

**EVALUATION OF COMPOUNDING TECHNIQUES FOR
SHORT FIBERS REINFORCED POLYCARBONATE**

Onuma Charoensuk

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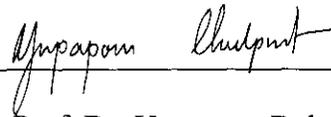
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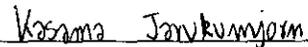
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งานวิจัยนี้ พอลิคาร์บอเนตเสริมแรงด้วยเส้นใย 3 ชนิดเตรียมโดยเครื่องบดผสมภายในเครื่องผสมแบบแกนเดี่ยวและแกนคู่ และการบดผสมโดยใช้เครื่องมือร่วมกัน ระบบอีพอกซีกับตัวทำแข็งอีดีเอสไอใช้เป็นสารช่วยปรับปรุงความเข้ากันได้ระหว่างเส้นใยกับพอลิคาร์บอเนต ผลการทดลองพบว่า ความยาวของเส้นใยในพอลิเมอร์ผสมไม่ขึ้นกับความยาวของเส้นใยเริ่มต้นทั้งระบบที่มีการใช้หรือไม่ใช้ระบบอีพอกซีเป็นสารช่วยปรับปรุงความเข้ากันได้ และการใช้เครื่องบดผสมภายในทำให้ขนาดของเส้นใยมีขนาดสั้นที่สุด ในขณะที่เครื่องผสมแบบแกนเดี่ยวทำให้ขนาดของเส้นใยยาวที่สุด

การใช้ระบบอีพอกซีที่ 1% ในการผสมพอลิคาร์บอเนตโดยใช้เครื่องบดผสมแบบแกนคู่พบว่า อุณหภูมิการบดงอมมีค่าลดลงแต่คุณสมบัติเชิงกลอื่นๆ มีค่าเพิ่มขึ้นและดีกว่าพอลิคาร์บอเนตที่ไม่มีอีพอกซี จากการทดสอบวิเคราะห์เชิงสถิติยืนยันผลการทดลองที่ได้อีกครั้ง และผลการทดลองยังพบอีกว่า เครื่องมือผสมแบบแกนคู่เป็นเครื่องมือผสมที่เหมาะสมที่สุด คุณสมบัติของพอลิคาร์บอเนตผสมจะมีค่าแตกต่างกันเมื่อใช้เครื่องมือผสมต่างกัน ผลการเสริมแรงพอลิคาร์บอเนตด้วยเส้นใยคาร์บอน เส้นใยเคฟลา และเส้นใยแก้วพบว่า ปริมาณเส้นใยที่ผสมที่เหมาะสมคือ เส้นใยคาร์บอน 5% เส้นใยเคฟลา 10% และเส้นใยแก้ว 15% โดยน้ำหนัก ทั้งระบบการผสมที่มีและไม่มีการช่วยปรับปรุงความเข้ากันได้อีพอกซี

เครื่องผสมแบบแกนคู่เป็นเครื่องผสมที่ให้ประสิทธิภาพการผสมเส้นใยกับพอลิคาร์บอเนตที่ดีที่สุด ซึ่งผลการทดลองสนับสนุนโดยการวิเคราะห์เชิงสถิติ และที่ปริมาณเส้นใยผสมที่ได้แต่ละชนิดพบว่า สารช่วยปรับปรุงความเข้ากันได้อีพอกซีทำให้คุณสมบัติเชิงกลของพอลิคาร์บอเนตที่เสริมแรงด้วยเส้นใยแก้วมีค่าเพิ่มขึ้นอย่างชัดเจน แต่พอลิคาร์บอเนตที่เสริมแรงด้วยเส้นใยคาร์บอนและเส้นใยเคฟลามีคุณสมบัติเชิงกลลดลงเมื่อมีการใช้ระบบอีพอกซีเป็นสารช่วยปรับปรุงความเข้ากันได้

การศึกษาโดยกล้องจุลทรรศน์แบบส่องกวาดพบว่า ระบบอีพอกซีที่ 1% สามารถทำหน้าที่เป็นตัวต้านทานการแตกหักสำหรับของผสมพอลิคาร์บอเนตได้ โดยเฉพาะพอลิคาร์บอเนตที่เสริม

เสริมแรงด้วยเส้นใยแก้วจะเห็นการยึดติดระหว่างเส้นใยกับพอลิเมอร์ที่ดีขึ้นอย่างชัดเจน ในขณะที่การใช้ฟอกซีเป็นสารช่วยปรับปรุงความเข้ากันได้ไม่ได้มีผลทำให้พอลิคาร์บอนเนตที่เสริมแรงด้วยเส้นใยคาร์บอน และเส้นใยเคฟลาสามารถยึดติดกันได้ดีขึ้น

สาขาวิชาวิศวกรรมพอลิเมอร์
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ลายมือชื่อนักศึกษา Onuma Charoensuk
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FIBRE REINFORCED/ POLYCARBONATE/ GLASS FIBRE/ KEVLAR FIBRE/
CARBON FIBRE/ COMPATIBILISER/ EPOXY SYSTEM/MIXER EVALUATION

The fibres reinforced polycarbonate(PC) were prepared by using internal mixer, single and twin screw extruders, and its combination. The epoxy/DDS system was used as compatibiliser. The final fibres length in the PC compound was not depended on the initial length for both uncompatibilised and 1% compatibilised systems. The internal mixer gave rise to the shortest final fibre length but single screw extruder led to the longest one.

The compatibilisation of PC blends using 1% epoxy and DDS system as compatibiliser mixed in twin screw extruder had shown the lower HDT but superior in mechanical properties than the neat PC. These results were confirmed by the statistical analysis. The study had also revealed that the twin screw extruder offers the best alternative for the mixing equipment. The properties of the PC compounds were varied when the difference mixers were used for both compound systems. For reinforcing PC with carbon, kevlar and glass fiber, the optimal fiber contents were observed at 5%, 10% and 15% by weight, respectively, for both systems.

The twin screw extruder was found to be the most effective mixer for compounding those fibres reinforced PC. The statistical analysis was used to support this outcome. At the optimal fibre contents, the epoxy compatibiliser had been

this outcome. At the optimal fibre contents, the epoxy compatibiliser had been successfully used to improve the mechanical properties of the fibre glass reinforced PC. However, the properties of carbon and kevlar fibres reinforced PC were not enhanced by adding the compatibiliser.

The SEM study had indicated that 1% of epoxy was an effective fracture toughener for the PC compound. It had also illustrated that 1% epoxy was successfully used as compatibiliser for the glass fibre reinforced PC as indicated by the adhesion between fibre and polymer phase. Nevertheless, it was not ineffectively used for the carbon and kevlar reinforcements.

School of Polymer Engineering

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Student's Signature Onuma Charoensuk

Advisor's Signature M. Uthairi

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CHAPTER I

INTRODUCTION

1.1 General Introduction

Mixing is a fundamental unit operation in the processing and fabrication of polymeric materials, as well as an essential operation during the polymerisation of neat or virgin polymers. The term compounding refers to the blending and homogenisation of ingredients such as reinforcing agents, fillers, colorants, stabilisers, flame retardants, curing agents, and other active ingredients into the polymer matrix, resulting in a mixture that can be fabricated, formed, molded and processed into a final product form (White, Coran, and Moet, 2001).

Polycarbonates(PCs) are high performance thermoplastic engineering polymers. They are polyesters in which the repeating unit corresponded to an ester of carbonic acid and dihydroxy compound. They have been prepared by polycondensation reactions of dihydroxy compounds, more frequently 2,2-bis-(4-hydroxyphenyl) propane known as bisphenol A and carbonic acid derivatives. The values of \overline{M}_n of the commercial PCs are normally in the range 20,000 g/mol to 50,000 g/mol and they have a pronounced effect on the melt index. There would be difficult to process when the viscosity was too high. The flexibility of the chain is restricted by the presence of carbonate polar groups and aromatic benzene rings leading to high melting temperature(T_m) ranging from 230°C to 250°C and the glass transition temperature(T_g) of 149°C. Melt processing temperature as high as around

230°C to 320°C are required and degradation can be easily occurred (Feldman and Barbalata, 1996). Despite the rather high cost, PCs have imposed themselves on the market by their outstanding impact strength, and excellent optical and thermal properties. They have found various applications in the optical equipments, automotives, electrical appliances, sporting goods, building structures and many other industries used.

One method to improve mechanical and thermal properties of PC is fibre reinforcement. Generally, three main types of fibres are supplied for engineering material; polymeric, ceramic and metallic fibres. Glass fibre is one type of ceramic fibres while kevlar and carbon fibres are derived from polymers. Basic difficulty for compounding the polymer is incompatibility between polymer matrix and added ingredients. Most polymer structures are immiscible with each other and form two-phase systems and consequently adhesion between the two-phases is low, rising in poor properties. The interaction between the components of immiscible blends can be modified with the use of compatibiliser, which usually acts at the interfaces of the immiscible components to improve the interfacial adhesion.

Mixing machinery can generally be classified into “batch” and “continuous” mixers. In batch mixers, all of the materials to be incorporated are placed in a mixing chamber containing one or more agitators or mixing rotors. The later one, the components to be incorporated is continuously added into the mixer and the uniform compounded stream exits from the machine. In industries, among various types of mixing equipment, internal mixer and twin screw extruder have most widely been used to prepare polymer blends (Lee and Han, 2000).

1.2 Research Objectives

The main objectives of this research study can be classified as follows;

- (i) To evaluate the mixing efficiency by means of improving the properties of fibre reinforced PC prepared by using internal mixer, single and twin screw extruders.
- (ii) To investigate the effect of processing conditions on the aspect ratio of the fibres employed.
- and (iii) To study the used of epoxy systems as compatibiliser in the polycarbonate compounds.

1.3 Scope and Limitation of the Study

In the experimental study, the short fibres reinforced PC were prepared by using internal mixer, single and twin screw extruders as the compounding machines. Four methods of the compounding techniques namely; Method I, Method II, Method III, and Method IV, are summarised in detail as shown in Table 1.1. Three types of short fibre; glass, kevlar, and carbon fibres were employed. The fibre contents added in the blends were 5%, 10%, and 15% by weight, respectively. The approximate initial fibre length 8 mm. was manually cut, using the scissor, from the unidirectional fibre bundle. The mechanical properties by means of tensile, flexural, and impact strength of the compounds were determined. Also, the thermal property, heat distortion temperature(HDT) was also analysed. The epoxy systems using solid epoxy resin, diglycidyl ether of bisphenol A(DGEBA), and 4,4'-diaminodiphenyl sulfone(DDS) as curing agent were used, at phr equal to 1.5, as compatibiliser to improve the compatibility of the compound. In accordance, the mechanical properties

of the compatibilised mixture could be enhanced.

Table 1.1 Summarisation of the compounding techniques.

Method	Method Description	Assignment
I	Compound using twin screw extruder.	TSE
II	Compound using internal mixer.	IM
III	High concentrate fibre/PC masterbatch prepared by internal mixer and then diluted in single screw extruder.	MB+SSE
IV	Compound using single screw extruder.	SSE

CHAPTER II

LITERATURE REVIEW

2.1 General Background

The bisphenol A based polycarbonate has been widely used as thermoplastic engineering material because of such desirable properties as high transparency, high impact strength, broad temperature resistance, and an excellent dimensional stability (Hagenaars *et al.*, 2002). However, PC has some draw back characteristics that limit its utilise in many applications, for instance, its exceptional toughness is not retained in thick molded sections or the presence of sharp notches or at low temperatures (Kayano *et al.*, 1996). In recent years, the need of new materials with improved specific properties has strongly affected the research and production of composite material. Most polymer structures are immiscible with each other and form two separated phase systems. The adhesion between the two phases is low and given rise to poor properties. The interaction between the components of the immiscible blends can be enhanced by using the compatibilisers. They must accomplish three tasks: (i) decrease the interfacial tension coefficient between the two immiscible polymers, thus making it possible to generate the desired morphology, (ii) stabilise the morphology during the phase formation stage in high stress fields, and (iii) improve adhesion between the phases in the solid state.

Epoxy resins find wide applications in the composites industries, which are due to their versatility, including their chemistry that allows tailoring the structure of

the monomers on the basis of the requested properties of the final products. For example, by using more functionalized epoxy monomers, they are possible to increase the glass transition temperature(T_g), and to improve the mechanical properties. On the other hand, more flexible curing agents lock the thermoset in a less constrained network having a lower T_g (Carfagna *et al.*, 2000). For this reason, the common epoxy system, DGEBA and DDS, could be used as compatibiliser to improve mechanical properties of short fibre reinforced PC.

2.2 Compatibilisation of the PC Blends

Polycarbonate(PC)/poly(acrylonitrile-butadiene-styrene)(ABS) blends compatibilised with both maleic anhydride(MA) grafted polypropylene and solid epoxy resin, bisphenol A type, were prepared and injection molded (Tjong and Meng, 2000). The effects of the compatibiliser additions on the morphology and mechanical properties of the blends were investigated. The tensile and Izod impact tests revealed that the addition of epoxy at 2 phr regarding to the 70/30 of MA-grafted PC/ABS blend led to a significant increased in both tensile ductility and impact strength. In this case, the impact strength of these compatibilised blends was close to that of PC.

Choi *et al.*(1997) studied the tensile properties, morphology and thermal behavior of poly(phenylene sulfide)(PPS)/PC binary system and PPS/PC/epoxy ternary system. It was observed that the viscosity average molecular weight(\overline{M}_v) of the PC fraction was decreased during the melt blending of the PC with PPS. By melt blending PPS and PC with epoxy as a compatibiliser, a shift of T_g of PC to a lower value could be somewhat prevented. It was also suggested that epoxy coupled the degraded PC chains generated by blending with PPS. The tensile strength and tensile

modulus of PPS/PC/epoxy were remarkably improved to more than 100% compare with those of the epoxy free system. In addition, it was found that the improvement of tensile properties was mainly due to the homogeneous morphology by blending of small amount of epoxy with PPS and PC.

2.3 Toughening of Epoxy Systems

Epoxy based on the diglycidyl ether of bisphenol A(DGEBA) has been toughened with polycarbonate(PC) and then cured with 4,4'-diaminodiphenylsulfone(DDS) and maleic anhydride(MA) (Rong and Zeng, 1996). The chemical interaction between the PC and epoxy resin during the melt mixing process and its effect on the physical characteristics of PC had been investigated prior to the curing process. They were found that the PC usually has no effect or has promoting effect on the curing reaction for amine hardener because of the *in situ* curing mechanism. The presence of quaternary ammonium salts catalysing the PC-epoxy reaction resulted in a two-step curing behavior, but its curing kinetics were similar to those of the neat resin. Moreover, they studied the phase separation and morphology of these blends (Rong and Zeng, 1997). It was found that the phase separation in the PC/epoxy blends was controlled by varying the composition and the PC/epoxy reaction and utilising curing agents of different reactivity. The internal fractionation change by PC chain scission during the melt blending process has an important influence on the crystallization. It was suggested that hydrogen bonding and grafting reaction of the PC/epoxy semi-IPN was the key to control phase separation and morphology.

Polycarbonate could react with the diglycidyl ether of bisphenol A(DGEBA) at 200°C through transesterification and addition reactions, resulting in degradation of PC chains with phenolic end groups and also in PC/DGEBA copolymers (Don and Bell, 1998). The curing agent used was an aromatic amine, diaminodiphenyl methane (DDM). They were found that the presence of a small amount of PC promoted the amine-epoxide reactions, probably due to the catalytic effect of the phenolic end groups in the PC chains. However, the PC did not alter the epoxy cure reaction mechanism. Additionally, they examined two different blending processes of DGEBA with PC, namely, solution-blending(SB) and melt-blending(MB) processes (Don *et al.*, 1999). It was indicated that during the melt-blending of an epoxy oligomer based on the DGEBA with PC at 200°C, the secondary hydroxyl groups in the DGEBA react with the carbonate groups in PC through transesterification, resulting in degraded PC chains with phenolic end groups and also in PC/DGEBA copolymers. The fracture toughness of epoxies was increased due to the incorporation of ductile PC chains into the epoxy network.

The chromatographic and spectroscopic variations of PC/epoxy blends during the hot melt process of dissolving PC in epoxy have been observed by Li *et al.* (1997). They reported that the characteristics of PC/epoxy blend were influenced by the following factors: (i) the minor components in the epoxy monomer, (ii) the ratio of PC to epoxy in the PC/epoxy blend, and (iii) the environment in which the blends were prepared. The water contained in the epoxy might hydrolyse the PC and yields two phenolic chain end groups and carbon dioxide. The alcoholic hydroxyls existing in epoxy may transesterify the original aromatic/aromatic carbonate of PC to aromatic/aliphatic and aliphatic/aliphatic carbonates. The α -glycol present in epoxy

would react with the carbonate of PC to form the cyclic carbonate structure. Whether the PC hydrolysis, transesterification or cyclization reactions proceed during the process of dissolving PC in epoxy depended on the minor components present in the epoxy monomer. Results showed that the oxirane of epoxy would not react with the carbonate of PC in the absence of a catalyst.

2.4 The Effect of Curing Agent on Epoxy/Amine Systems

More recently microwave curing has been shown to be a viable alternative as an accelerated curing system. Boey *et al.* (1999) investigated the effect of different curing agent under microwave reaction for epoxy systems. The three types of curing agents used were 4,4'-Diamino-di-phenyl-sulfone(DDS), 4,4'-Diamino-diphenyl-methane(DDM) and *m*-Phenylene diamine(m-PDA). Thermal cure temperatures were determined between the onset and midway to the peak temperature of the freshly prepared epoxy/amine system of the DSC exotherm curves. They found that a low degree of cure at a relatively low microwave power was observed in DGEBA/DDS system, and has been attributed to the sluggish reaction of DDS with epoxy, resulting in the entrapment of the functional group in the cross-link network. DDM and m-PDA were more reactive than DDS and gave relatively faster full curing time than DDS.

2.5 The Effect of Fibre Reinforcement

2.5.1 The Effect of Fibre Length and Fibre Content

Thomason and Vlug(1996) published that the influence of fibre length, 0.1 mm to 50 mm, and the concentration from 3%wt to 60%wt on the properties of glass reinforced polypropylene laminates. They found that laminate stiffness was

increased linearly with fibre concentration up to 40%wt. However, stiffness was virtually independent of fibre length above 0.5 mm. Predictions of tensile modulus using the Cox model correlated well with the experimental data. High concentrations, more than 40%wt, of long fibre resulted in fibre packing problems and an increase in void content that led to a reduction in modulus. The matrix molecular weight and the fibre sizing compatibility had little effect on the laminate stiffness.

2.5.2 Glass Fibre Reinforced Polycarbonate

Ho and Jeng(1997) investigated the tribological characteristics of short glass fibre reinforced polycarbonate composites. Various filler reinforcements were frequently added to the base materials to improve the wear and impact properties. They studied the wear behavior of polycarbonate reinforced with 20%wt of short glass fibre. The specimens were prepared under different molding conditions, with varied filling time, melt temperature, and mold temperature. The results showed that the wear volume loss in the sliding direction perpendicular to the melt flow direction were usually lower than that in the sliding direction parallel with the melt flow direction. The optimum injection molding conditions for lowest wear volume loss were at filling time of 5 sec., melt temperature of 290°C, and mold temperature of 80°C, when the sliding direction was parallel with the melt flow direction. The effect of wear volume loss was influenced significantly by the thickness of the frozen layer and the fibre orientation with respect to the sliding direction. Moreover, optimization of the injection molding process of short glass fibre reinforced polycarbonate composites using Grey relational analysis were studied by Chang *et al.* (2000). They indicated that three distinct layers, a frozen layer, a shear layer, and a core layer, were observed from the surface to the core for various injection molding conditions. The

fibre orientation was perpendicular to the melt flow direction in the frozen layer and the core layer, but it had the opposite direction in the shear layer.

2.5.3 Glass Fibre Reinforced Epoxy Systems

Kaynak *et al.* (2003) demonstrated flexibility improvement of short glass fibre reinforced epoxy by using a liquid elastomer. For this purpose, diglycidyl ether of bisphenol A(DGEBA) based epoxy matrix was modified with hydroxyl terminated polybutadiene(HTPB). The silane coupling agent(SCA) was also used to improve the interfacial adhesion between glass fibres and epoxy matrix. They reported that scanning electron microscope(SEM) emphasized the surface treatment of glass fibres with SCA, and HTPB modification of epoxy matrix improved flexural properties especially due to the strong interaction between fibres, epoxy, and rubber. It was also revealed that HTPB modification resulted in relatively round rubber domains in the epoxy matrix leading to increase in flexibility of the specimens.

2.5.4 Kevlar Fibre Reinforced Polycarbonate

Tanaka *et al.* (2001) had conducted a research on the development of a lightweight space debris shield using high strength fibres. Kevlar high strength fibre was one of potential materials from a viewpoint of strength, lightweight, and flexibility. There were showed that the developed the shield could stop the polycarbonate projectile with 13 mm in diameter, 1 gram in weight, and 6.9 km/sec in velocity. Adoption of the high strength fibre in the bumper materials may reinforce the protection capability and reduce the weight drastically.

2.5.5 Kevlar Fibre Reinforced Epoxy Systems

Thermal and dynamic mechanical properties of anhydride and diamine epoxy/kevlar composites were studied by Anton *et al.* (1999). Differential scanning

calorimetry(DSC) and dynamic mechanical thermal analysis(DMTA) techniques had been used to characterise different kevlar/epoxy composites. Tetrafunctional aliphatic amine and anhydride diglycidyl epoxy had been used as matrix, and different quantities of continuous kevlar fibre as reinforcement. They found that the kevlar fibres had different effect on curing kinetics and final thermal properties depended on epoxy matrix type. As kevlar content increased, DSC heat flow curves shifted to much lower temperatures in the anhydride matrix case, while there was very little difference in the diamine epoxy one. A significant decreasing in the glass transition temperature(T_g) was observed as kevlar content increased when anhydride matrix was used, while little change was observed in the reinforced diamine system.

2.5.6 Carbon Fibre Reinforced Polycarbonate

Carneiro *et al.* (1998) conducted the research on the production and assessment of polycarbonate composites reinforced with vapor-grown carbon fibres. Vapor-grown carbon fibres were produced from methane in a flow reactor. The fibres were compounded with polycarbonate in the co-rotating twin screw extruder and the composites were subsequently injection molded. They found that the tensile properties of the injection molded specimens were marginally better than those of the unreinforced polycarbonate, but the impact resistance was severely diminished by the addition of the carbon fibres. Furthermore, Huang and Young (1996) studied interfacial micromechanics in thermoplastic and thermosetting matrix carbon fibre composites. The interfacial micromechanics of carbon fibres in thermoplastic matrices, poly(methyl methacrylate)(PMMA) and polycarbonate(PC), had been investigated by determining the distribution of interfacial shear stress along fibres in the single fibre model composites using Raman spectroscopy. It was found that the

maximum values of interfacial shear strength for the PMMA and the PC model composites were 3.1 MPa and 8.4 MPa, respectively. They were much lower than the value of 30 MPa obtained for the same fibre in a thermosetting epoxy resin matrix. These low values of interfacial shear stress in thermoplastic systems could be explained by the lack of chemical bonding between the fibre and matrices, and possibly the effect of residual solvent.

2.5.7 Carbon Fibre Reinforced Epoxy Systems

Woo and Mao(1996) examined the interlaminar morphology effects on fracture resistance of amorphous polymer modified epoxy/carbon fibre composites. Effects of three amorphous thermoplastic polymers used as interlaminar toughening particles in carbon fibre/epoxy laminated composites had been compared. Surprisingly, incorporation of bisphenol A polycarbonate with its solvent-induced crystallinity during particulate preparation, induced only very little improvement in the modified composite system. The microstructural morphology at the PC/epoxy interlaminar region showed insoluble chunks of PC domains with crystals, the brittleness and lack of wetting with the epoxy phase adversely affected the macroscopic mechanical fracture behavior.

Using X-ray photoelectron spectroscopy(XPS) to investigate fibre/matrix chemical interactions in carbon fibre-reinforced composites was conducted by Weitzsacker *et al.* (1997). The degree of adhesion between the fibre and matrix had been recognized to be a critical factor in determining the performance of fibre reinforced composites. Indirect analysis of the matrix/fibre interface was possible using model compounds that represent functional groups present in the matrix system. Model compounds based on epoxy, polyimide, polyphenylene sulfide and vinyl ester

matrices had been investigated to characterise the chemical reactions at the fibre/matrix interface. XPS had been used to characterize both the carbon fibre surfaces and the reacted carbon fibre surfaces. The model compounds for the polyimide and polyphenylene sulfide matrices had been found to show little chemical interaction with the fibre surfaces while chemical reactions had been observed in the vinyl ester and epoxy resin model compound/fibre interfaces.

From the works reviewed, the use of solid epoxy as the compatibiliser for the fibre reinforced PC in order to improve its properties would be the prime interesting research point of views. The evaluation of mixers used to prepare the specimen and hence for the future processing equipments would also essential for technical and economical benefits.

CHAPTER III

EXPERIMENTAL METHODOLOGY

3.1 General Background

As previously mentioned that there were four techniques for PC/fibre compounding in this study and they were evaluated in term of their efficiency of mixing. The short fibre; glass, kevlar, and carbon fibres, were engaged as reinforcement. The fibre content of 5%, 10%, and 15% by weight were incorporated in the compound. The approximate initial fibre length of 8 mm was manually cut. The twin screw extruder was presumed and also found as the most favorable mixer in order to evaluate mixing efficiency. The epoxy rich system, in the other word branched chain , derived from solid epoxy diglycidyl ether of bisphenol A(DGEBA) cured with aromatic amine, 4,4'-diaminodiphenyl sulfone(DDS), at 1.5 phr was used as compatibiliser to increase the adhesion between fibre and polymer matrix. The details of the experimental procedures, the materials used, and also the material testing and characterisation will be testified.

3.2 Materials and Chemical Reagents

The main materials used in this study can be classified into three categories; (i) polymer matrix and compatibiliser, (ii) the fibrous reinforcements, and (iii) miscellaneous chemical reagents.

The PC pellets, grade S-3000R, used as matrix phase in this experiment were supplied from Thai Polycarbonate Co., Ltd. The basic properties of this matrix were provided by the manufacturer and also analysed in house. They are summarised in Table 3.1. It was dehumidified at 120°C under vacuum for 5 h before immediately transferring to processing machine. The solid epoxy prepolymer, diglycidyl ether of bisphenol A(DGEBA) called as EPOTEC YD-019 was purchased from Thai Epoxy and Allied Products Co., Ltd. It was also dehumidified at 100°C under vacuum for 5 h prior to use. The DDS used as high temperature curing agent system was supplied from Vantico Co., Ltd. Both epoxy and curing agent were used as received. The fundamental properties of these materials are summarised in Table 3.2. The calculated stoichiometry ratio, or phr, of this system is 2.35. However by using lower ratio, phr equal to 1.5 as applied in this research study, it would give rise to the epoxy rich cured product. Accordingly, highly branched chain structure would be derived.

The reinforcements, glass, kevlar, and carbon fibre supplied as fibre bundle were employed. They were supplied from Siam Mercantile Agencies Co., Ltd., DuPont™ Co., Ltd., and Pranda Co., Ltd., respectively. The characteristics of these fibres are presented in Table 3.3. These fibres were manually cut using ceramic scissor into the desired length and thoroughly coated with DDS. This was done by dissolving DDS in approximately 100 ml of acetone and well stirred with the fibre for 2 -3 min. Then, the solvent was removed by rotary evaporator at 70°C for 10 min and dried in vacuum oven at 80°C for 1 h. In case of using the reinforcements without DDS coating, the carbon and kevlar were dried in the oven at 200°C for 5 h and immediately transferred to the mixer. While the moisture residual in glass fiber was removed in the hot oven at 300°C for 5 h.

The A.R. grade Tetrahydrofuran(THF) was supplied by A.C.S. Xenon partnership Ltd. It was utilised as solvent for PC solution and used as received.

The Sodium Hexametaphosphate was supplied by Italmar (Thailand) Co., Ltd. It was used as glass fibre dispersant for particle size analysis, while the Nonylphenyl-Polyethylene Glycol, or Nonidet P40, was used as the kevlar and carbon fibres dispersant for particle size analysis. It was supplied by A.C.S. Xenon partnership Ltd. The commercial grade acetone was used as the non solvent for PC. It was supplied by Wittaya Srom Co., Ltd. Those reagents were used as received.

Table 3.1 The properties of the commercial PC, S-3000R.

Property	Value
¹ Glass Transition Temperature(T_g , °C)	150
¹ Melt Transition Temperature(T_m , °C)	230
² Bulk Density(g/ml)	1.23
² Melt Density(g/ml)	1.20
¹ Number Average Molecular Weight(\overline{M}_n , g/mol)	10,000
¹ Weight Average Molecular Weight(\overline{M}_w , g/mol)	27,000
² Viscosity Average Molecular Weight(\overline{M}_v , g/mol)	32,137
² Melt Flow Index(MFI at 300/2.16, g/10min)	13.20

¹Supplied from manufacturer

²In house analysis

Table 3.2 The properties of solid epoxy and curing agent.

Property	Value
EPOTEC YD-019:	
Epoxide Equivalent Weight (EEW, g/eq)	2,637
Softening Point (°C)	130-145
Viscosity at 25°C (40% in butyl carbitol, cPs)	7,490
Insoluble content (ppm)	5
DDS:	
Amine Hydrogen Equivalent Weight (AHEW, g/eq)	62
Melting Point (°C)	175

Table 3.3 The basic characteristics of the fibres.

Fibre	Supplier	Denier (g/9000m)	Density (g/ml)
Glass	Siam Mercantile Agencies Company Limited	19,813	2.50
Kevlar	Dupont TM Company Limited	2,989	1.44
Carbon	Pranda Company Limited	41,996	1.76

3.3 Mixing Equipments

There were three typical mixers employed in the study, twin screw extruder, internal mixer, and single screw extruder. They were used as individual and also in combination for each compounding technique as given earlier.

3.3.1 Twin screw Extruder

The BrabenderTM DSE 35/17D twin screw extruder used in this study was consisted of three segmented kneader blocks. The single hole palletized die was attached. For mixing only PC and short fibres, the barrel temperature at the feed zone to die was gradually controlled by means of electrical heated from 270°C to 290°C.

However, in case of using epoxy/hardener as compatibiliser in the compound ingredient, the barrel temperature was lowered to 230°C to 250°C, respectively. The screw speed was constantly controlled at 7 rpm. At this revolution, it gave rise to the residual mixing time, as refer to cure time for epoxy system, of approximately 2 min for the compatibilised system.

3.3.2 Internal Mixer

The HAAKE Rheomix PolyLab™ internal mixer was carried out in this study. It was comprised of two Roller counter-rotating rotors. For mixing the PC and short fibre, the mixing chamber was electrically and constantly controlled at 280°C. In order to retain the constant shear viscosity during the mixing, the sequences of the adding the ingredients were as followed, loading PC pellets and mixed until the PC was soften that would take about 1min. Then, adding a half of the DDS coated fibre with solid epoxy and mixed for 2 min. Finally, adding the less of the fibre and further mixed for 3 min. However for system without epoxy, adding a half of fibre at the 2nd minute of mixing time and the less at the 4th minute. To prevent and minimise the degradation of epoxy chain occurred during mixing, the mixing temperature was lowered to 230°C. The rotor speed was constantly controlled at 60 rpm, the optimum degree of fill was 70% and hence the calculated batch size is 260 g. The compound was then crushed into small shrunk by using jaw crusher.

In case of preparing the PC/fibres masterbatch for dilution in single screw mixer, the desired high concentrate fibres PC masterbatch was prepared by the internal mixer. The mixing manner was repeated as previously described except that the mixing time was shortened to 3 minutes.

3.3.3 Single Screw Extruder

The Betol, BC 32, single screw extruder was employed. It consisted of the Maddock mixing screw with the L/D ratio of 30 and the three holes palletized die were attached. For mixing the uncompatibilised PC and short fibres, the barrel temperature at the feed zone to the die was gradually increased from 270°C to 290°C. The screw speed was constantly controlled, by means of panel display, at 20 rpm. In this method, all the ingredients were dry mixed and continuously added into the extruder. The extrudated strands were cut using pelletiser into approximately 3 mm rod shape pellets. In case of mixing the epoxy compatibiliser in the compound ingredient, the barrel temperature was lowered to 230°C to 250°C. At this screw speed, it consequently gave rise to the curing time of approximately 2 min for the epoxy reaction.

3.4 Characterisation and Performance Testing

3.4.1 Dilution Viscometry

The intrinsic viscosity, $[\eta]$, of the polymer was determined by using dilution viscometer technique and hence the average viscosity molecular weight (\overline{M}_v) was calculated. The Viscosystem, AVS 360 from Schott Gerate Co., Ltd., was employed and the experiment was conducted using Ubbelohde capillary with the total measuring volume of 20.0 ml. The measuring temperature was constantly controlled at $25.0 \pm 0.1^\circ\text{C}$ using water circulation bath. Commonly, the initial concentration of 1.000%w/v PC soluted in THF was prepared in 10.00 ml volumetric flask. The solution was then transferred into the capillary and allowed to equilibrate for at least 5 min. The flow time was measured three times using the online photo encoder and the

mean average value was recorded. Then the initial PC solutions were diluted to 0.500%, 0.250%, and 0.125% with fresh THF, respectively. The viscosity average molecular weight (\overline{M}_v) was calculated according to Mark-Houwink-Sakurada equation as shown below.

$$[\eta] = K \overline{M}_v^a \quad (3.1)$$

Where, K and a are the constants. For the PC in THF solution at 25°C, these values are 0.00399 ml/g and 0.70 ml/g, respectively.

3.4.2 Fibre Length Determination

In order to compare between the initial and final length of the fibre in the compound obtained from each mixer regarding to the initial fibres length, the diffraction particle size analyser model Mastersizer S was employed to investigate the final average fibres length of the PC compound. The polarized optical microscope, Nikon Model Eclipses E660 POL with the compensator scale of 0.001 mm was also used to verify the length. The fibres sample were placed on slide plate and covered with cover plate. They were visually observed under polarized light with magnification of 10x50. The specimen fibre after mixing was obtained by solvent extraction of the PC compound using THF. The fibre was then filtered through the filter paper No.40, thoroughly washed with fresh THF several times, and then dried at 80°C for 1 h in the vacuum oven. For particle size analysis, the fibre and the given dispersant was suspended into the machine crucible. The suspended liquid was transferred into analyser cell of the machine. The helium-neon laser was used for the indentation light source. The result was reported as normal distribution curve and the average particle size was taken from the middle of the peak.

3.4.3 Scanning Electron Microscopy

The morphology of fractured surface of the compound specimens taken from unnotched Izod impact testing were examined using scanning electron microscope(SEM) model JSM 6400. The specimens were cut at the thickness of 5 mm below the fractured surface. It was placed on the sample holder and coated and adhered with silver paint. Fracture surface was again coated with gold by ion sputtering for 5 min.

3.4.4 Heat Distortion Temperature

Regarding to ASTM D648, the manual DTUL/VICAT from ATLAS with the standard load of 1820 kPa or 264 psi was applied to measure the heat distortion temperature(HDT) at heating rate of 120°C/h. Silicone oil was used as heating transfer media. The injected rectangular cross section specimen with 127 mm in length, 13 mm in depth, and 4 mm in width were tested. Three specimens were immersed under the calculated loading weight at the assigned standard load. The HDT was read from the thermometer when the specimen had been deflected to 0.25 mm or 0.010 in.

3.4.5 Izod Impact Testing

The Atlas Basic Pendulum Impact tester Model BPI, was used for Izod impact testing. The standard ASTM D256, Test Method A, was assigned to determine the impact strength of the compound specimen. The total impact energy of 5.4 J was selected. The unnotch test specimens with 12.7 mm in thickness, 64 mm in length, and 4 mm in width were prepared by injection molding. Ten specimens of each compound sample were tested. The impact resistance was reported as impact strength (kJ/m^2) that was the failure energy, divided by the cross section area of the sample.

3.4.6 Flexural and Tensile Measurement

The Instron Universal Testing Machine Model 5565 was used to measure the flexural and also tensile properties. The standard test method, ASTM D5943, with the three points bending test fixture was followed to determine flexural properties. The maximum load, maximum stress, and flexural modulus were resolved. The test specimen was constantly strained at the rate of 10 mm/min. The span length of 72.0 mm was calculated and adjusted. The injected test specimen having the same dimensions as described for the HDT testing was employed.

For determining tensile properties, the standard test ASTM D638 was followed. The tensile properties, tensile stress, strain at break, and Young's modulus (E) were reported. The constant strain rate of cross head speed was set at 5 mm/min. The dimension of dumbbell shaped specimens at 13 mm in width at narrow section, 20 mm in overall width, 60 mm in gauge length, 165 mm in overall length, and 3.7 mm in thickness were prepared by injection molding. The distance between the ends of the grips and the final gauge length were adjusted at 115 mm and 50 mm, respectively. Normally, five samples were conducted for each test.

3.4.7 Injection Molding Machine

The Chuan Lih Fa CLF-80T injection molding machine was used to prepare the test specimen. The barrel temperatures at the feed zone to nozzle were gradually raised from 270°C, 280°C, 280°C, and 290°C, respectively. The mold temperature was maintained at 80°C using silicone oil bath. The screw diameter of 35 mm with L/D ratio of 30 was equipped. For injection the rectangular test specimen, the injection speed of 82 cm³/sec, the injection pressure of 1,520 kg/cm², and the holding pressure of 640 kg/cm² were recorded. The cycle time was set at 10 sec for

feed time, 6 sec for injection time, 5 sec for holding pressure, 20 sec for cooling time, and 10 sec for mold open time. The shot size of the rectangular specimen was 8.0 g. On the other hand, for injection the dumbbell shaped test specimen the families mold, dumbbell and rectangular bar, gate balancing using the Moldflow™ was employed. The injection parameters were set at the injection speed of 46 cm³/sec, the injection pressure of 1,200 kg/cm², and the holding pressure of 1,200 kg/cm². The cycle time were adjusted at 15 sec for feed time, 6 sec for injection time, 5 sec for holding pressure, 20 sec for cooling time, and 10 sec for mold open time. The weight of the dumbbell specimen was 10.8 g. However, in case of injection of the compatibilised fibre reinforced PC, the barrel temperature at the feed zone to nozzle was lowered to 230°C, 240°C, 240°C, and 250°C, respectively.

CHAPTER IV

RESULTS AND DISCUSSION

4.1 The \overline{M}_v of Polycarbonate

PC is the most well known as one of moisture sensitive polymers. Its chain length would be hydrolysed and consequently shortened by heat and high shear after repeatedly passing through the processing equipments. In order to determine the degradation reaction of the PC, the viscosity average molecular weights (\overline{M}_v) of polymer using dilution viscometry technique, as described in Chapter III, were calculated in accordance with equations as shown in Appendix A. Table 4.1 shows the experimental values, it reveals that the molecular mass of the virgin PC is 32,137 g/mole and it is decreased depending on the processed machines. The maximum chain reduction at approximately 35% is found for the internal mixer due to its highest shear stress. In single and twin screw extruders, around 7-9% reduction is observed. It is indicated that these two mixers manifested lower shear stress during mixing process. Comparing the uncompatibilised with the compatibilised systems using the same mixer, the test results illustrate that the declination of the polymer chain obtained from the former system is less than the later one. This observation could be explained by the reaction between epoxy and polycarbonate chains that might be occurred during the melt processing as reported by Don and Bell(1998). The reaction would be accomplished the chain scission of the polymer. The processing variables could be additionally taken into account for the molar mass reduction. As expected, the internal

mixer was operated as high shear rate as 60 rpm but the lower shear rate, at 7 and 20 rpm, were controlled for twin and single screw mixers, respectively. These processing speeds would also play the significant affected and resulted in the molecular weight reduction of the PC. In addition, injection molding used to fabricate the compound specimen is further exaggerated the chain shortening of the polymer sample.

Table 4.1 The viscosity average molecular weight (\bar{M}_V) of the processed polycarbonates.

Processed PC	$[\eta]$ (ml/mol)	\bar{M}_V (g/mol)	% Reduction ¹
Virgin PC	0.57	32,137	-
Uncompatibilised PC:			
Internal mixer	0.42	20,775	-35.35
Single screw extruder	0.54	29,748	-7.43
Twin screw extruder	0.53	28,964	-9.87
Twin screw and injection molding ²	0.46	23,658	-26.38
Compatibilised PC:			
Internal mixer	0.40	19,376	-39.71
Single screw extruder	0.53	28,964	-9.87
Twin screw extruder	0.49	25,893	-19.43
Twin screw and injection molding ²	0.42	20,775	-35.35

¹as compared with the molecular weight of the virgin PC

²Injection molding was used to prepare the specimen.

4.2 Fibre Length Analysis

The fibre lengths retained in the reinforced PC blends were verified by using

the particle size analyser and also observed under polarized light optical microscope. In the early stage of this study, the initial fibres lengths used were varied from 4 to 8 mm, and the final fibre length resulted in the polymer mixture was determined. The experimental results of the 5% added of glass, kevlar, and carbon fibres in the reinforced PC that mixed by internal mixer and twin screw extruder are shown in Table 4.2. As from the basic hypothesis that the properties of the fibre reinforced PC must depend on the aspect ratio of the fibre in the final compound. The longer fibre length would result in the better mechanical properties.

Table 4.2 The fibre lengths of glass, kevlar, and carbon fibres before and after processed via the internal mixer and twin screw extruder.

Initial Fibre Length (mm)	Final Fibre Length of 5% Fibres Reinforced PC(mm)					
	Glass Fibre		Kevlar Fibre		Carbon Fibre	
	¹ IM	² TSE	¹ IM	² TSE	¹ IM	² TSE
4	0.04933±0.02	0.430±0.02	0.13325±0.06	0.450±0.02	0.13085±0.06	0.430±0.02
6	0.04995±0.01	0.450±0.02	0.13446±0.05	0.460±0.02	0.13118±0.06	0.440±0.03
8	0.05119±0.01	0.460±0.04	0.13886±0.05	0.480±0.03	0.13101±0.06	0.450±0.04
Average	0.050	0.447	0.136	0.463	0.131	0.440

¹analysed by particle size analyser

²observed under polarized optical microscope

The analysis results show that the average final length of the fibres mixed with PC via the internal mixer are approximately decreased to 0.05, 0.14, and 0.13 for the glass, kevlar, and carbon fibres, respectively, regardless to the initial length added. While the average fibre length mixed by twin screw extruder are approximately reduced to 0.45, 0.46, and 0.44 for the glass, kevlar, and carbon fibres, respectively

and also regardless to the initial length. As state earlier that internal mixer was operated at high shear rate while the twin screw extruder was processed at low shear rate. This declaration would be clearly explained that shorter fibre length is noticeable observed for the internal mixer and vice versa for the twin screw extruder. Therefore, it can be concluded that the final length of fibres retained in the compound are independent on the initial length of the fibres. These find out enlighten that only 8 mm initial fibre length was used through this research work.

It would had the argument that the fibre content added in the blends would have another affect on the fibre length reduction due to its increase in shear viscosity. Table 4.3 shows the length of the fibres obtained from the reinforced PC derived from the three mixers used in the study. By utilising the average initial length of 8 mm, the characterised results show that, as expected and for the given composition, internal mixer gives rise to the shortest final fibre length and single screw mixer leads to the longest one. It is also found that the longest length is obtained when using only 5% fibre loaded. Adding more than 5% loading would result in the shorter length and its length is insignificantly depended on the fibre content. The higher viscosity and consequently increasing in fibre-fibre shear force would be the most important role for the explanation. Vice versa adding 1% of the epoxy compatibiliser would decrease in the mixture viscosity and hence reducing the solid-solid shear force. This statement can be evidently observed, for all type of fibre used, from the longer final fibre length obtained in compatibilised system when compared with the compounds without adding epoxy compatibiliser.

Table 4.3 The final length of glass, kevlar, and carbon fibres from PC compounds mixed by using internal mixer, twin screw and single screw extruder, respectively.

Compounding Ingredient (%w/w)		IM ¹ (mm)		TSE ² (mm)		SSE ² (mm)	
		Uncompatibilised system	Compatibilised system	Uncompatibilised system	Compatibilised system	Uncompatibilised system	Compatibilised system
PC:GF	95: 5	0.05119	0.07166	0.460	0.490	2.050	2.260
	90:10	0.04925	0.06810	0.420	0.450	1.730	2.120
	85:15	0.04887	0.06665	0.340	0.420	1.700	1.980
PC:KF	95: 5	0.13886	0.15901	0.480	0.510	2.420	2.530
	90:10	0.13565	0.15410	0.450	0.490	2.140	2.410
	85:15	0.13410	0.15125	0.400	0.430	2.030	2.290
PC:CF	95: 5	0.13101	0.14919	0.450	0.510	1.990	2.180
	90:10	0.13036	0.14786	0.420	0.460	1.930	2.060
	85:15	0.14285	0.14535	0.310	0.430	1.600	1.930

¹analysed by diffraction particle size analyser

²analysed polarized optical microscope

4.3 Properties of PC and Compatibilised PC

The performance properties by means of thermal and mechanical properties of PC and its compatibilised using 1% of epoxy/DDS system and mixed by four mixing procedures as described previously, are summarized in Table 4.4. The results show that the HDT of compatibilised PCs are averagely 131°C. They are approximately

6°C lower than the reference PC obtained in the same processing manner that show the thermal property at 138°C. According to well known Fox's equation, the dilution effect of the miscible highly branched epoxy added, which has lower HDT than PC, could be explained.

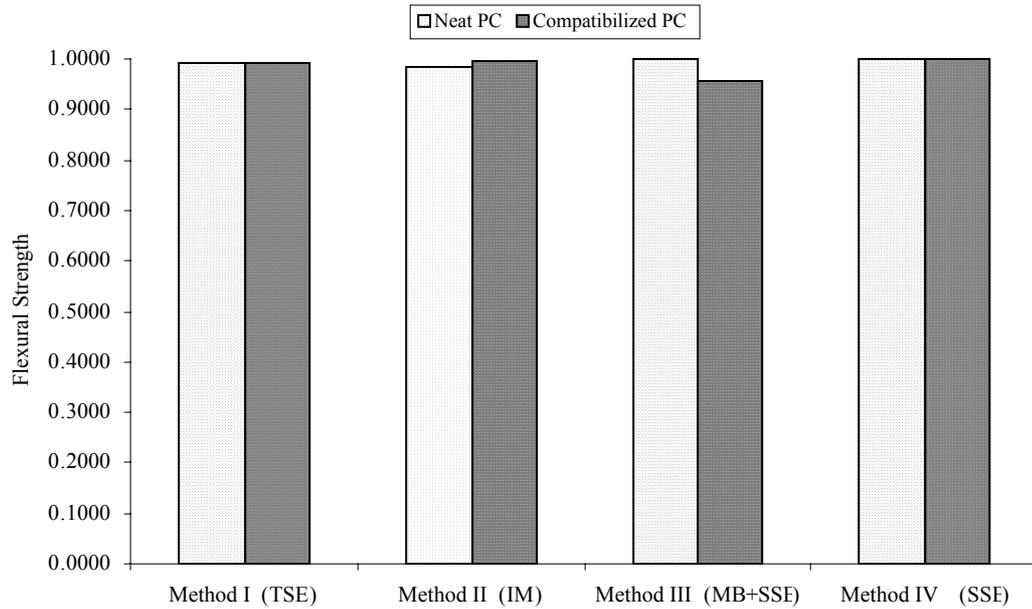
One of the drawback properties of PC is known as notched sensitive materials. Consequently, the unnotched specimens were exploited in the impact testing. By using the maximum impact energy applied, 5.4 J, the entire test samples were not broken during the test. These indicated that the impact strength of both PC specimens were greater than 106 kJ/m².

Flexural testing, using three points bending test fixture, was an experiment applied to evaluate the fracture toughness the compounds. The results in Table 4.4 review that the average tested values of flexural stress of the epoxy compatibilised PCs processed from the given methods are in the region of 99 MPa that are slightly higher than the reference PCs that are calculated at 98 MPa. These figures are independent on the mixing machines. Except for the sample obtained from method III, where masterbatch and single screw were used, that its value is noticeable lower than the virgin PC. It could be due to the vast degradation of the polymer or highly branched compatibiliser. As in the preparation procedure, it was repeatedly passed through the mixers. Thermal and shear induced degradation would be potentially occurred.

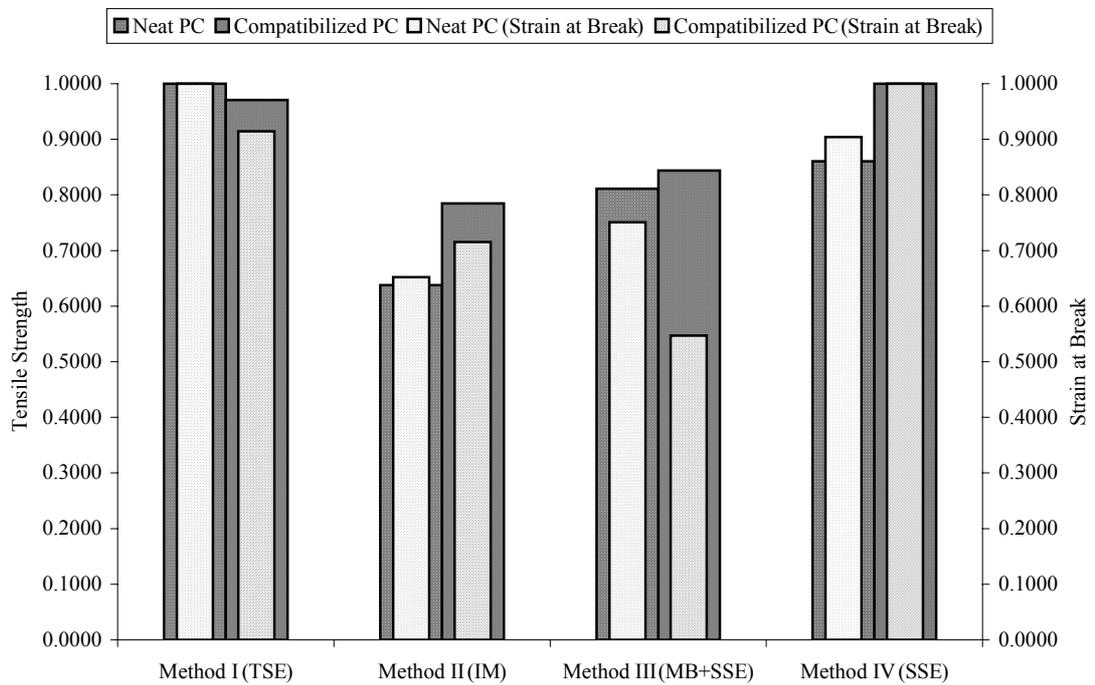
The tensile strength by means of ultimate tensile stress, % strain at break, and Young's modulus of the compatibilised PC are slightly higher than the value obtained from the neat PC for all parameters. Except for the system where the samples were prepared using masterbatch, Method III, which is again obviously lower. This

indication can also be explained by statement as mentioned before.

Taking both flexural and tensile testing into account, it could be pronounced that the compatibilised PC has superior properties than neat PC. That would be contributed from the fracture toughening of the epoxy added.



(a)



(b)

Figure 4.1 Normalised plot of the properties of PC and compatibilised PC;

(a) Flexural Property, and (b) Tensile Property.

Table 4.4 Thermal and mechanical properties of PC and PC with compatibiliser.

Properties	Method I (TSE)		Method II (IM)		Method III (MB+SSE)		Method IV (SSE)	
	PC	PC with Compatibiliser	PC	PC with Compatibiliser	PC	PC with Compatibiliser	PC	PC with Compatibiliser
HDT (°C)	138±0.0	133±0.0	137±0.6	132±0.6	138±0.6	131±0.6	138±0.6	131±0.6
Impact Strength (kJ/m ²)	na.*	na.	na.	na.	na.	na.	na.	na.
Flexural at Maximum Stress (MPa)	98.17±1.66	98.88±3.94	97.67±1.57	99.23±0.55	99.10±1.28	95.36±0.75	99.09±1.68	99.98±0.27
Ultimate Tensile Stress (MPa)	67±0.7	68±0.5	43±2.6	55±5.5	55±0.6	58±3.0	58±0.7	70±0.2
Tensile Strain at Break (%)	12±0.5	12±0.4	8±0.4	9±0.8	9±0.4	7.±0.5	10±1.4	12±0.4
E-Modulus (MPa)	940±22.6	957±40.8	901±14.4	963±9.9	968±25.4	991±28.0	978±9.8	981±10.1

*Unbreakable Sample

In order to verify the dependency of the mixing methods on the properties of the neat PC and the compatibilised PC compound, the Kruskal-Wallis ANOVA test as summarised in Appendix B was applied. Typically, two statistical hypotheses, null(H_0) and alternative(H_1) will be identified as shown below;

H_0 : The four mixing methods can not be differentially evaluated

and H_1 : One way or another, the mixing method can be differentially evaluated

If H_0 is accepted, it means that the properties of the compounds do not depend on the mixing procedure. Vice versa, accepting H_1 means using different mixer would cause in the variation of the properties of the final composite.

In this study, the level of significant(α) for accepting H_0 is given at 0.05 or 95% confidential. For example, using the 20 data samples of the flexural property by means of maximum stress(MPa), the tested data are summarized in Table 4.5.

From Table 4.5, the maximum stresses are ranked from the smallest, 95.59 MPa, assigned as R_1 to the largest, 100.94 MPa, R_{20} . The critical region of approximate size, $\alpha=0.05$, is corresponded to the values of T greater than the 0.95. The random sample(k) assigning as mixing methods is equal to 4 and hence the degree of freedom($k-1$) equal to 3 and there are no ties. The quantile of a chi-squared random variable, taken from Table C1 in Appendix C, is 5.660.

The calculated T using equation B5, as shown in Appendix B, is equal to 2.360. It is seen that the calculated T is smaller than the value given in Table C1 at 95% confidential. This calculation leads to the acceptance of H_0 . It means that the flexural property at maximum stress of the neat PC does not depend on the mixing methods.

Table 4.5 The ANOVA test of the flexural property of neat PC.

Method I (TSE)		Method II (IM)		Method III (MB+SSE)		Method IV (SSE)	
Flexural at Maximum Stress(MPa)	Rank (R_{ij})						
98.76	10	99.10	12	100.47	19	98.52	9
97.51	7	96.76	4	98.34	8	96.70	3
99.40	14	97.17	5	97.36	6	100.94	20
95.59	1	95.83	2	100.13	17	98.90	11
99.61	16	99.49	15	99.22	13	100.41	18
R_i	48		38		63		91
n_i	5		5		5		5
R_i^2/n_i	460.8		288.8		793.8		744.2

From the results obtained in Table 4.4 and using the statistical approach described above, the calculated T value for all mixing methods and properties can be determined and summarized in Table 4.6. The calculated T value obtained for the HDT and flexural properties are less than the critical T . Therefore, the H_0 are accepted. On the other hand, the calculated T obtained from the ultimate tensile strength, strain at break, and Young's modulus are greater than the critical T . Therefore, the H_1 are accepted. It means that using different mixers would cause variation in the final product properties.

Table 4.6 Summary of the Kruskal-Wallis test of neat PC properties prepared by Method I, Method II, Method III, and Method IV, respectively.

Tested Properties	R_i^2/n_i				df (k)	The calculated T	The critical T	Conclusion
	Method I (TSE)	Method II (IM)	Method III (MB+SSE)	Method IV (SSE)				
HDT	168.8	44.1	96.3	243.0	3	5.019	7.815	H_0 Accepted
Flexural at Maximum Stress	460.8	288.8	793.8	744.2	3	2.360	5.660	H_0 Accepted
Ultimate Tensile Stress	1620.0	45.0	320.0	845.0	3	17.857	5.660	H_1 Accepted
Tensile Strain at Break	1445.0	51.2	304.2	980.0	3	16.440	5.660	H_1 Accepted
E-Modulus	387.2	51.2	1008.2	1248.2	3	13.994	5.660	H_1 Accepted

Additionally, using Statistical Package for Social Science(SPSS) WindowsTM based commercial software was applied to confirm the statistical analysis. According to the SPSS calculation, if the level of significant(α) is greater than the given value, then, the hypothesis H_0 will be accepted. For the uncompatibilised PC, the exact conclusions are encountered as seen in Table 4.7.

Table 4.7 Summary results of the Kruskal-Wallis test of the properties of neat PC and compatibilised PC mixed by Method I to Method IV using SPSS programming.

Tested Properties		Heat Distortion Temperature (°C)	Flexural at Maximum Stress (MPa)	Ultimate Tensile Stress (MPa)	Tensile Strain at Break (%)	E-Modulus (MPa)
PC	Chi-Square	5.019	2.360	17.857	16.440	13.994
	df	3	3	3	3	3
	Sig.	0.170	0.501	0.000	0.001	0.003
	Conclusion	H_0 Accepted	H_0 Accepted	H_1 Accepted	H_1 Accepted	H_1 Accepted
Compatibilised PC	Chi-Square	8.250	9.656	16.097	17.331	5.126
	df	3	3	3	3	3
	Sig.	0.041	0.022	0.001	0.001	0.163
	Conclusion	H_1 Accepted	H_1 Accepted	H_1 Accepted	H_1 Accepted	H_0 Accepted

Table 4.7 shows the ANOVA test results obtained from the SPSS computer programming with the applied level of significant of 0.05. For the properties of compatibilised PC, it is found that the only calculated significant of Young's modulus is greater than the given value. The rests are less than the given significant values.

From this statistical determination, the preliminary conclusion could be drawn that for the system with compatibiliser their properties are largely depended on the mixing procedures. This prime conclusion would be described by the viscosity reduction and chemical reaction derived from epoxy added.

4.4 Optimisation of Fibre Contents in Reinforced PC Systems

4.4.1 Carbon Fibre

The carbon, kevlar, and glass fibre reinforced PC compounds with and without epoxy compatibiliser were mixed in twin screw extruder. The test specimens of each composition were injected in injection molding as described earlier. The optimal properties were used to validate the best possible fibres content for the PC systems. The twin screw extruder was assumed to be the most appropriate mixer by mean of minimal shear viscosity and homogeneity of the ingredient as mentioned earlier.

Table 4.8 shows the thermal and mechanical properties of carbon fibre reinforced PC with and without epoxy compatibiliser, respectively. The HDT of the samples without epoxy are slightly increased from 135°C to 137°C with increasing fibre content from 5% to 15%. Vice versa, the HDT of the other system are decreased from 136°C to 132°C with increasing fibre content.

The results summarized in Table 4.8 also indicate that the toughness by means of the impact strength, flexural at maximum stress of the PC reinforced with short carbon fibre with and without epoxy compatibiliser are dramatically decreased when more than 10% of the fibre were added.

Considering the tensile properties of the specimen prepared, the test results show that both ultimate tensile strength and strain at break are decreased as the fibre contents increased from 5% to 15% for both systems. However, the Young's modulus of the samples is visibly increased with increasing the fibre loading for the system without epoxy.

From the experimental data, especially the toughness and tensile properties, it could be concluded that over load the carbon fibre for the PC would reduce the toughness of the polymer. Based on the data obtained from this research study, the optimal carbon fibre loading for the PC would be 5% or less for the system with and without epoxy compatibilised.

4.4.2 Kevlar Fibre

Table 4.9 shows the properties resulted from testing of kevlar fibre reinforced PC with and without epoxy compatibiliser and the fibre content varied from 5% to 15%, respectively.

As expected that the HDT of the samples from both systems are slightly increased with increasing fibre content. The maximum values are found at 15% fibre content. The HDT of the epoxy added samples for each fibre loading are 1°C lower than the system without epoxy.

The results summarized in Table 4.9 also indicate that the toughness by means of the impact strength, flexural at maximum stress of the PC reinforced with short kevlar fibre with and without epoxy were increased from 5% loaded to 10%. However, the inferior properties are noticed for further 15% fibre loaded. The highest flexural and impact properties of both systems are established at 10% fibre reinforced samples.

Considering the tensile properties of the specimen obtained, the test results show that both ultimate tensile strength and strain at break are marginally decreased as the fibre contents increased from 5% to 10% but vastly decreased for further 15% fibre incorporation for both systems. On the other hand, the Young's modulus of the samples is insignificantly increased with increasing the fibre content for the system without epoxy. However, the reversed trend is observed for the epoxy compatibilised system. The tensile parameters point out that the higher fibre load the more brittle materials are obtained.

From the experimental data, especially the toughness and the tensile properties, it could be accomplished that over loaded the PC matrix with kevlar fibre would reduce the toughness of the polymer compound. Based on these research outcomes, the optimal kevlar fibre loading for the PC would be 10% for the system with and without epoxy compatibilised.

4.4.3 Glass Fibre

Table 4.10 illustrates the test results of 5%, 10%, and 15% fibre glass reinforced PC with and without epoxy compatibiliser, respectively. For system without epoxy addition, the HDT is gradually but insignificantly increased with the fibre contents. However, it is almost unchanged in the system adding epoxy.

Table 4.8 Thermal and mechanical properties of carbon fibre reinforced PC.

Specimen Compositions (%w/w)	Properties						
	HDT (°C)	Flexural at Maximum Stress (MPa)	Impact Strength (kJ/m ²)	Ultimate Tensile Stress (MPa)	Tensile Strain at Break (%)	E-Modulus (MPa)	
PC:CF	95:5	112.87±10.35	71.59±4.42	49±1.8	5±0.3	1050±24.7	
	90:10	46.79±3.53	34.89±2.71	23±2.8	3±0.4	1048±28.0	
	85:15	48.17±3.12	16.47±1.06	36±3.3	4±0.6	1107±58.9	
PC:CF:ES	95:5:1	125.00±3.93	62.21±2.07	60±6.5	4±0.2	1087±35.3	
	90:10:1	30.22±4.23	7.89±0.48	50±9.8	4±0.3	1060±55.0	
	85:15:1	25.29±5.56	6.18±0.65	32±3.3	3±0.4	1164±48.1	
Remark: PC = Polycarbonate, CF = Carbon Fibre, ES = Epoxy System							

Table 4.9 Thermal and mechanical properties of kevlar fibre reinforced PC.

Specimen Compositions (%w/w)		Properties						
		HDT (°C)	Flexural at Maximum Stress (MPa)	Impact Strength (kJ/m ²)	Ultimate Tensile Stress (MPa)	Tensile Strain at Break (%)	E-Modulus (MPa)	
PC:KF	95:5	134±1.0	100.77±0.61	68.57±1.25	64±2.5	9±1.4	1063±39.8	
	90:10	135±0.0	106.54±2.44	72.31±2.81	66±1.2	8±0.4	1115±27.6	
	85:15	136±1.0	93.96±8.74	49.60±2.43	50±9.8	5±1.5	1169±60.7	
PC:KF:ES	95:5:1	133±0.6	106.64±0.25	50.27±1.95	68±1.0	10±0.6	1083±9.0	
	90:10:1	134±0.6	108.10±2.68	69.22±2.01	65±3.0	9±1.7	1016±52.8	
	85:15:1	135±1.0	61.70±9.18	32.74±2.33	34±4.2	4±0.6	1054±57.5	
Remark: PC = Polycarbonate, KF = Kevlar Fibre, ES = Epoxy System								

Table 4.10 Thermal and mechanical properties of glass fibre reinforced PC.

Specimen Compositions (%w/w)		Properties						
		HDT (°C)	Flexural at Maximum Stress (MPa)	Impact Strength (kJ/m ²)	Ultimate Tensile Stress (MPa)	Tensile Strain at Break (%)	E-Modulus (MPa)	
PC:GF	95:5	135±1.0	95.60±0.63	Unbreak	53±8.8	9±2.5	1146±36.8	
	90:10	137±0.0	103.44±0.47	65.49±1.78	62±0.6	9±0.2	1212±34.1	
	85:15	138±0.6	106.18±0.56	66.51±1.21	64±1.4	9±0.4	1240±54.3	
PC:GF:ES	95:5:1	136±0.0	102.61±1.58	Unbreak	66±1.3	10±0.3	1084±45.4	
	90:10:1	136±0.6	108.05±2.34	76.43±1.59	68±1.4	10±0.5	1165±67.4	
	85:15:1	137±0.0	110.02±0.32	79.31±1.33	68±0.5	10±0.1	1213±39.7	
Remark: PC = Polycarbonate, GF = Glass Fibre, ES = Epoxy System								

The impact and flexural properties that describe the fracture toughness of the materials, are also rose with the content for the both samples. Within this experiment, the maximum figures are attained at 15% except for the impact strength where unbreakable test results are evidenced at 5%. The impact and flexural figures of the compatibilised system are estimably 15% and 5% superior than the uncompatibilised.

Ultimate tensile stress and Young's modulus are increased with increasing the glass contents except for the strain at break that is constant. In comparison between the system with and without compatibiliser, the stress from the later system is approximately 20% higher than the former one. Further increasing in fibre glass loading, for example more than 40%, would create high void content in the blends and hence inferior the mechanical properties(Thomason and Vluc, 1996). Based on this research data, it could be stated that the optimal glass fibre loading for the PC would be 15% for both systems with and without epoxy compatibilised.

With in the system studied and from the data discovered, the optimal fibre concentration that could be added into the PC blends are 5%, 10% and 15% for carbon, kevlar and glass fibre for both systems, respectively.

4.5 Mixing Evaluation

The results from previous section found that the optimal fibre content of the PC compound with and without epoxy compatibiliser are 5%, 10%, and 15% for carbon, kevlar, and glass fibres, respectively. In this section the heat distortion temperature(HDT), flexural at maximum stress, impact strength, ultimate tensile stress, tensile strain at break, and Young's modulus at the specified content will be

used to evaluate the mixing efficiency of the mixers employed. Later of this topic, both compatibilised and uncompatibilised systems will further be compared in order to evaluate the significant of the epoxy compatibiliser used in this study.

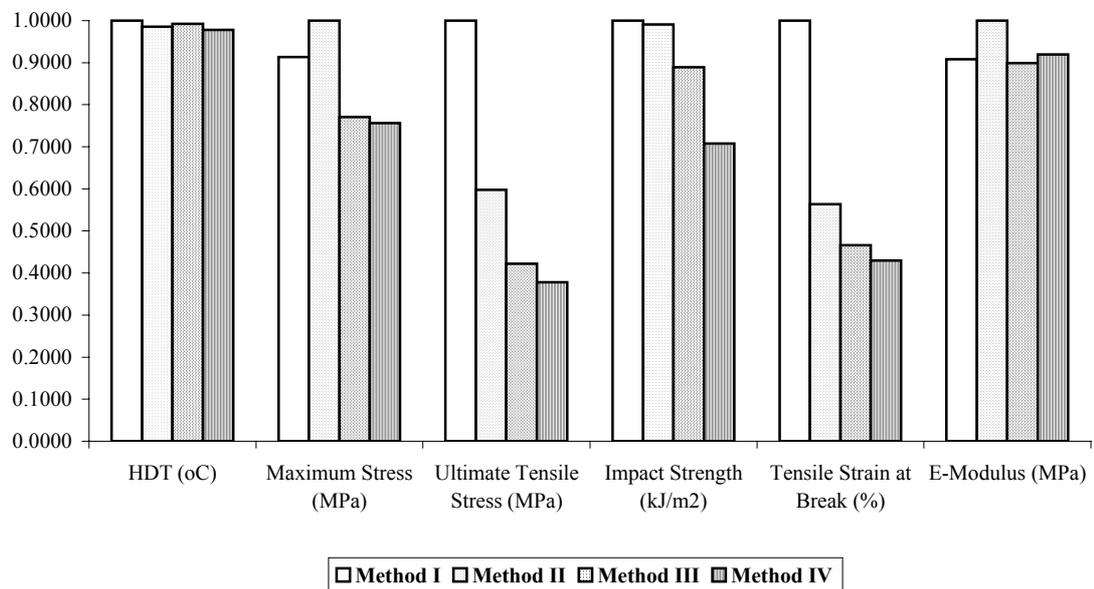
4.5.1 The Mixers

The PC compound with 5% carbon, 10% kevlar, and 15% glass fibre with and without adding the compatibiliser were prepared in twin screw extruder, Method I, the internal mixer, Method II, from in house masterbatch and diluted in the single screw extruder, Method III, and finally in single screw extruder only, Method IV, respectively. The thermal and mechanical properties of the compound obtained from those mixing methods are summarized in Table 4.11 to Table 4.13 and also they are plotted on the normalised scale shown in Figure 4.2 to Figure 4.4, correspondingly.

Table 4.11 and Figure 4.2 show the tested properties and the normalized plot of the uncompatibilised 5% carbon short fibre reinforced PC. The maximum HDT, impact strength, ultimate tensile stress and strain at break of 135°C, 71.6 kJ/m², 49 MPa and 5%, respectively, is obtained when the twin screw extruder was engaged. However, the highest values for flexural stress and Young's modulus at 123.5 MPa and 1156 MPa are seen when internal mixer was employed. In contradictory, the lowest values, indicated inferior properties, are found for mixing in the single screw extruder. These found out are in agreement with the previous stated hypothesis that less reduction in molar mass and fibre length, due to the less shear stress derived from the mixer, would contribute to the better properties.

Table 4.11 Thermal and mechanical properties of 5% carbon fibre reinforced PC.

Properties	5 %wt Carbon Fibre			
	Method I (TSE)	Method II (IM)	Method III (MB+SSE)	Method IV (SSE)
HDT(°C)	135±1.0	133±0.6	134±0.6	132±0.6
Flexural Stress(MPa)	112.9±10.4	123.5±1.9	95.2±1.5	93.4±2.2
Impact Strength(kJ/m ²)	71.6±4.4	70.9±1.0	63.7±2.2	50.7±1.1
Ultimate Tensile Stress(MPa)	49±7.8	29±1.1	21±1.0	18±0.7
Tensile Strain at Break(%)	5±0.4	3±0.3	2±0.1	2±0.2
Young's Modulus(MPa)	1050±24.7	1156±40.0	1040±20.0	1063±8.2

PC:Carbon Fibre 95:5 %(w/w)**Figure 4.2** Normalised plot of the properties of 5% carbon fibre reinforced PC.

From the previous section, the \overline{M}_V of PC and the final fibre length obtained from the internal mixer had significantly decreased when compared with the

other mixers especially comparing with twin screw extruder. These results could be used to explain the contribution of the mixer on the final properties of the PC compounds. The mixer that retains the molecular weight and the fibre length of the compound will superior effect on the properties of the final product. With in the acceptable experimental errors, it can be pronounced that the twin screw extruder are the most appropriate mixer for mixing PC with 5% carbon fibre.

Table 4.12 and Figure 4.3 also show the tested properties and the normalized plot of the 10% kevlar short fibre reinforced PC without the epoxy compatibiliser. The data obtained show that both thermal, by means of HDT, and mechanical properties, by means of impact, flexural and tensile tests, of the samples derived from twin screw extruder are 135°C, 72.3 kJ/m², 106.5 MPa, 66 MPa, 8% and 1095 MPa, respectively. There are more superior than the other mixing methods. Considering the toughness parameters, impact, flexural stress and strain at break, the data indicate that the compound obtained from twin screw extruder has higher toughness than mixed in the other mixers. This tendency is similar to those observed for mixing PC with carbon fibre. It could be strengthen the conclusion that twin screw mixer are the most suitable mixer for compounding PC and the fibres.

Table 4.12 Thermal and mechanical properties of 10% kevlar fibre reinforced PC.

Properties	10 %wt Kevlar Fibre			
	Method I (TSE)	Method II (IM)	Method III (MB+SSE)	Method IV (SSE)
HDT(°C)	135±0.0	133±0.6	134±0.6	133±0.6
Flexural Stress(MPa)	106.6±2.5	103.7±0.8	97.2±1.7	87.2±8.9
Impact Strength(kJ/m ²)	72.3±2.8	71.7±1.4	63.0±1.0	55.5±1.3
Ultimate Tensile Stress(MPa)	66±1.2	23±5.5	32±2.5	34±5.4
Tensile Strain at Break(%)	8±0.4	3±0.7	4±0.6	4±1.1
Young's Modulus(MPa)	1095±39.4	931±51.1	1015±26.8	1009±49.9

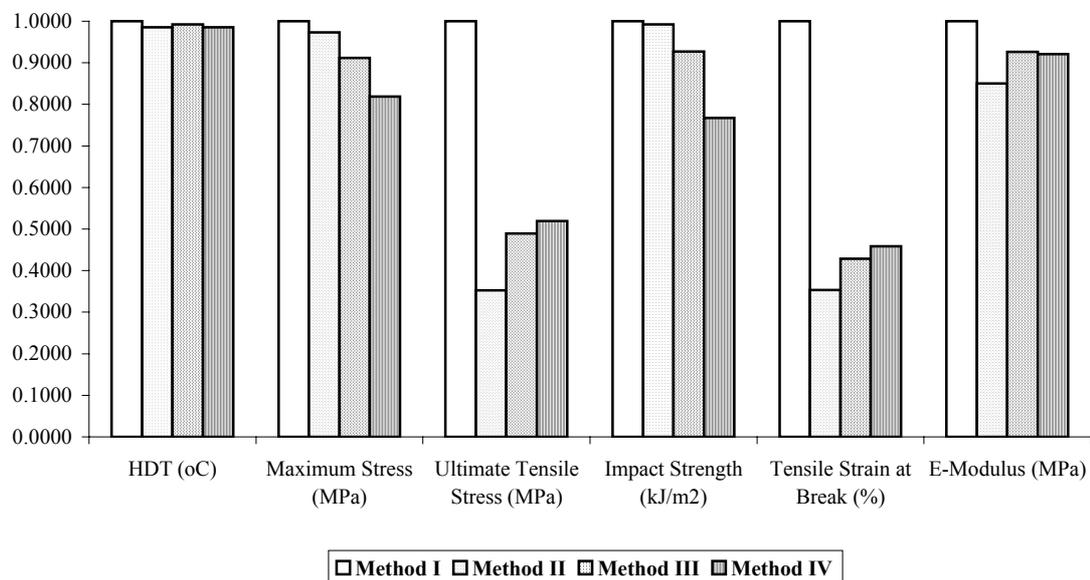
PC:Kevlar Fibre 90:10 %(w/w)**Figure 4.3** Normalised plot of the properties of 10% kevlar fibre reinforced PC.

Table 4.13 and Figure 4.4 review the tested results of the 15% fibre glass reinforced PC prepared by four mixing methodologies. Unlike the above discussions,

it is observed that the properties are independent on the mixers. For example, highest values of the tensile properties of the compound are detected when processed in twin screw extruder. However, the HDT, flexural stress and impact strength are inconsistent with the mixing methods. In majority, however, compounding by using twin screw extruder had overall and distinct superior properties than the others. It is again confirmed that twin screw extruder is the best mixer for mixing PC with carbon, kevlar and glass fibre.

From the observation conducted in this study, the general conclusion could be drawn that twin screw extruder is the most appropriate mixer for mixing the PC with the short fibres. The mixer itself minimises the reduction of chain length and also retaining the fibre length during the processing.

Table 4.13 Thermal and mechanical properties of 15% glass fibre reinforced PC.

Properties	15 %wt Glass Fibre			
	Method I (TSE)	Method II (IM)	Method III (MB+SSE)	Method IV (SSE)
HDT(°C)	138±0.6	137±0.6	137±0.6	136±0.6
Flexural Stress(MPa)	106.2±0.6	102.6±0.7	107.7±0.8	108.4±1.6
Impact Strength(kJ/m ²)	66.5±1.3	71.1±2.5	63.3±1.4	65.0±1.5
Ultimate Tensile Stress(MPa)	64±1.4	47±3.6	61±2.7	55±5.1
Tensile Strain at Break(%)	9±0.4	5±0.6	7±0.9	6±1.3
Young's Modulus(MPa)	1240±54.3	1132±82.7	1146±90.9	1229±31.0

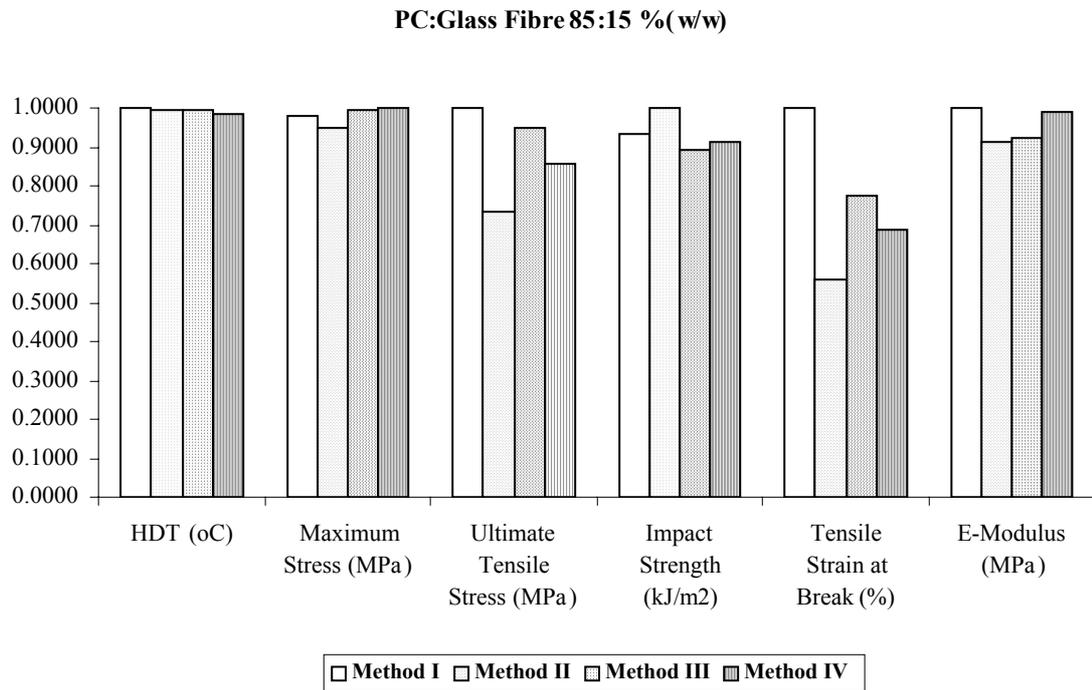


Figure 4.4 Normalised plot of the properties of 15% glass fibre reinforced PC.

4.5.2 Statistical Process Analysis

To emphasize the discussion statements composed earlier, the SPSS computer programming and Kruskal-Wallis calculation as previously described was examined to exclude the twin screw extruder from the other mixers. The statistical test results were presented in Table 4.14(a) to Table 4.14(c).

As state that if the null hypothesis(H_0) is accepted, it would mean that the properties of the compound are not depended on the excluded mixers. However, accepting the alternative hypothesis(H_1) shows the dependency of the properties on the compounding process. Table 4.14(a) shows the statistical testing for 5% carbon fibre added into the compound. The test results reveal that the H_1 are accepted at 95% confidential for all properties testing. These conclusions indicate that there is property variations due to the mixers used. As seen from the previous section and gain the

supportive evidence that the twin screw extruder was the most suitable mixer for mixing 5% carbon fibre with the PC.

Statistical testing for 10% kevlar reinforced PC is summarised in Table 4.14(b). The outcomes show that both testing of HDT and tensile strain at break where the H_0 is accepted. Accepting H_1 is found for the rest of property testing. It is accepted that these two properties are insignificantly influenced by the molar mass of polymer especially for the PC. Omitting these two test results, it could again sustained the judgment made that the twin screw mixer is the prime choice of mixing 10% kevlar with the polymer.

The similar statistical testing conclusions as seen for the kevlar mixture are presented for the 15% glass fibre mixed with PC and summarised in Table 4.14(c). The H_0 of HDT and Young's modulus properties testing are accepted. It is difficult to explained why the Young's modulus is independent on the mixers. The clarification could be hypothesised that the altering in the modulus due to the chain length would be compensated by high reinforcement concentration. More over, the test conclusions are majority voted for the twin screw extruder.

From the statistical analysis, it gains more confident to wrap up that twin screw extruder is among the most appropriate mixer for incorporating the carbon, kevlar and glass fibre with the bisphenol A based polycarbonate.

Table 4.14(a) The Kruskal-Wallis test of 5% carbon fibre reinforced PC properties.

Properties		HDT(°C)	Flexural Stress(MPa)	Impact Strength(kJ/m ²)	Ultimate Tensile Stress(MPa)	Tensile Strain at Break(%)	E-Modulu (MPa)
Chi-Square		9.472	14.840	25.389	17.583	16.816	14.246
df		3	3	3	3	3	3
Sig.	All methods	0.024	0.002	0.000	0.001	0.001	0.003
	Except Method I(TSE)	0.033	0.006	0.000	0.002	0.005	0.002
Conclusion		H_1 Accepted	H_1 Accepted	H_1 Accepted	H_1 Accepted	H_1 Accepted	H_1 Accepted

Table 4.14(b) The Kruskal-Wallis test of 10% kevlar fibre reinforced PC properties.

Properties		HDT(°C)	Flexural Stress(MPa)	Impact Strength(kJ/m ²)	Ultimate Tensile Stress(MPa)	Tensile Strain at Break(%)	E-Modulu (MPa)
Chi-Square		8.927	16.897	25.469	14.497	12.440	14.337
df		3	3	3	3	3	3
Sig.	All methods	0.030	0.001	0.000	0.002	0.006	0.002
	Except Method I(TSE)	0.135	0.002	0.000	0.037	0.221	0.027
Conclusion		H_0 Accepted	H_1 Accepted	H_1 Accepted	H_1 Accepted	H_0 Accepted	H_1 Accepted

Table 4.14(c) The Kruskal-Wallis test of 15% glass fibre reinforced PC properties.

Properties		HDT(°C)	Flexural Stress(MPa)	Impact Strength(kJ/m ²)	Ultimate Tensile Stress(MPa)	Tensile Strain at Break(%)	E-Modulu (MPa)
Chi-Square		7.607	15.046	23.300	16.621	14.417	5.583
df		3	3	3	3	3	3
Sig.	All methods	0.055	0.002	0.000	0.001	0.002	0.134
	Except Method I(TSE)	0.141	0.008	0.000	0.005	0.039	0.181
Conclusion		H_0 Accepted	H_1 Accepted	H_1 Accepted	H_1 Accepted	H_1 Accepted	H_0 Accepted

4.5.3 Compatibiliser

In this research work the epoxy system using solid epoxy, YD-019, cured with DDS under stoichiometric ratio, phr equal to 1.5, was used as the compatibiliser in the compounding. The thermal and mechanical properties of the compatibilised and uncompatibilised PC compounds reinforced with short fibres at the desired content were evaluated. The values obtained from the standard tests are summarized in Table 4.15 to Table 4.17, respectively.

The properties of the PC reinforced with 5% short carbon fibre with and without adding the epoxy system is shown in Table 4.15. The HDT indicates that the compatibilised compounds had approximately 1-2 °C lower than the uncompatibilised ones for all mixing methods. The reduction in thermal characteristic could be due to the dilution effect of the highly branched epoxy chain in the PC matrix. Moreover,

when considering the mechanical properties of the PC compounds, impact and flexural strength, and tensile elongation, the tested results show that there are no obvious improvements or incompetence between two compounding systems. These results will be explained by the SEM analysis later on. Likewise, there is no dependency on the mixers.

Table 4.16 illustrates the test figures deriving from 10% kevlar reinforced PC prepared by four different methods. Similar trend that observed from PC mixed 5% carbon fibre is repeated. The HDT of compatibilised compound is a few degrees lower than the uncompatibilised one and it is not depended on mixing procedures. Comparing between two systems, incorporating highly branched epoxy into the PC mixed with 10% kevlar has generally inferior properties than the system without epoxy. There is also no dependency among the mixing modes.

Finally, the Table 4.17 shows the test data resulting from 15% fibre glass mixed with PC. Generally, the HDT of the fibre glass reinforced PC is slightly higher than that found in the other fibres for both compounding system. However, the temperature obtained from compatibilised compound still a few degrees lower than the other one. Conversely to those reinforced systems, the mechanical properties, flexural and tensile, of the system having epoxy are obviously superior than the compound with no epoxy added. Especially, in the mixing systems using internal mixer, method II, masterbatch process, method III, and single screw extruder, method IV, where the improvements of properties by using compatibiliser are significantly observed. These improvements could be due to fact that the epoxy system would increase the adhesion between the fibre and the dispersing polymer phase. This proposal will be confirmed the SEM photographs later in this chapter.

From the above results, adding the epoxy into the PC compounds not only reduce the shear viscosity of the mixing ingredient but, ambiguously, also induce the chemical reaction between the compatibiliser and the polymer. The reaction would persuade the chain shortening and consequently encounter for the inferior properties as seen in the carbon and kevlar. Nevertheless, the shorter chain length due to the reaction but retaining the fibre aspect ratio as reducing the shear viscosity and increasing in the adhesion bonding between the reinforcement and the polymer matrix as seen for the fibre glass would overcome the former effect. These phenomenons would assist the strength properties of the final PC reinforced compound.

Table 4.15 The thermal and mechanical properties of carbon fibre reinforced PC with and without compatibiliser.

Properties	5 %wt Carbon Fibre															
	Method I (TSE)				Method II (IM)				Method III (MB+SSE)				Method IV (SSE)			
	With		Without		With		Without		With		Without		With		Without	
		%change		%change		%change		%change		%change		%change		%change		%change
Heat Distortion Temperature (°C)	136±0.6	+0.74	135±1	-3.10	129±1	-3.10	133±0.6	-3.10	132±0.6	-3.10	134±0.6	-1.52	130±0.6	-1.54	132±0.6	-1.54
Flexural at Maximum Stress (MPa)	125.00 ±3.93	+9.70	112.87 ±10.35	-55.10	79.63 ±1.65	-55.10	123.51 ±13.93	-55.10	87.66 ±8.21	-55.10	95.16 ±1.46	-8.56	81.17 ±9.63	-15.10	93.43 ±2.24	-15.10
Impact Strength (kJ/m ²)	62.21 ±2.07	-15.08	71.59 ±4.42	-18.77	59.72 ±0.63	-18.77	70.93 ±0.99	-18.77	63.60 ±1.22	-18.77	63.68 ±2.23	-0.12	50.65 ±0.89	-0.02	50.66 ±1.12	-0.02
Ultimate Tensile Stress (MPa)	61±6.5	+19.67	49±1.8	+25.64	39±2.6	+25.64	29±1.1	+25.64	34±5.4	+25.64	21±1.0	+38.24	27±1.7	+33.33	18±0.7	+33.33
Tensile Strain at Break (%)	4±0.2	-25.00	5±0.4	0.00	3±0.3	0.00	3±0.3	0.00	3±0.4	0.00	2±0.1	+33.33	3±0.4	+33.33	2±0.2	+33.33
E-Modulus (MPa)	1087 ±35.3	+3.40	1050 ±24.7	+1.78	1177 ±88.8	+1.78	1156 ±40.0	+1.78	1044 ±50.7	+1.78	1040 ±20.0	+0.38	1024 ±59.4	-3.91	1064 ±8.2	-3.91
Remark: with = carbon fibre reinforced PC with compatibiliser, without = carbon fibre reinforced PC without compatibiliser																

Table 4.16 The thermal and mechanical properties of kevlar fibre reinforced PC with and without compatibiliser.

Properties	10 %wt Kevlar Fibre											
	Method I (TSE)			Method II (IM)			Method III (MB+SSE)			Method IV (SSE)		
	With	Without	%change	With	Without	%change	With	Without	%change	With	Without	%change
Heat Distortion Temperature (°C)	134±0.6	135±0.0	-0.75	131±0.6	133±0.6	-1.53	133±0.6	134±0.6	-0.75	132±0.6	133±0.6	-0.76
Flexural at Maximum Stress (MPa)	108.10 ±2.68	106.54 ±2.44	+1.44	102.67 ±0.42	103.68 ±0.77	-0.98	103.66 ±1.20	97.16 ±1.73	+6.27	103.34 ±2.42	87.23 ±8.89	+15.59
Impact Strength (kJ/m ²)	69.22 ±2.01	72.31 ±2.81	-4.46	67.56 ±1.48	71.75 ±1.45	-6.20	60.33 ±0.72	67.03 ±0.96	-11.10	51.23 ±0.97	55.46 ±1.35	-8.26
Ultimate Tensile Stress (MPa)	65±3.0	66±1.2	-1.54	24±1.0	23±5.5	+4.17	34±5.3	32±2.5	+5.88	55±6.1	34±5.4	+38.18
Tensile Strain at Break (%)	9±1.7	8±0.4	+11.11	2±0.1	3±0.7	-50.00	4±0.9	4±0.6	0.00	6±1.2	4±1.1	+33.33
E-Modulus (MPa)	1016 ±52.8	1116 ±27.6	-9.84	1004 ±31.5	931 ±51.1	+7.27	1009 ±57.4	1015 ±26.8	-0.59	996 ±7.0	1009 ±49.9	-1.30
Remark: with = kevlar fibre reinforced PC with compatibiliser, without = kevlar fibre reinforced PC without compatibiliser												

Table 4.17 The thermal and mechanical properties of glass fibre reinforced PC with and without compatibiliser.

Properties	15 %wt Glass Fibre											
	Method I (TSE)			Method II (IM)			Method III (MB+SSE)			Method IV (SSE)		
	With	Without	%change	With	Without	%change	With	Without	%change	With	Without	%change
Heat Distortion Temperature (°C)	137±0.0	138±0.6	-0.73	136±0.6	137±0.6	-0.74	135±0.6	137±0.6	-1.48	135±0.6	136±0.6	-0.74
Flexural at Maximum Stress (MPa)	110.02 ±0.32	106.18 ±0.56	+3.49	115.28 ±1.43	102.62 ±0.73	+10.98	112.39 ±1.22	107.72 ±0.85	+4.16	120.95 ±1.16	108.40 ±1.56	+10.38
Impact Strength (kJ/m ²)	79.31 ±1.33	66.51 ±1.27	+16.14	91.53 ±1.03	71.08 ±2.50	+22.34	88.34 ±1.60	63.33 ±1.40	+28.31	82.14 ±1.62	64.99 ±1.47	+20.88
Ultimate Tensile Stress (MPa)	68±0.5	64±1.4	+5.88	73±0.3	47±3.6	+35.62	70±0.3	61±2.7	+12.86	73±1.1	55±5.1	+24.66
Tensile Strain at Break (%)	10±0.1	9±0.4	+10.00	8±0.2	5±0.6	+37.50	9±0.3	7±0.9	+22.22	9±0.4	6±1.3	+33.33
E-Modulus (MPa)	1213 ±39.7	1240 ±54.3	-2.23	1257 ±28.4	1132 ±82.7	+9.94	1239 ±39.4	1146 ±90.9	+7.51	1278 ±110.0	1229 ±31.0	+3.83
Remark: with = glass fibre reinforced PC with compatibiliser, without = glass fibre reinforced PC without compatibiliser												

In order to validate the above statement, the two tails t -test statistical method, was applied to differentiate the properties of the short fibres reinforced PC of both systems by the mathematical mean that;

$$\frac{\sum(x_i - y_i)}{N} = 0 \quad (4.1)$$

$$\frac{\sum x_i}{N} - \frac{\sum y_i}{N} = 0 \quad (4.2)$$

then
$$\bar{X} - \bar{Y} = 0 \quad (4.3)$$

where x_i = the properties of the compound with compatibiliser.

y_i = the properties of the compound without compatibiliser.

and N = number of specimens testing.

Typically, two statistical hypothesizes, null (H_0) and alternative (H_1) will be identified as shown below;

if H_0 : $\bar{X} - \bar{Y} = 0$, accepted it means that x and y is analogous,

and if H_1 : $\bar{X} - \bar{Y} \neq 0$, accepted it means that x and y is, somehow, difference.

According to the t -test method, the t value can be computed. It is know that if t value fall into the range of $-t_{1-\alpha/2} < t < t_{1-\alpha/2}$, where $1-\alpha$ is degree of confidentiality, the H_0 is accepted. However, the H_0 would be rejected when calculated t lies outside the calculated t value, $-t_{1-\alpha/2} > t > t_{1-\alpha/2}$.

In this study the degree of confidential to accept H_0 is set at 95% and, therefore, the level of the significant (α) is 5% or 0.05. The null hypothesis, H_0 , will be accepted when the calculated t is felt between $t_{0.975}$ and $-t_{0.975}$.

The calculated t can be determined by using the equation 4.4 as show below.

$$t = \frac{(\bar{X} - \bar{Y})}{S_P \sqrt{1/n_x + 1/n_y}} \quad (4.4)$$

$$S_P = \sqrt{\frac{(n_x - 1)S_x^2 + (n_y - 1)S_y^2}{(n_x + n_y - 2)}} \quad (4.5)$$

and

$$S_i = \frac{\sum(x_i - \bar{X})^2}{n_i - 1} \quad (4.6)$$

where S_i is the variant of i -data and $(n_x + n_y - 2)$ is the degree of freedom used in the test.

The statistical test results of PC reinforced with the short glass fibre are summarized in Table D1 to Table D4 in Appendix D. Only one pair of data point, HDT property of glass fibre reinforced PC with and without compatibiliser mixed by Method I is shown. By using the equation 4.4 and the degree of freedom of 4 the t value equal to -2.012. From the statistical table for the t -test as shown in Appendix C, it reveals that the critical value of t at degree of freedom 4 and level significant of 5%, $\alpha/2$ equal to 0.025, are ± 2.78 . In comparison between the calculated t and the value obtained from the referee table, it is found that the calculated t is felt onto the H_0 hypothesis region, $\pm t_{(1-\alpha/2)}$. As the result, H_0 is accepted. It means that the HDT property of glass fibre reinforced PC with and without compatibiliser is analogous.

By using SPSS commercial computer programming to assist and confirm the statistical calculation shown earlier, the exact conclusion is encountered. According to the SPSS calculation, the level of significant, Sig., will be identified. If value of the Sig. of the calculated t , $|t|$, which is also calculated from the equation 4.4,

is greater than the given level of significant, then, the hypothesis H_0 will be accepted. By using the computer programming, shown in Appendix D, the Sig., 2-tailed, of the data for the HDT property of glass fibre reinforced PC is 0.184. The applied level of significant for this test is 0.05. Therefore, it is concluded that the Sig. value is greater than the level of significant. Taken the result from the previous manually calculation t -test and also from the SPSS commercial software, it could strongly suggest that the HDT property of glass fibre reinforced PC with compatibiliser is identical to the HDT obtained from the glass fibre reinforced PC without compatibiliser. Statistically, this statement is true with the 95% degree of confidence.

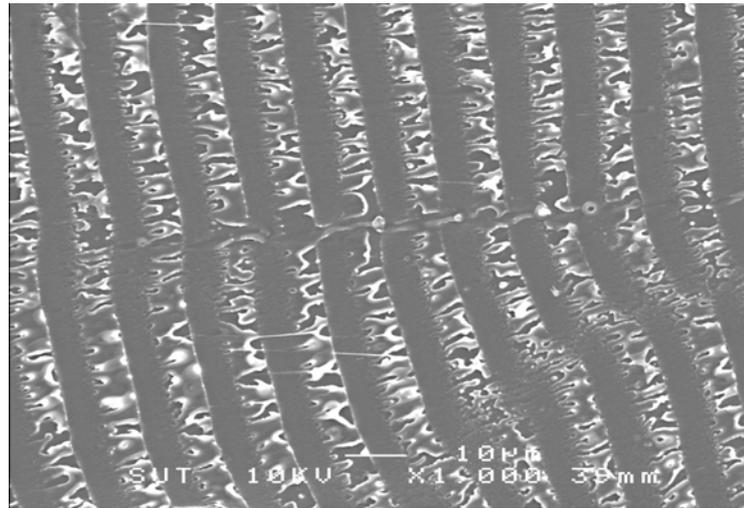
The rest of the statistical property testing for the fibre glass reinforced PC, the alternative hypothesis, H_1 , are mostly accepted except testing for the Young's modulus. It means that the properties of the compatibilised compound are differed from the uncompatibilised material and it is independency on the mixers. However, for the kevlar and carbon fibre systems, with great difficulty, the general conclusions obtained from the statistical investigation can not be made.

From both the results obtained directly experimental testing and also from the statistical, it can be pronounced that the properties of glass fibre reinforced PC are improved when epoxy is used as the compatibiliser for all mixing methods. Excepting for the HDT and Young's modulus that are not differentiated. The properties of kevlar and carbon fibre reinforced PC are slightly decreased when adding epoxy as the compatibiliser and the interior are depended on the mixers employed.

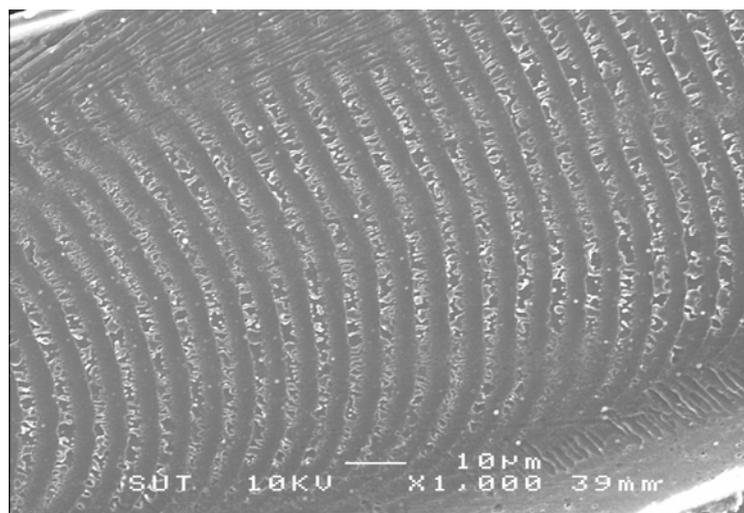
4.6 Morphological Investigation

The SEM micrographs of the fractured surface, obtained from impact testing, of the PC, PC with compatibiliser and fibres reinforced PC with and without compatibiliser, processed through the twin screw extruder, at the magnification of X1000 are shown in Figure 4.5 to Figure 4.8, respectively.

The SEM micrographs of the neat PC and epoxy compatibilised PC are shown in Figure 4.5. It is visually observed that the miscibility between epoxy and PC is obtained. It is also clearly seen from the fracture surface of the neat PC that it has larger and rougher crack propagated trace than the epoxy compatibilised polymer. This picture reveals that and the failure propagation took place in the virgin PC faster than the other. It might be indicated that highly branched epoxy added inhibit the crack progression and hence toughening the polymer. This statement is confirmed by the better properties of the compatibilised PC than the reference PC as describe earlier. From the micrograph and the test results, it could be repeatedly concluded that the fracture toughening properties of the PC is further accomplished by incorporating 1% of the highly branched epoxy cured with DDS.



(a)



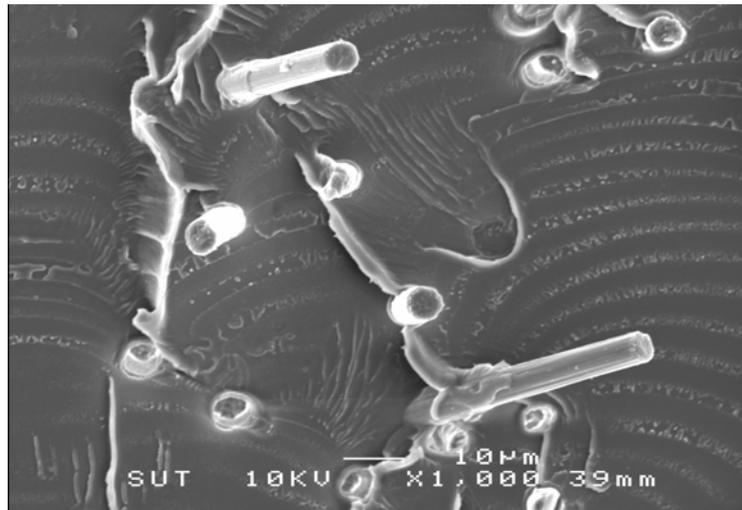
(b)

Figure 4.5 SEM micrographs of the fractured surfaces of (a) neat PC and (b) PC with epoxy compatibiliser at X1000.

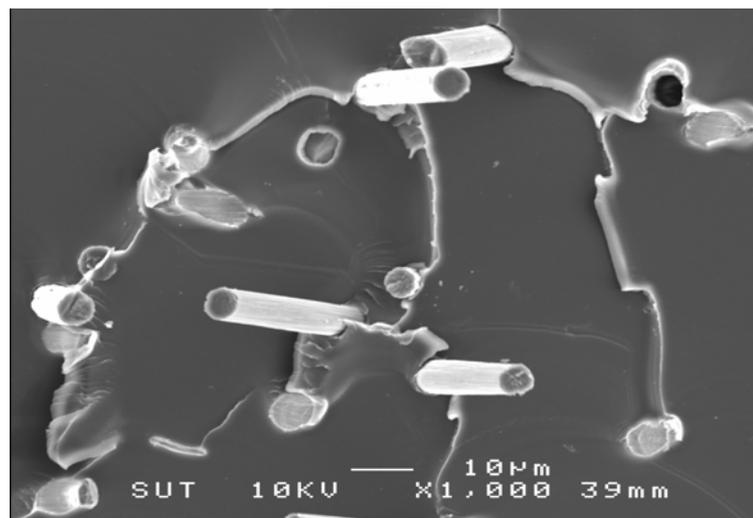
Figure 4.6 illustrates the SEM micrographs of 5% carbon fibre reinforced PC without and with epoxy and mixed by twin screw extruder, respectively. From the picture obtained from two systems, there are the traces for both the fibre pull out and fibre debonding, especially in the compatibilised sample. It is also noticed that there is

evidently space between fibre and the dispersed phase. This observation indicates that the adhesion between the fibre and the polymer matrix can not be crucially enhanced by adding the epoxy. Consequently, the strength properties can not be improved as revealed in the previous section.

Figure 4.7 shows the SEM micrographs of fracture surface of PC reinforced with 10% kevlar fibres without and with compatibiliser, respectively. There is a trace of fibril and the end of filament yarn. It designates the fibre fracture of the high toughness kevlar yarn. This is probably contributed to fracture toughness improvement of the polymer compound. However, having the close inspection at fibre/polymer matrix adhesion phases, it is clearly distinguished, especially for the system without epoxy, the incompatibility between the fibre and the polymer phase. This inhomogeneity can not be much improved by adding the epoxy as the clearly slit still preserve and act as the void deflected for the sample. This phenomenon would easily introduce the fibre pull out mechanism. In accordance, the fracture toughness can not be enhanced. The explanation is in agreement with mechanical test results as introduced previously.

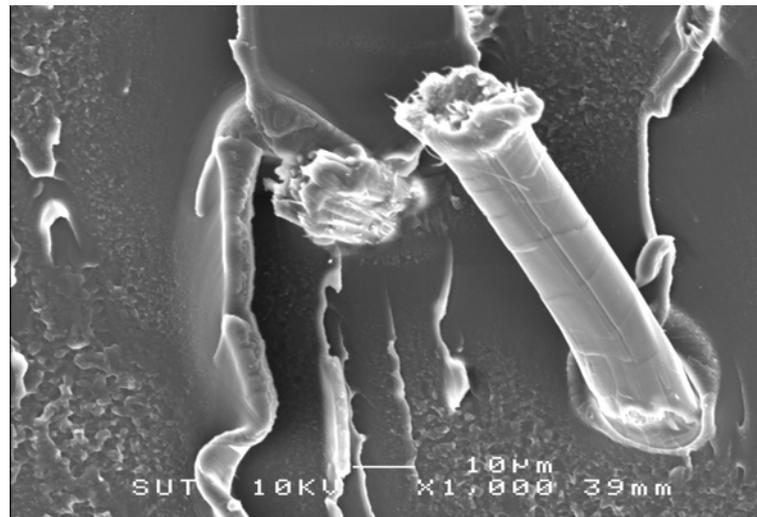


(a)

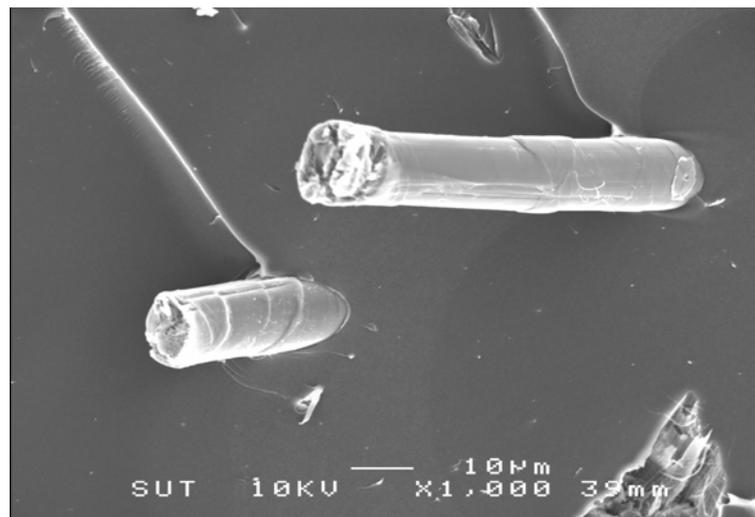


(b)

Figure 4.6 SEM micrographs of the fractured surfaces of 5% carbon fibre reinforced PC of (a) without and (b) with epoxy compatibiliser at X1000.

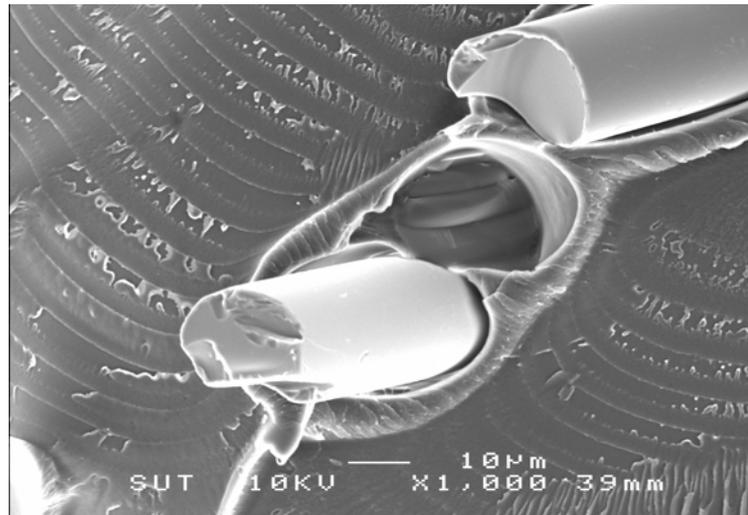


(a)

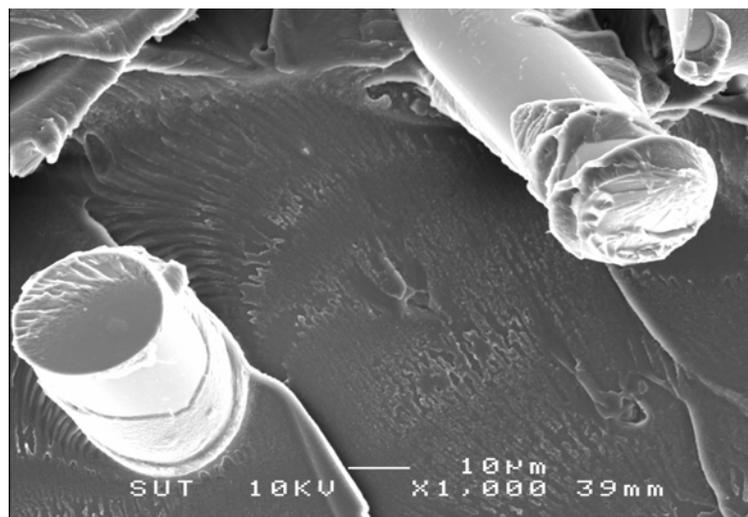


(b)

Figure 4.7 SEM micrographs of the fractured surfaces of 10% kevlar fibre reinforced PC of (a) without and (b) with epoxy compatibiliser at X1000.



(a)



(b)

Figure 4.8 SEM micrographs of the fractured surfaces of 15% glass fibre reinforced PC of (a) without and (b) with epoxy compatibiliser at X1000.

Finally, Figure 4.8 shows the SEM of 15% glass fibre added into the PC compounds without and with epoxy mixed by the twin screw machine, respectively. It is clearly seen that there are fibre fracture and delamination of fibre and polymer matrix in the uncompatibilised sample. Vice versa, only fibre fracture is observed in

the compatibilised system. The delamination would easily initiate the fibre pull out during the fracture mechanism. Therefore, lower fracture toughness would be found. On the other hand, including the epoxy compatibiliser into the compound would enhance the adhesion force between the reinforcement and dispersed polymer. Consequently, higher fracture toughness would be recovered. This conclusion is emphasized by the outcome of the mechanical testing from the earlier section which showed that the compatibilised and fibre glass reinforced PC had superior mechanical properties than the uncompatibilised system.

From the SEM study, a clear conclusion can be made and confirmed that the 1% epoxy compatibiliser were successfully used for the fibre glass reinforced PC. It plays the important role for improving the adhesion between fibre and polymer phase. Nevertheless, there was ineffectively used for the carbon and kevlar ones.

CHAPTER V

CONCLUSIONS

The main aim of this research work including evaluating the mixing efficiency by means of improving of the mechanical properties of fibres reinforced PC prepared by using internal mixer, single and twin screw extruders. Investigation the effect of processing conditions on the aspect ratio of the fibres, and the compatibilisation of epoxy systems in the compounds are also promptly discussed.

From the fibre length analysis, it has been shown that the final fibres length in the PC compound did not depended on the initial length for both uncompatibilised and 1% compatibilised systems. The internal mixer gave rise to the shortest final fibre length but single screw mixer lead to the longest one.

The compatibilisation of PC blends using 1% epoxy and DDS system as compatibiliser mixed in twin screw extruder has shown the lower HDT but superior in mechanical properties than the neat PC. The statistical analysis has been used to strengthen the experimental result. The study has also revealed that the twin screw extruder offers the best alternative for the mixing equipment. The properties of the PC compounds have been varied when the difference mixers were used for both compounding systems. The viscosity reduction and chemical reaction between PC and epoxy has been the prime suspect for the variation.

For reinforcing PC with carbon, kevlar and glass fiber, the optimal fiber contents have been suggested at 5%, 10% and 15% by weight for both systems.

The twin screw extruder has been found the most effective mixer for compounding those fibres reinforced PC. It minimised the chain reduction and also retained the fibre aspect ratio. The statistical analyses have been used to confirm this conclusion.

At the optimal fibre contents, the epoxy compatibiliser has been successfully used to improve the mechanical properties of the fibre glass reinforced PC. However, the properties of kevlar and carbon fibres reinforced PC have not been enhanced by adding the compatibiliser.

The SEM study has indicated that 1% of epoxy was an effective fracture toughener for the PC compound. It has also illustrated that 1% epoxy was successfully used as compatibiliser for the fibre glass reinforced PC as indicated by the adhesion between fibre and polymer phase. Nevertheless, it has not ineffectively used for the carbon and kevlar reinforcements.

Recommendation For Further Work

The main interesting objectives for the further studied related to this research study should be followed:

- (i) To find the appropriate compatibiliser content for kevlar and carbon fibres systems.
- (ii) To investigate the effect of other types of epoxy compatibilisers on the properties of the compounds.
- and (iii) To study effective of epoxy/DDS system at higher concentration.

Research Publication

Parts of this work were published and being presented in the following conferences;

Charoensuk, O. and Meekum, U. (2003). Development of compounding techniques for short fibre reinforced polycarbonate. In **International conference in advances in petrochemicals and polymers in the new millennium** (p.116). Bangkok, Thailand.

Charoensuk, O. and Meekum, U. (2004). Compatibilisation of polycarbonate reinforced with short glass fibre using epoxy system. In **The 4th national symposium on graduate research** (p.73). Chiang Mai, Thailand.

Meekum, U. and Charoensuk, O. (2005). Polycarbonate reinforced short fibres compatibilised with highly branched epoxy system. In **31st Congress on science and technology of Thailand**. Nakhon Ratchasima, Thailand.

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APPENDIX A

THE VISCOSITY MOLECULAR WEIGHT RELATIONSHIP

The Viscosity-Molecular Weight Relationship

The limiting viscosity number $[\eta]$ of a solution which has long been called the intrinsic viscosity is defined as

$$[\eta] = \lim_{c \rightarrow 0} \frac{\eta - \eta_0}{\eta_0 c} \quad (\text{A1})$$

in term of the solvent viscosity, η_0 , the solution viscosity, η , and the solute concentration, c . The concentration, c , is expressed in grams of solute per milliliter of solution or, more frequently, in grams of solute per 100 milliliters of solution, the limiting viscosity number being given in the reciprocal of these unit. Here, following the IUPAC, it is adopted the former unit. The quantity $[\eta]$ of a polymer solution is measure of the capacity of a polymer molecule to enhance the viscosity, which depends on the size and the shape of the polymer molecule. Within a given series of polymer homologs, $[\eta]$ increases with the molecular weight by means of viscosity average molecular weight (\overline{M}_V), hence it is a measure of \overline{M}_V . Experimentally, it is expressed by Mark-Houwink-Sakurada equation:

$$[\eta] = K \overline{M}_V^a \quad (\text{A2})$$

it is now well established that for linear, flexible polymer, under special conditions of temperature or solvent. Where K is a constant, and a is called a Mark-Houwink-Sakurada exponent. Note that K and a are different from polymer to polymer and can depend on the solvent as well.

Table A2 The solvent and the constant value of polymers.

Polymer	Solvent	Temp. (°C)	$K (\times 10^3)$ (ml/g)	a
Polycarbonate (PC)	Tetrahydrofuran	25	39.9	0.70
	Chloroform	25	30.1	0.74
	Methylene Chloride	25	11.9	0.80
Poly(ethylene) (PE)	Decalin	135	62.0	0.70
	Tetralin	130	51.0	0.72
	p-Xylene	105	51.0	0.73
Poly(propylene) (PP)	-	-	-	-
Atactic	Benzene	25	27.0	0.71
Isotactic	Decalin	135	11.0	0.80
syndiotactic	Heptane	30	31.2	0.71
Poly(styrene) (PS)	-	-	-	-
Atactic	Benzene	25	7.8	0.75
Isotactic	Benzene	30	10.6	0.74
Head-to-Head	Tetrahydrofuran	25	53.0	0.61
Ring	Cyclohexane	40	55.6	0.50
Poly(methyl methacrylate) (PMMA)	Acetone	25	5.3	0.73

Source: Brandrup, J., Immergut, E. H., and Grulke, E. A. (1999)

The viscosity measurement by dilution-solution viscometry can be explained in the section 3.4.1 Chapter III. Equation A3, A4, A5, and A6 are used to measure the intrinsic viscosity of the experiment in Mark-Houwink-Sakurada equation.

$$\text{Relative Viscosity; } \eta_{rel} = \frac{\eta_0}{\eta_0} \approx \frac{t}{t_0} \quad (\text{A3})$$

$$\text{Specific Viscosity; } \eta_{sp} = \frac{\eta - \eta_0}{\eta_0} = \frac{t - t_0}{t_0} \quad (\text{A4})$$

$$\text{Reduced Viscosity; } \eta_{red} = \frac{t - t_0}{t_0 c} = \frac{\eta_{sp}}{c} \quad (\text{A5})$$

$$\text{Inherent Viscosity; } \eta_{inh} = \frac{\ln \eta_{rel}}{c} \quad (\text{A6})$$

Where t_0 is solvent flow time in the unit of second, t is solution flow time in the unit of second, and c is the concentration of solution in the unit of grams per deciliter.

APPENDIX B

THE KRUSKAL-WALLIS TEST MANNER

The experimental can be strengthened results by applying the nonparametric statistical methods. Comparisons with parametric methods, nonparametric methods are based on some of the same assumptions on which parametric methods are based, such as the assumption that the sample is a random sample. Nonparametric methods are perfectly robust for distribution assumptions on the population, because they are equally valid for all distributions (Conover, 1999). Even if a parametric test does not depend critically on an assumption that samples come from a distribution in a particular family, when in doubt they may prefer a nonparametric test needing weaker assumptions. More importantly, nonparametric methods are often the only ones available for data that simply specify order, ranks, or count of numbers of events or of individuals in various categories. Nonparametric methods are not assumption-free. In most statistical problems what can deduce, by either parametric or nonparametric methods, depends upon what assumptions can validly be made (Sprent and Smeeton, 2001).

The procedure for the Kruskal-Wallis test is conducted in the following manner:

1. Data

The data consist of k random samples of possibly different sizes. Denote the i th random sample of size n_i by $X_{i1}, X_{i2}, \dots, X_{in_i}$. Then the data may be arranged into columns.

Sample 1	Sample 2	Sample 3
$X_{1,1}$	$X_{2,1}$		$X_{k,1}$
$X_{1,2}$	$X_{2,2}$		$X_{k,2}$
.....
X_{1,n_1}	X_{2,n_2}		X_{k,n_k}

Let N denote the total number of observations

$$N = \sum_{i=1}^k n_i \quad (\text{B1})$$

Assign rank 1 to the smallest of the totality of N observations, rank 2 to the second smallest, and so on to the largest of all N observations, which receives rank N . Let $R(X_{ij})$ represent the rank assigned to X_{ij} . Let R_i be the sum of the ranks assigned to the i th sample.

$$R_i = \sum_{j=1}^{n_i} R(X_{ij}) \quad i = 1, 2, \dots, k \quad (\text{B2})$$

Compute R_i for each sample.

If the ranks may be assigned in several different ways because several observations are equal to each other, assign the average rank to each of the tied observations.

2. Assumptions

1. All samples are random samples from their respective populations.
2. In addition to independence within each sample, there is mutual independence among the various samples.
3. The measurement scale is at least ordinal.
4. Either the k population distribution functions are identical, or else some of the populations tend to yield large values than other

populations do.

3. Test Statistic

The test statistic T is defined as

$$T = \frac{1}{S^2} \left(\sum_{i=1}^k \frac{R_i^2}{n_i} - \frac{N(N+1)^2}{4} \right) \quad (\text{B3})$$

where N and R_i are defined in equation B1 and B2, respectively, and where

$$S^2 = \frac{1}{N-1} \left(\sum_{\substack{\text{all} \\ \text{Ranks}}} R(X_{ij})^2 - N \frac{(N+1)^2}{4} \right) \quad (\text{B4})$$

If there are no ties S^2 simplifies to $N(N+1)/12$, and the test statistic reduces to

$$T = \frac{12}{N(N+1)} \sum_{i=1}^k \frac{R_i^2}{n_i} - 3(N+1) \quad (\text{B5})$$

If the number of ties is moderate there will be very little difference between equation B3 and B5, so the simpler equation B5 may preferred.

4. Null Distribution

The exact distribution of T is given by Table C1 in appendix C for $k=3$ and $n_i \leq 5$, but in general the exact distribution is too cumbersome to work with. Therefore the chi-squared distribution with $k-1$ degrees of freedom is used as an approximation to the null distribution of T.

5. Hypotheses

H_0 : All of the k population distribution functions are identical

H_1 : At least one of the populations tends to yield larger observations than at least one of the other populations

Reject H_0 at the level α if T is greater than its $1-\alpha$ quantile from the null distribution. If $k=3$, all of the sample sizes are 5 or less, and there are no ties, the

exact quantile may be obtained from Table C1 in Appendix C. When there are ties, or when exact tables are not available, the approximate quantiles may be obtained from Table C2 in appendix C, the chi-squared distribution with $k-1$ degree of freedom. Reject H_0 at the level α if T exceeds the $1-\alpha$ quantile thus obtained. The p-value is approximately the probability of a chi-squared random variable with $k-1$ degrees of freedom the observed value of T .

6. Multiple Comparisons

If, and only if, the null hypothesis is rejected, we may use the following procedure to determine which pairs of populations tend to differ. We can say that populations i and j seem to be different if the following inequality is satisfied:

$$\left| \frac{R_i}{n_i} - \frac{R_j}{n_j} \right| > t_{1-(\alpha/2)} \left(S^2 \frac{N-1-T}{N-k} \right)^{1/2} \left(\frac{1}{n_i} + \frac{1}{n_j} \right)^{1/2} \quad (\text{B6})$$

where R_i and R_j are the rank sums of the two samples, $t_{1-\alpha/2}$ is the $(1-\alpha/2)$ quantile of the t distribution obtained from Table C3 in Appendix C with $N-k$ degree of freedom, S^2 comes from equation B4, and T comes from equation B3 or B5. This procedure is repeated for all pairs of populations. The same α level is usually used here as in the Kruskal-Wallis test.

APPENDIX C

STATISTICAL TABLES

Table C1 Quantiles of the Kruskal-Wallis Test Statistic for Small Sample Sizes^a

Sample Sizes	$W_{0.90}$	$W_{0.95}$	$W_{0.99}$
2, 2, 2	3.7143	4.5714	4.5714
3, 2, 1	3.8571	4.2857	4.2857
3, 2, 2	4.4643	4.5000	5.3571
3, 3, 1	4.0000	4.5714	5.1429
3, 3, 2	4.2500	5.1389	6.2500
3, 3, 3	4.6000	5.0667	6.4889
4, 2, 1	4.0179	4.8214	4.8214
4, 2, 2	4.1667	5.1250	6.0000
4, 3, 1	3.8889	5.0000	5.8333
4, 3, 2	4.4444	5.4000	6.3000
4, 3, 3	4.7000	5.7273	6.7091
4, 4, 1	4.0667	4.8667	6.1667
4, 4, 2	4.4455	5.2364	6.8727
4, 4, 3	4.7730	5.5758	7.1364
4, 4, 4	4.5000	5.6538	7.5385
5, 2, 1	4.0500	4.4500	5.2500
5, 2, 2	4.2933	5.0400	6.1333
5, 3, 1	3.8400	4.8711	6.4000
5, 3, 2	4.4946	5.1055	6.8218
5, 3, 3	4.4121	5.5152	6.9818
5, 4, 1	3.9600	4.8600	6.8400
5, 4, 2	4.5182	5.2682	7.1182
5, 4, 3	4.5231	5.6308	7.3949
5, 4, 4	4.6187	5.6176	7.7440
5, 5, 1	4.0364	4.9091	6.8364
5, 5, 2	4.5077	5.2462	7.2692
5, 5, 3	4.5363	5.6264	7.5429
5, 5, 4	4.5200	5.6429	7.7914
5, 5, 5	4.5000	5.6600	7.9800

Source: Conover (1999)

^aThe null hypothesis may be rejected at the level α if the Kruskal-Wallis test statistic, given by equation B5 in Appendix B, exceeds the $1-\alpha$ quantile given in the table.

Table C2 Chi-Squared Distribution^a

	p=0.750	0.900	0.950	0.975	0.990	0.995	0.999
k = 1	1.323	2.706	3.841	5.024	6.635	7.879	10.83
2	2.773	4.605	5.991	7.378	9.210	10.60	13.82
3	4.108	6.251	7.815	9.348	11.34	12.84	16.27
4	5.385	7.779	9.488	11.14	13.28	14.86	18.47
5	6.626	9.236	11.07	12.83	15.09	16.75	20.51
6	7.841	10.64	12.59	14.45	16.81	18.55	22.46
7	9.037	12.02	14.07	16.01	18.48	20.28	24.32
8	10.22	13.36	15.51	17.53	20.09	21.96	26.13
9	11.39	14.68	16.92	19.02	21.67	23.59	27.88
10	12.55	15.99	18.31	20.48	23.21	25.19	29.59
11	13.70	17.28	19.68	21.92	24.73	26.76	31.26
12	14.85	18.55	21.03	23.34	26.22	28.30	32.91
13	15.98	19.81	22.36	24.74	27.69	29.82	34.53
14	17.12	21.06	23.68	26.12	29.14	31.32	36.12
15	18.25	22.31	25.00	27.49	30.58	32.80	37.70
16	19.37	23.54	26.30	28.85	32.00	34.27	39.25
17	20.49	24.77	27.59	30.19	33.41	35.72	40.79
18	21.60	25.99	28.87	31.53	34.81	37.16	42.31
19	22.72	27.20	30.14	32.85	36.19	38.58	43.82
20	23.83	28.41	31.41	34.17	37.57	40.00	45.32
21	24.93	29.62	32.67	35.48	38.93	41.40	46.80
22	26.04	30.81	33.92	36.78	40.29	42.80	48.27
23	27.14	32.01	35.17	38.08	41.64	44.18	49.73
24	28.24	33.20	36.42	39.37	42.98	45.56	51.18
25	29.34	34.38	37.65	40.65	44.31	46.93	52.62
26	30.43	35.56	38.89	41.62	45.64	48.29	54.05
27	31.53	36.74	40.11	43.19	46.96	49.64	55.48
28	32.62	37.92	41.34	44.46	48.28	50.99	56.89
29	33.71	39.09	42.56	45.72	49.59	52.34	58.30
30	34.80	40.26	43.77	46.98	50.89	53.67	59.70
40	45.62	51.81	55.76	59.34	63.69	66.77	73.40
50	56.33	63.17	67.50	71.42	76.15	79.49	86.66
60	66.98	74.40	79.08	83.30	88.38	91.95	99.61
70	77.58	85.53	90.53	95.02	100.4	104.2	112.3
80	88.13	96.58	101.9	106.6	112.3	116.3	124.8
90	98.65	107.6	113.1	118.1	124.1	128.3	137.2
100	109.1	118.5	124.3	129.6	135.8	140.2	149.4

Source: Conover (1999)

^aThe entries in this table are quantiles W_p of a chi-Squared random variable W with k degree of freedom, selected so $P(W \leq W_p) = p$ and $P(W > W_p) = 1-p$.

Table C3 The t Distribution^a

Degree of Freedom	p=0.6	0.75	0.90	0.95	0.975	0.99
1	0.325	1.000	3.078	6.314	12.706	31.821
2	0.289	0.816	1.886	2.920	4.303	6.965
3	0.277	0.765	1.638	2.353	3.182	4.541
4	0.271	0.741	1.533	2.132	2.776	3.747
5	0.267	0.727	1.476	2.015	2.571	3.365
6	0.265	0.718	1.440	1.943	2.447	3.143
7	0.263	0.711	1.415	1.895	2.365	2.998
8	0.262	0.706	1.397	1.860	2.306	2.896
9	0.261	0.703	1.383	1.833	2.262	2.821
10	0.260	0.700	1.372	1.812	2.228	2.764
11	0.260	0.697	1.363	1.796	2.201	2.718
12	0.259	0.695	1.356	1.782	2.179	2.681
13	0.259	0.694	1.350	1.771	2.160	2.650
14	0.258	0.692	1.345	1.761	2.145	2.624
15	0.258	0.691	1.341	1.753	2.131	2.602
16	0.258	0.690	1.377	1.746	2.120	2.583
17	0.257	0.689	1.333	1.740	2.110	2.567
18	0.257	0.688	1.330	1.734	2.101	2.552
19	0.257	0.688	1.328	1.729	2.093	2.539
20	0.257	0.687	1.325	1.725	2.086	2.528
21	0.257	0.686	1.323	1.721	2.080	2.518
22	0.256	0.686	1.321	1.717	2.074	2.508
23	0.256	0.685	1.319	1.714	2.069	2.500
24	0.256	0.685	1.318	1.711	2.064	2.492
25	0.256	0.684	1.316	1.708	2.060	2.485
26	0.256	0.684	1.315	1.706	2.056	2.479
27	0.256	0.684	1.314	1.703	2.052	2.473
28	0.256	0.683	1.313	1.707	2.048	2.467
29	0.256	0.683	1.311	1.699	2.045	2.462
30	0.256	0.683	1.310	1.697	2.042	2.457
40	0.255	0.681	1.303	1.684	2.021	2.423
60	0.254	0.679	1.296	1.671	2.000	2.390
120	0.254	0.677	1.289	1.658	1.680	2.358
∞	0.253	0.674	1.282	1.645	1.960	2.326

Source: Conover (1999)

^aThe entries in this table are quantiles W_p of the t distribution for various degrees of freedom. Quantiles W_p for $p < 0.5$ may be computed from the equation

$$W_p = -W_{1-p}$$

Note that $W_{0.50} = 0$ for all degrees of freedom.

APPENDIX D

STATISTICAL ANALYSIS RESULTS

Mean Comparison Analysis by Using Mathematical Calculation and the SPSS Program

Carbon Fibre

Table D1 Independent sample *t*-test of the carbon fibre reinforced polycarbonate between with and without compatibiliser by mixing Method I (TSE).

Properties	Average mean value		df	Sig. (2-tailed)	The Calculated <i>t</i>	The Critical <i>t</i>	Conclusion
	With ES	Without ES					
Heat Distortion Temperature (°C)	135.67	135.00	4	.387	+1.101	±2.776	H_0 Accepted
Flexural at Maximum Stress (MPa)	125.00	112.87	8	.057	+0.340	±2.306	H_0 Accepted
Impact Strength (kJ/m ²)	62.21	71.59	14	.000	-2.324	±2.145	H_1 Accepted
Ultimate Tensile Stress (MPa)	60.58	48.89	8	.014	+2.609	±2.306	H_1 Accepted
Tensile Strain at Break (%)	3.94	4.72	8	.004	-12.983	±2.306	H_1 Accepted
E-Modulus (MPa)	1087.00	1050.40	8	.098	+0.059	±2.306	H_0 Accepted

Remark : ES = Epoxy System

Table D2 Independent sample *t*-test of the carbon fibre reinforced polycarbonate between with and without compatibiliser by mixing Method II (IM).

Properties	Average mean value		df	Sig. (2-tailed)	The Calculated <i>t</i>	The Critical <i>t</i>	Conclusion
	With ES	Without ES					
Heat Distortion Temperature (°C)	129.00	132.67	4	.010	-6.033	±2.776	H_1 Accepted
Flexural at Maximum Stress (MPa)	79.63	123.51	8	.002	-5.506	±2.306	H_1 Accepted
Impact Strength (kJ/m ²)	59.72	70.93	14	.000	-30.094	±2.145	H_1 Accepted
Ultimate Tensile Stress (MPa)	38.92	29.20	8	.000	+3.103	±2.306	H_1 Accepted
Tensile Strain at Break (%)	2.78	2.66	8	.520	+2.002	±2.306	H_0 Accepted
E-Modulus (MPa)	1177.01	1156.47	8	.655	+0.006	±2.306	H_0 Accepted

Remark : ES = Epoxy System

Table D3 Independent sample *t*-test of the carbon fibre reinforced polycarbonate between with and without compatibiliser by mixing Method III (MB+SSE).

Properties	Average mean value		df	Sig. (2-tailed)	The Calculated <i>t</i>	The Critical <i>t</i>	Conclusion
	With ES	Without ES					
Heat Distortion Temperature (°C)	132.33	134.33	4	.013	-7.356	±2.776	H_1 Accepted
Flexural at Maximum Stress (MPa)	87.67	95.16	8	.111	-0.249	±2.306	H_0 Accepted
Impact Strength (kJ/m ²)	63.60	63.68	14	.935	-0.043	±2.145	H_0 Accepted
Ultimate Tensile Stress (MPa)	34.46	20.65	8	.004	+3.060	±2.306	H_1 Accepted
Tensile Strain at Break (%)	2.70	2.20	8	.048	+6.875	±2.306	H_1 Accepted
E-Modulus (MPa)	1044.30	1039.98	8	.866	+0.004	±2.306	H_0 Accepted

Remark : ES = Epoxy System

Table D4 Independent sample *t*-test of the carbon fibre reinforced polycarbonate between with and without compatibiliser by mixing Method IV (SSE).

Properties	Average mean value		df	Sig. (2-tailed)	The Calculated <i>t</i>	The Critical <i>t</i>	Conclusion
	With ES	Without ES					
Heat Distortion Temperature (°C)	130.33	131.67	4	.047	-4.928	±2.776	H_1 Accepted
Flexural at Maximum Stress (MPa)	81.17	93.43	8	.045	-2.295	±2.306	H_1 Accepted
Impact Strength (kJ/m ²)	50.66	50.66	14	.992	0.000	±2.145	H_0 Accepted
Ultimate Tensile Stress (MPa)	27.31	18.49	8	.000	+7.004	±2.306	H_1 Accepted
Tensile Strain at Break (%)	2.64	2.03	8	.021	+8.769	±2.306	H_1 Accepted
E-Modulus (MPa)	1023.55	1063.52	8	.208	-0.025	±2.306	H_0 Accepted

Remark : ES = Epoxy System

Kevlar Fibre

Table D5 Independent sample *t*-test of the kevlar fibre reinforced polycarbonate between with and without compatibiliser by mixing Method I (TSE).

Properties	Average mean value		df	Sig. (2-tailed)	The Calculated <i>t</i>	The Critical <i>t</i>	Conclusion
	With ES	Without ES					
Heat Distortion Temperature (°C)	133.67	135.00	4	.057	-2.732	±2.776	H_0 Accepted
Flexural at Maximum Stress (MPa)	108.10	106.54	8	.363	+0.374	±2.306	H_0 Accepted
Impact Strength (kJ/m ²)	69.22	72.31	14	.026	-2.281	±2.145	H_1 Accepted
Ultimate Tensile Stress (MPa)	65.05	66.14	8	.486	-0.263	±2.306	H_0 Accepted
Tensile Strain at Break (%)	9.27	8.44	8	.344	+0.650	±2.306	H_0 Accepted
E-Modulus (MPa)	1015.60	1115.51	8	.009	-2.477	±2.306	H_1 Accepted

Remark : ES = Epoxy System

Table D6 Independent sample *t*-test of the kevlar fibre reinforced polycarbonate between with and without compatibiliser by mixing Method II (IM).

Properties	Average mean value		df	Sig. (2-tailed)	The Calculated <i>t</i>	The Critical <i>t</i>	Conclusion
	With ES	Without ES					
Heat Distortion Temperature (°C)	130.67	132.67	4	.013	-7.356	±2.776	H_1 Accepted
Flexural at Maximum Stress (MPa)	102.67	103.68	8	.041	-3.663	±2.306	H_1 Accepted
Impact Strength (kJ/m ²)	67.56	71.75	14	.000	-3.907	±2.145	H_1 Accepted
Ultimate Tensile Stress (MPa)	24.18	23.32	8	.747	+0.064	±2.306	H_0 Accepted
Tensile Strain at Break (%)	2.53	2.98	8	.245	-1.815	±2.306	H_0 Accepted
E-Modulus (MPa)	1003.63	931.38	8	.053	+0.058	±2.306	H_0 Accepted

Remark : ES = Epoxy System

Table D7 Independent sample *t*-test of the kevlar fibre reinforced polycarbonate between with and without compatibiliser by mixing Method III (MB+SSE).

Properties	Average mean value		df	Sig. (2-tailed)	The Calculated <i>t</i>	The Critical <i>t</i>	Conclusion
	With ES	Without ES					
Heat Distortion Temperature (°C)	132.67	133.67	4	.101	-2.678	±2.776	H_0 Accepted
Flexural at Maximum Stress (MPa)	103.66	97.16	8	.000	+4.370	±2.306	H_1 Accepted
Impact Strength (kJ/m ²)	60.33	67.03	14	.000	-17.938	±2.145	H_1 Accepted
Ultimate Tensile Stress (MPa)	33.96	32.36	8	.564	+0.123	±2.306	H_0 Accepted
Tensile Strain at Break (%)	3.62	3.62	8	.994	0.000	±2.306	H_0 Accepted
E-Modulus (MPa)	1009.02	1014.72	8	.848	-0.004	±2.306	H_0 Accepted

Remark : ES = Epoxy System

Table D8 Independent sample *t*-test of the kevlar fibre reinforced polycarbonate between with and without compatibiliser by mixing Method IV (SSE).

Properties	Average mean value		df	Sig. (2-tailed)	The Calculated <i>t</i>	The Critical <i>t</i>	Conclusion
	With ES	Without ES					
Heat Distortion Temperature (°C)	131.67	132.67	4	.101	-2.678	±2.776	H_0 Accepted
Flexural at Maximum Stress (MPa)	103.34	87.23	8	.013	+2.454	±2.306	H_1 Accepted
Impact Strength (kJ/m ²)	51.23	55.46	14	.000	-5.826	±2.145	H_1 Accepted
Ultimate Tensile Stress (MPa)	54.69	34.35	8	.001	+2.954	±2.306	H_1 Accepted
Tensile Strain at Break (%)	6.52	3.87	8	.008	+2.936	±2.306	H_1 Accepted
E-Modulus (MPa)	996.02	1008.79	8	.600	-0.011	±2.306	H_0 Accepted

Remark : ES = Epoxy System

Glass Fibre

Table D9 Independent sample *t*-test of the glass fibre reinforced polycarbonate between with and without compatibiliser by mixing Method I (TSE).

Properties	Average mean value		df	Sig. (2-tailed)	The Calculated <i>t</i>	The Critical <i>t</i>	Conclusion
	With ES	Without ES					
Heat Distortion Temperature (°C)	137.00	137.67	4	0.184	-2.012	±2.776	H_0 Accepted
Flexural at Maximum Stress (MPa)	110.02	106.18	8	0.000	+25.782	±2.306	H_1 Accepted
Impact Strength (kJ/m ²)	79.30	66.51	14	0.000	+15.154	±2.145	H_1 Accepted
Ultimate Tensile Stress (MPa)	68.54	64.41	8	0.002	+4.350	±2.306	H_1 Accepted
Tensile Strain at Break (%)	9.94	9.17	8	0.008	+12.050	±2.306	H_1 Accepted
E-Modulus (MPa)	1212.84	1239.92	8	0.397	-0.018	±2.306	H_0 Accepted

Remark : ES = Epoxy System

Table D10 Independent sample *t*-test of the glass fibre reinforced polycarbonate between with and without compatibiliser by mixing Method II (IM).

Properties	Average mean value		df	Sig. (2-tailed)	The Calculated <i>t</i>	The Critical <i>t</i>	Conclusion
	With ES	Without ES					
Heat Distortion Temperature (°C)	136.33	136.67	4	.519	-1.250	±2.776	H_0 Accepted
Flexural at Maximum Stress (MPa)	115.30	102.63	8	.000	+13.189	±2.306	H_1 Accepted
Impact Strength (kJ/m ²)	91.53	71.08	14	.000	+9.093	±2.145	H_1 Accepted
Ultimate Tensile Stress (MPa)	72.78	47.26	8	.000	+3.544	±2.306	H_1 Accepted
Tensile Strain at Break (%)	8.46	5.13	8	.000	+17.935	±2.306	H_1 Accepted
E-Modulus (MPa)	1257.16	1132.47	8	.025	+3.040	±2.306	H_1 Accepted

Remark : ES = Epoxy System

Table D11 Independent sample t -test of the glass fibre reinforced polycarbonate between with and without compatibiliser by mixing Method III (MB+SSE).

Properties	Average mean value		df	Sig. (2-tailed)	The Calculated t	The Critical t	Conclusion
	With ES	Without ES					
Heat Distortion Temperature (°C)	134.67	136.67	4	.013	-7.356	±2.776	H_1 Accepted
Flexural at Maximum Stress (MPa)	112.39	107.72	8	.000	+6.253	±2.306	H_1 Accepted
Impact Strength (kJ/m ²)	88.47	63.33	14	.000	+22.689	±2.145	H_1 Accepted
Ultimate Tensile Stress (MPa)	70.21	60.96	8	.001	+2.922	±2.306	H_1 Accepted
Tensile Strain at Break (%)	9.35	7.09	8	.003	+6.699	±2.306	H_1 Accepted
E-Modulus (MPa)	1239.35	1146.05	8	.084	+0.025	±2.306	H_0 Accepted

Remark : ES = Epoxy System

Table D12 Independent sample *t*-test of the glass fibre reinforced polycarbonate between with and without compatibiliser by mixing Method IV (SSE).

Properties	Average mean value		df	Sig. (2-tailed)	The Calculated <i>t</i>	The Critical <i>t</i>	Conclusion
	With ES	Without ES					
Heat Distortion Temperature (°C)	134.67	135.67	4	.101	-2.678	±2.776	H_0 Accepted
Flexural at Maximum Stress (MPa)	120.95	108.40	8	.000	+10.084	±2.306	H_1 Accepted
Impact Strength (kJ/m ²)	82.14	64.99	14	.000	+14.217	±2.145	H_1 Accepted
Ultimate Tensile Stress (MPa)	73.42	55.23	8	.001	+2.582	±2.306	H_1 Accepted
Tensile Strain at Break (%)	9.27	6.28	8	.004	+4.115	±2.306	H_1 Accepted
E-Modulus (MPa)	1278.87	1229.46	8	.381	+0.009	±2.306	H_0 Accepted

Remark : ES = Epoxy System

BIOGRAPHY

Onuma Charoensuk was born in Chachoengsao east of Thailand. She earned her undergraduate degree in Polymer Engineering at the state university at Nakhon Ratchasima, Suranaree University of Technology, before continued her graduate degree in Polymer Engineering at the same university. Her expertise includes the field of Polymer Mixing and Compounding. During graduate study she presented three papers entitled of “Development of Compounding Techniques for Short Fiber Reinforced Polycarbonate” in An International Conference on “Advances in Petrochemicals and Polymers in the New Millenium” on July 22-25, 2003 Bangkok Thailand, “Compatibilisation of Polycarbonate Reinforced with Short Glass Fiber Using Epoxy System” in the 4th Grad Research Conference on August 10-11, 2004 Chiang Mai Thailand, and “Polycarbonate reinforced short fibers compatibilized with highly branched epoxy system” in 31st Congress on Science and Technology of Thailand on October 18-21, 2005 Nakhon Ratchasima Thailand.