

STUDY THE EFFECT OF SCREW SPEED AND TEMPERATURE PROFILE ON
STRUCTURE, THERMAL AND PHYSICAL PROPERTIES OF MELT BLENDED
POLYAMIDE 6/MMT NANOCOMPOSITES

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ABSTRACT

In this thesis, the effect of screw speed and temperature profile during the extrusion of polyamide 6/montmorillonite nanocomposites was discussed. The clay content (1 to 5 of weight percentage), screw speed (100 rpm to 300 rpm) and temperature barrel profile of co rotating twin-screw extruder were varied. Polyamide 6 (PA6) and montmorillonite (MMT) were melt blended under on/off and automatic heating of extrusion for a comparative study. The morphology of PA6/MMT nanocomposite was determined through the application of x-ray diffraction (XRD) and field emission scanning electron microscope (FESEM), while the thermal properties of the sample were analyzed from the thermogravimetric analysis (TGA). The differential scanning calorimetry (DSC) is used to determine the physical characteristic of PA6/MMT nanocomposite. Optimal dispersion of MMT occurred at low content of clay. Exfoliated structure was consistently obtained for PA6-1, while the collapse of *d*-spacing was mainly observed for PA6-3 and PA6-5. The presence of agglomerated MMT for PA6-5 proved the tendency of MMT in agglomeration at higher MMT content. The thermal stability of PA6/MMT nanocomposite was improved only at 1 wt. % of MMT and incorporation of more MMT reduced it further. The on/off heating method increased the thermal stability of PA6-1, while the effect was varied for PA6-3 and PA6-5. The on/off heating extruded PA6-1 possessed lower value of crystallinity but melting and crystallization temperature was measured at similar value as automatic heating extruded PA6-1.

ABSTRAK

Di dalam tesis ini, kesan kelajuan skru dan profil suhu semasa penyemperitan poliamida 6/montmorillonite nanokomposit telah dibincangkan. Kandungan tanah liat (1 hingga 5 peratus berat), kelajuan skru (100 rpm ke 300 rpm) dan suhu profil tong bersama penyemperit skru berkembar telah divariasi. Poliamida 6 (PA6) dan montmorilonit (MMT) telah diproses di bawah kaedah penyemperitan pemanasan on/off dan automatik untuk kajian perbandingan. Morfologi komposit nano PA6/MMT telah ditentukan melalui penggunaan x-ray pembelauan (XRD) dan penggunaan bidang mikroskop imbasan elektron (FESEM), manakala sifat haba sampel dianalisis daripada analisis termogravimetri (TGA). Kalorimeter pengimbasan pembezaan (DSC) digunakan untuk menentukan ciri-ciri fizikal komposit nano PA6/MMT. XRD sampel menunjukkan bahawa struktur exfoliasi telah diperolehi secara konsisten pada berat satu peratus, manakala kejatuhan saiz d terutamanya diperhatikan untuk PA6-3 dan PA6-5. Kehadiran gumpalan MMT untuk PA6-5 membuktikan kecenderungan MMT untuk pengumpulan pada kandungan MMT yang lebih tinggi. Kestabilan terma komposit nano PA6/MMT bertambah baik hanya pada satu berat peratus dan penambahan MMT pada kadar yang lebih tinggi hanya mengurangkannya lanjut. Kaedah pemanasan on/off meningkatkan kestabilan terma PA6-1, manakala untuk PA6-3 dan PA6-5 kesannya adalah berbeza-beza. Penghabluran untuk PA6-1 yang diproses semasa pemanasan on/off mempunyai nilai yang lebih rendah tetapi takat lebur dan suhu penghabluran adalah sama seperti PA6-1 yang diproses di bawah pemanasan automatik.

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CHAPTER I

BACKGROUND OF RESEARCH

1.1 INTRODUCTION

Ever since the success of full-scale commercialization of polyamide based nanocomposite from polyamide 6 and MMT in the plastic market (Kojima et al., 1993), there have been many studies by researcher. The purpose of those studies is to increase the number of polymer hybrid in the nano-scale that could be formed for industrial applications. Polyamide 6 (PA6) (Fornes et al., 2004), polyamide 66 (PA66) (Shen et al., 2004), polyamide 12 (PA12) (Phang et al., 2004), polyurethane (PUR) (Psarras et al., 2007), polypropylene (PP) (Prashantha et al., 2008), polyethylene terephthalate (PET) (Anand et al., 2007), polyethylene (PE) (Li et al., 2010), polymethylmethacrylate (PMMA) (Lee et al., 2006), polyvinyl chloride (PVC) (Awad et al., 2009), and polystyrene (PS) (Zhao and Samulski, 2006) are some synthetic polymers that could be converted into a polymer layered silicates nanocomposite (PLSN) by the addition of layered silicates in their matrices. The target in mind for the material researcher is to provide the industries with a material of different characteristic improvement to work with. So far, Europe and the USA remain as the major player in the nanocomposite market. However, the report by Global Industry Analysts, Inc (2011) predicts in the future that the Asian Pacific will join the market if based on the progress of nanotechnology knowledge among the researchers and industry community in the region.

Through an intercalation of polymer from solution, in-situ intercalative polymerization method, sol-gel method and direct melt-mixing, dispersion and distribution of layered silicates through an exfoliation mechanism could be realistically

achieved (Tanahashi, 2010). If the exfoliation step succeeds then during the synthesizing, an increase in the properties of PLSN after the addition of nano scaled MMT as an additive into the polymer could come into observation.

Generally, PLSN could be fabricated by using in-situ intercalative polymerization method or a pre-solution method, which for commercialization purpose requires a significant amount of solvent and solution (Sinha Ray and Okamoto, 2003). In contrast, a melt blending by extruder could be used to manufacture a PLSN in a mass quantity without the usage of solvent. With mechanical mixing by an extruder, there are no critical attention needed on the waste-treatment of released solution and solvent into the environment. In addition, this strategy, which could be conducted through the usage of single screw- or twin screw-extruder and even a mixer could impressively offers a degree of freedom to the plastic industries in terms of type of base polymers and nano-scaled filler since direct melt mixing is based on top-down approach. Furthermore, as extrusion is already been established as one of polymer manufacturing process and is comfortably compatible with available plastic technologies like injection molding, there should be no risk of adaption problem to be faced by industries with regarding to the economic value and 'know-how'.

Although direct mechanical mixing by an extruder could be practiced to fabricate PLSN, there are still many measurements need to be done to overcome some engineering difficulty caused by technical limit of extruder and the error as well as barrier in the strategy of direct mixing compounding. One of the factors contributed to unsuccessful attempt of manufacturing PLSN by using an extruder compounding is the thermal-degradation suffered by melt blended of PLSN due to the over-exposed of heating originated from external heat by extruder heat-plate and friction occurred between cylinder wall and material (Rauwendaal, 2010). The total processing temperature that is higher than the thermal stability of compounded organic theoretically lead to the thermal degradation (Pramoda et al., 2003; VanderHart et al., 2001).

Since dispersion mechanism of melt-compounding strategy depend mainly on the strength specific energy input, U to break the aggregated filler into nano-scale particles (Favis et al., 1990), there could be a possibility for the manufacturer to reduce

the risk of over-exposed to heat by running the melt blending of PLSN under certain processing condition of extrusion. The specific energy input (U) is defined as products of shear stress, shear strain and mean residence time as demonstrated by Eq. (1.1) (Rauwendaal, 1981), where σ_{12} is the shear stress, $\dot{\gamma}$ is the shear strain, and \bar{t} is the mean residence time. From the equation, it is evidently shows that shears stress, shear strain and mean residence time are directly proportional to specific energy input.

$$U \approx \sigma_{12} \dot{\gamma} \bar{t} \quad (1.1)$$

The manual thermal control of extrusion used during this research is comparable with the on/off controller effect for heating and cooling system of extruder. The power to the extruder would be switched off if the actual temperature is above the set point, while completely on if the actual temperature is below the set point (Rauwendaal, 2010). The heating system of extruder was manually controlled to secure the melt blending of PA6/MMT at temperature well below melting point of PA6. This approach is believed could minimize the risk of thermal degradation for compatibilizer in MMT due to the low melt temperature. In contrast, the automatic heating control system that was used throughout this work employs fuzzy logic system to enhance thermal performance of extruder. Since this fuzzy logic system is focusing more on barrel set temperature control rather than melt temperature, there is a risk that thermal degradation of organic modifier of MMT might still occurs when the melt temperature exceeding the onset degradation temperature of organic component in MMT (Abeykoon et al., 2011). Generally, it was found from the previous works that the different process setting and melt flow temperature could affect the thermal homogeneity of molten compound (Abeykoon et al., 2012; Abeykoon et al., 2011; Kelly et al., 2006; Brown et al., 2004; Rasid and Wood, 2003). As a result, a change in the thermodynamic stability of PLSN could be observed if the processing parameters involved during an extrusion are varied (Paci et al., 2010). In this research, the focus is more on the influence of processing condition on the characteristic of PA6/MMT nanocomposites rather than the advantages and disadvantages possessed by different type of temperature controller. Thus, during the melt blending of PA6/MMT nanocomposites, the thermal control of used extruder would be alternated between manual (on/off) and automatic to observe the effect of this processing condition on the properties of PA6/MMT nanocomposites.

This thesis is written to provide the reader a chance to look into the effect of processing condition on the manufacturing aspect of PLSN. Simultaneously, the explanation about the influences of shear intensity and temperature barrel profile of twin screw extruder under on/off and fuzzy thermal control of extrusion on the dispersion behavior of layered silicate in the polyamide-based material would be in detail structured. PA6 was preferred as matrix polymer in this research primarily because of the accessibility of this material to the plastic industry besides its naturally already good mechanical, chemical and thermal properties while MMT which belongs to a group of layered silicate, acts as nano scaled filler for the composite. The change in the thermal stability of manual thermal control extruded sample of PA6/MMT and the exfoliation rate of platelets including the crystalline behavior of the PA6/clay after the addition of layered silicates into the pristine polymer since the effectiveness of nano additives are discussed further in chapter 4.

1.2 PROBLEM DESCRIPTION

In discussing the strategy of PLSN fabrication by direct mechanical mixing or melt blending, the thermal degradation suffered by organic modified MMT during the extrusion has been strongly affected the properties of extruded PLSN. The decomposition and degradation of materials involved during the compounding processing of PLSN could be rooted to thermal degradation due to many factors involved. An excessive heat originated from high shear stresses produced by high speed of screw and friction between material and extruder's inner wall with the heat from extruder heating plate that simultaneously flow into the compounded material is recognized as one aspect that would lead to the thermal breakdown of PLSN. The combined effect of shear stress and temperature in blending is concluded as an issue that causes degradation in PLSN, not just a temperature alone (VanderHart et al., 2001).

The low thermal stability of the component of organic modified layered silicate (OMLS) mainly alkyl ammonium compound is also reported to be contribute to the degradation of PNC. From the thermogravimetric analysis (TGA), the breakdown of the component of OMLS is recorded at 155 °C under non-oxidative environment and at a higher temperature; a major thermal degradation has being observed (Fornes et al., 2003). Due to the chemical change in the structure of surfactant during the decomposition, the thermodynamic stability of melt intercalation could has been interrupted and consequently the level of exfoliation of platelets as well as the interfacial bonding could be affected and this leads to the low mechanical and physical properties of PNC. A decomposition of OMLS that contains dimethyl dehydrogenated-tallow ammonium ion released a free amine that consisted of one methyl and two-tallow substituent. The degradation of surfactant itself may bring an undesirable chemical side effect during the compounding (Xie et al., 2001).

Besides a thermal degradation, a mechanical decomposition that occurred during the compounding is initiated by a too high strength stresses of screw could resulting in a molecular weight reduction of polymer as well. The rheological properties of PLSN could be affected and as mentioned by Cho and Paul (2001) and Dennis et al. (2000; 2001) in their works, the melt processing condition that leads to the degradation of polymer could significantly affects the exfoliation rate of OMLS.

1.3 RESEARCH OBJECTIVE

The main target of this research is to study on the influence of extrusion processing condition to the thermal stability and the structure of melt blended PLSN. The superiority of PLSN is measured indirectly from the MMT dispersion in the matrices, which could be determined by employing thermal and morphological characterization. Since the thermal behaviour, crystallinity and dispersion performance of PNC could be significantly influenced by a certain processing parameter during a melt compounding of extruder (Dennis et al., 2000, 2001), a variation of temperature profile and screw speed is used to investigate the change of thermal stability, morphology and crystalline behavior of PA6/MMT nanocomposites after the varied processing condition of extrusion. The correlation between the extruder processing parameter and the dispersion rate in the PLSN would be examined in order to draw the optimum and reproducible processing parameter that could be conducted in order to fabricate and replicate the high performance grade PLSN. The following aspects have been focused in this research:

1. To evaluate the effect of screw speed and temperature profile on the morphology of melt blended PA6/MMT nanocomposites
2. To study the effect of screw speed and temperature profile on the thermal stability of melt blended PA6/MMT nanocomposites
3. To outline the influence of screw speed and temperature profile on physical structure of melt blended PA6/MMT nanocomposites

1.4 SCOPES OF RESEARCH

By underlining the step taken by author to achieve the objective of the proposed research, a general scope or an experimental framework of this thesis was established. Following are the designed scopes that assisted and guiding the author to the achievement of research objective as well as completion of this thesis:

Phase I

1. The preparation of PA6/MMT nanocomposites was conducted on the co- rotating twin screw extruder due to the better mixing performance than single screw extruder.
2. Polyamide 6 (PA6) was selected as main polymer and organic modified MMT is the nano additive.
3. During the preparation of PA6/C20A nanocomposites, clay content, a series of screw speed (100 rpm, 200 rpm and 300 rpm) and temperature barrel profile of extruder (from 195 °C to 224 °C are varied).

Phase II

1. The thermal stability of collected samples PA6/MMT is investigated by using thermogravimetric analysis (TGA).
2. The structure of PA6/MMT is objectively characterized by using x-ray diffraction (XRD) and visually interpreted through the application of field emission scanning electron microscopy (FESEM).
3. By utilizing differential scanning calorimetry (DSC), the physical structure of PA6/C20A changes after certain extrusion condition are outlined.

1.5 SIGNIFICANCE OF RESEARCH

In order to reduce the risk of thermal degradation of compounded material through a twin-screw extruder, the melt blending of polyamide 6/clay nanocomposite should be operated under certain processing condition. The dispersion mechanism of twin-screw extruder, the dispersive and distributive flow of molten PLSN respectively is relied heavily on the specific energy input, U , as the products of a shear stress, shear strain and a mean residence time (Favis et al., 1990). From this observation, it is hypothesized that an on/off control of extrusion does not give any negative influence on the dispersion pattern of extruded PA6/MMT nanocomposites.

However, the effectiveness of an adiabatic extrusion by twin-screw extruder should be examined in order to study the influence of adiabatic melt mixing on the thermodynamic stability of PLSN. Due to the accessibility of extruder, which is commonly used in plastic sector/industry to manufacture conventional micro- or macro composite, it is an understandable advantage to use a direct melt-mixing by a twin-screw extruder during the fabrication of PLSN because of the good compatibility with an already established technology like injection molding. The lack of specialist needed to operate an extruder is also another benefit of this approach. Thus, the accumulated result from this research reckoned to be important for us due to the listed factors below:

1. To provide a future reference for the organization or person that interested in melt blending of PLSN by twin-screw extruder.
2. To establish the effect of extrusion under different thermal control approach on the properties of PLSN.
3. To synthesize a PA6 based nanocomposites with higher thermal stability for industrial application.

1.6 SUMMARY

Direct melt mixing by twin-screw extruder is presented as an alternative strategy to the in-situ intercalative polymerization, template synthesis and solution intercalation method in the assembly of PA6/MMT nanocomposites. The usage of solution and/or solvent could be eliminated, which is the main advantage of this approach. However, it is important to remember that physical bonding which occurs between polymer and nano-scaled filler during the melt-compounding may or may not produce nanocomposite. As a result, a screw speed and a residence time of PA6/MMT nanocomposite in an extruder are to be considered as an important parameter due to the impact of shear stresses and elongation flow created to the dispersion and distribution progress of platelets in polymer matrix. In the research, an on/off thermal control was conducted by running the twin-screw extruder with previously heated element plate of extruder that has just been off-switched. The purpose of this approach is to secure a melt blending of PA6/MMT at temperature below melting point of PA6 as the risk of thermal degradation for organic component in MMT could be reduced. A series of pre-determined process parameter of twin-screw extruder would varied in order to observe the effect of extruder processing variable under adiabatic on the thermal stability and dispersion behavior of PA6/MMT nanocomposite. The extruded polymer nanocomposite would then be pelletized, collected and packaged for an intended thermal, morphology and physical characterization later.

CHAPTER II

LITERATURE REVIEW AND DISCUSSION

2.1 INTRODUCTION

The preparation of PLSN through direct melt-mixing route tends to expose the degradation and decomposition risk for organic component in OMLS. Normally, the processing temperature would be higher than the thermal stability of organoclay allowed due to the combination of shear stress, melt viscosity and barrel temperature during the compounding. The decomposition of surfactant in the polymer matrices would induce modification in the properties of melt-mixed PLSN as a result of change of chemical structure of OMLS (Leszczyńska et al., 2007). Therefore, it is essential for researcher to develop other strategy of melt-intercalation of PLSN that can be utilized to reduce the thermal degradation and decomposition rate of organic modifier of OMLS during the high-melting temperature polymer processing (above 180°C). In this chapter, the literatures that are relevant for this current research work were discussed as the general characteristic of layered-silicates (LS) and PLSN would be included. In addition, the analysis techniques used for the characterizing of PA6/MMT nanocomposite in the later stage of this research work would also be academically explained.

2.2 POLYMER NANOCOMPOSITE

The fabrication of polymer based nanocomposite gains its greater impetus and significant attention from academician, scientist and governments via the breakthrough of research by Toyota Research Centre & Development in the early 90s (Kojima et al., 1993). In addition, Giannelis and co-workers have made an important discovery; where after a series of conducted experiment had deduced that the possibility of fabrication of nanocomposite by melt intercalation could be actually realized by application of melt compounding (Giannelis et al., 1990). Polymer-nanocomposite term itself is further elaborated by Utracki (2004), who explains the definition of a polymer-nanocomposite as a composite that demonstrate a dispersion of nano-particle in the matrices of polymer with one dimension of this particle must be less than 100 nm in size.

In discussing the level of progress for polymer-nanocomposite, it is not really quite a speculation to any person that routinely followed the development of nano-material, when published journal and scientific paper that related to the field of nanotechnology are continuously increasing from year after year (Maniara and Krisnakant, 2004). The growing number for literatures involved with the research of polymer-nanocomposite is directly demonstrates the constant exploring of knowledge in nanocomposite field by experts in order to understand the properties, characteristic and best fabrication approach especially in the field of polymer nanocomposite.

Nowadays, a layered silicate is regarded as a popular and favorite nano-additive to be used during the fabrication of polymer nanocomposite by the industries due to the abundance number of silicates in nature as well as economically manufacturing price. The phosphates, manganates, titanates, cement hydrates, bidimensional zeolites and silicic acid are other materials that have same-layered properties like layered silicates that belong to the group of phyllosilicates and also used as nanofillers (Annand-Bergaya, 2008). Although a conventional polymer-composite is widely used and already established its role in the broad spectrum of consumerism whether by industries or even by a housewives , the higher loading of additives required by this composite in order to achieve the same characteristic as a nanocomposite clearly shows the weakness of unadventurous feature of ordinary composite. This higher contain of additives more

CHAPTER I

BACKGROUND OF RESEARCH

1.1 INTRODUCTION

Ever since the success of full-scale commercialization of polyamide based nanocomposite from polyamide 6 and MMT in the plastic market (Kojima et al., 1993), there have been many studies by researcher. The purpose of those studies is to increase the number of polymer hybrid in the nano-scale that could be formed for industrial applications. Polyamide 6 (PA6) (Fornes et al., 2004), polyamide 66 (PA66) (Shen et al., 2004), polyamide 12 (PA12) (Phang et al., 2004), polyurethane (PUR) (Psarras et al., 2007), polypropylene (PP) (Prashantha et al., 2008), polyethylene terephthalate (PET) (Anand et al., 2007), polyethylene (PE) (Li et al., 2010), polymethylmethacrylate (PMMA) (Lee et al., 2006), polyvinyl chloride (PVC) (Awad et al., 2009), and polystyrene (PS) (Zhao and Samulski, 2006) are some synthetic polymers that could be converted into a polymer layered silicates nanocomposite (PLSN) by the addition of layered silicates in their matrices. The target in mind for the material researcher is to provide the industries with a material of different characteristic improvement to work with. So far, Europe and the USA remain as the major player in the nanocomposite market. However, the report by Global Industry Analysts, Inc (2011) predicts in the future that the Asian Pacific will join the market if based on the progress of nanotechnology knowledge among the researchers and industry community in the region.

Through an intercalation of polymer from solution, in-situ intercalative polymerization method, sol-gel method and direct melt-mixing, dispersion and distribution of layered silicates through an exfoliation mechanism could be realistically

achieved (Tanahashi, 2010). If the exfoliation step succeeds then during the synthesizing, an increase in the properties of PLSN after the addition of nano scaled MMT as an additive into the polymer could come into observation.

Generally, PLSN could be fabricated by using in-situ intercalative polymerization method or a pre-solution method, which for commercialization purpose requires a significant amount of solvent and solution (Sinha Ray and Okamoto, 2003). In contrast, a melt blending by extruder could be used to manufacture a PLSN in a mass quantity without the usage of solvent. With mechanical mixing by an extruder, there are no critical attention needed on the waste-treatment of released solution and solvent into the environment. In addition, this strategy, which could be conducted through the usage of single screw- or twin screw-extruder and even a mixer could impressively offers a degree of freedom to the plastic industries in terms of type of base polymers and nano-scaled filler since direct melt mixing is based on top-down approach. Furthermore, as extrusion is already been established as one of polymer manufacturing process and is comfortably compatible with available plastic technologies like injection molding, there should be no risk of adaption problem to be faced by industries with regarding to the economic value and 'know-how'.

Although direct mechanical mixing by an extruder could be practiced to fabricate PLSN, there are still many measurements need to be done to overcome some engineering difficulty caused by technical limit of extruder and the error as well as barrier in the strategy of direct mixing compounding. One of the factors contributed to unsuccessful attempt of manufacturing PLSN by using an extruder compounding is the thermal-degradation suffered by melt blended of PLSN due to the over-exposed of heating originated from external heat by extruder heat-plate and friction occurred between cylinder wall and material (Rauwendaal, 2010). The total processing temperature that is higher than the thermal stability of compounded organic theoretically lead to the thermal degradation (Pramoda et al., 2003; VanderHart et al., 2001).

Since dispersion mechanism of melt-compounding strategy depend mainly on the strength specific energy input, U to break the aggregated filler into nano-scale particles (Favis et al., 1990), there could be a possibility for the manufacturer to reduce

the risk of over-exposed to heat by running the melt blending of PLSN under certain processing condition of extrusion. The specific energy input (U) is defined as products of shear stress, shear strain and mean residence time as demonstrated by Eq. (1.1) (Rauwendaal, 1981), where σ_{12} is the shear stress, $\dot{\gamma}$ is the shear strain, and \bar{t} is the mean residence time. From the equation, it is evidently shows that shears stress, shear strain and mean residence time are directly proportional to specific energy input.

$$U \approx \sigma_{12} \dot{\gamma} \bar{t} \quad (1.1)$$

The manual thermal control of extrusion used during this research is comparable with the on/off controller effect for heating and cooling system of extruder. The power to the extruder would be switched off if the actual temperature is above the set point, while completely on if the actual temperature is below the set point (Rauwendaal, 2010). The heating system of extruder was manually controlled to secure the melt blending of PA6/MMT at temperature well below melting point of PA6. This approach is believed could minimize the risk of thermal degradation for compatibilizer in MMT due to the low melt temperature. In contrast, the automatic heating control system that was used throughout this work employs fuzzy logic system to enhance thermal performance of extruder. Since this fuzzy logic system is focusing more on barrel set temperature control rather than melt temperature, there is a risk that thermal degradation of organic modifier of MMT might still occurs when the melt temperature exceeding the onset degradation temperature of organic component in MMT (Abeykoon et al., 2011). Generally, it was found from the previous works that the different process setting and melt flow temperature could affect the thermal homogeneity of molten compound (Abeykoon et al., 2012; Abeykoon et al., 2011; Kelly et al., 2006; Brown et al., 2004; Rasid and Wood, 2003). As a result, a change in the thermodynamic stability of PLSN could be observed if the processing parameters involved during an extrusion are varied (Paci et al., 2010). In this research, the focus is more on the influence of processing condition on the characteristic of PA6/MMT nanocomposites rather than the advantages and disadvantages possessed by different type of temperature controller. Thus, during the melt blending of PA6/MMT nanocomposites, the thermal control of used extruder would be alternated between manual (on/off) and automatic to observe the effect of this processing condition on the properties of PA6/MMT nanocomposites.

CHAPTER III

RESEARCH METHODOLOGY

3.1 INTRODUCTION

The grade of polymer as well as type of organoclay used during this research would be described in this chapter. In addition, the varied process parameter during the on/off and automatic heating of extrusion and screw configuration of twin-screw extruder would also be included to outline the change of processing condition taken in order to study the effect of extrusion system on the properties of PA6/MMT nanocomposite.

3.2 MATERIALS

During the preparation of PLSN by varied extrusion processing condition, an industrial grade of polyamide 6 (PA6) was used as the polymer matrix while marketed organic modified MMT under the registered name of Cloisite[®]20A (C20A) would be incorporated as nano scaled filler. The characteristic and properties of PA6 and MMT would be explained below.

3.2.1 Polyamide 6 (PA6)

In order for this research to be relevant to the industry, a commercial grade of PA6 under the trademark name of ZISAMIDE®TP210 and was manufactured by Zig Sheng Industrial Co. Ltd.(China) was employed as the polymer base in this study. The melt flow index is valued at 172 g/min while the melting temperature was confirmed at 224 °C from DSC measurement. The physical and chemical features of PA6 are listed in the Table 3.1.

Table 3.1: Physical and chemical properties of PA6

Physical state	solid
Form	granules
Colour	undyed
Odour	odourless
Specific gravity (g/cc)	1.14
Ignition temperature (°C)	Min. 395 °C
Melting point (°C)	220-225°C
Solubility in water	insoluble
Density at 20 °C (kg/m ³)	1130 kg/m ³

3.2.2 Montmorillonite (MMT)

In this thesis, MMT was selected as nano scaled filler. The MMT was supplied by Southern Clay Products (Rockwood additives, Ltd). The modifier concentration for this type of organic-modified nanoclay is determined to be at 95 mequiv/ 100g of clay, whereas for MMT with registered name, C30B, the concentration of modifier is found to be less (90 mequiv/100g of clay). The higher values of modifier concentration in selected MMT might work in both ways, as this positively could lead to the better exfoliation rate of nanoclays platelets in the host polymer or negatively; increase the thermal degradation rate of PA6/MMT nanocomposites due to the great number of low thermal stability of alkyl ammonium tails. The composition of the hydrogenated tallow alkyl chains was agreed on ~65 % C18; ~30 % C16; ~5 % C14. A Cloisite 20A is recognized as $M_2 (HT)_2$, where M represents methyl while HT is for hydrogenated tallow, respectively. The physical and chemical traits of selected MMT would be listed in the Table 3.2 while the chemical arrangement of the modified MMT would be portrayed in Figure 3.1 respectively.

Table 3.2: physical and chemical attributes of selected MMT

Physical state	solid
Form	powder
Colour	off-white
Odour	odourless
Specific gravity (g/cc)	1.77
Auto-ignition temperature (°C)	190 °C thin film ignition
Solubility in water	insoluble
Dry particle sizes	2-13 μ m