ISOTHERMAL CRYSTALLIZATION STUDY AND MECHANICAL PROPERTIES OF POLYPROPYLENE/POLYSTYRENE/STYRENE-ETHYLENE-BUTADIENE-STYRENE BLENDS

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I want to dedicate this thesis to my parents, family, husband and my lovely childrens Amnah and Ammar. Without their help, understanding and encouragement it simply never would have been. Thanks for the love.

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ABSTRACT

An understanding of the crystallization process kinetics is important for the selection of processing parameters and product properties control. In this study, the isothermal crystallization of polypropylene/polystyrene/styrene-ethylene-butadiene-styrene (PP/PS/SEBS) blends were evaluated using differential scanning calorimetry (DSC). The blends were prepared by using Brabendar twin screw extruder and samples were then injection molded. 10-15 mg samples were quenched from the melt stage to a range of crystallization temperatures (Tc) between 134-138 ℃. That samples were held at these selected temperatures until the calorimeter response returned to the baseline. Isothermal crystallization data were then analyzed using *Avrami* kinetics model and *Hoffman-Weeks* theory. The increase of amorphous PS and SEBS content in system blends led to the decreases in the crystallization rate. The region covered for Avrami exponent, n, was between 1.97 to 3.2 (±0.1), indicating that heterogeneous nucleation of spherulites occurred and the growth of spherulites was between two-dimensional and three-dimensional for primary crystallization process of the blends. Equilibrium melting temperature ($Tm°$) obtained through *Hoffman-Weeks* theory was found to decrease with increasing PS and SEBS contents. The presence of amorphous polymer could disrupt the packing of the crystalline component in the blend, resulting in defective, lower melting point crystallites. The mechanical properties determined from tensile, flexural and impact test revealed that loading of SEBS gave a synergistic effect on flexural properties, and higher loading of SEBS produced superior impact properties. The best stiffness–toughness balance was obtained from the blend of PP/PS 90/10 at 5 phr SEBS loading which also gave fastest crystallization. Morphology studies by scanning electron microscope (SEM) showed finer dispersion of PS with the presence of SEBS into PP/PS blends. SEBS confirmed the improvement of blend compatibility.

ABSTRAK

Pemahaman tentang kinetik proses penghabluran adalah penting bagi memilih parameter pemprosesan dan kawalan sifat produk. Kajian ini membincangkan penghabluran isoterma adunan polipropilena/polistirena/stirena-etilina-butadiena-stirena (PP/PS/SEBS) yang dinilai menggunakan kaedah pengimbasan kalorimeter kebezaan (DSC). Adunan disediakan menggunakan mesin penyemperit skru berkembar berjenama Brabendar diikuti penghasilan sampel secara acuan suntikan. 10-15 mg sampel telah disejukkan secara cepat dari tahap leburan kepada julat suhu penghabluran (Tc) antara 134-138 ℃. Suhu dipilih dibiarkan sehingga kalorimeter kembali ke garis dasar. Data yang diperoleh dari kajian penghabluran isoterma dianalisa menggunakan model kinetik *Avrami* dan teori *Hoffman-Weeks*. Peningkatan kandungan amorfus PS dan SEBS ke dalam sistem adunan mengakibatkan penurunan kadar penghabluran. Eksponen Avrami, n, di antara 1.97 to 3.2 (±0.1) menandakan berlakunya penukleusan heterogenus sferulit berlaku dan pertumbuhan sferulit adalah di antara dua dimensi dan tiga dimensi untuk proses penghabluran utama adunan. Keseimbangan suhu lebur $(Tm°)$ yang diperoleh daripada teori *Hoffman-Weeks* didapati mengurang dengan peningkatan PS dan kandungan SEBS. Kehadiran polimer amorfus boleh mengganggu dan merencatkan padatan kristal dalam campuran dan suhu lebur yang lebih rendah diperoleh. Keputusan sifat mekanikal daripada ujian regangan, lenturan dan hentaman menunjukkan penambahan SEBS pada skala kecil mengakibatkan kesan sinergi pada sifat lenturan manakala penambahan pada skala besar memberi kesan sifat unggul pada kekuatan hentaman. Keseimbangan kekakuan-kekuatan yang terbaik diperoleh daripada adunan PP/PS 90/10 pada kandungan 5 phr SEBS yang juga memberi masa penghabluran terpantas berbanding adunan lain. Kajian morfologi dari SEM menunjukkan kehadiran SEBS menjadikan fasa penyebaran PS baik dan sekata. SEBS terbukti meningkatkan keserasian dalam adunan.

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LIST OF ABBREVIATIONS

ASTM	-	American society for testing and materia*ls*
AVM	-	Aromatic vinyl monomer
DSC	-	Differential scanning calorimeter
EB	-	Ethylene-butadiene
EOC	-	Ethylene–octene copolymer
EPDM	-	Ethylene propylene diene monomer
EPR	-	Ethylene-propylene rubber
GPC	-	Gel permeation chromatograph
h	-	Hour
HDPE	-	High density polyethylene
HDPE	-	High density polyethylene
iPP	-	Isotactic polypropylene
LLDPE	-	Linear low density polyethylene
MAH	-	Maleic anhydride
MFI	-	Melt flow index
min	-	Minute
mLLDPE	-	Metal-locene-catalyzed linear low density polyethylene
mPP	-	Maleic anhydride grafted PP
MRSF	-	Modified rape straw flour
PC	-	Polycarbonate
PE	-	Polyethylenes
PES	-	Poly(ether sulphone)
PET	-	Polyethylene terepthalate
phr	-	Parts per hundred ratio
PMMA	-	Poly (methyl methacrylate)

PP	-	Polypropylene
PPA	-	Aromatic vinyl monomer-grafted PP
PPE	-	Polyphenylene ether
PP-g-PS	-	Polypropylene grafted polystyrene
PPS	-	Poly (phenylene sulfide)
PS	-	Polystyrene
PTFE	-	Polytetrafluoroethylene
rpm	-	Revolution per minute
SAXS	-	Small angle X-ray scattering
SBS	-	Styrene-butadiene-styrene
SEBS	-	Styrene-ethylene-butadiene-styrene
SEM	-	Scanning electron microscope
SMI	-	Styrene maleimide
t	-	Time
TEM	-	Transmission electron microscopy
THF	-	Tetrahydrofuran
UV	-	Ultraviolet
Tc	-	Crystallization temperature
Tg	-	Glass temperatutre
Tm	-	Melting temperature
$Tm°$	-	Equilibrium melting temperature

LIST OF SYMBOLS

%	-	Percent
ΔHm	-	Heat of melting
cm	-	Centimeter
g	-	Gram
J	-	Joule
k	-	Crystallization rate constant
K	-	Kelvin
Kg	-	Nucleation constant
kg	-	Kilogram
kV	-	kilovott
m	-	Meter
mg	-	miligram
mm	-	millimeter
MPa	-	MegaPascal
n	-	Avrami exponent
^{o}C	-	Degree Celsius
s	-	second
$t_{1/2}$	-	Crystallization half-life
wt %	-	Weight percent
Xt	-	Relative crystallinity
Xc	-	Degree of crystallinity

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CHAPTER 1

INTRODUCTION

1.1 Background of the study

The commercial significance of semi-crystalline polypropylene (PP) is well known and confirmed by a large number of producers throughout the world [1] . PP is commodity polymer most widely used in different applications such as automotive, packaging and insulator [2]. This thermoplastic was highly consumption because of its well-balanced physical and mechanical properties and their easy processability at a relatively low cost. However, it exhibits a relatively low modulus and stiffness compared to engineering plastic [3]. Semi-crystalline polymers like PP exhibit both crystalline and amorphous phases. PP structure and properties are strongly dependent on the tacticity or the stereoregularity of the polymeric chains. Isotactic polypropylene (iPP) is a rigid thermo-plastic with stereoregular structures competent to form helices and pack into crystals, resulting in highest crystallinity. Due to irregular structure, atactic polypropylene (aPP) is an amorphous gum-like polymer. The aPP form has lowest crystallinity and sydiotactic polypropylene (sPP) crystllinity are between them [4].

Reinforcement presence in PP matrix will influences the crystalline morphology. This is because the crystalline morphology affects the mechanical properties of PP [5]. The structure of semi-crystalline polymer also was controlled by the mechanism and crystallization kinetics [6]. Crystallization occurs during manufacturing of the polymeric material products. The understanding of its

crystallization kinetics and process conditions is very essential for end product property control and design of processing operation [3]. A faster crystallization process will lead to a shorter fabrication time consumed in the injection molding machine, where it will also directly lower the end part cost [2]. Therefore, research on the crystallization behavior is most significant not only theoretically but also practically. It is very attractive to study the crystallization kinetics in order to understand the properties of processed products and indirectly will optimize the blend composition.

The crystallization kinetics of PP or other single polymer component has been studied [4, 7-10]. Studies on crystalline/crystalline blend system also was being explored and discuss previously such as high-density polyethylene (HDPE)/linear low-density polyethylene (LLDPE), LLDPE /modified polypropylene [11] and poly(ethylene terephthalte)/poly(trimethylene terephthalate) blends (PET/PTT) [12]. It is intriguing to study the crystallization behavior of blends between iPP which is semi-crystalline with amorphous PS. This semi-crystalline/amorphous system is much easier to study since only one polymer component shows crystals rather than crystalline-crystalline blends system. Nevertheless, PP/PS blend is immiscible because of unfavorable molecular interactions of the structural difference between the dispersed phase and the continuous phase.

Raghu *et al.* [13], found that for the uncompatibilized 70/30 PP/PS blends, the morphology seemed to be distinguished by a large domain size of the dispersed phase with relatively large particles protruding from the matrix. Researchers have studied that styrene-ethylene-butadiene-styrene (SEBS) is one of the effective compatibilizers, as the styrene and ethylene butylene (EB) blocks of SEBS are miscible with PS and PP [14-17]. It has been proven by a previous study by Halimahtudaliana *et al.* [18], which reported that cavities of smaller and more uniform size can be obtained by adding SEBS to this PP rich blend. Toughness of the PP matrix was increased by adding SEBS elastomer, which can also as impact modifier.

Crystallization studies of uncompatibilized PP/PS blends were reported by Adewole *et.al* [14]. Crystallinity decreased with increasing PS content in the blends, probably due to kinetic limitation to crystal growth imposed by the presence of the PS. Melting temperature of PP decrease to the diluents effect of PS. PP spherulite size also decreases as the PS concentration increases [19]. Primary nucleation process of the PP/PS blends facilitates with addition of non-crystallizable component such as, EPR and SEBS. It increased the level of crystallinity of blends [14]. In a study on crystallization of PP/SEBS blends, Gupta *et. al* [20] concluded that at low loading SEBS induces a decrease in crystallinity and an increase in spherulite size. In the region of higher SEBS content crystallinity decreases while spherulite size increases. There were reduction in enthalpy of melting (ΔHm) and enthalpy of crystallization (ΔHc) of compatibilized PP/PS blends with incorporation of styrene-isoprene-styrene (SIS), styrene-butadiene-styrene (SBS) and styrene-butadiene-rubber (SBR) [13].

Isothermal crystallization kinetics that is related to semi-crystalline of PP blends was investigated by other researchers. Jianglei Qin *et.al* [1] analyzed the isothermal crystallization kinetics using Avrami equation of the polypropylene (PP)/metallocene-catalyzed linear low density polyethylene (mLLDPE) blends. Avrami exponent shows the heterogeneous crystallization nucleation of the blends, the enlargement of spherulites is nearly three-dimensional, and the crystallization mechanism of PP is not affected greatly by mLLDPE. The Avrami exponents from the blends are higher compared to pure PP because mLLDPE helps PP to form perfect spherulites. The crystallization rates of PP are reduced because the addition of mLLDPE will decrease the crystallization temperature of PP [21]. PP resulted in melting-point depression at lower high density polyethylene (HDPE) contents. HDPE was able to penetrate the PP phase sufficiently at lower HDPE contents to reduce the number and size of regions of high segment density, thus delaying the nucleation and subsequent crystallization of the PP phase [22].

Jean Hong Chen and Yu-Lun Chang [23] reported on isothermal crystallization of iPP and aPP. They concluded that with small amounts of a aPP, the aPP molecules promoted the mobility of iPP molecues and reduced entanglement

between iPP molecules. It led to an increasing crystallization ability of iPP, whereas with larger amount of aPP, the decrese in Xc of the iPP blends. This may have been due to the larger amount of diluents aPP action that suppresses the amount of nucleus. The isothermal crystallization kinetics of PP and polyamide (PA) blends at composition 70/30 have been studied with and without the addition of maleic anhydride functionalized polypropylene at loading 1, 3, 5 and 10% by weight percent. The reported result gave in reducing crystallization rate because of the PA act as diluents effect component that influenced by the presence of PP [24]. On the contrary, the crystallization rate of PP in the blends is higher than the corresponding value for pure PP. This effect is higher in the blends without compatibilizer. This behaviour is related to the nucleating activity by the PA component.

The mechanical, morphology and rheological properties of PP/PS/SEBS blends have been reported by other researchers [7, 25-27]. However, the literature reports no previous work on the isothermal crystallization kinetic analysis for these blends i.e PP/PS/SEBS. Therefore, in the present work, efforts have been made to study the crystallization kinetics in terms of blends composition and SEBS loading at 5 and 25 phr loading. The crystallization kinetics is important in order to control rate of crystallization and determine change in material properties of various composition polymer blends. Isothermal crystallization kinetics was analyzed according to Avrami's equation. While, Hoffman-Weeks theory was applied to determine equilibrium melting temperature ($Tm°$).

1.2 Problem Statement

Crystallization study on single PP and uncompatibilize PP blends were reported previously [14, 23]. However, there is no study being reported on compatibilized PP blend based on the effect of compatibilizer (i.e. SEBS) loading and blends compositions towards PP's crystallization kinetics. It is known that mechanical properties of the blend samples were determined by crystallization rate. Therefore, crystallization study is very interesting and significant to investigate since it has potential to control the properties of end product.

The focus of this study is to investigate PP/PS/SEBS blends under isothermal crystallization kinetics and analyzed using Avrami approach. This study provided further information on crystallization kinetics of compatibilized semi-crystalline-amorphous blend systems and also the relationship between the crystallization kinetics and the mechanical properties of the blends.

1.3 Research objectives

The most important aim of this research is to study the isothermal crystallization kinetics of PP/PS/SEBS blends and relationship on its mechanical properties. The overall objective can be further divided into:

1. To investigate the equilibrium melting temperature of PP/PS/SEBS blends using Hoffman-Weeks approach and isothermal crystallization kinetics by Avrami's theory.

2. To study the effect of blend compositions, SEBS loadings and relationship of crystallization kinetics on the mechanical properties of PP/PS/SEBS blends.

1.4 Scope of the research

The scope of this research:

i. Dynamic scanning test by using the DSC which determines the thermal parameters of melting temperature (Tm) and crystallization temperature (Tc).

ii. Conducting isothermal crystallization test using DSC to observe the crystallization kinetics and analyzing it by using Avrami and Hoffman-Weeks theory.

iii. Sample preparation of PP/PS (wt%) blends with SEBS loading at 5 and 25 phr, and increment interval of PS about 10% of each interval by using Brabendar twin screw extruder.

iv. Investigate the mechanical properties on tensile (ASTM D638), flexural (ASTM D790), impact test (ASTM D256) and morphology throughout scanning electron microscope **(**SEM).

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