

# **POLYPROPYLENE ORGANOCCLAY NANOCOMPOSITE**

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## ABSTRACT

In large exploration of nanotechnology, polymer based nanocomposite have become major area of current scientific research and industrial application. As reported, by 2009, it is estimated the market size of polymer nanocomposite for industrial purposes is about 48 million pounds. Polymer nanocomposite is invented to meet the purpose of improving the properties of the polymer itself such as mechanical properties, thermal properties, conductivity and many more. In this study, pure material of polymer, Polypropylene (PP) and Sodium (Na<sup>+</sup>) content organoclay with different ratios of PP/Na<sup>+</sup> were synthesized by melt intercalation polymerization with the objective of producing polymer nanocomposite with enhanced mechanical properties specifically, tensile and hardness properties. The predispersed master batch of different ratio of PP/Na<sup>+</sup> nanocomposite were shaped into a pellet form by using a twin screw extruder before molded into a dog-bone form by hot press machine. The samples are varies by weight percent starting with pure Polypropylene, 1%, 3% and 5% of cloisite Na<sup>+</sup>. The samples were characterized by Fourier Transform Infrared (FTIR) Spectroscopy, tested mechanically using Universal Testing machine for tensile properties and hardness properties via Brinell Hardness Test. FTIR studies illustrate that Na<sup>+</sup> organoclay successfully intercalated into the Polypropylene layer. The resulting composites indicated about 70% increases in the modulus for tensile, compared to pure Polypropylene. In tensile test, a maximum load is applied on the sample until it reaches on its break point. While for hardness test it's about 24% increases, compared to pure Polypropylene. Hardness was measured on a Rockwell type instrument modified to measure the depth of the unrecovered indentation. A good mixing process and dispersion of Na<sup>+</sup> can ensure a high increment of the mechanical properties. Samples also need to be fully dried, so that less bubbles, which affecting the data, can be produced during hot press molding process.

## ABSTRAK

Di dalam eksplorasi dunia nanoteknologi yang luas, polimer nanokomposit telah menjadi suatu tarikan utama di dalam kajian saintifik dan juga di dalam aplikasi industri pada hari ini. Seinggakan ada laporan mengatakan, sehingga tahun 2009, dijangkakan saiz pasaran bagi polimer nanokomposit mampu menjangkau sehingga 48 milion pound. Polimer nanokomposit dihasilkan adalah bertujuan untuk memenuhi keperluan semasa termasuk meningkatkan kualiti polimer itu sendiri sebagai contoh seperti peningkatan dari segi mekanikal, termal, konduktiviti, dan lain-lain. Bagi kajian ini, Polipropylene (PP) dicampur bersama Natrium ( $\text{Na}^+$ ) dengan komposisi  $\text{Na}^+$  terhadap PP yang berbeza-beza melalui kaedah '*melt intercalation*' dengan tujuan untuk meningkatkan keupayaan polimer ini, terutamanya dari segi mekanikalnya iaitu sifat kebolehtarikan dan sifat kekerasan polimer. Sampel mentah setelah dicampurkan bersama akan dibentuk menjadi palet melalui alat '*twin screw extruder*'. Kandungan sampel adalah berbeza mengikut kandungan  $\text{Na}^+$  iaitu bermula dengan Polipropylene asli (0%  $\text{Na}^+$ ), 1%, 3%, dan 5%. Kesemua sampel kemudiannya dikaji menggunakan *Fourier Transform Infrared (FTIR) Spectroscopy* dan diuji secara mekanikal melalui mesin '*Universal Testing Machine*' untuk melihat sifat kebolehtarikan polimer dan juga untuk sifat kekerasan polimer melalui ujikaji Brinell. Ujikaji FTIR menunjukkan  $\text{Na}^+$  telah berjaya dimasukkan ke dalam lapisan Polipropylene. Komposit yang terhasil menunjukkan peningkatan sebanyak 70% bagi sifat kebolehtarikan polimer. Manakala bagi sifat kekerasan, peningkatan sebanyak lebih kurang 24% berjaya dicatatkan. Kekerasan polimer ini diukur berdasarkan kedalaman lekukan yang terhasil. Proses pencampuran yang baik antara polypropylene dan  $\text{Na}^+$  mampu menghasilkan penyebaran  $\text{Na}^+$  secara menyeluruh yang akhirnya dapat meningkatkan sifat-sifat mekanikal polimer tersebut. Tidak lupa juga semasa proses acuan, kesemua sampel perlu dipastikan kering sepenuhnya supaya buih-buih yang mampu memberi kesan terhadap perolehan data tidak terhasil.

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**LIST OF ABBREVIATIONS**

|                    |   |  |
|--------------------|---|--|
| ASTM               | - | American Standard Testing Method               |
| CEC                | - | Cation exchange capacity                       |
| FTIR               | - | Fourier Transform Infrared                     |
| HB                 | - | Hardness Brinell number                        |
| ISO                | - | International Organization for Standardization |
| MMT                | - | montmorillonite                                |
| PEO                | - | Poly (ethylene oxide)                          |
| PLSNs              | - | Polymer-layered silicate nanocomposites        |
| PP                 | - | Polypropylene                                  |
| PP-MA              | - | Polypropylene Maleic Anhydride                 |
| PP-Na <sup>+</sup> | - | Polypropylene-Sodium organoclay                |
| PVA                | - | Poly (vinyl alcohol)                           |
| SEM                | - | Scanning Electron Microscopy                   |
| TEM                | - | Transmission Electron Microscopic              |
| TM                 | - | Tensile Modulus                                |
| TS                 | - | Tensile Strength                               |

XRD - X-Ray Diffractometer

**LIST OF NOMENCLATURE**

|            |   |                              |
|------------|---|------------------------------|
| $\delta$   | - | Stress                       |
| $\partial$ | - | Strain                       |
| $F$        | - | Applied force                |
| $D$        | - | Indentation diameter         |
| $\Delta F$ | - | Changes in force applied     |
| $\Delta L$ | - | Changes in length            |
| $A_i$      | - | Initial cross-sectional area |
| $L_i$      | - | Initial length               |
| $F_b$      | - | Force at break point         |
| $L_b$      | - | Elongation at break point    |

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## CHAPTER 1

### INTRODUCTION

#### 1.1 Background of study

Humans have taken advantage of the versatility of polymers for centuries in the form of oils, tars, resins, and gums. However, it was not until the industrial revolution that the modern polymer industry began to develop. In 1843, Charles Goodyear succeeded in producing a useful form of natural rubber throughout a process known as "vulcanization" <sup>[5]</sup>. The introduction of these revolutionary materials began an explosion in polymer research that is still going on today. Nowadays, with a large dose of exploration, polymer has come to knock the door of nanotechnology, which is a very pleasure technology that brings the human's life to the other one whole new of dimension. Polymer nanocomposite seizes the promise of advances that exceed those achieved in recent decades in composite material. The nanostructure created by a nanophase in polymer matrix represents a major option to the structure of typical polymer composites.

Composites are structures in which two (or more) materials are combined to produce a new material whose properties would not be attained by conventional means. The continuous constituent of the composite, the matrix, serves to surround the other phases and provides bulk form to the material. The major structural constituents are typically fibers or particles. Fiber-filled composites are often laminates, formed from

stacks of individual plies or lamina, which provide continuous in-plane reinforcement. The strength and rigidity of a composite can be controlled by varying the shape, amount, surface functionality and orientation of the major structural constituent in the matrix. This ability to tailor properties, combined with the inherent low density of composites and their relative ease of fabrication, makes these materials extremely attractive alternatives for many applications, such as boats, sports equipment, automotive components, biomedical products, underground pipes, and aircraft/aerospace structures.

What differentiates nanocomposites material from classical composites is the scale of control of fabrication, processing and performance, which can be achieved nearly down to the atomic scale. Nano-scale filler that introduced to this field, somehow increase the focused properties up to an extent of numbers. This improvement might as well affect another industry such as agricultural, military, automotive, textile, and construction industries. Most of the commercial interest in nanocomposites has been focused on thermoplastics. They can be broken into two groups: less expensive commodity resins and the more expensive (and higher performance) engineering resins. One of the goals of nanocomposites was to permit substitution of more expensive engineering resins with a less-expensive commodity resin nanocomposite. Substituting a nanocomposite commodity resin with equivalent performance as a more expensive engineering resin should yield overall cost saving.

Nanocomposites have proven to be more difficult to manufacture than first anticipated, but new materials in pilot plants and laboratories may be able to live up to much of their initial promise. Greater understanding of the chemistry driving the formation of nanocomposites has enabled researchers to discover practical production methods for these materials. Nanocomposites offer improvements in several of the properties of thermoplastics including tensile strength, modulus, barrier and heat distortion temperature. If a nanocomposite could offer these improvements at no additional cost, then it quickly would replace a large percentage of unfilled thermoplastics. Unfortunately, improved performance of a nanocomposite compared to a thermoplastic comes with an increase in price.

## **1.2 Objective of study**

The aims of this study are to produce Polypropylene with Na<sup>+</sup> as nanocomposite, as the nanofiller in order to enhance the mechanical properties, which are the tensile hardness properties of the Polypropylene produced.

## **1.3 Scope of the study**

In order to achieve the objective of the study, these numbers of scopes will be entertained:

- i. To produce Polypropylene with Na<sup>+</sup> organoclay as nanofiller using in-situ polymerization
- ii. To characterize Polypropylene with Na<sup>+</sup> as nanocomposite using Fourier Transform Infrared (FTIR) Spectroscopy
- iii. To study the mechanical properties which are tensile and hardness properties of the Polypropylene/Na<sup>+</sup> polymer nanocomposite.

## **1.4 Problem statement**

Nanocomposites manufactured by the melt compounding technique have not yet attained the level of performance obtained by adding the Na<sup>+</sup> filler in situ during the polymerization stage of making the polymer. It is believed this is due to the less than optimum level of exfoliation achieved during the melt-compounding step.



From the literature and through the work of this study it is shown that the compounding parameters and mixing screw profile are important variables however a most important variables the pre treatment of the clay itself. Since the clay is an aggregate of thousands of individual platelets, it is critical that the individual platelets be separated to some extent prior to compounding. If the platelets are not separated, it is unlikely that the shear forces generated during the compounding process will be great enough to overcome the forces holding the aggregates together. Hence, less than optimum exfoliation or dispersion will result. The process of opening up the spaces (gallery) between the plates is called intercalation.

The first problem becomes finding and applying the right clay treatment allowing for intercalation and then having the intercalant is compatible enough with the host polymer so properties are enhanced without the sacrifice of other properties. Such as higher strength but not at the expense of embrittling the plastic.

The second problem is being able to melt compound these prepared clays to get maximum dispersion without degrading the polymer. Ultimately the problem to be solved is how to fabricate a thermoplastic nanocomposite yielding enhanced properties.

Despite of advanced properties reported, many difficulties appear when manufacturing nanocomposite materials. The most critical obstacles to successful commercialization are access to nanocomposite formulation and process technology. It is known that not all polymers are well suited nanocomposite development. Compatibility is the keyword. Actually, there is many issue concerning the control of nanocomposite structure and the understanding of the structure-property relationships in order to ensure the desired property improvement are unsolved, which strongly minimize the industrial application.

## 1.5 Rationale and Significance

This research is designed in purpose of increasing the properties of Polypropylene, which reinforced with the existence of Na<sup>+</sup> as nanocomposite. With this extremely upgraded version of Polypropylene, a lot of industry will benefit from it. It can be the agricultural industry, as well as for the automotive industry. In other word, increasing properties of Polypropylene will increase the quality of the product produced from these industry.

Frankly, although this research consumed a usage of Na<sup>+</sup> nanocomposite that known as a quite expensive material, the product, PP-Na<sup>+</sup> nanocomposite not actually having price hike due to the usage of Na<sup>+</sup> nanocomposite that is very small, as low as 0.5%. There will be an increment of the price, but in acceptable range.

## 1.6 Layout of the Thesis

Chapter 2 begins with the introduction of Polymer nanocomposite. This is then followed by the description of Polypropylene and Na<sup>+</sup> organoclay.

Chapter 3 starts with the explanation on preparing and producing the sample. All the related method on product characterization and testing will be discussed on detail in this chapter. For characterization, Fourier Transform Infrared (FTIR) Spectroscopy was used. While for testing part, Universal Testing machine is used for tensile testing, and Brinell test is used for hardness testing.

Chapter 4 commence with the data gained from experiment ran before. The data then is analyzed thoroughly and presented via graph and all related diagram. In this chapter, we can see how Na<sup>+</sup> organoclay affecting the structure in Polypropylene that brings us to the enhanced properties of Polypropylene nanocomposite, with Na<sup>+</sup> as nanofiller. The enhanced properties of PP/Na<sup>+</sup> nanocomposite are showed by the data collected from the tensile and hardness test.

Chapters 5 conclude all the experimentation process and data obtained from the study, as well as some recommendations for future works are presented.

## CHAPTER 2

### LITERATURE REVIEW

#### 2.1 Introduction

The first use of the concepts found in 'nano-technology' was in "There's Plenty of Room at the Bottom," a talk given by physicist Richard Feynman at an American Physical Society meeting at Caltech on December 29, 1959 ([www.inventors.about.com](http://www.inventors.about.com)). Basically, nanotechnology is the study of the controlling of matter on an atomic and molecular scale. This technology deals with structures of the size 100 nanometers or smaller in at least one dimension, and involves developing materials or devices within that size.

Nanotechnology is not new to polymer science as prior studies before the age of nanotechnology involved nanoscale dimensions but were not specifically referred to as nanotechnology until recently. Phase separated polymer blends often achieve nanoscale phase dimensions; block copolymer domain morphology is usually at the nanoscale level; asymmetric membranes often have nanoscale void structure, miniemulsion particles are below 100 nm; and interfacial phenomena in blends and composites involve nanoscale dimensions. Even with nanocomposites, carbon black reinforcement of elastomers, colloidal silica modification and even naturally occurring fiber (e.g.,

asbestos-nanoscale fiber diameter) reinforcement are subjects that have been investigated for decades. Almost lost in the present nanocomposite discussions are the organic – inorganic nanocomposites based on sol–gel chemistry which have been investigated for several decades (Mark J.E et al, 1984)

## **2.2 Polypropylene**

Polypropylene (PP) is one of the classes of thermoplastic polymer. It is made by chemical industry and used in wide range of function including packaging, textiles, stationary, laboratory equipment and even for automotive component. During the period from 1974 to 1999, the rate of consumption of PP increased from 7% to 12% annually (Michael J. Balow, 2003)

This high growth rate of PP consumption has required the production capacity to keep up with the growing demand. Every now and then there is always a research came out with a new perspective in improving Polypropylene itself, hence enhanced the properties of these polymer so that it can meet up with the growing demand in the market.

### **2.2.1 Synthesis of Polypropylene**

PP is produced commercially from olefin (alkene) monomers which contain a reactive carbon to carbon double bond (Environmental Information Document, September 2009). As many other polymer, Polypropylene start with a monomer which is Propylene. The final product consisting of many thousands of bound propylene units is called the Polypropylene.

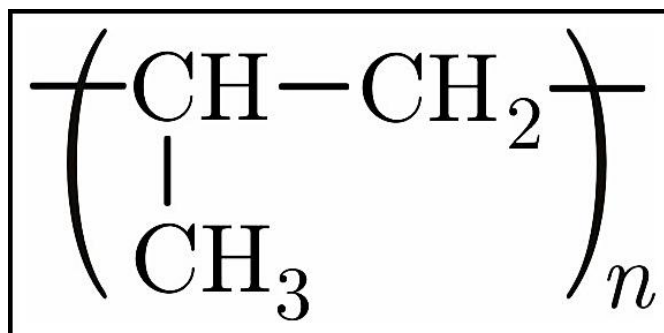


Figure 2.1: Molecular structure of Propylene

Polypropylene can be made from the monomer propylene by Ziegler-Natta polymerization and by metallocene catalysis polymerization.

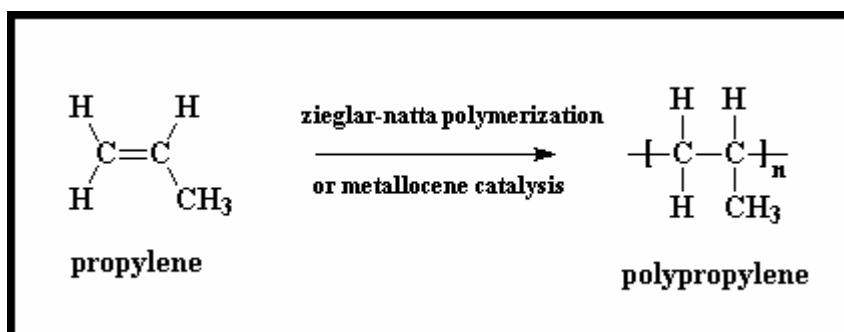


Figure 2.2: Chemical polymerization of Polypropylene

### 2.2.1.1 Ziegler-Natta Polymerization

The origin of the Ziegler-Natta catalysis dates back to 1953 when Ziegler discovered, at Muelheim, that some transition-metal compounds, eg,  $\text{TiCl}_4$ , used in combination with aluminium alkyls, could polymerize ethylene into linear poly-ethylene under mild conditions of pressure and temperature. In 1954, Ziegler's concept was applied by Natta, at the Polytechnic of Milan, to the stereo selective polymerization of propylene into a partially crystalline and stereoregular, previously unknown and unexpected polymer: isotactic polypropylene. These scientific breakthroughs, were quickly translated into industrial realities by Hoechst in 1955 and Montecatini in 1957 (Kissin Y.V, 2008), with the manufacture and

commercialization of high-density polyethylene and isotactic polypropylene, respectively.

Ziegler-Natta catalysts are prepared by reacting certain transition metal halides with organometallic reagents such as alkyl aluminum, lithium and zinc reagents (Beat Weidmann et al, 1983). The catalyst formed by reaction of triethylaluminum with titanium tetrachloride has been widely studied, but other metals (e.g. V & Zr) have also proven effective. The following diagram presents one mechanism for this useful reaction.

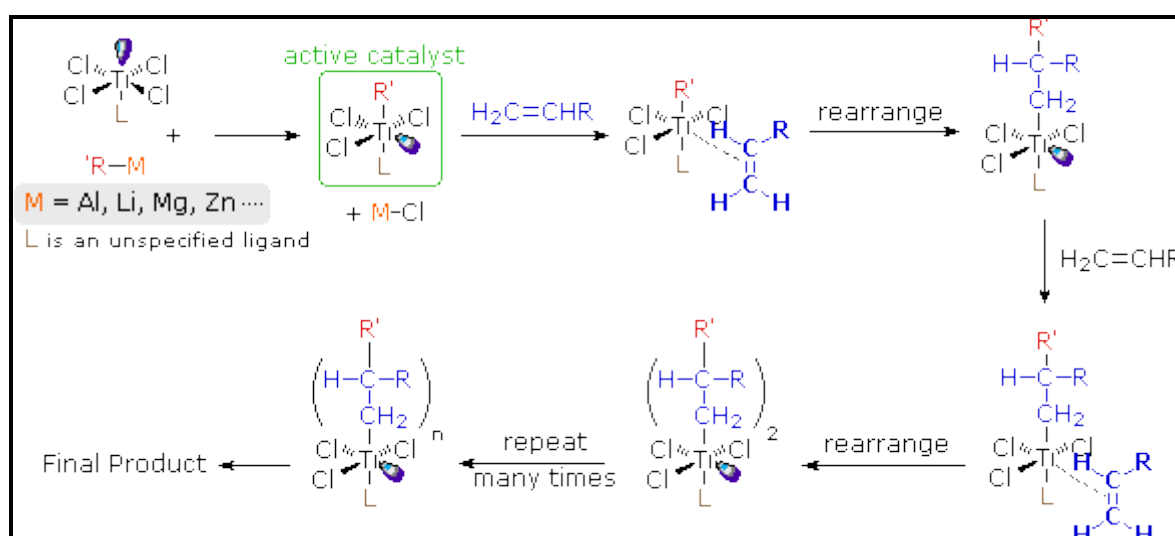


Figure 2.3: Mechanism of Ziegler-Natta polymerization

Others have been suggested, with changes to accommodate the heterogeneity or homogeneity of the catalyst. Polymerization of propylene through action of the titanium catalyst gives an isotactic product; whereas, vanadium based catalyst gives a syndiotactic product. But direct polymerization of polar comonomers using Ziegler-Natta catalysts is limited to Lewis bases, which lead to catalyst deactivation, polymer degradation and comonomer homopolymerization (Santeri Paavola et al, 2005).

### 2.2.1.2 Metallocene catalysis polymerization

The second class of Ziegler-Natta catalysts is metallocenes. They are soluble in aromatic hydrocarbons and usually contain two components, a metallocene complex and a special organometallic cocatalyst, MAO, [-O-Al-CH(CH<sub>3</sub>)<sub>n</sub>]. The idealized metallocene catalysts have the composition Cp<sub>2</sub>MCl<sub>2</sub> (M = Ti, Zr, Hf) such as titanocene dichloride. Bochmann, Hill and Kissin found that the organic ligands are derivatives of cyclopentadienyl. In some complexes, the two Cp rings are linked with bridges, like -CH<sub>2</sub>-CH<sub>2</sub>- or >SiPh<sub>2</sub>.

Depending of the type of their cyclopentadienyl ligands, metallocene catalysts can produce either isotactic or syndiotactic polymers of propylene or other 1-alkenes (Bochmann and Alt, 2000).

### 2.2.2 Physical Properties of Polypropylene

Provided below is the data gained from previous study about Polypropylene. This table included the physical and mechanical properties of PP:

**Table 2.1: Properties of Propylene**

| Property                                       | Value     |
|--|-----------|
| <b>PHYSICAL</b>                                |           |
| Specific gravity                               | 0.905     |
| Specific volume (in. <sup>3</sup> /lb.)        | 30.8-30.4 |
| Water absorption, 24 hours, 1/8 inch thick (%) | 0.01-0.03 |
| <b>MECHANICAL</b>                              |           |
| Tensile strength (psi)                         | 5,000     |
| Elongation (%)                                 | 10-20     |
| Tensile modulus (10~5 psi)                     | 1.6       |
| Flexural modulus (10~5 psi)                    | 1.7-2.5   |
| Impact strength, Izod (ft-lb/in. of notch)     | 0.5-2.2   |
| Hardness, Rockwell R                           | 80-110    |

Another form of PP produced in much lower volumes as a byproduct of semicrystalline PP production and having very poor mechanical and thermal properties, is a soft, tacky material used in adhesives, sealants, and caulk products.



The above two products are often referred to as “isotactic”(crystallizable) PP and “atactic” (noncrystallizable) PP, respectively. In this study, it is focused on increasing the properties related to tensile properties and hardness properties of Polypropylene by mix it with Sodium Montmorillonite.

### **2.2.3 Application of Polypropylene**

By now the major global markets for PP are dominated by flexible and rigid packaging (together 37%), followed by automotive, electric/electronic, and appliances (21%). Impressively, in third place with 18% are textile applications (Ellis et al, 2002).

Polypropylene for ages has been developed over and over again. By now, polypropylene has already been developed for medical purposes. It has been used in hernia and pelvic organ prolapse repair operations to protect the body from new hernias in the same location. A small patch of the material is placed over the spot of the hernia, below the skin, and is painless and is rarely, if ever, rejected by the body. However, a polypropylene mesh will erode over the uncertain period from days to years. Therefore, the FDA has issued several warnings on the use of polypropylene mesh medical kits for certain applications in pelvic organ prolapse, specifically when introduced in close proximity to the vaginal wall due to a continued increase in number of mesh erosions reported by patients over the past few years (FDA Public Notification, 2008).

### **2.3 Nanofiller/nanocomposite**

Composite materials are materials having two or more distinct phases such that a better combination of properties is achieved (Agarwal and Callister, 1997). The constituents must be chemically and physically different and separated by a distinct interface. The composite consists of a matrix, which is continuous and