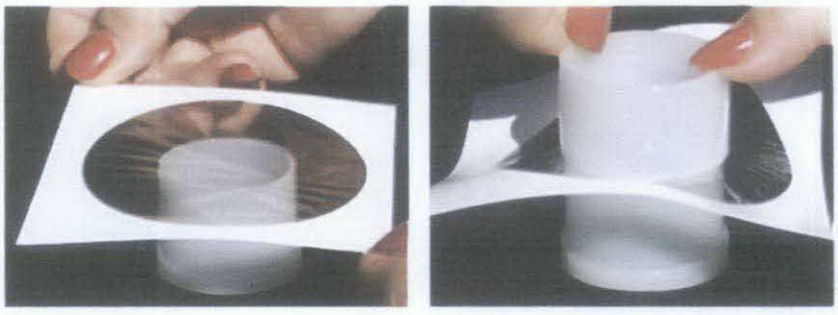


Figure 5: Mylar used as XRF window film [22]

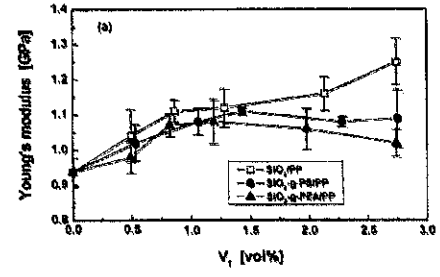
1.3 Significance of the Project

This project will demonstrate how tensile strength of PP can be improved so that it can have high quality and producibility as same as Mylar. With that result, the producibilty of window film for XRF can be improved. It was found beforehand that the tensile strength and other mechanical properties of PP can be improved by adding nanoparticles materials. A low nanoparticles loaded polymer composites with improved mechanical performance can be prepared by conventional compounding technique in which the nanoparticles are pregrafted by some polymers using irradiation. To examine the applicability of the approach, a tougher PP was compounded with nanosilica by industrial-scale twin screw extruder and injection molding machine in the present work. The results of tensile tests indicated that the nanoparticles can simultaneously provide PP with stiffening, strengthening and toughening effects at rather low filler content (typically 0.5% by volume). The presence of grafting polymers on the nanoparticles improves the form ability of the composites.[2] Thus, the improvement mechanical and physical properties of PP will improve all plastic products in industry such as bottle, dustbin, pipe, jug and chair.

By adding nanoparticles in PP will enhance the degree of intermolecular bonding between adjacent chains. This restricts the number of solute molecules that can fit into these chain locations. So polymer degradation can be avoided. In this study silica sand nanoparticles will be used as one of the raw materials to fully utilize the abandon resource. Other than that, the potential of silica sand nanoparticles usage when combined with PP will also be analyzed to provide useful data for engineering design purpose in the future. Figure 6 shows some of products in industry that made from plastic.

Figure 6: Products made from plastic [10][11][12]

Table 2: Summary of literature review

No	Author(s) + Title	Findings	Remarks
1	**Tensile performance improvement of low nanoparticles filled-polypropylene composites** (Chun Lei Wu, Ming Qiu Zhang: Zhongshan University,China)	**Materials** • Isotactic polypropylene(PP) • Silica(particle size 15nm) • Styrene and ethyl acrylate (as grafting monomers) **Methods** • Irradiation grafting on nano-SiO_2 • Two-roll mill(for compounding) • Injection molding • Tensile test by Hounsfield-5KN universal testing machine • Scanning Electron Microscopy/SEM(observation) **Results** • Nanoparticles can simultaneously provide PP with stiffening, strengthening and toughening effects at a rather low filler content (typically 0.5% by volume) • Improves the tailorability of the composites • A high interfacial stiffness corresponds to a high composite modulus • The increased amount of grafting polymers further increases the modulus mismatching of the filler and the matrix, and reduces the stiffening effect of SiO_2, leading to the drop of Young's moduli of the composites at high filler regime • Tensile strength reduced because of poor filler/matrix bonding between the grafting polymers and the matrix	There are some improvement of stiffness, strength and toughness when adding low silica nanoparticles (typically 0.5% by volume) into PP. This journal relates with the study which is to study on the mechanical properties of the composites. One of the methods which is injection molding also relates with the study. 

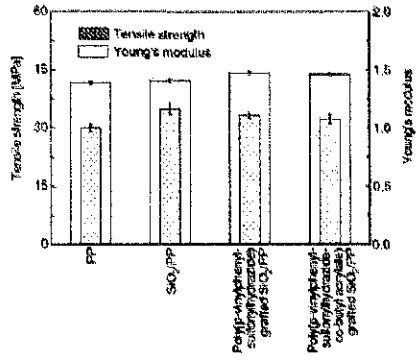
<table>
<tr>
<td>2</td>
<td>Polypropylene-Nanosilica-Filled Composites: Effects of Epoxy-Resin-Grafted Nanosilica on the Structural, Thermal, and Dynamic Mechanical Properties
(Chaganti Srinivasa Reddy, Chappal Kumar Das: Indian Institute of Technology, India)</td>
<td>Materials
• Thermoplastic polypropylene (PP)
• Nano-SiO_2 (particle size 50-60nm)
• Epoxy resin liquid diglycidyl eter of bisphenol A
• Tin(II) chloride and metyl isobutyl

Methods
• Epoxy-resin grafting on nano-SiO_2
• Corotating, twin screw, sigma internal mixer (for compounding)
• Injection molding
• Tensile test by Hounsfield HS 10KS universal testing machine
• Wide-angle X-ray diffraction/WAXD, Transmission Electron Microscopy/TEM, Dynamic Mechanical Analysis/DMA and Scanning Electron Microscopy/SEM (observation)

Results
• Better dispersion of epoxy-resin-grafted nanosilica (ENS) in PP matrix (PP-ENS) compared to polypropylene-nanosilica (PP-NS)
• Higher thermal stability for PP-ENS than PP-NS
• Increase of elastic modulus and glass transition temperature for PP-ENS with respect to PP-NS</td>
<td>This journal compared the PP-ENS with PP-NS composites (with 2.5 wt% of both NS and ENS). The authors found that PP-ENS composites have better structural, thermal and mechanical properties compared to PP-NS composites. Internal mixer will be used to combine PP with silica sand nanoparticles. The mechanical properties on this journal relate with the study.

TABLE V
Results of the Tensile Test
<table><tr><th>Sample configuration</th><th>Tensile strength (MPa)</th><th>Elongation at break (%)</th><th>Elastic modulus (MPa)</th><th>Toughness (MJ/m^3)</th></tr><tr><td>PP</td><td>25.97</td><td>10</td><td>107.81</td><td>2.64</td></tr><tr><td>PP-NS</td><td>14.96</td><td>5</td><td>207.83</td><td>0.40</td></tr><tr><td>PP-ENS</td><td>27.28</td><td>11</td><td>198.62</td><td>2.95</td></tr></table></td>
</tr>
<tr>
<td>3</td>
<td>Interfacial effects in nano-silica/polypropylene composites fabricated by in-situ chemical blowing
(L. F. Cai, Y. L. Mai: Guangdong Public Laboratory of Chemical Engineering, China)</td>
<td>Materials
• Isotactic polypropylene (PP) homopolymer
• Nano-SiO_2 (average particle size 12nm)
• Silane coupling agent (γ-methacryloxypropyltrimethoxy silane)
• 4-styrenesulfonic acid sodium salt hydrate
• Butyl acrylate (BA) monomer

Methods
• Grafting of poly(p-vinylphenyl-sulfonylhydrazide-co-butyl acrylate)
• Ungrafted or grafted nanosilica compounded with PP by using mixer</td>
<td>The authors used mixer to combined nanosilica (1.00/2.63 weight ratio or 0.38%) with PP. This method quite similar with the study on methodology part. Internal mixer will be used to combine PP with silica sand nanoparticles. (refer Methodology)</td>
</tr>
</table>

		• Fourier-transform infrared /FTIR, Transmission Electron Microscopy /TEM(observation) • Injection molding • Tensile test by Hounsfield HS 10KS universal testing machine • Impact test by Atlas advanced pendulum impact device **Results** • Enhancement of impact strength of the nano composites • Tensile strength increased due to interfacial effect between the grafted polymer and PP matrix • Young's modulus nearly same for all composites because the stiffness is nearly the same	
4	**The Structure and Physical Properties of Polypropylene and Thermoplastic Olefin Nanocomposites Containing Nanosilica** (Yiqun Liu, Marianna Kontopoulou: Department of Chemical Engineering, Queen's University, Kingston, Canada)	**Materials** • Polypropylene (PP) homopolymer • Ethylene-octane copolymer polyolefin elastomer • Maleated PP • Nano-SiO_2 (specific surface of $150m^2/g$) **Methods** • Composites with 0-7 wt% nanosilica were prepared by using mixing chamber • Compression molding • Tensile and flexural test by using Instron 3369 universal tester • Impact test by using Instron BLI impact tester • TEM, SEM and X-ray Diffraction/XRD (observation) **Results** • Tensile moduli, impact and flexural properties of TPO/nanosilica composites showed improvements at low loadings of nanosilica, indicating a good balance of stiffness and toughness	The authors found that both nanosilica filled samples had higher flexural modulus and flexural stress than the unfilled TPO. This mechanical properties relate with the study Flexural properties of TPO/nanosilica composites Sample | Flexural modulus (MPa) | Flexural stress[a] (MPa) TPO | 797 ± 24 | 25.5 ± 0.5 TPO–5 wt% SiO_2 | 920 ± 39 | 28.4 ± 1.3 TPO–5 wt% mSiO_2 | 942 ± 64 | 28.7 ± 1.6 TPO composition (PP/PP-g-MAn)/POE 80/20. [a] Measured at 5% strain.

3 METHODOLOGY

3.1 Materials

Silica sand nanoparticles (SiO_2) filled with polypropylene (PP) composite samples is required for the research. Raw materials needed to produce the tensile and flexural specimen are polypropylene (PP) as the polymer matrix and particles of Silica sand nanoparticles as the reinforcement.

3.2 Tools & Equipments

The following are tools and equipments that will be used in the experiment:

i) ZetaSizer, Nano ZS (ZEN 3600) (Malvern) nano particle analyzer
ii) Field Emission Scanning Electron Microscope (FESEM)
iii) X-Ray fluorescene (XRF)
iv) Internal mixer (Thermo Haake PolyLab System)
v) Granular machine
vi) Injection molding machine
vii) Universal Tensile Testing machine (5 KN) LLOYD Instruments
viii) Mettler Toledo AX205 density measurement instrument

3.3 Sample Preparation & Experiment

3.3.1 Required Samples

There will be a minimum of 5 samples required, namely:

1. Pure PP
2. PP + 5% of silica sand nanoparticles
3. PP + 10% of silica sand nanoparticles
4. PP + 15 % of silica sand nanoparticles
5. PP + 20% of silica sand nanoparticles

3.3.2 Samples Dimension Requirements

Each sample above will be cut to obtain dimension which is 25mm x 3mm x 216mm. This dimension corresponds to the required sample size for testing under ASTM, D 5083 – 96. [23]

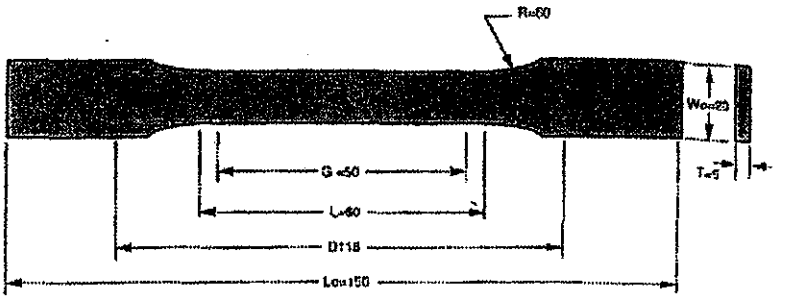


Figure 7: Specimen according to ASTM (D 5083 - 96) [23]

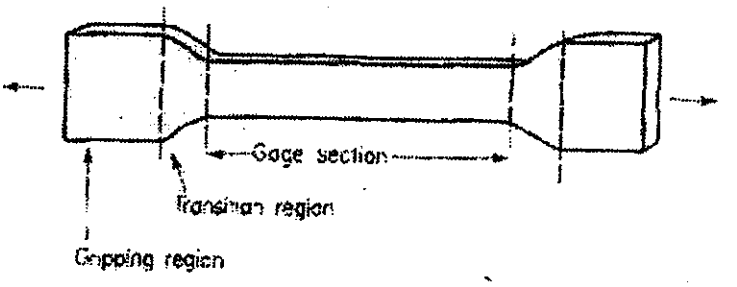


Figure 8: Specimen according to standards and then used for testing [23]

3.3.3 Preparation of Silica Sand Nanoparticles

The silica sand will be grounded to nanoparticles by using ball mill machine with zirconia (beads) ball as grinding media for 6 hours (Figure 9). Figure 10 shows the silica sand that have been grounded into nanoparticles. The production of silica sand nanoparticles (with average size less than 100nm) was confirmed by using the ZetaSizer, Nano ZS (ZEN 3600) (Malvern) nano particle analyser. The silica sand nanoparticles were produced (lab-scale) in UTP Mechanical Engineering Department. The characteristics of silica sand nanoparticles were shown in Figure 11 and Table 3.

Figure 9: Ball mill machine

Figure 10: Silica sand nanoparticles

Based on the result shown in Figure 11, the average size for the silica particles was found 78.82 nm which was qualified to be considered as SiO_2 nanoparticles. Photon correlation spectroscopy (PCS) or dynamic light scattering (DLS), is a technique used to determine particle size by examining the diffusion rates (i.e. Brownian motion) of suspended particles. These results are then verified microscopically through FESEM. The FESEM image in Figure 12 shows the morphology, size and distribution of the SiO_2 sand naoparticles. It can be seen that some of SiO_2 particulates have fused together resulted from squeezing action of the ball-milling process. The after ball-milled SiO_2 nanoparticles formed wide range of shape from irregular or rod-like aggregates and agglomerates to rounded nanoparticles.

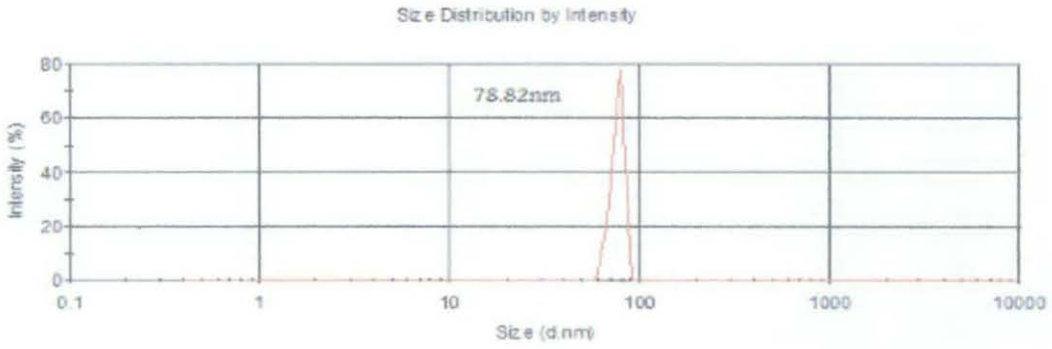


Figure 11: The average particle size produced by size distribution of intensity

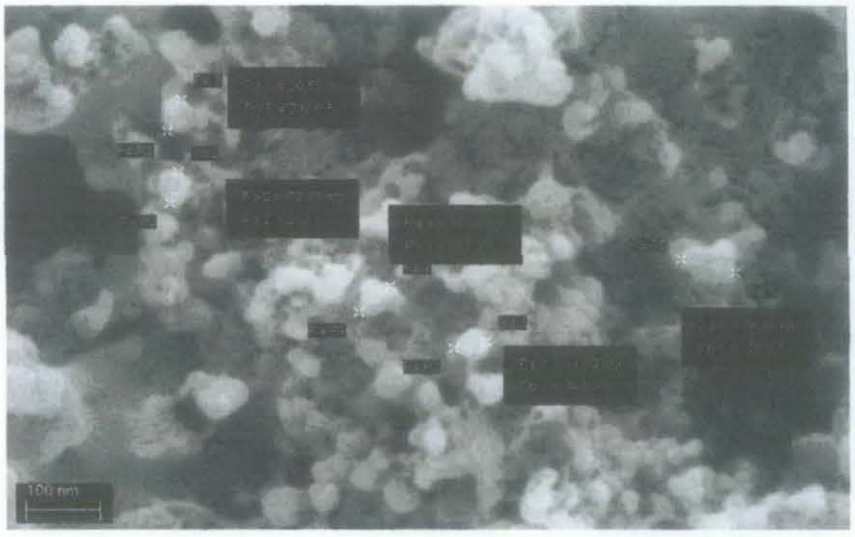


Figure 12: FESEM image of the silica sand nanoparticles

The chemical composition of silica sand then was analyzed by using XRF analysis following STG2-S4-Check measurement method. The result was shown in Table 3 below.

Table 3: Chemical composition of Tronoh silica sand nanoparticles

Compound	Weight percentage (wt%)
Al_2O_3	2.99
SiO_2	95.22
P_2O_5	0.77
K_2O	0.095
CaO	0.139
TiO_2	0.16
Fe_2O_3	0.121

3.3.4 Mixing Process

Before mixing with PP, silica sand nanoparticles was heated at 200 °C to remove the possible absorbed water for 5 hours. Then the silica sand nanoparticles were weighted according to the different weight percentage (5, 10, 15 and 20 wt%) before mix with PP. The mixing of nanosilica sand with PP was carried using internal mixer (Thermo Haake PolyLab System) under Roller-Rotors R3000 with 50 rpm and 190 °C temperature for 10 minutes (Figure 13). Figure 14 shows the sample that been produced from the internal mixer.

Figure 13: Internal mixer

Figure 14: Sample from internal mixer

3.3.5 Granular Shape Production

After mixing process the granular shape of the mixed particles were produced by using granular machine (Figure 15). Figure 16 shows the sample produced from the granular machine.

Figure 15: Granular machine

Figure 16: Granular shape of PP+SiO_2 nanoparticles

3.3.6 Composites Production

The composites with 10, 15 and 20wt% also been produced by the same process. Figure 17 shows all the composites with different wt%.

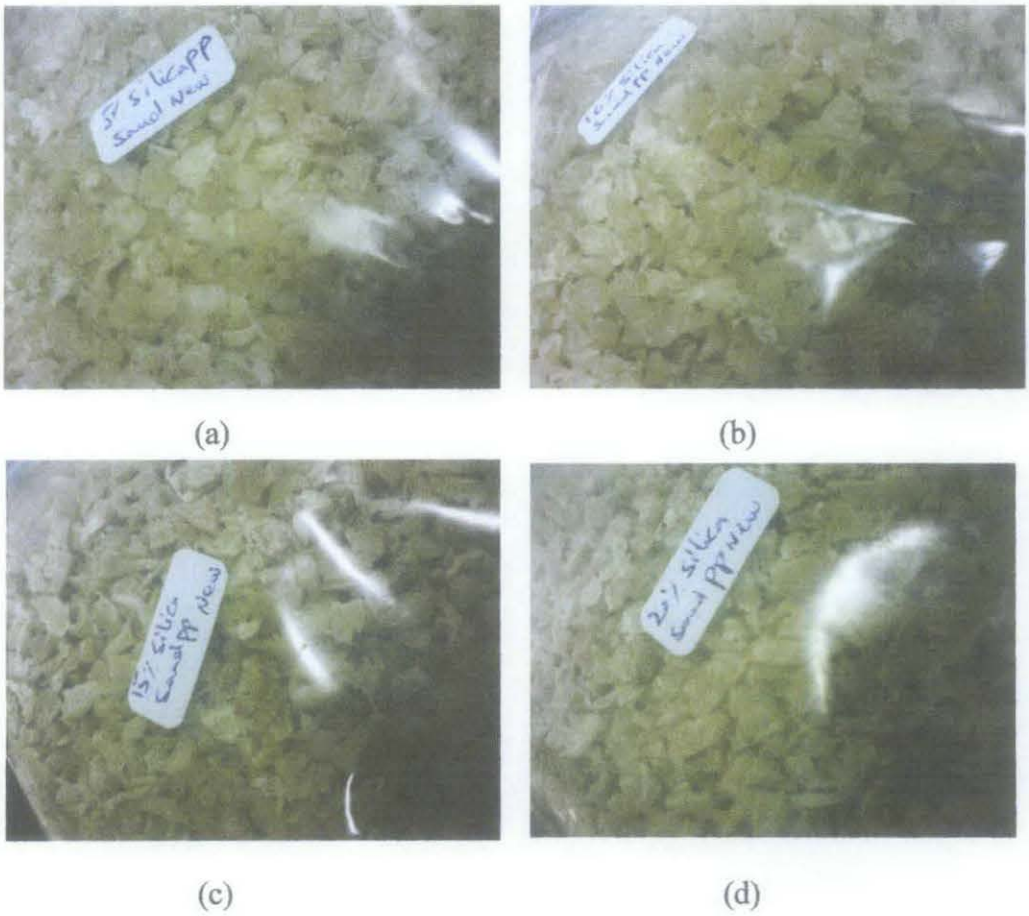


Figure 17: PP composites with (a) 5wt% SiO_2, (b) 10wt% SiO_2, (c) 15wt% SiO_2, (d) 20wt% SiO_2

3.3.7 Dog-bone-shaped Tensile Bar Production

The resultant particles were moulded into dog-bone-shaped tensile bars (ASTM D638-97) with an injection molding machine at 215 °C. Figure 19 shows the tensile bars that have been produced from injection molding machine (Figure 18).

Figure 18: Injection molding machine

Figure 19: Dog-bone-shaped tensile bars
(From left: pure PP, 5wt%, 10wt%, 15wt%, and 20wt% SiO_2+PP)

Tensile and flexural properties of the composites were measured by using Universal Tensile Testing machine (5 KN) LLOYD Instruments, LR 5K with a cross head speed of 50mm/min. The densities of the composites will be measured by Mettler Toledo AX205 density measurement instrument. The PP matrix composites with 5, 10, 15 and 20 wt% of silica sand nanoparticles will be produced as the specimens.

Figure 20 shows the flow chart of the project based on procedure of experiment. The flow chart is same for all 5, 10, 15 and 20wt% composites.

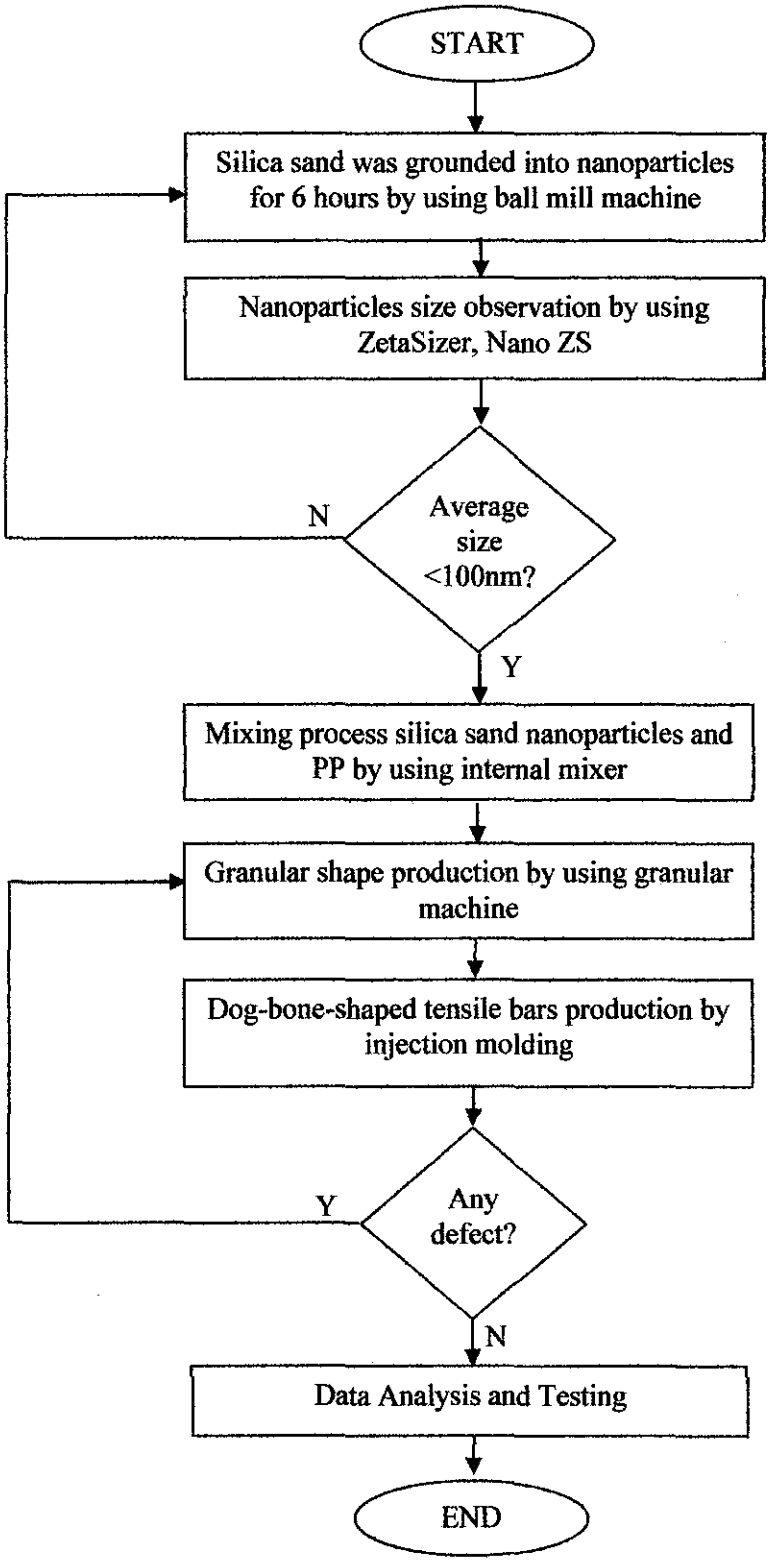


Figure 20: Project flow chart

3.4 Project Planning

Table 4: Project planning for Final Year Project I

No	Activities	Week													
		1	2	3	4	5	6	7	8	9	10	11	12	13	14
1	**Selection of Project Title**	■	■												
2	**Preliminary Research Work**														
	• Literature review		■	■	■	■	■	■	■	■	■	■	■	■	■
	• Preliminary report preparation		■	■	■										
	• Submission of preliminary report				▲										
3	**Project work**														
	• Preparation of silica nanoparticles by using ball mill machine				■	■	■								
	• Heating process of silica nanoparticles					■	■								
	• Mixing process by using internal mixer						■	■							
	• Granular shape production by using granular machine							■	■						
	• Dog-bone-shaped tensile bars production by using injection molding								■	■	■				
4	**Progress report and seminar**														
	• Data gathering and analysis							■	■	■	■				
	• Submission of progress report								▲						
	• Seminar								▲						
5	**Project works continues**														
	• Tensile test by using universal testing machine									■	■				
	• Physical properties (densities) analysis										■	■			
	• Data gathering and analysis										■	■			
6	**Interim report**														
	• Discussion of data										■	■	■		
	• Submission of interim report													▲	
7	**Oral presentation**														▲

Mid-Semester Break

■ Process

▲ Key milestone

Table 5: Project planning for Final Year Project II

No	Activities	Week														
		1	2	3	4	5	6	7		8	9	10	11	12	13	14	15
1	**Project Work Continues**																
	• Mixing process by using internal mixer	■															
	• Granular shape production by using granular machine		■	■													
	• Dog-bone-shaped tensile bars production by using injection molding			■													
	• Flexural test by using universal testing machine			■	■												
	• Microstructure analysis by using Field Emission Scanning Electron Microscope (FESEM)					■	■										
2	**Progress Report**																
	• Data gathering and analysis						■	■									
	• Submission of progress report									▲							
3	**Project Work Continues**																
	• Data gathering and analysis									■	■						
4	**Pre-EDX**																
	• Poster Preparation										■	■					
	• Submission of Poster												▲				
5	**Project Work Continues**																
	• Preparation of Dissertation										■	■	■	■			
	• Preparation of Technical Paper												■	■			
6	**Dissertation and Technical Paper**																
	• Submission of Draft Report													▲			
	• Submission of Dissertation (soft bound)														▲		
	• Submission of Technical Paper														▲		
7	**Oral Presentation**															▲	
8	**Hard Bound Dissertation Submission**																▲

Mid-Semester Break

■ Process

▲ Key milestone

4 RESULTS AND DISCUSSION

After the samples had been produced, several tests took places to measure the mechanical and physical properties of the composites.

4.1 Tensile Test

The dog-bone-shaped tensile specimens of PP-SiO_2 composites were tested to get the tensile strength and Young's modulus. Tensile test was conducted by using Universal Tensile Testing machine (5KN) LLOYD Instruments, LR 5K (Figure 21). The result of the tensile testing was shown in Table 6.

Table 6: Result for tensile test

Silica Sand+PP Composites	Max Load (N)	Tensile Strength (MPa)	Young's Modulus (MPa)
Pure PP	1505.0907	37.6273	254.4127
5% Silica Sand+PP	1221.7070	30.5427	242.7426
10% Silica Sand+PP	1193.5399	29.8385	274.3043
15% Silica Sand+PP	1239.2493	30.9812	259.4165
20% Silica Sand+PP	1219.0814	30.477	293.6561

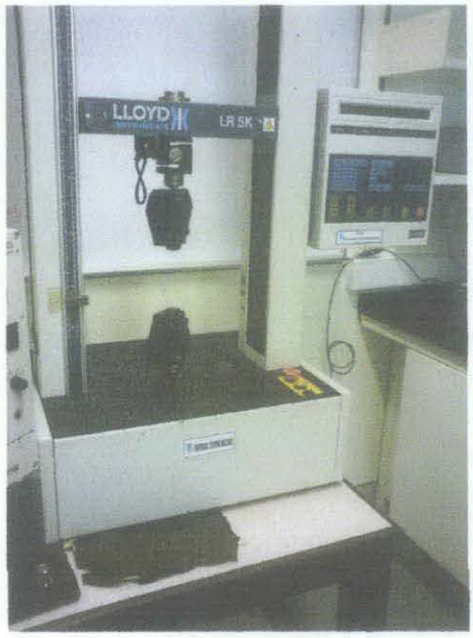


Figure 21: Universal Tensile Testing machine

Figure 22 shows the fracture tensile test samples of PP-SiO_2 composites. A strong filler/matrix adhesion would lead to enhanced strength of particulate composites.[2] According to their consideration, it is known that the improvement of tensile strength of the composites is due to interfacial interaction. The improvement of interfacial interaction will increase the tensile strength of the composites. However, the addition of other particles in PP composites does not always increase the tensile strength. Based on the result obtained, tensile strength of PP-SiO_2 composites decreased with the addition of silica sand nanoparticles and become slightly constant for different addition wt% of silica sand nanoparticles as shown in Figure 23. This was due to poor bonding at the interface between PP and silica sand nanoparticles. The low of interfacial interaction will result the decrease in tensile strength.

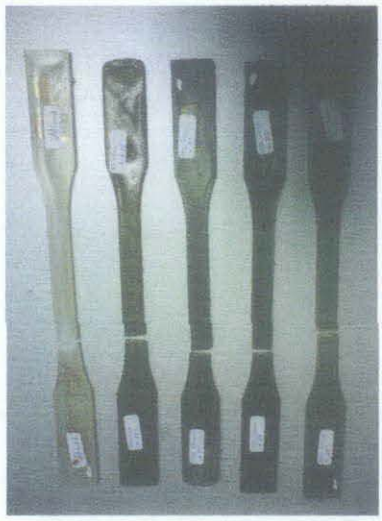


Figure 22: Fracture tensile test samples of PP-SiO_2 composites (pure PP, 5wt%, 10wt%, 15wt%, 20wt% SiO_2+PP)

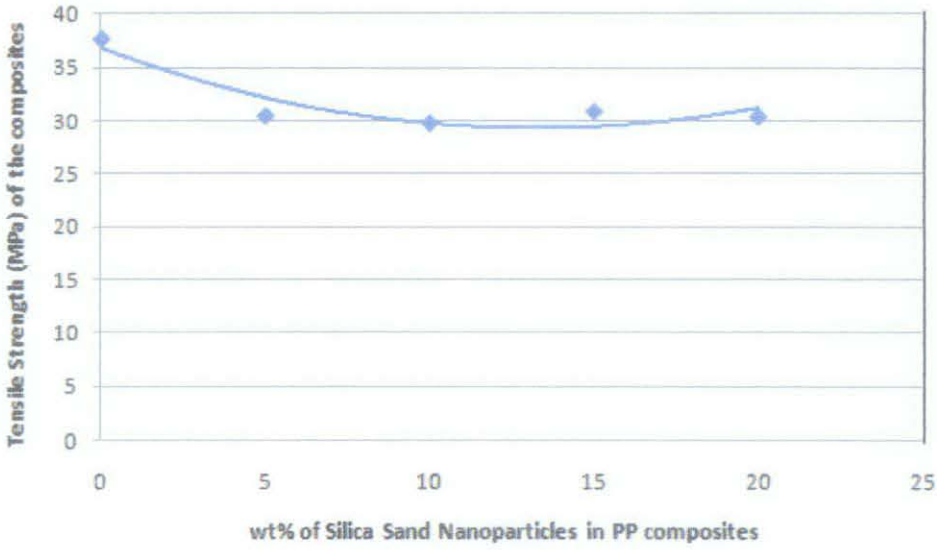


Figure 23: Tensile strength of PP-SiO_2 composites

There is an increasing trend for Young's modulus with the addition of silica sand nanoparticles in PP composites as shown in Figure 24. This is because the addition of silica sand nanoparticles increased the interfacial stiffness of the composites. Usually the capability of composite interface to transfer elastic deformation depends to a great extent upon the interfacial stiffness and static adhesion strength. A high interfacial stiffness corresponds to a high composite modulus.[1] As interfacial stress transfer efficiency depends on the stiffness of the interphase, higher interfacial stiffness favors improvement of the composites modulus.[2]

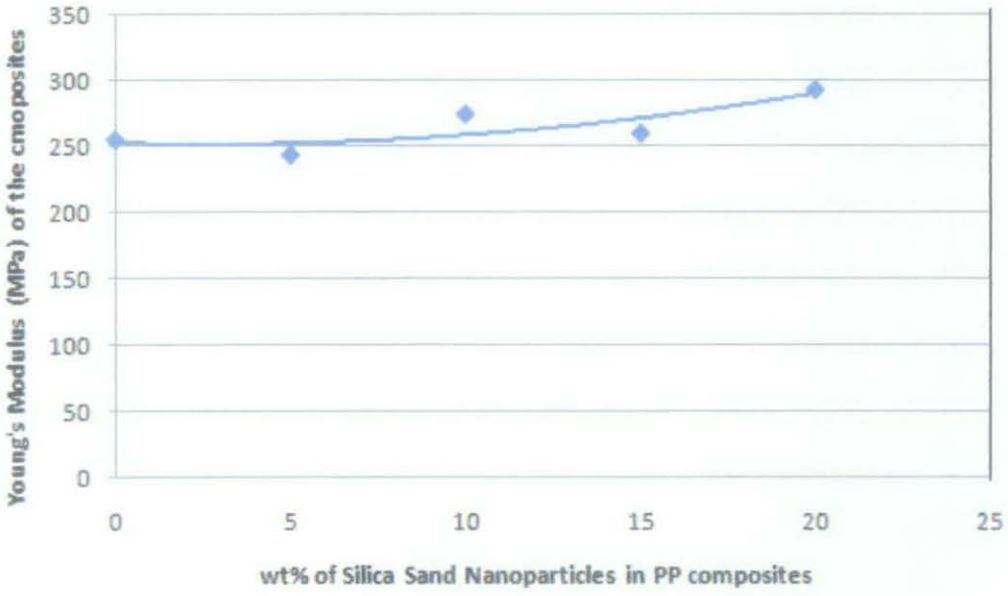


Figure 24: Young's Modulus of PP-SiO_2 composites

4.2 Density Measurement

The small parts of the samples then were cut to get the density measurement (Figure 25). From the graph as shown in Figure 26 an increasing trend of density was observed with the increasing content of silica sand nanoparticles in PP. It is because silica sand (SiO_2) is denser than PP and causes an enhancement of density of PP when added as reinforcement. The densities of PP based SiO_2 are given in Table 7.

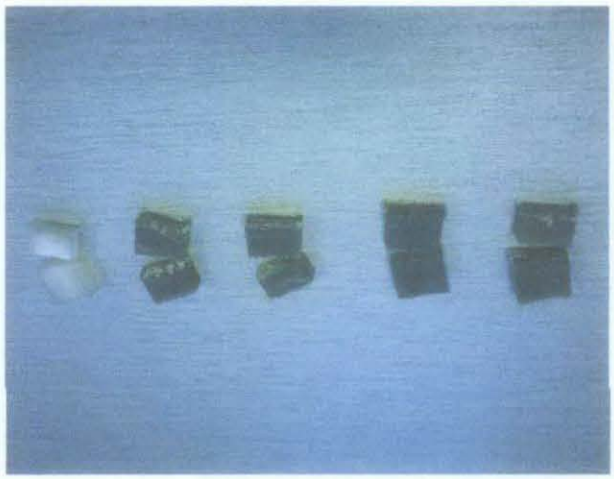


Figure 25: Samples of PP-SiO_2 composites for density measurement (pure PP, 5wt%, 10wt%, 15wt%, and 20wt% SiO_2+PP)

Table 7: Density measurement for PP composites with different wt% of SiO_2

Composites/Density	1st reading (g/cm^3)	2nd reading (g/cm^3)	Average (g/cm^3)
Pure PP	0.892	0.852	0.872
5% Silica Sand+PP	0.873	0.855	0.864
10% Silica Sand+PP	0.883	0.899	0.891
15% Silica Sand+PP	0.932	0.876	0.904
20% Silica Sand+PP	0.959	1.006	0.983

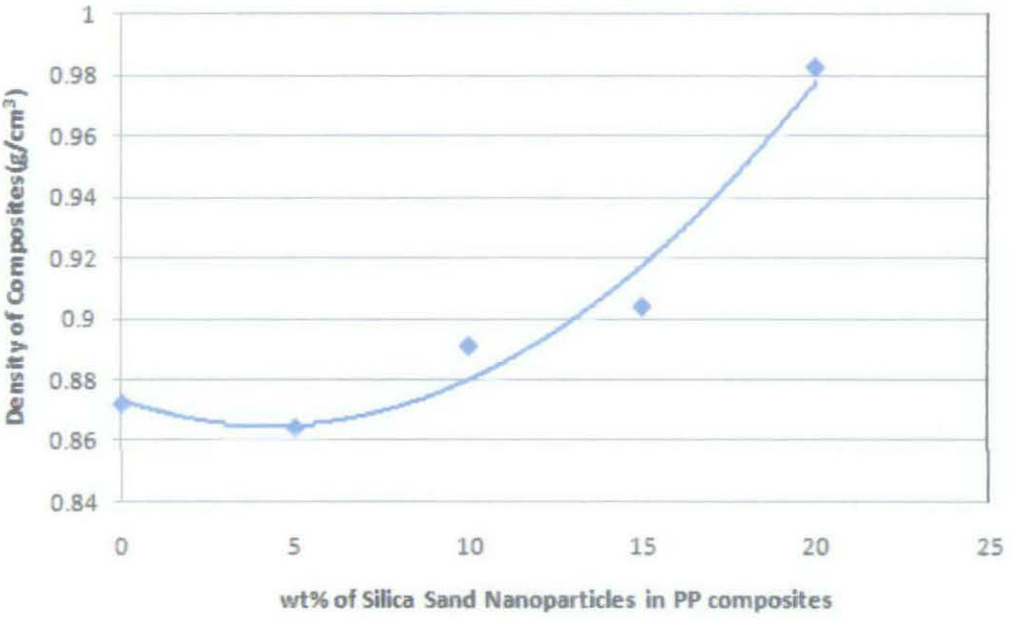


Figure 26: Density analysis of PP-SiO_2 composites

4.3 Flexural Test

The dog-bone-shaped tensile specimens were cut into rectangular shaped as shown in Figure 27. Flexural test was conducted according to ASTM standard (D790-03) by using Universal Tensile Testing machine (5KN) LLOYD Instruments, LR 5K (Figure 28). The result of the flexural testing was shown in Table 8 below.

Table 8: Result of flexural strength test

Batch Reference	Wt% of nanosilica	Maximum Bending Stress at Maximum Load (MPa)
PP	0	19.894
PP+5%nanosilica	5	19.847
PP+10%nanosilica	10	20.018
PP+15%nanosilica	15	19.963
PP+20%nanosilica	20	20.09

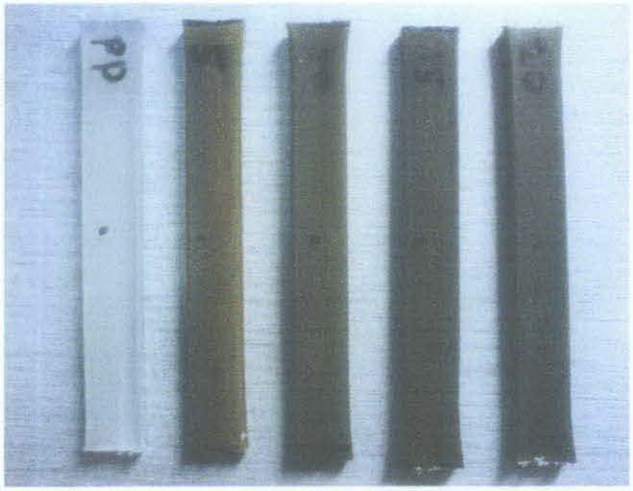


Figure 27: Flexural test samples of PP-SiO_2 composites (pure PP, 5wt%, 10wt%, 15wt%, and 20wt% SiO_2+PP)

Figure 28: Universal Testing machine used for flexural test

Based on the graph obtained in Figure 29, the increase of weight percentage (wt%) of silica sand nanoparticles increase the flexural strength of the composites. The tensile moduli, impact and flexural properties of thermoplasticolefin blend (TPO)/nanosilica composites showed improvements at low loadings of nanosilica, indicating a good balance of stiffness and toughness.[16] According to the statement, the increase of flexural strength is due to good adhesion between PP and silica sand nanoparticles. When the bonding is strong enough, the flexural strength of particulate composites can be higher than pure PP.

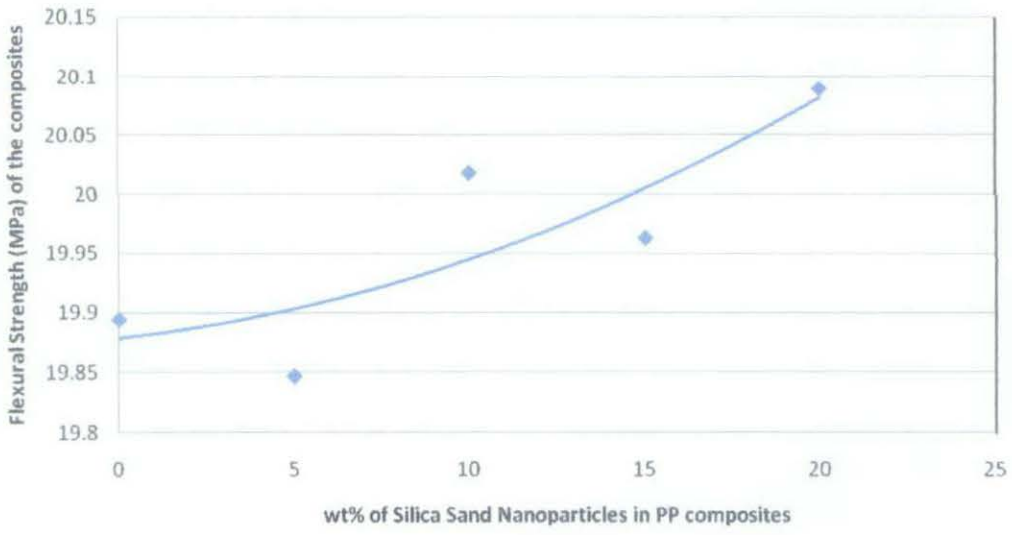


Figure 29: Flexural strength of PP-SiO_2 composites

4.4 FESEM Analysis of Fracture Surface of Tensile Samples

The fracture surface of 5, 10, 15 and 20 wt% of PP-SiO_2 composites tensile samples were observed by using Field Emission Scanning Electron Microscope (FESEM). Some of the white zones in FESEM analysis show that large portion of the polymer matrix is surrounded by the silica sand nanoparticles cause plastic deformation and cohesive failure of the composites. In case of 5wt% less agglomeration take place and fracture areas are not deeper as compared to 20wt% of silica sand nanoparticles, where the fracture areas are deeper and small due to large agglomeration of the nanoparticles (as shown in Figure 30). This was due to agglomeration between PP and silica sand nanoparticles thus decrease the tensile strength and result in brittle fracture.

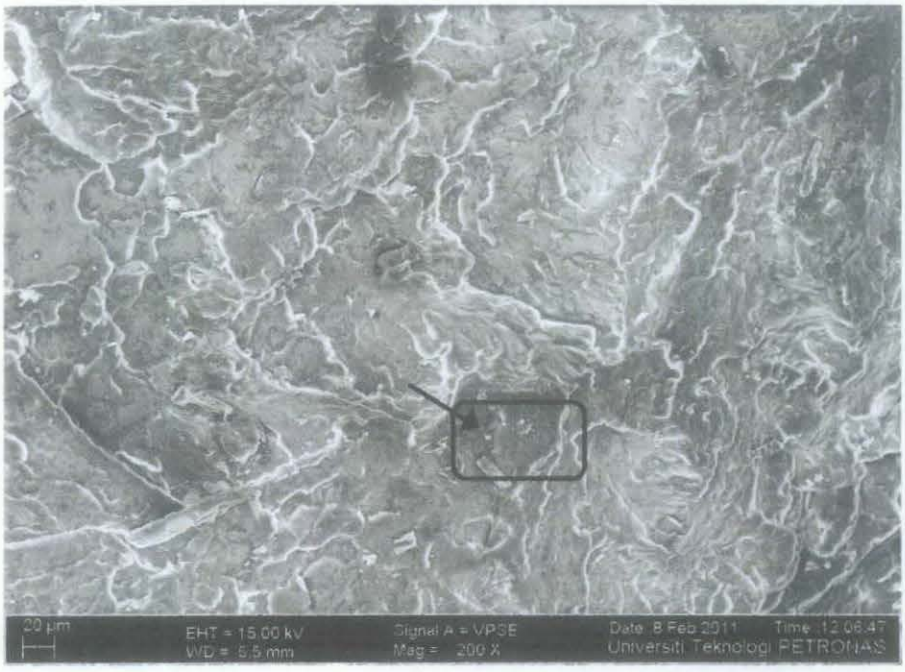


Figure 30(a): Fracture surface analysis of PP-SiO_2 nanoparticles composites with 5wt% SiO_2

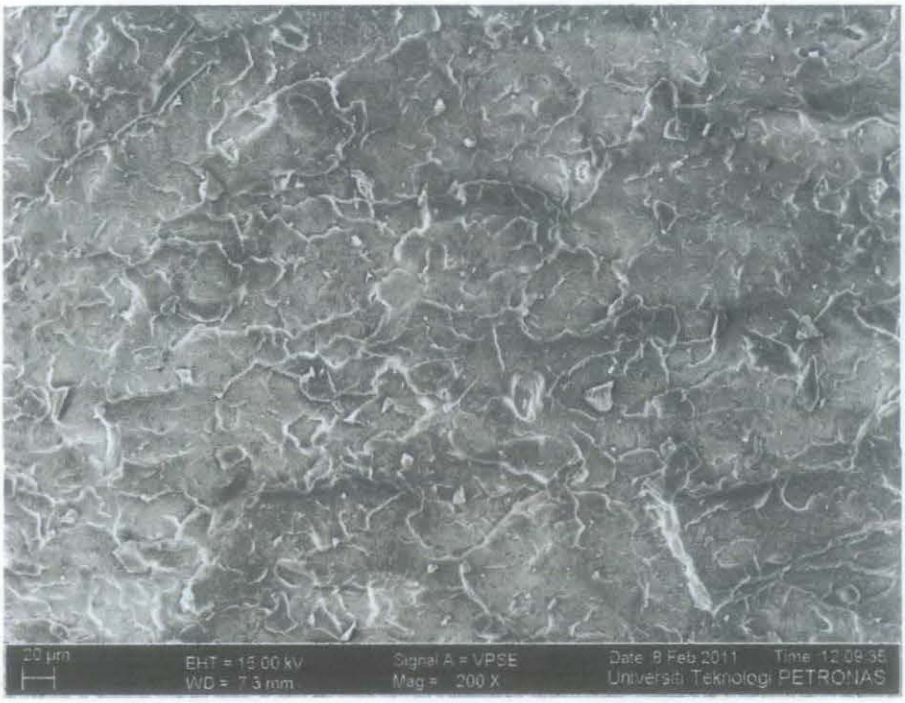


Figure 30(b): Fracture surface analysis of PP-SiO_2 nanoparticles composites with 10wt% SiO_2

Figure 30(c): Fracture surface analysis of PP-SiO_2 nanoparticles composites with 15wt% SiO_2

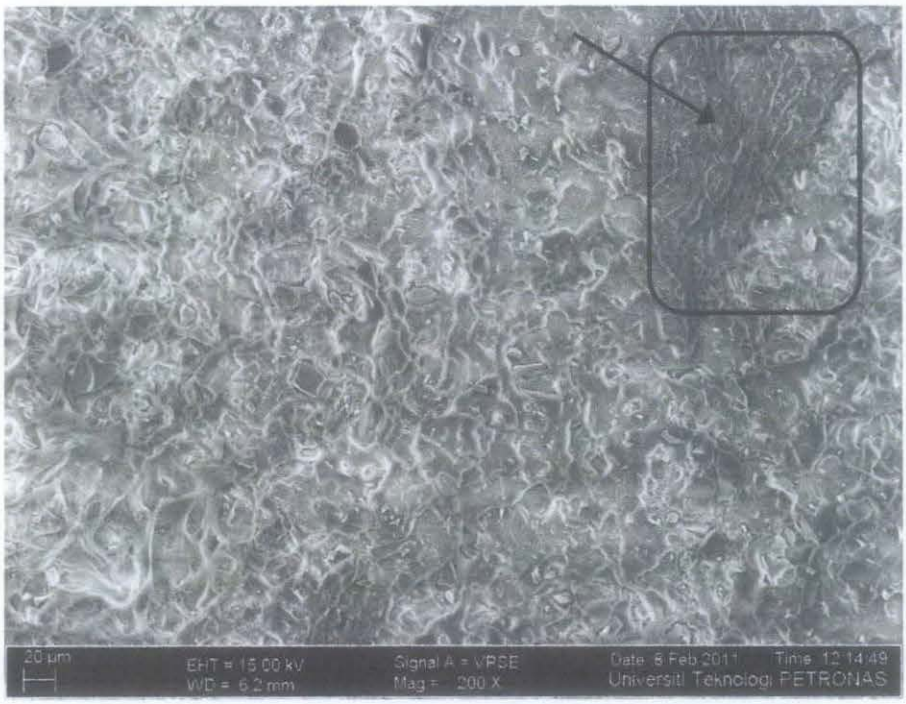


Figure 30(d): Fracture surface analysis of PP-SiO_2 nanoparticles composites with 20wt% SiO_2

5 CONCLUSION AND RECOMMENDATIONS

5.1 Conclusion

Over this report, the author has done additional researching of data and information from various resources in order to do this project successfully. The needed materials already obtained at the material laboratory and the testing machines and equipment are also ready to be used.

Sample preparation plays very important role in determining physical and mechanical properties of nanosilica composite. Each specimen need to be prepared exactly the same way each other. Appropriate sample preparation increases level of particle dispersion inside the system and could yield better results.

The effects of silica sand nanoparticles in PP increased its density and flexural strength but slightly decreased its tensile strength. From experimental values, PP matrix with 20% addition of nanosilica could be proposed as the optimum mixture ratio. In this project, although pure PP recorded the highest tensile strength value, common experimental trend in polymer nanocomposites can be observed from 5% to 20% particle addition. The addition of silica sand nanoparticles to PP causes an enhancement in physical properties: density from $0.872 g/cm^3$ to $0.983 g/cm^3$. Thus, the objective of this project, which is to study on effects of nanosilica sand addition on physical and mechanical properties of polypropylene (PP), had been achieved.

5.2 Recommendations

The further study and analysis can be done to get better understanding about the project. Other properties such as thermal properties can be studied in the future to develop the project.

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