SCHOOL OF MATERIALS AND MINERAL RESOURCES ENGINEERING

UNIVERSITI SAINS MALAYSIA

EFFECT OF ELECTRON BEAM IRRADIATION OF HIGH DENSITY POLYETHYLENE/WASTE TIRE POWDER (HDPE/WTP) BLENDS ON PROCESSABILITY, TENSILE PROPERTIES AND MORPHOLOGICAL PROPERTIES.

By

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Dissertation submitted in partial fulfillment

of the requirement for the degree of Bachelor of Engineering with Honours

(Polymer Engineering)

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DECLARATION

I hereby declare that I have conducted, completed the research work and written the dissertation entitled "Effect Of Electron Beam Irradiation To High Density Polyethylene/Waste Tire Powder (HDPE/WTP) Blends On Processability, Tensile Properties And Morphological Properties.". I also declare that it has not been previously submitted for the award of any degree or diploma or other similar title of this for any examining body or University.

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Sincerely,

Khairunnisa Azalin Binti Azami

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LIST OF ABBREVIATIONS

HDPE	High Density Polyethylene
WTP	Waste Tire Powder
SEM	Scanning Electron Microscopy
POE-*g*-MA	Maleic Anhydride-Grafted Ethylene-Octene Copolymer
PE-*g*-MA	Maleic Anhydride-grafted Polyethylene
PE-LD	Low-Density Polyethylene
EFB	Empty Fruit Bunches
OPF	Oil Palm Fronds
OPT	Oit Palm Trunk
EB	Electron Beam

KESAN PANCARAN RADIASI ELEKTRON BAGI POLIETILINA BERKETUMPATAN TINGGI DIADUN DENGAN SISA SERBUK TAYAR TERHADAP KEBOLEHPROSESAN, SIFAT-SIFAT TENSIL DAN SIFAT-SIFAT MORFOLOGI

ABSTRAK

Mengitar semula sisa seperti getah masih menjadi masalah semasa. Menyelamatkan sumber dan tenaga utama haruslah dipandang serius. Proses mengitar semula yang murah serta mesra alam harus diutamakan. Pada masa kini, industri getah dan penggunaan bahan polimer meningkat selari dengan perindustrian yang berkembang pesat. Oleh kerana bahan-bahan polimer tidak mudah terurai, pelupusan polimer sisa merupakan masalah alam sekitar yang utama. Selain daripada itu, penambahbaikan sifat ketahanan polietilina berketumpatan tinggi perlu dipelajari. Untuk mengitar semula sisa tayar dan menambahbaik sifat-sifat polietilina berketumpatan tinggi, polietilina berketumpatan tinggi diadun bersama sisa getah dan pancaran radiasi electron telah dijalankan. Sisa tayar yang dipilih adalah dalam bentuk serbuk dan ditapis untuk mendapatkan saiz yang kurang daripada 3 mikron. Ia berfungsi sebagai pengisi manakala polietilena berketumpatan tinggi bertindak sebagai matriks. Kandungan pengisi adalah pelbagai iaitu 0. 1. 2. 3 dan 5 peratus. Pancaran radiasi elektron telah dijalankan untuk mengkaji perbezaan perilaku adunan selepas melalui rawatan radiasi dan tanpa melalui rawatan radiasi. Pemantauan kebolehprosesan, ujian tegangan dan analisis kegagalan telah dilakukan untuk mengkaji sifat-sifat adunan. Secara keseluruhan, hasil menunjukkan bahawa kandungan pengisi yang pelbagai

mempengaruhi proses, sifat tegangan dan morfologi adunan. Rawatan radiasi juga meningkatkan kualiti sifat adunan.

EFFECT OF ELECTRON BEAM IRRADIATION OF HIGH DENSITY POLYETHYLENE/WASTE TIRE POWDER (HDPE/WTP) BLENDS ON PROCESSABILITY, TENSILE PROPERTIES AND MORPHOLOGICAL PROPERTIES

ABSTRACT

Recycling of waste is still a current issue. Saving primary sources and energy should be a priority. Inexpensive recycling which is environmentally friendly should be preferred. Nowadays, the rubber industry and the consumption of polymeric materials increase in parallel to the rapid industrialization and civilization. As polymeric materials do not decompose easily, disposal of waste polymers is a major environmental problem. Other than that, the improvement of the toughness of HDPE also need to be studied. In order to recycle the waste and improve the properties of HDPE, HDPE was blended with WTP and EB irradiation was conducted. The waste tire selected was in powder form and sieved to get smaller size than 3 micron. It acts as filler while the high density polyethylene acts as matrix. The fillers content were varied which are 0, 1, 2, 3 and 5 percent. Electron beam irradiation was conducted to study the differences behaviour of the blends under radiation treated and without radiation treated. Processability monitoring, tensile test and failure analysis were carried out to investigate the properties of the blends. Overall, the results show that the varied content of fillers affected the processability, tensile properties and morphology of the blends. The radiation treatment had also improved the properties of the blends.

CHAPTER 1

INTRODUCTION

1.1 Research Background

In the world of engineering, the concept of making people's life easy and solving their difficulty is an important key portion to become successful engineers. As the world's population grows and becomes more industrialized, the number of tires produced, and eventually discarded, is growing rapidly. Figure 1.1 below illustrates the world's rubber consumption, a majority of which is used in the production of tires.

Experts predict that the number of automotive and bicycle tires produced in 2015 will reach three billion, which directly correlates to the amount waste tires that will be generated (Rubber World Magazine, 2012). Studies have shown that scrap tire rubbers can be used as aggregates in concrete mixtures and as modifiers in bituminous composites.

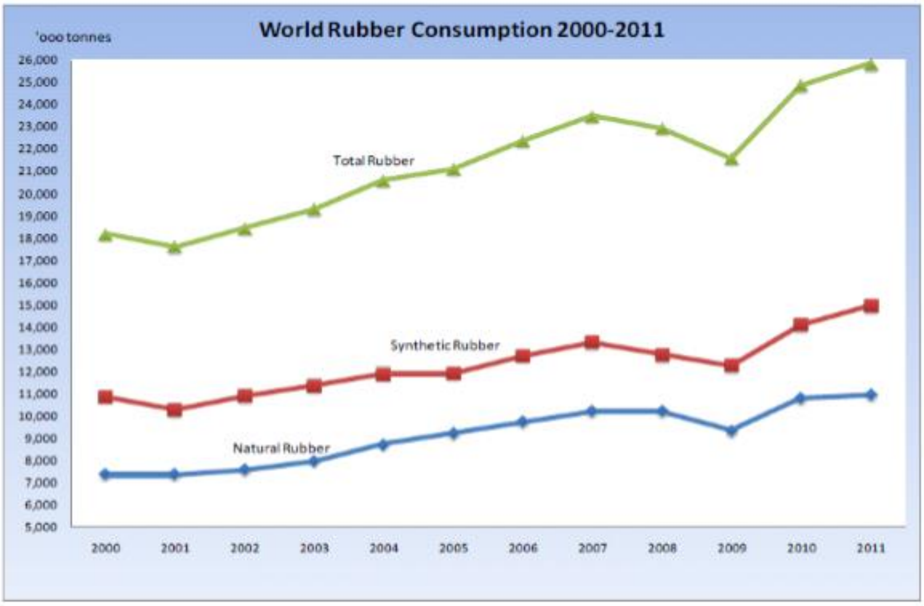

Figure 1.1: - World Rubber Consumption, 2000-2011 (NR Rubber Statistics, 2011)

As to resolve this abundance of waste tires, there are research made to study the compatibilizer in waste tire powder and low-density polyethylene blends and the blends modified asphalt. As the result, with the increasing ratio of waste tire powder (WTP) to low-density polyethylene (LDPE), the hardness and tensile strength of the WTP/LDPE blends decreased while the elongation at break increased. (Wiley, 2010).

Five kinds of compatibilizers, such as maleic anhydride-grafted polyethylene (PE-*g*-MA), maleic anhydride-grafted ethylene-octene copolymer (POE-*g*-MA), maleic anhydride-grafted linear LDPE, maleic anhydride-grafted ethylene vinyl-acetate copolymer, and maleic anhydride-grafted styrene-ethylene-butylene-styrene, were incorporated to prepare WTP/LDPE blends, respectively (Wiley, 2010).

Other than that, there are also research on thermoplastic elastomer blends based on waste rubber and low-density polyethylene. Materials based on waste rubber from discarded tires and low-density polyethylene (PE-LD) have been studied. The blends were prepared via compounding of molten PE-LD with ground rubber (average particle size 0.4 mm) in an internal mixer. The products behaved like thermoplastic elastomers. To improve mechanical properties of the blends, various compatibilisers (dicumyl peroxide, sulphur system) were added.The best materials had tensile strength 8.6 MPa and elongation at break 260% (Zdenek, 2011).

Lastly, I also found the research on possibility of using waste tire composites reinforced with oil palm frond as construction materials. Based on all researches and journals available, there are many fibres have been used to replace the role of wood raw materials in composite production. This also solves the environmental pollution that cause by those materials. Numbers of study have utilized the oil palm biomass which is its empty fruit bunches (EFB), trunk (OPT) and stem but none of it have mentioned about the trimmed oil palm fronds (OPF). There is abundance of trimmed OPF been produced yearly and it has high potential to be used in wood-based industries.

1.2 Problem statement

Nowadays, the fast developments of the economy especially with broaden of technologies apply everyway makes the demand of the automobiles will keep increasing. The problem that occurs due to this demand is the abundance of the waste rubber tires while the sources of the rubber itself will decrease. Based on the statistic data that have been analyzed, approximately 1.5 billion tires are discarded every year worldwide (Jun et all, 2008). This problem occurred due to the short lifetime of the tires.

Therefore, one of the biggest problems is how to reduce environmental pollution that may cause by the waste rubber tires. Thereby, it is necessary to develop some methods for reused and recycling the waste tires. Since most of the composition of the tires is rubber, it has properties such as high strength, high resistance to the abrasion, durable, elastic and anti-caustic that have high potential to be recycle as new raw materials for further applications.

As the number of waste tire keeps increasing daily, this research focused on the application of the waste tires to be reused as a fillers material. The properties of the waste tires need to be investigated in order to be reused for application such garden decoration, tree guard, shock absorbent and fences according to the properties requirements for the choosen applications.

Other than that, this study also focused on the improvement of the inferior properties of HDPE by blending with waste tire powder (WTP) and EB irradiation. The High Density Polyethylene was chosen as the matrix because the blending of thermoplastic polymer like polyethylene has the ability to flow under certain conditions (supported usually by the action of heat and/or pressure), so that it can be shaped into products at acceptable cost (Sienkiewicz, 2012). HDPE has known have the high stiffness, while WTP have more elastic properties. Blending of HDPE with WTP, improved the compatibilization of the blends and treatment with EB irradiation are expected to improve the toughness of the resulted blends (Suganti, 2016).

Lastly, radiation processing of polymeric materials involves treatment of polymeric material with ionizing radiation to modify their physical and chemical properties. Properties of polymeric materials can be modified by irradiation as it is bound to crosslink, degrade, grafted or cured when subjected to ionizing radiation (Cheng, 2012). The usage of ionizing radiation in developing a sustainable management of polymeric waste by manipulating the crosslinking and chain scission yield is a new and emerging field of application. In this study, electron beam (EB) irradiation was used onto HDPE/WTP blends to enhance the properties. The efficiency of EB irradiation in improving the properties of HDPE/WTP blends has been reported.

1.3 Research objective

The main objectives for this project:

I. To study the effect of different compositions of High Density Polyethylene/Waste Tire Powder (HDPE/WTP) blends on processability, tensile properties and morphological properties.

II. To compare the effect of High Density Polyethylene/Waste Tire Powder (HDPE/WTP) blends on tensile properties and morphological properties with and without treatment of electron beam irradiation.

1.4 Thesis Outline

This thesis is consisting of five consecutive chapters. Chapter 1 describes a concise introduction, problem statement and objectives of the research. In this chapter reader are expected to get the general idea of the whole content of this thesis writing. Readers are also being introduced with several important keywords for further and easy understanding. Next is chapter 2 which comprises a full review on the formation of polymer blend, fundamental concepts of electron beam irradiation and a brief explanation on tests that will be conducted in this project.

Furthermore, chapter 3 explains the information on the experimental procedures that is used in this research. In this chapter it also comprised the experimental design and the method of testing that involves. It covers a brief explanation on the characterization equipment, their operation principles and sample preparation. In this research, the characterizing being used is Scanning Electron Microscope (SEM). Moreover, chapter 4 show to the readers about the experimental results and brief discussion on the processability, tensile properties and morphological properties of polymer blend. Finally, in chapter 5 is devoted to the conclusions of this research work and suggestions for future work.

CHAPTER 2

LITERATURE REVIEW

2.1 Recycling

A lot of waste is generated from automobiles and one of these wastes is used tires. The powder of these used tires can be used as a substitute of raw material for the production of rubber. Granules of rubber can be obtained in various final grain sizes. This has become meaningful because processed rubber is becoming more acceptable on the market due to increasing raw material prices. Waste tire rubber powder is widely used to build the playground and highway road. It is also material of rubber product. The sales profit depends naturally on the quality of the output material and the pricing structure depends on processing that is as efficient as possible (Atulesh, 2011).

Manufacturing of rubber powder from used tires is a three-stage processing primarily shredding, after that granulation and lastly fine grinding through which high-quality materials for recycling are ultimately produced (Atulesh, 2011).

The very first step involved in the recycling of tires is shredding. Shredding means separation of wire and mesh from tire also breaking them into pieces. This can be obtained by a machine with moving parts in which used tires are put and steel or iron made wires are separated out from the tire. Then, tire is torn to pieces. The machine is very versatile which can also be used for shredding of all kinds of input materials and is well suited for different industries (Atulesh, 2011).

The diameter of rotors ranges from 457 mm - 850mm to 2000 mm width which are driven by either one or two oversized gearboxes. There is a well integrated hydraulic power pack into the machine housing which is used to save space and protect it from damage but is still easy to access or remove for maintenance. In this machine shredders are usually designed for a wide range of applications and which can also be used in industries such as in-house and general recycling, electronic waste and post consumer waste handling. Apart from the tyre waste input materials can be all types and forms of plastics such as lumps, pipes, film, bales, woven bags, electronic waste like cables and ICBs, paper, wood and other organic materials. In some cases where the raw material or waste is of small size, can be sent directly to the next step granulation (Atulesh, 2011).

The granulators are used in the next step of this recycling process in which pieces of waste tyres are grinded in the large sized granulators to produce large quantity of granules. Granulators are developed as slow-running grinders for applications in the injection and blow molding sector. The material which has to be granulated is fed via a sound-absorbing feed hopper which is available in a wide range to suit the application. The slow-speed granulators which are designed of this range are commonly mounted on either low or high level base frames. With these numerous options, the slow speed granulators can be tailored to an extremely wide range of applications (Atulesh, 2011).

The last step involved in the production of rubber powder from tyre waste is to convert the granulated material into the fine powder. This is done by means of pulverizes. Pulverizes are high speed, precision grinders which are used for the processing of medium hard, impact resistant and friable materials. The granulated material is introduced through the centre of a vertically fixed grinding disc which is mounted concentrically with an identical high speed rotating disc to make the fine powder of it. Some typical applications are the pulverizing of plastic products, tube, edge trim materials, film waste. The waste generated from the food, chemical and pharmaceutical industry can also be pulverized in order to make the fine powder. The material to be pulverized is. Inside the pulverizer the centrifugal force acts on the material to be pulverized and carries the material through the grinding area and the resulting powder is collected with a blower and cyclone system which is fine rubber powder or the other product of which waste material use (Atulesh, 2011).

2.2 High Density Polyethylene

Polyethylene has variety types including one of them is High Density Polyethylene (HDPE). HDPE is widely used in packaging application such as manufacturing bottles to hold household, industrial and automotive chemicals such as liquid detergent bleach. This is because HDPE has several captivating properties such as low permeability, stiffness and high environmental stress crack. In general, HDPE is linear polymer with the chemical composition of polymethylene $(CH_2)_n$ which makes it chemically the closest in the structure to the pure polyethylene (Peacock, 2000).

HDPE has a comparatively high density compared to other polymers, with a specific gravity of 0.95. HDPE is relatively hard and resistant to impact and can be subjected to temperatures of up to 120°C without being affected. It has regular backbone of great flexibility, impending disentanglement needed for crystallization but it’s regularly allows close packing and it crystallizes readily by up to 90%. HDPE is not autoclavable which are used to sterilise products using high pressures and temperatures. It is also recognizable by its opaque or translucent appearance due to its high crytallinity.

Since HDPE is known as high degree of crystallinity, it also perform in tensile strength properties. The HDPE yields in discrete region and thin down to form necking when stretched perpendicular to its orientation direction. (Peacock, 2000).

The basic deformation mechanism of unmodified HDPE is shear yielding, although crazing has been observed in some cases. Shear yielding leads to a permanent change in the dimensions or shape and implies translational motions of the HDPE chains, reaching great deformations because of the molecular entanglements which act as resistant points. Shear yielding can be observed in a localized o diffused way, depending on the magnitude of the zone affected by this process. In semicrystalline polymers such as HDPE, shear yielding is a process localized in the vicinity of the crystalline areas, as described in Figure 2.1. The existence of amorphous zone allows the crystal to show slight distortions (e.g., rotation, shear and intralamellar slipping) with reversible characteristics.

The increase of deformation provokes that the localized deformation of the crystals is more marked, leading to a destruction of lamellar aggregates and a irreversible rearranging of the polymer chains. Finally the amorphous zones and the crystals are oriented in the tensile direction leading to a fibrous structure. The localized shear yielding can also be manifested as a consequence of in homogeneities and instabilities of geometrical origin, superficial and/or internal defects, which take place in the deformation process, promoting the concentration of plastic deformation.

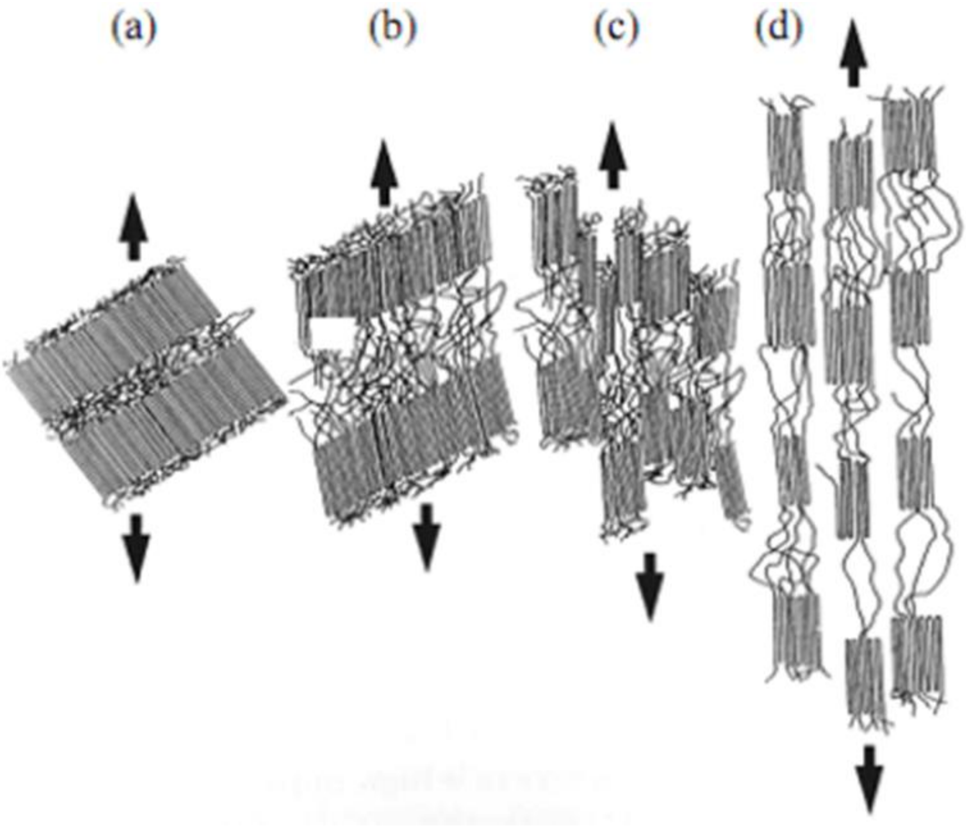

Figure 2.1: Schematic representation of the plastic deformation: (a) no deformation, (b) chain motion inside lamellae, (c) lamellae fragmentation, and (d) tension alignment (Oswald, et. Al., 2003)

Crazes can be observed in HDPE. Crazing consists of the generation of a system of interpenetrated microvoids as shown in Figure 2.2 which are developed on the perpendicular plane to the main tensile direction. These microvoids are stabilized by microfibres of material that does not coalesce. The microfibres in the craze act as bridges in a microcrack, allowing the load to be transmitted, stabilizing the craze and giving rise to an enhancement of resistance. The rupture of the fibrils commonly leads to the microvoid coalescence and thus leading to cracks and ulterior fracture in a brittle manner.

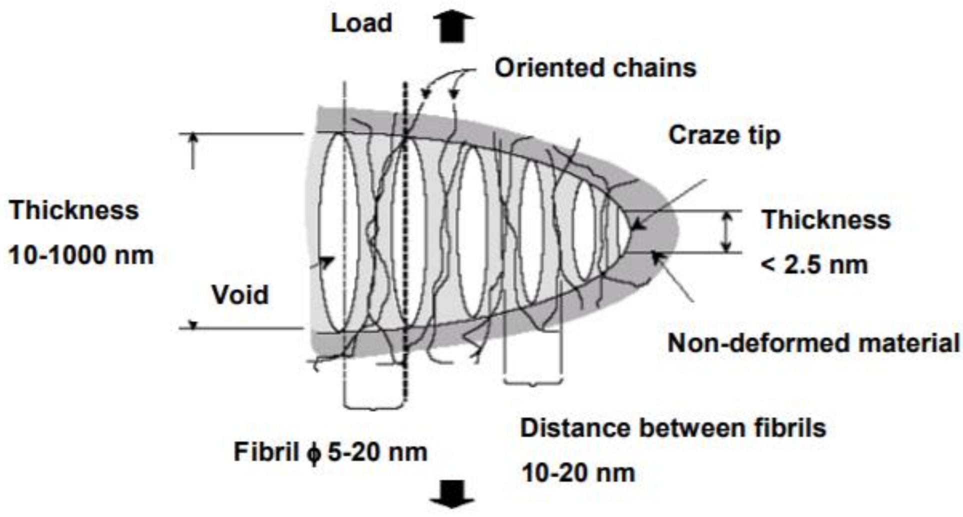

Figure 2.2. Representation of the ideal structure of a craze.

It should be taken into account that the semicrystalline nature of HDPE implies that the fracture behaviour of polypropylene is strongly dependant on the crystalline structure and superstructure (crystalline form, lamellae dimension, crystallite size, crystallinity, spherulite size) (Varga, 1991) along with the molecular characteristics (molecular mass and its distribution) and processing-induced morphology.

2.3 Waste Tire Powder (WTP)

It is necessary for the academia and researchers to develop methods and ways to minimizes and recycling the waste tires because the proportion of these waste tires being recycled are remains negligible (Yang et al, 2004). (Jun et al, 2008) stated that waste tire rubber is an ideal raw material for the functional composite panel because it possesses some unique properties: excellent energy absorption, characteristically large elastic deformation, better sound insulation, and durability and abrasion resistance, anti-caustic and anti-rot.

Scrap tire rubber powder can be obtained from tires through two principal processes: (1) ambient, which is a method in which scrap tire rubber is ground or processed at or above ordinary room temperature and (2) cryogenic, a process that uses liquid nitrogen to freeze the scrap tire rubber until it becomes brittle and then uses a hammer mill to shatter the frozen rubber into smooth particles (California, 2003).

Tire is made up mainly by rubber. Its constitution varies a little between the car tires and heavy truck tires. Rubber consists of a complex mixture of elastomers, polyisoprene, polybutadiene and stirene-butadiene. Stearic acid (1.2%), zinc oxide (1.9%), extender oil (1.9%) and carbon black (31.0%) are also important components of tires (B.A Black, 1994). In Table 2.1, chemical composition of the typical rubber powder is presented.

Table 2.1: The typical rubber powder characteristic

Material/element	Mass percentage
Rubber	54%
Carbon black	31%
Extended oil	1.9%
Oxidize zinc	1.9%
Sulfur	1.2%
Additives	10%

2.4 Polymer Blend

All new materials attract interest on the basis of its property, processing and cost performance. Polymer blend can be considered as one of the desire choices in enhance the material properties and develop material with various performances. The interest on polymer blending had been rose up because of cost effective route compared to the synthesis of the new polymer. Generally, blends represent less expensive route than synthesis of the new polymer because of the less involved chemistry. Modulus is property that can be expected to obey some relationship for blends where the weighing function of composition will be sensitive to phase morphology (Paul and Barlow, 1979).

Polymer blends are mixture of at least two macrospecies and/or copolymer. (LA Utracki, 2002). In polymer blending, it is crucial to know how well the material combines where it is depending on the sign of the free energy of mixing, blends either miscible or immiscible. The mixing two or more polymer always lead to an immicisble system. As the result, the material performance is poor and impact strength are effected. To improve performance of immiscible blends, usually they need to be compatibilized.

There are three aspects of compatibilization (L A Utracki, 2002):

1. Reduction of the interfacial tension that facilities fine dispersion.
2. Stabilization of morphology against its destructive modification during the subsequent high stress and strain processing.
3. Enhancement of adhesion between phase in the solid state, facilitating the stress transfer, hence improving the mechanical properties of the product.

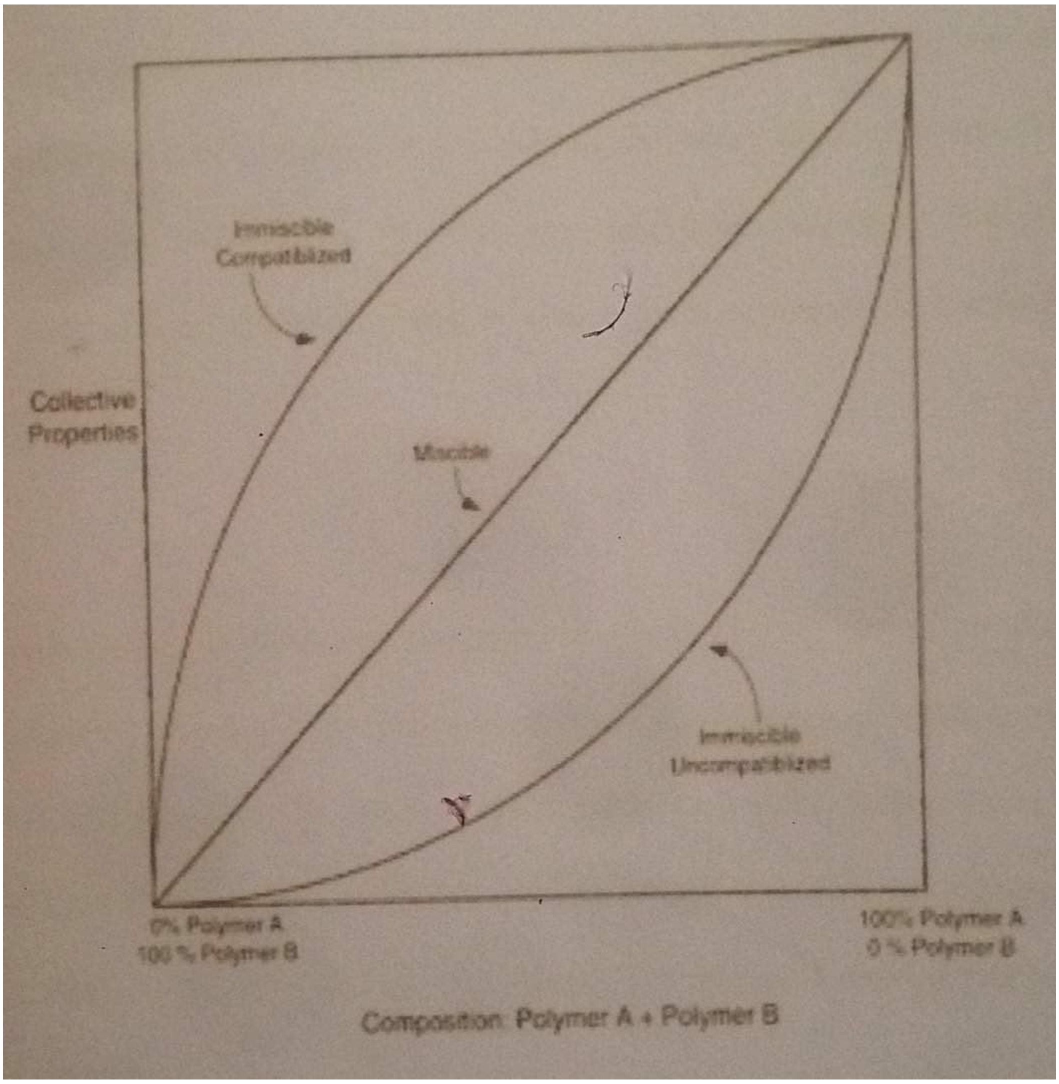

Figure 2.2: Potential effect on polymer blend properties as component concentration changes (LA Utrachi, 2002).

The purpose of combining two or more polymers is to achieve in the blend a combination of favourable properties fom each polymer. Figure ____ shows ideal expected property combinations from blending two polymers that are either miscible (solid center line), immiscible and uncompatibilized (bottom line) or immiscible and compatibilized (top line) (L A Utracki, 2002).

Two immiscible polymers which are blended without good compatibilization will obtain a mixture with bad physical properties compared to the single polymer. Besides, the blend may has poor structural integrity and heat stability due to no mechanism in stabilizing a dispersion of one polymer in a matrix of the other. In some cases., the blend might be appearing delaminated. For the two immiscible polymers that are blended with the good compatibilization, there is a synergistic combination property from each polymer.

The most prior properties of blends including impact strength, stiffness, elongation and others because it usually used as a structural materials. The polymeric systems can be divided into britte or ductile. A brittle polymer tends to fail by crazing mechanism and have low crack initiation as well as propagation energy. The ductile polymer failure dua to fail by yielding, have high crack intiation energy and low crack propagation energy.

2.5 Electron Beam Irradiation

The simultaneous growth of both the polymer industry and nuclear technology in the f:irst two decades after world war II, has resulted in an outburst of papers concerning the interaction of high energy radiation with polymerie materials. Several hooks cover most results from this period and still act as important references nowadays (Makhblis, 1975). Also more up to date reviews with the latest developments on modification of homopolymers and blends, dosimetry and equipment appeared (Ungar, 1981) or are in preparation.

In irradiation of polymerie materials, two objectives exist: the study and development of polymers with a high resistance to radiation (e.g. application in nuclear power reactors) vs. the search for materials with a high radiation sensitivity to obtain better properties. The distinct interest in irradiation of long chain polymers is related to the fact that, in contrast to low molar mass species, large changes in the physical and mechanical behaviour can be obtained at relatively low doses.

For example, crosslinking can be induced via irradiation, involving only a few chemical changes per macromolecule, whereas the properties change completely. The same effect can usually be obtained by thermal energy (peroxide crosslinking/vulcanisation) but in this case the whole system has to be heated in order to induce only a few chemical changes per macromolecule. Especially for curing of coatings on substrates, radiation at ambient temperature is fast and efficient without the necessity of heating the whole system to the reaction temperature.

Radiation technology is also used for sterilization, pasteurization, de-infestation and de-contamination of microorganisms. This technology also covers the use of radiation to remove the colour, odour, microorganisms in liquids, solid matter and gas and induce the chemical and biological changes in the system to clean up pollution.

Radiation processing is a method of treatment of products and materials using radiation or ion energy. This method is used to alter the chemical or biological characteristics of the product to improve the products use and value or to reduce its impact on the environment. Among the most important commercial applications are renovation of plastic and rubber materials, sterilization of medical equipment and consumer goods, pasteurization and food preservation and reducing environmental pollution.

Radiation processing with an electron beam offers several distinct advantages when compared with other radiation sources, particularly γ-rays and x-rays. The process is very fast, clean and can be controlled with much precision. There is no permanent radioactivity since the machine can be switched off. In contrast to γ-rays and x-rays, the electron beam can be steered relatively easily, thus allowing irradiation of a variety of physical shapes. The electron beam radiation process is practically free of waste products and therefore is no serious environmental hazard.

These are not only capable of converting monomeric and oligomeric liquids, but also can produce, due to cross-linking, major changes in the properties of solid polymers. The cross-linking level can be adjusted by the irradiation dosage.

The absorbed dosage means the value of energy of ionizing radiation absorbed by a unit of mass of the processed material. The unit of absorbed dose is 1 Gray (1 Gy = 1J/kg). The main difference between beta and gamma rays is in their different abilities to penetrate the irradiated material. Gamma rays have a high penetration capacity. The penetration capacity of electron rays depends on the energy of the accelerated electrons. Due to electron accelerators, the required dosage can be applied within seconds, whereas several hours are required in the gamma radiation plant as shown in Figure 2.2 (Drobny, 2003).

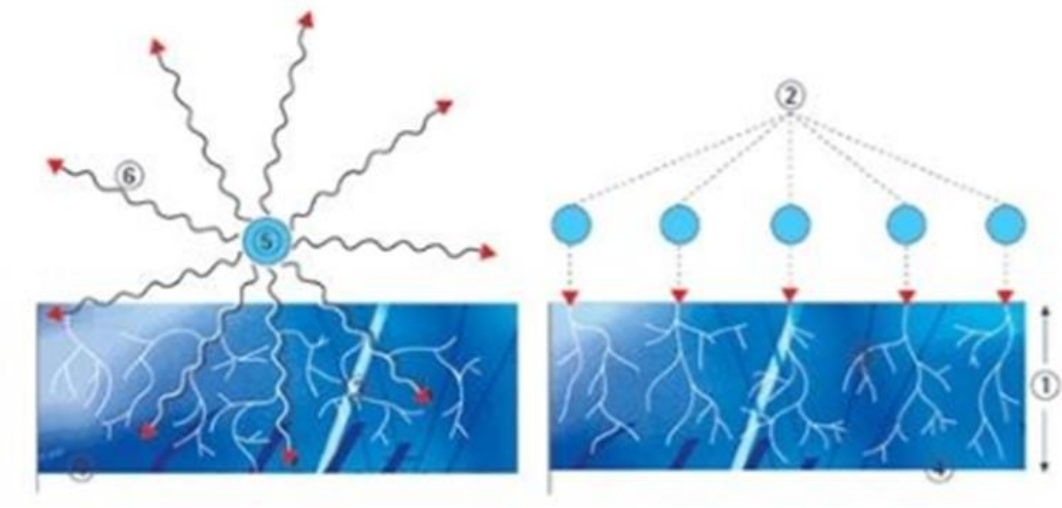

Figure 2.3: Design of Gamma Rays (a) and Electron Rays (b),

1 – Penetration depth of an electron, 2 – Primary electron, 3 – Secondary electron,

4 – Irradiated material, 5 – Encapsulated Co – 60 Radiation source, 6 – Gamma Rays

Irradiation of polyolefins, particularly the family of polyethylenes, represents an important segment of the radiation processing. Polyolefins can be irradiated in many forms, such as pellets and powder, films, extruded and molded parts or as wire and cable insulation (Drobny, 2003). Radiation cross-linking usually improves strength, reduces creep, contributes to chemical resistance improvement and in many cases improves tribological properties.

2.6 Tensile test

The tensile elongation and tensile modulus indicated the strength in the material. The purpose to conduct tensile test is to measure the ability of the material to withstand the force that tend to pull it apart and to determine to what extend the material stretches before breaking (Vishu, 2007). Tensile modulus can be related to the stiffness of material which can be determined from the stress-strain curve.

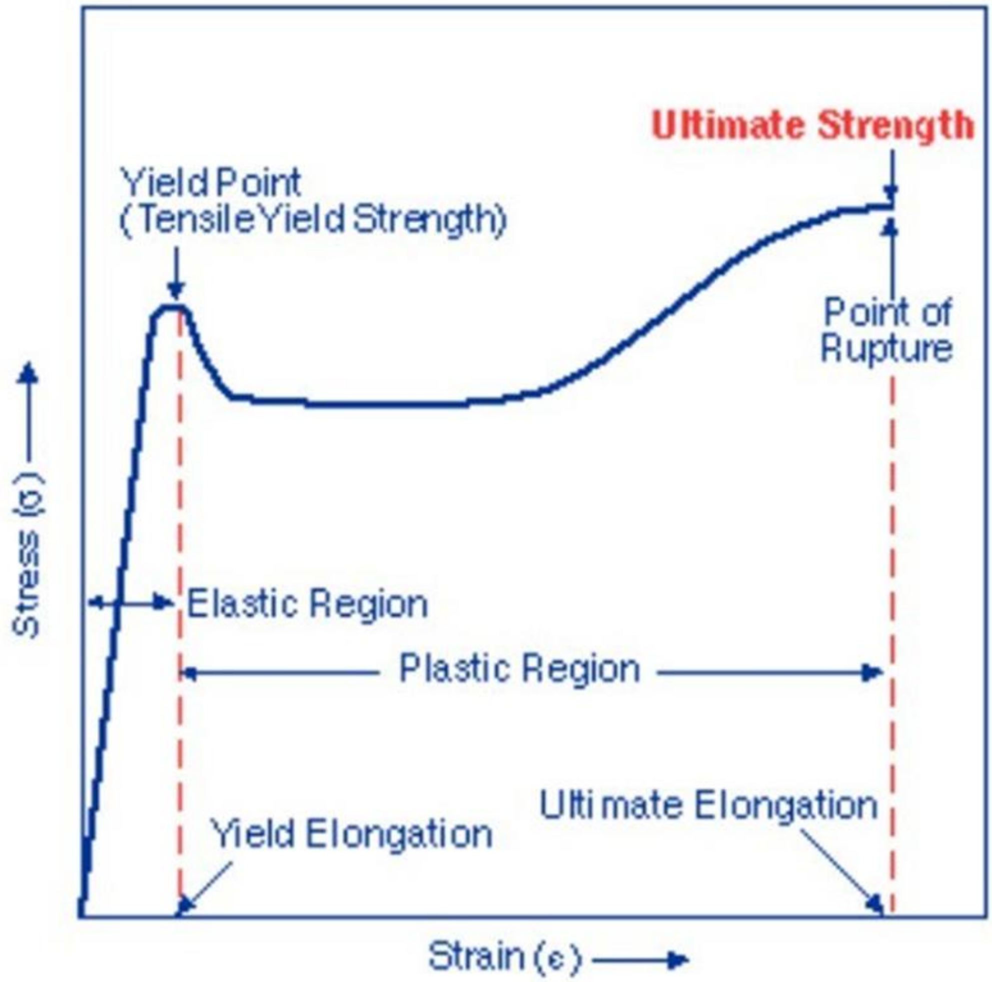

Figure 2.4: Typical stress-strain curve