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RECYCLING OF WOOD PLASTIC COMPOSITES PREPARED FROM
POLY (VINYL CHLORIDE) AND WOOD FLOUR

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A Thesis Submitted in Partial Fulfillment of the Requirements
for the Degree of Master of Engineering Program in Chemical Engineering

Department of Chemical Engineering

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จกัถกฤษณ์ เสนีตันติกุล : การนำผลิตภัณฑ์ประกอบแต่งไม้พลาสติกที่เตรียมจากพอลิไวนิลคลอไรด์และผงไม้กลับมาใช้ใหม่ (RECYCLING OF WOOD PLASTIC COMPOSITES PREPARED FROM POLY (VINYL CHLORIDE) AND WOOD FLOUR) อ. ที่ปรึกษาวิทยานิพนธ์หลัก : อ.ดร.สิริจุฑารัตน์ โควาวิสารัช, 94 หน้า.

งานวิจัยนี้มีจุดประสงค์เพื่อศึกษาการนำกลับมาใช้ใหม่ของผลิตภัณฑ์ประกอบแต่งไม้พลาสติกที่เตรียมจากพอลิไวนิลคลอไรด์ (พีวีซี) และผงไม้ การวิจัยมี 2 ส่วน ส่วนแรกศึกษาอัตราส่วนที่เหมาะสมของการนำเศษไม้พลาสติกจากโรงงาน (Scrap) ผสมกับเม็ดไม้ใหม่ (freshfeed, FF) ซึ่งเป็นเม็ด PVC ผสมผงไม้แล้ว โดยผสม Scrap : FF ในอัตราส่วนต่างๆ คือ 0:100, 20:80, 30:70, 40:60, 60:40, 80:20 และ 100:0 การขึ้นรูปไม้พลาสติกใช้เครื่องอัดรีดแบบสกรูคู่ จากนั้นตรวจสอบสมบัติเชิงกล เหนียวและความร้อนและเชิงกายภาพ เพื่อวิเคราะห์หาอัตราส่วนที่เหมาะสมที่สุดซึ่งให้ไม้พลาสติกที่มีสมบัติใกล้เคียงกับไม้ที่ผลิตจาก FF 100% มากที่สุด การวิจัยนี้พบว่าส่วนผสม Scrap : FF ที่ 30:70 เป็นอัตราส่วนซึ่งเหมาะสมที่สุดสำหรับการนำกลับมาใช้ใหม่โดย มอดูลัสภายใต้แรงดึง แรงดัดโค้ง แรงกด และการทนต่อแรงดึง แรงดัดโค้ง แรงกดต่างมีระดับใกล้เคียงกับของไม้พลาสติกที่ผลิตจากเม็ดไม้ใหม่ งานวิจัยส่วนที่ 2 ศึกษาอิทธิพลของจำนวนรอบในการนำ Scrap กลับมาใช้ใหม่ถึง 7 รอบ พบว่า สมบัติภายใต้แรงดัดโค้ง แรงดึงและแรงกด ลดลงในรอบที่ 2 ของการนำกลับมาใช้ใหม่ แต่ยังคงรักษาระดับเกือบจะคงที่ต่อไปจนถึงรอบที่ 5 การขึ้นรูปซ้ำถึง 7 รอบเริ่มลดถอยในด้านการดัดโค้ง ผลการศึกษาน้ำหนักโมเลกุลและการกระจายตัวของน้ำหนักโมเลกุลของเมทริกซ์พีวีซีในไม้พลาสติกที่นำกลับมาใช้ใหม่พบว่า มีระดับเกือบคงที่จนถึงการนำกลับมาใช้ใหม่ในรอบที่ 5 พบว่าน้ำหนักโมเลกุลเฉลี่ยโดยจำนวนและน้ำหนักโมเลกุลเฉลี่ยโดยน้ำหนักมีระดับลดลงเพียง 14% และ 11% แต่ในรอบที่ 7 น้ำหนักโมเลกุลเฉลี่ยทั้งสองลดลงถึง 35% และ 30% ทั้งนี้ คาดว่าแรงเฉือนเชิงกลที่เกิดขึ้นระหว่างกระบวนการอัดรีดไม้พลาสติกในแต่ละรอบมีผลไปตัดสายโซ่โมเลกุลของพีวีซี ทำให้น้ำหนักโมเลกุลเฉลี่ยลดลง ส่งผลให้สมบัติเชิงกลของไม้พลาสติกลดลงเมื่อถึงรอบที่ 7 แต่สมบัติด้านแรงกระแทกมีระดับเกือบคงที่ การนำไม้พลาสติกกลับมาใช้ใหม่ไม่มีผลต่ออุณหภูมิเปลี่ยนสถานะคล้ายแก้ว อุณหภูมิการสลายตัวและการดูดซึมน้ำของไม้พลาสติก การทดสอบสมบัติด้านการไหลได้ผลสอดคล้องกับน้ำหนักโมเลกุลเฉลี่ยที่ลดลง โดยค่าทอร์คสำหรับการไหลของไม้พลาสติกเมื่อหลอมมีระดับลดลงเมื่อนำกลับมาใช้ใหม่ในรอบที่ 7 ผลการวิจัยนี้ชี้ให้เห็นว่าสามารถนำไม้พลาสติกกลับมาใช้ใหม่เป็นผลิตภัณฑ์เดิมได้ถึง 5 รอบ โดยยังคงสมบัติใกล้เคียงไม้พลาสติกที่ขึ้นรูปจากเม็ดไม้ใหม่.

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JAKKID SANETUNTIKUL: RECYCLING OF WOOD PLASTIC COMPOSITES PREPARED FROM POLY (VINYL CHLORIDE) AND WOOD FLOUR. ADVISOR: SIRIJUTARATANA COVAVISARUCH, Ph.D. 94 pp.

The present study emphasized on closed-loop recycling of wood plastic composite (WPC) prepared from poly (vinyl chloride) and wood flour. The research consisted of two parts. The first part concentrated on re-processing industrial scrap of WPC profiles and its mixture with freshfeed (FF) consisting of PVC filled with wood flour. Different weight ratio of the WPC scrap and its freshfeed pellets was dry blended and re- extruded as recycled-WPC profiles at 0:100, 20:80, 30:70, 40:60, 60:40, 80:20 and 100:0 by using an industrial scale twin screw extruder. It was found that the scrap could be mixed up to 30 wt% without losing in the flexural, tensile, compressive properties, suggesting that the 30:70 was the most appropriate mix ratio. The second part focused on the influences of the number of recycling passes for up to 7 passes. A drop in the mechanical properties was notable at the second pass, and it remained relatively unchanged until the fifth pass. Flexural properties were reduced on the seventh pass of recycling. Estimation of the average molecular weights and the molecular weight distribution of the PVC matrix in the recycled WPC revealed that they remained relatively constant until the fifth pass of recycling, the \bar{M}_n and the \bar{M}_w were reduced by 14% and 11% respectively at the fifth pass. Significant decrement of both the \bar{M}_n and the \bar{M}_w by 35% and 30% was evident on the seventh pass of recycling. Upon re-processing, molecular chain scission was believed to have occurred as a consequence of the shear stress introduced repeatedly on the PVC melt during each re-processing pass. The impact property was found to remain relatively unchanged. Thermal properties such as glass transition temperature (T_g) and degradation temperature (T_d) as well as water absorption behavior were not affected by the recycling process. Rheological results in terms of the fusion torque of the recycled WPC were in agreement with the changes in the molecular weight as aforementioned, the fusion torque was notably lowered at the seventh pass of recycling. The present research revealed that the WPC could be recycled repeatedly up to five passes without critically affecting the mechanical, thermal and physical properties of the recycled WPC.

Department :..Chemical Engineering...Student's Signature.....

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CHAPTER I

INTRODUCTION

1.1 General Introduction

Majority of the commercially wood-plastic composites (WPC) in Thailand have been produced by mixing Poly (vinyl chloride) (PVC) with wood flour although those made from Polyethylene (PE) and wood flour are also emerging. Compared with natural solid wood, WPC offers many advantages. They include weathering and insect resistance. WPC can also be transformed easily and quickly to be the required products with lower processing cost than that required by natural wood. Wood plastic composite is also lighter than natural wood. In countries with high labour cost, wood plastic composites are gaining more popularity because they need not be painted as coloring of the composites can be done during their production process. The facts that WPCs help reducing deforestation and thermoplastic WPCs can be recycled have drawn extensive attention from the public and environmentalist worldwide.

The use of WPC is widespread and growing because it can be used effectively as natural wood substitutes. The growth rate of WPC application is approximately 14% in North America and Europe and 10% in Asia. [1] WPC are environmentally friendly material favorably conforming to governmental policy of many countries in Europe, America and Asia on promoting conservation of forest and utilization of recycled material. Wood-plastic composite is perceived as one of the most interesting plastic products, rapid growth rate is anticipated during next decade, approximately 75% of the WPCs are expected to be used in the construction industries, the remaining 25% will mostly be applied in furniture and at a much lesser extent in the automotive industries. [2]

Recent statistics indicated a drastic reduction of forest area in Thailand. In fact, the present forest area of 137,600 million m² accounts for only approximately 26% of the country total area. Current domestic demand for wood products is increasing to more than 3 million m³ per year. As a result, Thailand has to import nearly 99% of the total natural wood consumption within the country. This contributes to a trade deficit

of about 16,000 million Bath per year to foreign countries such as Laos, Burma, Indonesia, Chinese, Brazil, etc. [53] Current research conducted by the Royal Forest Department has verified that demands for wood are continually increasing in Thailand while forestation is rapidly diminishing. If Thai consumers can use wood-plastic composites to replace natural wood from forest, their needs to import natural wood will be reduce vastly. Literature survey revealed numerous research efforts had been well placed in the areas on improving the properties and the fabrication of WPCs, but there is yet lack of research attempts on recycling of the PVC-based WPC. Only investigations on the WPC prepared from recycled post-consumer PP or PE with ‘fresh’ wood flour were performed and documented. [49]

The current worldwide economic crisis has plunged many countries, Thailand included, to encounter economic problems. Utilization of natural resources sparingly to their utmost benefits is thus an important strategy while the country suffers economic turmoil. One such route is to recycle WPC waste. Recycling reduces the raw material cost, it can help WPC manufacturers to gain competitive advantage while also conserving environment. Hence, recycling of WPC waste is deemed appropriate in terms of economy, utilization and management of natural resource and environment.

1.2 Objectives of the Present Study

Industrial waste of PVC/wood flour wood-plastic composite created within a WPC processing plant is used as feedstock in the present study. It was obtained from two sources, i.e. the scrap of the WPCs created during the production process and the rejected items during quality inspection. The main objectives of this study are listed as follows:

- i) To find the most appropriate mixing ratio of the WPC scrap to the WPC freshfeed for close-loop recycling of WPC.
- ii) To investigate the influences of the number of re-processing passes upon recycling of the mixture of WPC scrap and WPC freshfeed in (i) WPC.

1.3 Scopes of the Research

Profiles of WPC for floor and deck applications were prepared from PVC filled with 20 phr of wood flour by using an industrial scale twin screw extruder. Industrial scrap of WPC profiles was collected for this study. The scrap consisted mainly of waste from the start-up process and the profiles rejected during quality inspection. All the collected scrap was pulverized and stored to avoid impurities. The current study consists of two parts as follows:

Part 1: Re-processing of mixture of WPC scrap and WPC freshfeed

This part concentrates on re-processing the grinded scrap of WPC profiles and its mixture with freshfeed PVC filled with wood flour. Mixtures with different weight ratio of the industrial WPC scrap (IW):WPC freshfeed (FF) pellets were dry blended and re-extruded as recycled WPC profiles at 0:100, 20:80, 30:70, 40:60, 60:40, 80:20 and 100:0 respectively. Mechanical, physical and thermal properties were evaluated to determine the most appropriate mix ratio of the IW:FF.

Part 2: Re-processing of the most appropriate mixture of IW:FF selected from part 1 up to seven passes

The fact that some of the re-processed WPC profiles may be refused and end up as waste again motivates the second part of the present study which involved re-processing the WPC profiles produced from the formulation of the IW:FF selected in Part 1. Re-processing of profiles was conducted repeatedly by using the industrial scale twin screw extruder by up to seven passes. The mechanical tests to investigate the tensile, flexural, compressive, and impact properties were conducted while the physical properties such as average molecular weight, density and water absorption characteristics were also studied. Thermal property in term of the glass transition temperature of the WPC was measured by dynamic mechanical analysis. Microscopic observation of the recycled WPC was carried out by using a Scanning Electron Microscope (SEM).

CHAPTER II

THEORY

2.1 Plastic Waste

Much of the plastic waste generated in residential, commercial and industrial areas has become a serious environmental problem due to the non-biodegradable characteristic of plastics. Disposal of plastic waste by landfills induces environmental and space problems. Incineration of plastic waste leads to air pollution and global warming damages. Recycling of plastic waste can avoid all of the aforementioned problems. The advantages of plastic recycling are energy saving, reduction in disposal costs and resource conservation. However, management procedures for plastic waste recycling are not straightforward and could be costly in some cases, so recycled plastics can not compete equally with virgin plastics. In addition, the properties of recycled plastics might be poor, so the application for recycled plastics is still limited.

2.1.1 Elimination of Plastics Waste

Plastic products tend to have long-life span. Approximately 80 percent of all the plastic products have as long as 20 - 100 years of life time, most of these products are construction materials such as pipe and furniture. About 10 percent are medium-life products, i.e. shoes, clothing, gardening applications. They are items used for many times before they are thrown away. The last 10 percents are short-life or one-time used plastic products such as medical equipments, medicine packaging, food packaging materials, cling films, oil bottles, beverage bottles and cosmetics bottles, etc. [1] After use, many plastic products can be disposed by means of incineration, recycling or sanitary landfill, as stated in section 2.1.

2.1.1.1 Incineration

Incineration is one option for disposing of a large percentage of municipal solid waste. It is also a disposal method of post-consumer plastic waste which is similar to the waste conversion to thermal energy by burning the waste combined with municipal waste in incinerator. Plastics, paper, and other flammable material are firstly separated from the waste. In practice, higher-value plastics that are easily sorted, such as PET bottles, are manually separated. [4] In Thailand, there are quite a few incinerators for municipal waste in certain provinces such as Phuket, Surat-Thani, and Rayong, but most of the existing incinerations are for biological or infectious waste from medical area. Currently, both energy recovery and some non-energy recovery incinerators are widely used in Japan, USA and in Europe. [5] Similarly to the waste conversion method, proper pollution control system is also required to ensure the human health, safety and clean environment.

2.1.1.2 Landfill

Landfill is deemed as the last option in terms of conservation of resources and environment. Proper sanitary landfill must be authorized. Prevention system such as those on flooding, unexpected smell and aesthetic aspect must also be introduced. [6] According to the sanitary principles, landfilling is possibly done either by digging down to the depth of soil layers or by landfilling above the ground level, or the combination of both methods may be chosen, depending on the general condition of the geographical terrain of the area in which landfill will be made. In general, only a small proportion of plastics, about 1-2 percent of the total volume of the waste, makes its way to landfill. Most plastic products have long durable period, inherent stability and inertness, making landfilling a safe means of disposal. Moreover, plastic such as PVC is often used as the lining material for landfill sites because it is so stable, it prevents any toxic or dangerous substances present in the landfill from leaching out, especially into groundwater. [7] The fact that, both post-industrial and post-consumer plastic scraps can be recycled and many plastic end products are typically in long-life applications, landfilling is therefore the last option for disposal when the other means is not possible.

2.1.1.3 Recycling

Not all plastics are recyclable. Examples of plastics commonly recycled such as Polyethylene (PE-both high density and low density polyethylene), Polypropylene (PP), Polystyrene (PS), Polyethylene terephthalate (PET) and Poly(vinyl chloride) (PVC). They are "Thermoplastic", which soften when heated and harden when cooled. With this property, plastic products are practically subjected to be reused or recycled many times.

Most plastics can be recycled but a proper collection method is needed in order to make recycling easy. There are several alternative methods of collection for discarded plastic objects such as drop-offs, deposit return and curbside collection. The main problem for plastic recycling is mixed plastic waste since various types of plastics melt at different temperatures. If a plastic which melts at a high temperature is mixed with another plastic that melts at a lower temperature, the appearance and properties of the recycled material is altered signifying that the re-processing ability would be poor. A collection method can be used for preliminary sorting plastic wastes, for example, plastic objects can be source separated by both households and commercial groups. The international code system for recycling provided by the Society of the Plastic Industry (SPI) is widely used to identify different types of plastics. The code is a three-sided triangular arrow with a number in the center and letters underneath as shown in **Figure 2.1**. It is intended for molding into or imprinting on plastic products.



Figure 2.1: SPI International code systems for recycling

Polyethylene terephthalate (PET) has good barrier properties, clarity and toughness. The main applications for PET are in food packaging, carbonated beverages and non-carbonated beverages. High density polyethylene (HDPE) is used for many packing applications because it provides good stiffness, excellent moisture barrier properties and excellent chemical resistance. The applications for HDPE are rigid bottles, food container, buckets and crates. Poly(vinyl) chloride (PVC) is very versatile plastic because it can be compounded with stabilizers, plasticizers and other additives. Major applications for PVC are pipes, conduits, wires, bottles and packaging films. Low density polyethylene (LDPE) has good toughness and flexible characteristics. Major applications for LDPE are packaging, agricultural and squeezed consumer bottles. Polypropylene (PP) has high temperature resistance, good chemical resistance and medium gloss. It is often used in the industries of food container, straws, pipes and household appliances. Polystyrene (PS) is a hard, transparent and brittle thermoplastic polymer. It is widely used in many applications because of its low cost and easy processability, for example, in packaging industry, disposable products, housewares and toys. Other plastics refer to those besides the six commodity plastics classified above by number and including thermoset resins.

Table 2.1: Applications of plastics products.

Applications	Examples of plastic products
Construction materials	pipe, fitting, water drain, window and door profile, wallpaper, flooring
Automotive parts	automobile upholstery, automobile console
Electrical equipment	electrical cable, socket, LED
Food packaging materials	water bottle, food packs, cling films
Medical equipment	surgery glove, blood container bag
Others	child playthings, shoes, credit card, furniture, sofa, bathroom curtains

There are two types of recycling namely closed-loop and opened-loop recycling. For the closed-loop recycling, plastic producer has to collect the plastic products when they are due to be thrown away. This can be done through good consumer collection strategies such as renting the product or giving the consumer money for the products returned. A more popular route involves recycling the waste produced during the manufacturing process. Production waste such as imperfect moldings that have to be off-cuts each product when it is removed from the mold is easily collected. Plastic producers can then recycle the collected plastic waste and use the resulting recycled material to produce the same product again. [8, 9] For the open-loop recycling, use plastic products are gained from a variety of sources such as post consumer plastic waste or through municipal waste. The plastic is then recycled to produce new unrelated plastic products.

2.2 Composite Materials

Composite material is composed of two or more distinct phases and its bulk properties are significantly different from those of any of the constituents. The primary phase, known as the matrix phase, has a continuous character. It is usually the more ductile and less hard phase which holds the dispersed phase and shares a load with the dispersed phase. The second phase is placed within the matrix in a discontinuous form, is called the dispersed phase. It is usually stronger than the matrix, therefore it is sometimes called the reinforcing phase. The constituents can be organic, inorganic or metallic (synthetic and naturally occurring) in the form of the particles, rods, fibers, plates, foams, etc.

Many commercial polymer materials are composites. There are many reasons for using composite materials rather than the simple homogeneous polymers. Some of these reasons are:

- i) Increased stiffness, strength, and dimensional stability
- ii) Increased heat deflection (or distortion) temperature
- iii) Reduced permeability to gases and liquids
- iv) Increased toughness or impact strength

- v) Increased mechanical damping
- vi) Modified electrical properties
- vii) Reduced cost

The properties of composite materials are governed by the properties of the components, by the shape of the filler phase, by the morphology of the system, and by the nature of the interface between the phases. Thus a great variety of properties can be obtained with composites just by modification of one of these items. An important property of the interface that can greatly affect the mechanical behavior is the strength of the interfacial bonding between the phases. [10, 11]

2.2.1 Composite materials classification

Composites can be classified in many manners. One simple classification scheme is to separate them according to reinforcement forms.

2.2.1.1 Particulate-reinforced composites

Particulate-reinforced composites consist of particles dispersed in a matrix. The reinforcement is considered to be a “particle” if all of its dimensions are roughly equal. Particulate may have any shape, configuration, or size. They may be powdered, beads, rods, or crystalline. They may be metallic, ceramic, manmade, or natural materials. Concrete and wood particleboards are two familiar examples of particulate composites. Pieces of ceramic particles are placed in a metallic matrix and used as tough, abrasion resistant cutting tools. Metallic flakes have been added to improve electrical properties and provide some degree of radiation shielding in polymer composites. Generally, particles in polymer usually extend rather than reinforce the material. These are usually referred to as "filled" systems. Because filler particles are included for the aim of cost reduction rather than reinforcement, these composites are not generally considered to be particulate composites. Nonetheless, in some cases the filler will also reinforce the matrix material. [11, 12]

2.2.1.2 Fiber-reinforced composites

Fiber-reinforced composites are composed of reinforced fibers in a matrix. They contain reinforcements having lengths greater than their cross-sectional dimensions. They can be further divided into those containing discontinuous and continuous fibers. In discontinuous fibers case, the bond between the fibers and the matrix is broken at the fiber's ends, and thus carry less stress than at the middle part of the fiber while, continuous fibers can carry stresses along its length. [11-13]

2.2.1.3 Laminar composites

Laminar composites are composed of layers of materials held together by matrix binder, sandwich and honeycomb component as well as the term high-pressure laminates are included. Complicating the definition of a composite as having both continuous and discontinuous phases is the fact that in a laminar composite, neither of these phases may be regarded as truly continuous in their dimensions. [11-14]

2.2.2 Matrix

Matrix is an essentially homogeneous resin or polymer material in which the fiber system of composite is embedded. Both thermoplastic and thermoset resin may be used. The purpose of composite matrix is to bind the fiber together by virtue of its cohesive and adhesive characteristics, to transfer load to and between fibers and to protect them from environments and handlings. Nonetheless, the matrix resin provides many essential functions. In addition to those mentioned above, the matrix keeps the reinforcing in the proper orientation and position so that they can carry the intended loads, distribute the loads more or less even among the fiber, provide resistance to crack propagation and damage, and provide all of the interlaminar shear strength of the composite. Moreover, the matrix generally determines overall service temperature limitations of the composite, and may also control its environmental resistance. [16]

Poly(vinyl chloride), PVC

Polyvinyl chloride, PVC, is a thermoplastic material with the chemical formula of $\text{CH}_2=\text{CHCl}$ as shown in **Figure 2.2**.

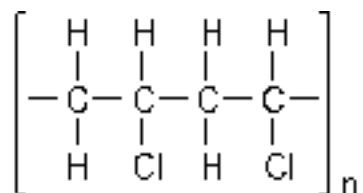


Figure 2.2: Chemical structure of Poly (vinyl chloride) (PVC).

PVC is one of the world's major polymers. It had a number of disadvantages; raw PVC polymer decomposes at about $100\text{ }^\circ\text{C}$, which is far below its melting point of $150\text{-}200\text{ }^\circ\text{C}$. PVC degrades in the presence of light and UV. It has poor mechanical properties and a relatively low softening temperature, between 70 and $80\text{ }^\circ\text{C}$. PVC resins are normally produced by one of the three polymerization procedures namely:

- i) Suspension polymerization (accounting about 80% of the total commercial production)
- ii) Emulsion (about 10- 15%)
- iii) Bulk or mass and solution polymerization (about 10%).

For suspension polymerization, vinyl chlorides are polymerized in an aqueous batch suspension. Emulsion PVC resins are polymerized in the presence of water-soluble, free radical initiator and characterized by high polymerization rates leading to high molecular weight resins. This type of PVC is normally less expensive than other forms of PVC, but requires higher amount of lubricants. [17] Bulk polymerized PVC resins are particles having very porous structure with a large surface. The basic repeat unit of PVC polymer chain is $[-\text{CH}_2-\text{CHCl}-]$. [24] PVC polymer resins have been reported to possess many attractive advantages but some imperfections do remain with virgin PVC. i.e., susceptibility to heat degradation, relatively high melts viscosity, comparably low heat deflection temperature, limited

strength and stiffness. To counteract these imperfections, some modifications are required with original PVC materials by adding small amount of a few additives such as heat or thermal stabilizer, plasticizers, impact modifiers, lubricants, fillers, etc. [17, 18] The functions of these additives can be summarized as follows:

- i) Heat stabilizers: to protect PVC against heat or shear degradation when process at elevated temperatures (above 170°C).
- ii) Plasticizers: to enhance the melt behavior of PVC resins and consequently improves the processability of PVC compounds. In addition, they help to convert hard, inherently brittle PVC resins into the compositions with varying degrees of softness and flexibility.
- iii) Lubricants: to reduce the frictional effects with the melt (external lubricant) ie., the friction at, and adhesion to, working surfaces as the composition is being processed. In addition, they lower the inter-particle and inter-molecular friction during processing (internal lubrication) and reduce the effective melt viscosity and heat build-up
- iv) Filler: to reduce material cost and enhance certain properties, particularly the stiffness. However, the addition of fillers has a deleterious effect on reducing the tensile strength, elongation at break, impact strength, and on increasing the hardness and density of the material. [19, 20]

Approximately 75% of PVC worldwide is produced by the suspension polymerization process, the polymerization of which is carried out inside vinyl chloride monomer (VCM) droplet dispersing in water. The PVC resin produced is in the form of porous particles with 100-150 μm in diameter. [21]

Table 2.2: Properties of rigid PVC. [12]

Property	Rigid PVC
Specific gravity	1.32-1.44
Tensile strength (MPa)	37.92-55.16
Tensile modulus (GPa)	2.83
Flexural strength (MPa)	41.37-110.32
Flexural modulus (GPa)	2.41-4.14
Compressive strength (MPa)	68.95-75.84
Water absorption after 24 h (%)	0.1
Weather stability	Good for light colors

PVC is a thermoplastic, which can soften and melt at elevated temperatures. Thus, by the application of heat and pressure, PVC may be extruded or molded into any desired shape. Processing can be formed from 180 to 200°C. PVC is uniquely responsive to functional additives, which permit the generation of rigid and flexible products, useful in novelties at low cost and in designed engineering applications. PVC is used in a wide range of applications because of its combined physical properties of:

- Compounding versatility as rigids or flexible.
- High modulus (unplasticized)
- Ease of fabrication.
- Low flammability.
- Low cost.

Because of the compounding versatility of PVC, consequently, PVC can be used in many applications such as wire insulation, tube, film, sheet, footwear, toys, hose, and many other applications.

2.2.3 Fillers

Filler is a finely divide solid that is added to a liquid semisolid, or solid composition to modify the composition's properties and reduce its cost. Filler are usually classified according to their source, function, composition or morphology, as shown in **Table 2.3**. [22] However, none of these classification schemes is entirely adequate owing to overlap and ambiguity of their categories. Filler can constitute either a major or a minor part of a composition. The structure of the filler particles can range from irregular masses to precise geometrical form such as spheres, polyhedrons, or short fiber. [35] An understanding of physical properties of filler and associated function is most important in applying filler technology. Mineral fillers represent over 80% of a filler market primarily because of their low cost.

Table 2.3: Classification of fillers.

Method of classification	Examples
Source	Natural (mineral), synthesis
Function	Pacification, reinforcement
Composition	Carbonate, silicate, lignin
Morphology	Hollow, sphere, fiber, platelet

In the plastics industry, the term filler refers to particulate materials that are added to plastic resins relatively large (over 5 %) volume loadings. At present, the performance specifications for most compounded plastics products are less strict that for other filled products, such as elastomers and paints, and cost reduction is the primary objective. [36]

Wood flour (WF)

Wood flour is a finely ground wood, dried wood product, fibrous in structure that acts as reinforcing materials for plastics. It is made mostly from softwood, chiefly pine and spruce, but hardwood maple or ash flour are used where no wood resin content is desired. Wood containing essential oils, such as cedar, are not

suitable. Wood flour is made from sawdust, chip and shavings by grinding in a buhrstone mill, and has the appearance of wheat flour.

Compared to inorganic filler, wood flour filler offer many advantages such as lower cost, little damage during processing, lower density, abundant renewable resource, biodegradable in nature and no health hazard. [23] The components of natural fibres are cellulose, hemicellulose, lignin, pectin, waxes and water-soluble substances, with cellulose, hemicellulose and lignin as the basic components with regard to the physical properties of the fibers. The percentage composition of each of these components varies for different fiber. It is composed of fibers of cellulose (40–50%), hemicellulose (15–25%) impregnated with lignin (15–30%).

Bulk density of wood flour is typically around 0.1-0.3 g/cm³. Aspect ratio of wood flour (length of thickness of fibers) is typically between 3:1 and 5:1. Specific gravity (density) of wood is about 1.3-1.4 g/cm³. When ashes (commonly at temperature between 525 and 575 °C), wood often leaves 0.25-0.50% of mineral residues. Wood Flour and sawdust do not melt but rather decompose above 190°C. This is, again, due to a more susceptible to temperature lignin and hemicellulosics, and because pure cellulose decomposes above 240 °C. [24]

2.3 Polymer Processing

2.3.1 Twin-screw extrusion

Twin-screw extrusion is used extensively for mixing, compounding, or reacting polymeric materials. The flexibility of twin-screw extrusion equipment allows this operation to be designed specifically for the formulation being processed. Twin screw extruders have already been mentioned as a type of extrusion equipment that is valuable with certain resins, especially those that are sensitive to heat such as PVC. The use of twin screws, which operate side by side in tandem, requires different linkages and barrel than would be used single screw operation. Therefore, an extruder cannot be converted from a single screw to a twin screw machine.

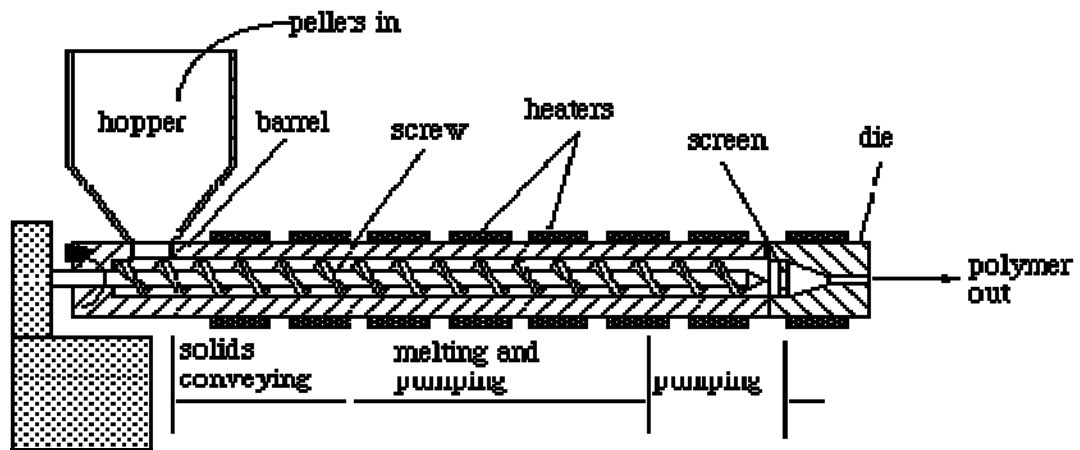


Figure 2.3: A simplified extruder diagram. [25]

Twin screw extruder is divided into co-rotating and counter rotating types. As the names indicate, the difference is in whether the two screws rotate in the same or in opposite direction, i.e. both clockwise or counterclockwise, or one in each sense. Twin screw extruder acts as positive displacement pumps with little dependence on friction, and this is the main reason for their choice for heat sensitive materials such as PVC. [26]

i) Co-Rotating Twin Screw Extruder : The co-rotating twin screw extruder transfers the melt from the screw channel of one screw to the other screw. The conveying mechanism, drag force is comparable to that found in the single screw extruder. By being transferred from one screw channel to another, the melt does follow a longer path and is subjected to higher shear. The co-rotating twin screw extruder finds application primarily in compounding. **Figure 2.4 (a)** presents the co-rotating twin screw extruder.

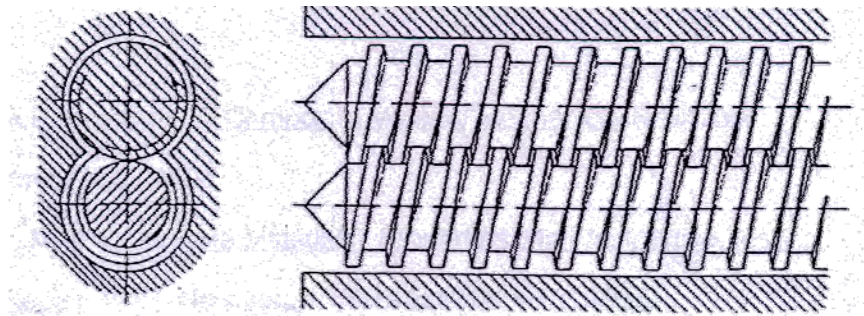


Figure 2.4: (a) Co-rotating twin screw extruder. [26]

ii) Counter-Rotating Twin Screw Extruder : Each screw segment forms a close chamber that conveys the melt material from the hopper to the end of the screw without any change with neighboring chambers. Drag forces are not needed for this conveying, with the result that little dispersion induced heating occurs. Heating occurs largely via the heater bands on the barrel, which can be controlled and permit gentle heating of sensitive materials. While this conveying and melting mechanism assures well controlled, gentle handling of the material, the melt is usually inhomogeneous and insufficiently plasticated so that the use of mixing elements is also be recommended here. **Figure 2.4 (b)** shows the counter-rotating twin screw extruder.

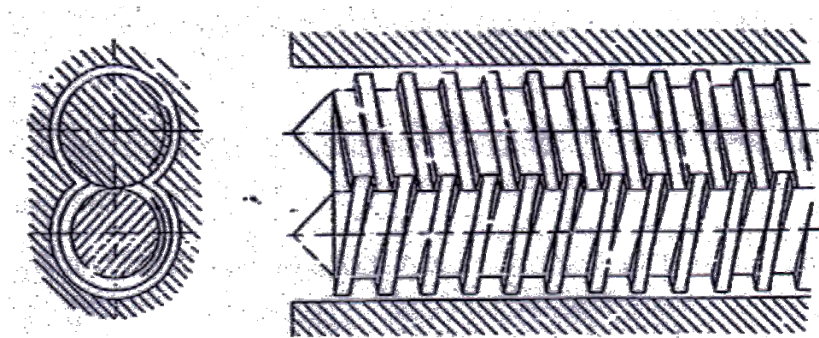


Figure 2.4: (b) Counter-rotating twin screw extruder. [26]

In addition, the configurations of the screws themselves may be varied using forward conveying elements, reverse conveying elements, kneading blocks, and other designs in order to achieve particular mixing characteristics.

2.4 Polymer Characterizations

2.4.1 Mechanical characterizations

2.4.1.1 Tensile test

Tensile test is one of the most fundamental type of mechanical tests. As shows in **Figure 2.5**, a curve will result showing how the polymer reacted to the forces being applied. The point of failure is of much interest and is typically called the ultimate strength. For most tensile testing of materials, the initial portion of the

test, the relationship between the applied force, or load, and the elongation the specimen exhibits is linear. In this linear region, the line obeys the relationship defined as "Hooke's Law" where the ratio of stress to strain is a constant, or $E = \sigma / \epsilon$. E is the slope of the line in this region where stress (σ) is proportional to strain (ϵ) and is called the Modulus of Elasticity or Young's Modulus. One of the properties can determine about a material is its ultimate tensile strength (UTS). This is the maximum load the specimen sustains during the test. The UTS may or may not equate to the strength at break. This all depends on what type of material you are testing such as brittle, ductile, or a substance that even exhibits both properties.

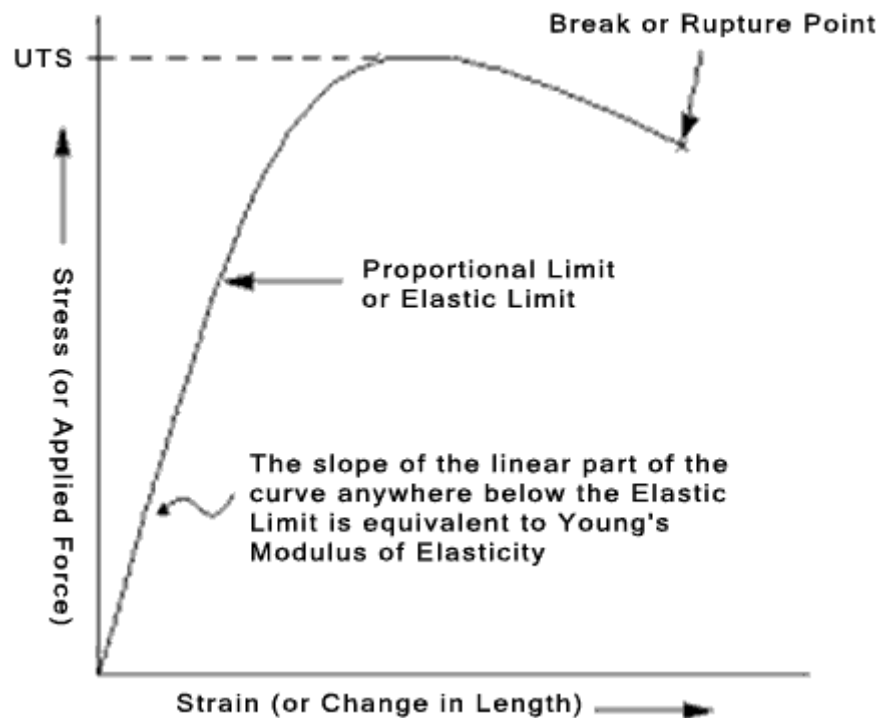


Figure 2.5: Stress-strain curve of tensile properties.

2.4.1.2 Flexure test

The flexure test method measures behavior of materials subjected to simple beam loading. It is also called a transverse beam test with some materials. Maximum fiber stress and maximum strain are calculated for increments of load. Results are plotted in a stress-strain diagram. Flexural strength is defined as the maximum stress in the outermost fiber. This is calculated at the surface of the

specimen on the convex or tension side. Flexural modulus is calculated from the slope of the stress vs. deflection curve. If the curve has no linear region, a secant line is fitted to the curve to determine slope. A flexure test produces tensile stress in the convex side of the specimen and compression stress in the concave side. This creates an area of shear stress along the midline. To ensure the primary failure comes from tensile or compression stress the shear stress must be minimized. This is done by controlling the span to depth ratio; the length of the outer span divided by the height (depth) of the specimen. For most materials $S/d=16$ is acceptable. Some materials require $S/d=32$ to 64 to keep the shear stress low enough. Flexure testing is often done on relatively flexible materials such as polymers, wood and composites. There are two test types; 3-point test and 4-point test. In a 3-point test the area of uniform stress is quite small and concentrated under the center loading point. In a 4-point test, the area of uniform stress exists between the inner span loading points (typically half the outer span length).

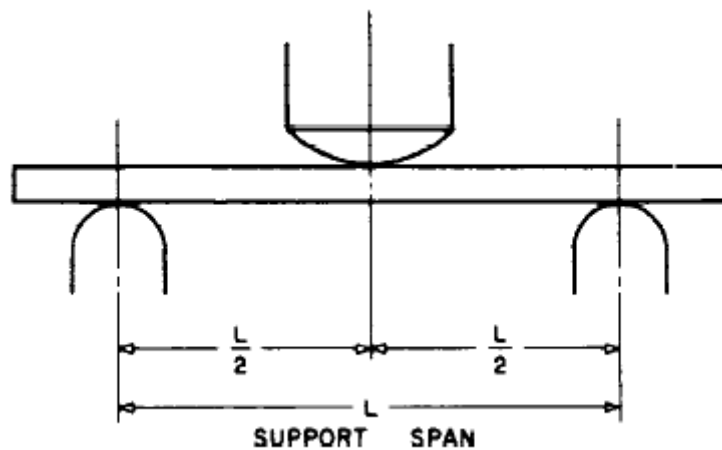


Figure 2.6: Three- point bending test.

2.4.1.3 Compressive test

A compression test determines behavior of materials under crushing loads. The specimen is compressed and deformation at various loads is recorded. Compressive stress and strain are calculated and plotted as a stress-strain diagram which is used to determine elastic limit, proportional limit, yield point, yield strength and, for some materials, compressive strength. **Figure 2.7** illustrates the modes of deformation in compression testing. (a) Buckling, when $L/D > 5$. (b)

Shearing, when $L/D > 2.5$. (c) Double barreling, when $L/D > 2.0$ and friction is present at the contact surfaces. (d) Barreling, when $L/D < 2.0$ and friction is present at the contact surfaces. (e) Homogenous compression, when $L/D < 2.0$ and no friction is present at the contact surfaces. (f) Compressive instability due to work-softening material.

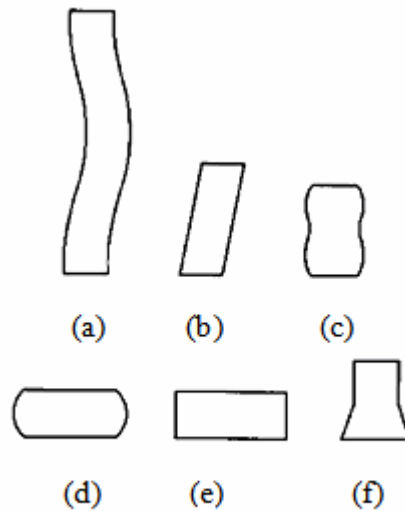


Figure 2.7: The modes of deformation in compression testing.

2.4.1.4 Notched izod impact test

Notched izod impact is a single point test that measures a polymer resistance to impact from a swinging hammer. Izod impact is defined as the kinetic energy needed to initiate fracture and continue the fracture until the specimen is broken. Izod specimens are notched to prevent deformation of the specimen upon impact. This test can be used as a quick and easy quality control check to determine if a material meets specific impact properties or to compare materials for general toughness. **Figure 2.8** illustrates the specimen is clamped into the impact test fixture with the notched side facing the striking edge of the hammer. The hammer is released and allowed to strike through the specimen. The result of the Izod test is reported in energy lost per unit of specimen thickness at the notch (such as ft-lb/in or J/cm) or the results may be reported as energy lost per unit cross-sectional area at the notch (ft-lb/in² or J/m²).

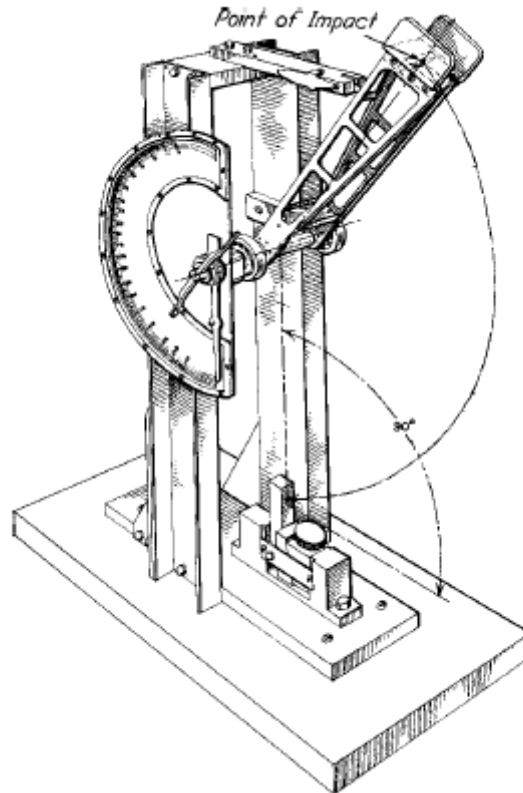


Figure 2.8: impact machine.

2.4.2 Thermal characterizations

2.4.2.1 Dynamic mechanical analysis (DMA)

DMA is a technique used to study and characterize materials. It is most useful for observing the viscoelastic nature of polymers. Two methods are currently used. One is the decay of free oscillations and the other is forced oscillation. Free oscillation techniques involve applying a force to a sample and allowing it to oscillate after the force is removed. Forced oscillations involve the continued application of a force to the sample. An oscillating force is applied to a sample of material and the resulting displacement of the sample is measured. The test specimen is clamped between the movable and stationary fixtures, and then enclosed in the thermal chamber. Frequency, amplitude, and a temperature range appropriate for the material being tested are input. The elastic modulus (or storage modulus, G'), viscous

modulus (or loss modulus, G'') and damping coefficient (Tan D) is determined as a function of temperature, frequency or time.

2.4.2.2 Thermogravimetric analysis (TGA)

TGA is a type of testing that is performed on samples to determine changes in weight in relation to change in temperature. The analyzer usually consists of a high-precision balance with a pan loaded with the sample. The sample is placed in a small electrically heated oven with a thermocouple to accurately measure the temperature. The atmosphere may be purged with an inert gas to prevent oxidation or other undesired reactions. A computer is used to control the instrument. The percent weight loss of a test sample is recorded while the sample is being heated at a uniform rate in an appropriate environment. The loss in weight over specific temperature ranges provides an indication of the composition of the sample, including volatiles and inert filler, as well as indications of thermal stability. TGA is commonly employed in research and testing to determine characteristics of materials such as polymers, to determine degradation temperatures, absorbed moisture content of materials, the level of inorganic and organic components in materials, decomposition points of explosives, and solvent residues. It is also often used to estimate the corrosion kinetics in high temperature oxidation.

2.4.2.3 Brabender torque rheometer

The Brabender torque rheometer, an instrument which continuously measures the torque exerted in shearing a polymer or compound specimen over a wide range of shear rates and temperatures, including those conditions anticipated in actual plant practice. The instrument records torque, time and temperature on a graph called a plastogram. The results provide information with regard to processability of an experimental compound and the effects of additives and fillers. It also measures and records lubricity, plasticity, scorch, cure, shear and heat stability and polymer consistency

The Brabender torque rheometer comprises a measuring cell in the form of an enclosed kneading chamber, in which two interlocking and counter-rotating Z-shaped kneading arms are installed. The speed of these kneading arms can

be varied, as can the temperature of the measuring cell either by cooling or heating. It is the torque which is measured, based on the premise that plasticity constitutes the capability of a ceramic body to have a certain resistance to deformation during the kneading process. With the use of the Brabender torque rheometer, the fundamental interdependence between plasticity and moisture content for ceramic bodies can be determined very precisely. This applies similarly to the interdependence between plasticity and temperature, or the dependency of plasticity on additives. The plasticity readings obtained from the Brabender torque rheometer expressed in the form of torque figures are not identical with the plasticity figures relevant to extrusion. This also holds true for those measured values determined by the penetrometer or other measuring methods. Yet the Brabender torque rheometer is ideally suited for the development or optimization of bodies which need to be plastified prior to extrusion by means of continuous or intermittent kneading.

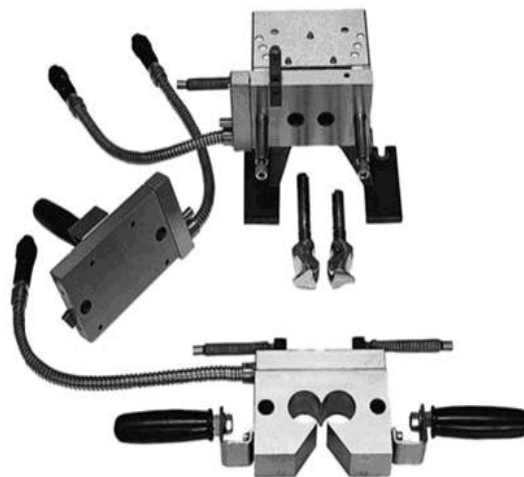


Figure 2.9: Disassembled of Brabender torque rheometer

2.4.3 Physical characterization

2.4.3.1 Gel permeation chromatography (GPC)

Gel permeation chromatography (GPC) is a separation technique based on hydrodynamic volume (size in solution). Molecules are separated from one another based on differences in molecular size. This technique is often used for polymer molecular weight determination. GPC, which is used when an organic

solvent is used as a mobile phase. A broader term which encompasses both of these separation methods is size exclusion chromatography (SEC).

GPC is accomplished by passing the sample through a porous media. Larger molecules can not access some of the pores and exit the column more rapidly. Smaller molecules penetrate into more of the porous structure and elute at longer retention times. **Figure 2.10** shows an example of this process and the path which two molecules of different size take when passing through a GPC column. The Larger molecules can not pass some of the pores of the gel while the smaller molecules fits into all the pores of the gel increasing the time required passing through the column. It is this filtration effect which causes separation by size. GPC is the most commonly used method for polymer molecular weight and molecular weight distribution or molecular mass distribution, which is defined as M_w divided by M_n and gives an indication just how narrow a distribution is. The process is done by first analyzing a series of standards of known molecular weight. The retention time for these standards is then used to create a calibration curve. The retention time for an unknown material can then be determined based on the retention volume at which it elutes. GPC differs from most other methods of chromatography in that separations are accomplished without interactions between the stationary phase and the sample. Interactions are undesirable and prevent a purely size based separation. This can be contrasted with the more common reverse phase chromatography (RP) in which separation is accomplished based on differences in the hydrophobic (van der waals) interactions between the column and sample components.

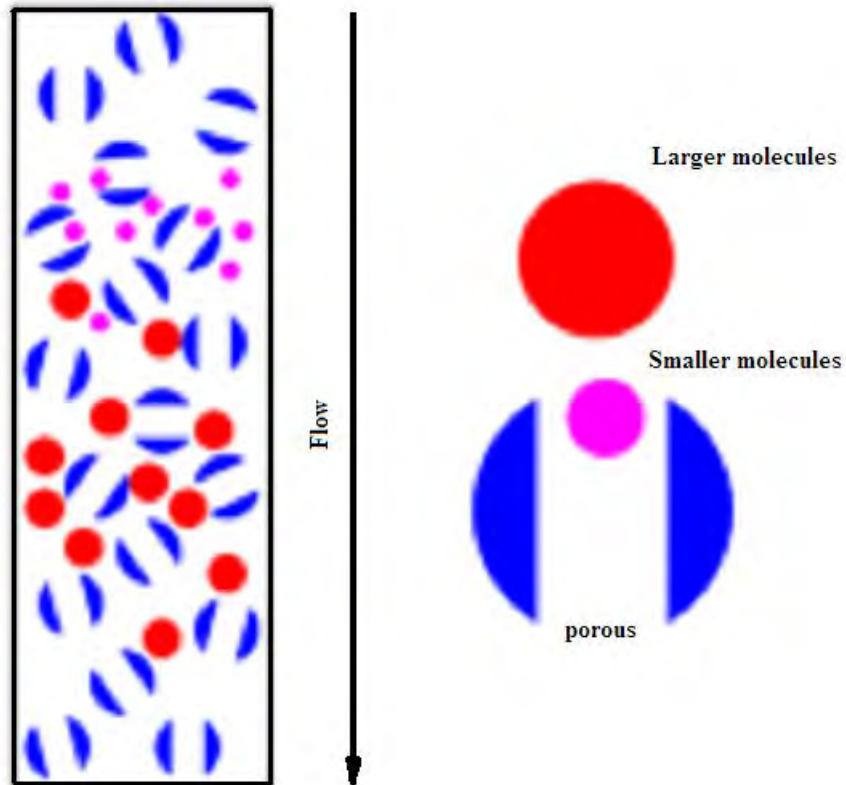


Figure 2.10: The path of two molecules of different size when passing GPC column

Different average values can be defined depending on the statistical method that is applied.

- (i) Number-average molecular weight

$$\bar{M}_n = \frac{\sum n_i M_i}{\sum n_i} = \frac{\sum w_i}{\sum (w_i / M_i)}$$

where

n_i = number of molecules with molecular weight M_i

w_i = weight fraction of material having molecular weight M_i

- (ii) Weight-average molecular weight

$$\bar{M}_w = \frac{\sum w_i M_i}{\sum w_i}$$

(iii) Z -average molecular weight

$$\bar{M}_Z = \frac{\sum w_i M_i^2}{\sum w_i M_i}$$

(iv) Z+1 -average molecular weight

$$\bar{M}_{Z+1} = \frac{\sum w_i M_i^3}{\sum w_i M_i^2}$$

Methods for determining molecular weights include measurements of osmotic pressure, light scattering, sedimentation, equilibrium, dilute solution viscosity, freezing points, vapor pressure

2.4.4 Morphology characterization

2.4.4.1 Scanning electron microscope (SEM)

SEM is a type of electron microscope that creates various images by focusing a high energy beam of electrons onto the surface of a sample and detecting signals from the interaction of the incident electrons with the sample surface. The surface of the sample is sputter coated in a vacuum with an electrically conductive layer of gold. The coated dry sample is now placed in a vacuum so that the electron beam can move without interference. Electricity is passed through the wire and then focused by magnets onto the sample. When the electrons from the gun strike the surface coating of gold, electrons are reflected back off the specimen to a detector, this is transmitted to a TV screen where the image is viewed and photographed. The SEM is used to examine biological materials (such as micro-organisms and cells), a variety of large molecules, metals and crystalline structures, and the characteristics of various surfaces.

CHAPTER III

LITERATURE REVIEW

In this chapter, the literature reviews on recycling of plastics are summarized.

Takahashi, K. *et al* [38] studied the performance of the blend of 25% post-consumer recycled blow molded HDPE bottles and 75% virgin HDPE. They found that the addition of the 25% recycled HDPE to the virgin polymer slightly decreased the melting temperature, heat of fusion, dynamic modulus and tensile impact energy. The small changes in the properties of the blended HDPE with 25% recycled content were not expected to significantly affect the performance of the base HDPE polymer.

Dintcheva, N. T. *et al.* [39] studied recycling of plastics blend for packaging. The recycled material is a blend of low density polyethylene (LDPE) and linear low density polyethylene (LLDPE). It was found that the mechanical and the rheological properties of the recycled HDPE depended on the processing condition, temperature and apparatus. The elongation at break and the viscosity of the recycled HDPE prepared in a closed mixer was lower than those of the HDPE re-processed in a single-screw extruder and a twin-screw extruder. High processing temperature and high residence times strongly enhanced the degradation processes and reduced the mechanical properties, particularly the elongation at break. The addition of ethylene vinyl acetate copolymer (EVA) in the recycled materials resulted in an improvement of the elongation at break. A possible use of this recycled plastic was for the production of low pressure pipes but the properties were lower than those of virgin pipe grade polyethylene.

Loultcheva, M. K. *et al.* [40] studied recycling of HDPE containers. The rheological and mechanical properties of recycled HDPE were found to depend on the re-processing conditions. They found that the viscosity of recycled HDPE at low shear rate increased with increasing number of extrusions due to the formation of chain branching. High residence time in the extruder led to a decrease of mechanical

properties, particularly the elongation at break. In addition, the elongation at break and the tensile strength of the HDPE recycled in a twin-screw extruder was higher than that of the HDPE recycled in a single-screw extruder. The addition of stabilizers in HDPE helped by maintaining the initial properties of the HDPE even after several recycling cycles.

Zahavich, A. T. P *et al.* [41] studied the effects of multiple extrusions on the properties of HDPE recycled from blown bottles. The complex viscosity at low frequency of recycled HDPE increased with an increasing number of extrusions. They used the cross over point of G' and G'' (storage and loss modulus) to measure the relative changes in the molecular weight distribution (MWD). It was indicated that the MWD of both the recycled and the virgin HDPE decreased with increasing extrusion passes. From the DSC results, the degree of crystallinity of the recycled HDPE decreased as the extrusion passes increased from zero to four passes. It was reported that degradation mechanisms such as chain scission and chain branching could be considered to be responsible for the decline in crystallinity.

Steenkamer, D. A. *et al.* [42] studied the recyclability of a cyclic thermoplastic composite which contained 58.7% by weight of glass fiber and Polybutylene terephthalate (PBT) as matrix. The composite was recycled by using a single-screw extruder. Re-extruded PBT/glass fiber was exposed to room temperature soaking in distilled water. After 0, 72, 168, and 312 hours of soaking, they were removed and wiped with dry cloth. Characterization was performed by using a gel permeation chromatography (GPC) as well as conducting a tensile test. The results showed an initial drop in molecular weight, tensile modulus, tensile strength, and elongation at break in unsoaked and soaked PBT/glass fiber for the 72 hours of exposure. However, the properties of those soaked for 168 and 312 hours were quite steady.

Xu, Q. W. *et al.* [43] studied the properties of recycled Rodrun LC-5000 liquid crystalline polymer (LCP) and polycarbonate (PC) binary composite with the weight ratio of LCP : PC at 30 : 70. Reprocessing was conducted by using a reciprocating screw injection molding machine. The study showed that the Young's modulus of the re-processed LCP/PC composites decreased slightly when the number of recycling increased. Moreover, the tensile strength and the tensile strain were enormously

reduced. However, the melt flow rate (MFR) of the recycled LCP/PC binary composite increased significantly with the number of the recycling. This was related to the decreasing molecular weight of the PC component. Microscope observation showed the average diameter of the LCP droplets was smaller and the surface of the composite was smoother when the number of recycling increased.

Carroll, D. R. et al. [44] studied the structural properties of Duraboard® plastic lumber made from recycled plastic, sawdust, and some other proprietary additives. The tests were carried out at -23.3°C to simulate winter and 40.6°C to simulate summer conditions. The result showed that mechanical properties namely the compressive strength, compressive modulus, flexural strength, flexural modulus, tensile strength, tensile modulus, and shear strength at -23.3°C were much more than those obtained at 40.6°C . Furthermore, the tests were done to compare the mechanical properties between plastic lumber and wooden lumber at normal temperature. The results showed that the plastic lumber had more shear strength, and was probably less likely to crack and split.

Dintcheva, N. T. et al. [45] studied the characterization and re-processing of greenhouse films made of polyethylene. They found that the degree of crystallinity of the post-consumer film was higher than that of the virgin film. The viscosity, the tensile strength and the elongation at break of the post-consumer film was lower than that of the virgin film. They claimed that this was due to the decrease of the molecular weight of postconsumer film, caused by photo-oxidative degradation during use. In addition, the mechanical properties decreased with the number of re-processing and with increasing level of photo-oxidative degradation.

Bertin, S. et al [46] studied recycling of mixed plastic waste composed of low-density polyethylene (LDPE) and polypropylene (PP). Both plastics were post-consumer waste. Blends of the virgin polymers had been prepared to compare the mechanical properties of both the virgin and the regenerated materials. Blending was carried out by using a single-screw and a twin-screw extruders. In this work, LDPE was mixed with PP at the ratio 90 : 10 by weight. The results showed that the twin-screw extrusion of both the virgin and the regenerate materials led to more homogeneous LDPE/PP blends with better mechanical properties (modulus, tensile

strength and elongation at break) than using the single-screw extrusion. Furthermore, compatibilizers such as ethylene–propylene copolymer (EPM), ethylene-propylene-diene monomer (EPDM), PE-g-poly (2-methyl-1, 3-butadiene) were incorporated to the LDPE/PP blends. These copolymer compatibilizers were forced to have sharp effects on some mechanical properties such as elongation at break and charpy impact energy.

Selke, S. E. et al. [47] prepared wood plastic composites containing recycled low density polyethylene (LDPE), high density polyethylene (HDPE), polypropylene (PP), polyethylene terephthalate (PET) and polyester mixed with wood fiber and compared the effects of using virgin plastics and recycled plastics as matrix. The composites were extruded by using Baker-Perkin co-rotating twin-screw extruder. The results showed that the composites made from recycled plastics had higher tensile strength than those made from the virgin ones. This mean that the difference between the recycled and the virgin plastics used as the matrix had slight effects on the wood plastic composites.

Giraldi, A. et al. [48] studied the thermal and the mechanical properties of recycled polyethylene terephthalate (PET) reinforced with glass fiber. They focused on the influences of two variables in the extrusion process namely the screw speed and the torque level. The composition of the glass fiber:PET:antioxidant were 30:69.5:0.5 by weight sequentially. The study showed that high level of screw torque was a significant factor leading to an increase in the impact strength of the recycled composites while the high screw speed of 200 rpm was a significant factor leading to an increase in the Young's modulus.

CHAPTER IV

EXPERIMENTAL

The experimental work of this research is directed towards evaluation of changes in the mechanical, thermal and physical properties upon closed-loop recycling and re-processing of thermoplastic WPC produced from PVC matrix and wood flour.

4.1 Experimental Procedure

4.1.1 Preparation of raw material for processing

Industrial scrap of WPC profiles extruded from poly(vinyl chloride) filled with 20 phr of wood flour for floor and deck applications was collected as raw material for this study. The collected scrap consisted mainly of waste from the start-up process and those rejected during quality inspection.

The scrap of PVC/wood flour wood-plastic profiles was grinded by using a pipe/profile granulators (ZERMA 50). Grinding was conducted until the scrap granules were close in size to the WPC freshfeed pellets. The dimensions of the granules were about 3-5 mm, as shown in **Figure 4.1**.

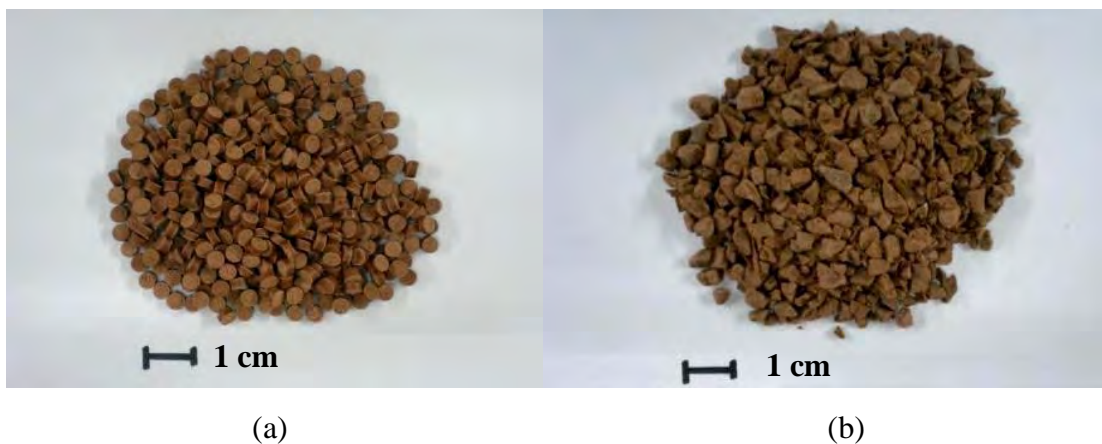


Figure 4.1: Pellets of PVC/wood flour (a) fresh feed and (b) grinded WPC scrap

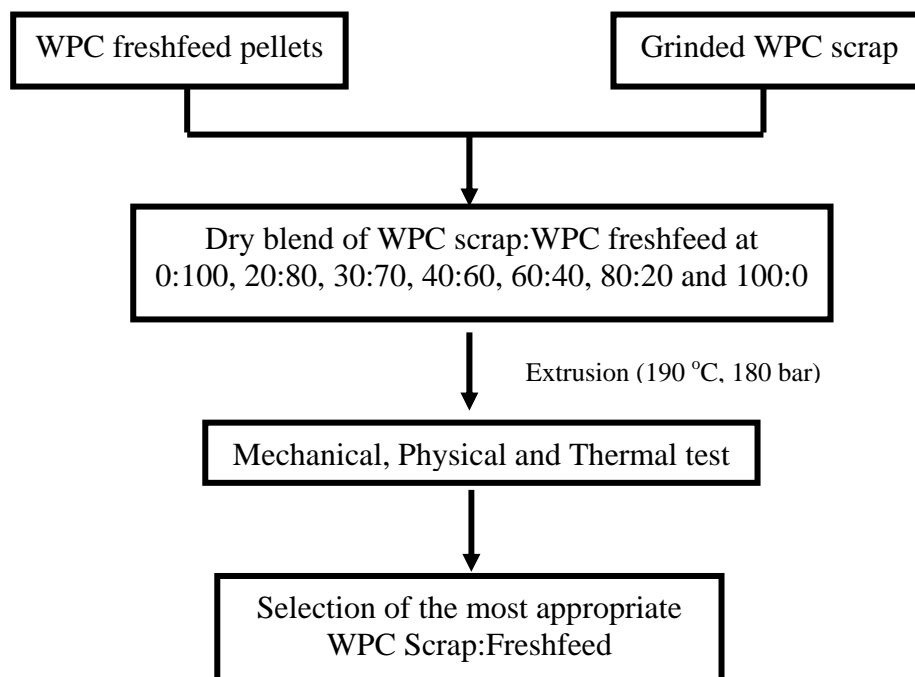
4.1.2 Preparation of wood plastic composites

The experimental work on recycling wood plastic profiles of the PVC/wood flour composites consisted of two parts as detailed below.

Part 1: To evaluate the appropriate ratio for recycling of WPC scrap:WPC freshfeed.

This part concentrated on re-processing the mixture of industrial scrap of PVC/wood flour WPC profiles with its freshfeed. Different weight ratio of the grinded WPC scrap:WPC freshfeed pellets was dry blended and re-extruded as recycled WPC profiles at 0:100, 20:80, 30:70, 40:60, 60:40, 80:20 and 100:0, as shown schematically in **Figure 4.2 (a)**. Re-processing of WPC profiles was conducted by using an industrial scale twin screw extruder. The mechanical, physical and thermal properties of the WPC produced from each mixture were evaluated and compared to determine the most appropriate mix ratio of WPC scrap:WPC freshfeed.

PART I



(a)

Part 2: To investigate the effects of number of re-processing passes on recycled WPC mixture.

In reality, there is opportunity whereby some of the re-processed WPC profiles may be rejected and become industrial WPC scrap again, so the second part of this study focuses on the effects of re-processing the WPC profiles produced from the selected formulation of the WPC scrap:WPC freshfeed in Part 1. Re-processing of profiles was conducted repeatedly for up to seven passes by using the industrial scale extruder, as shown schematically in **Figure 4.2 (b)**.

PART II

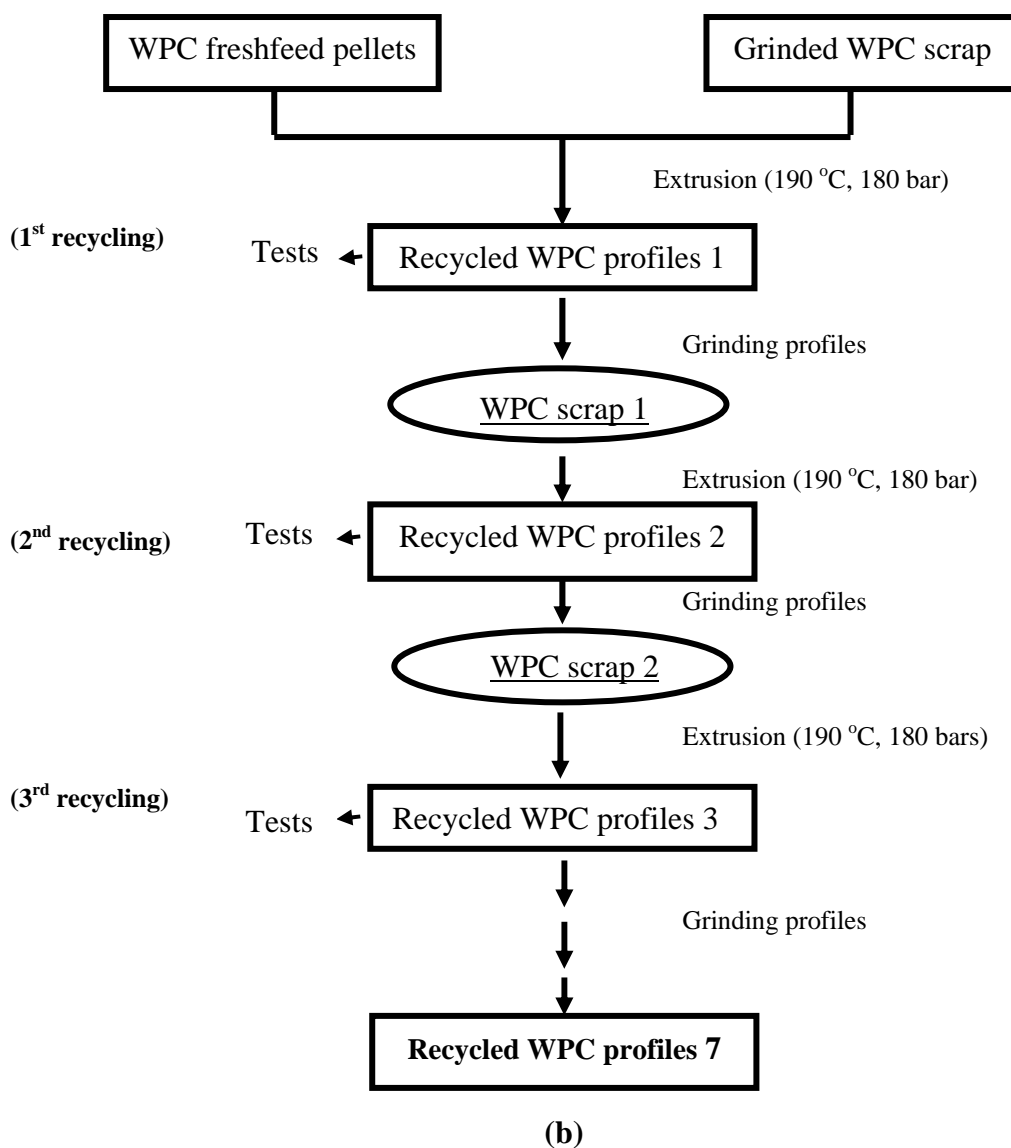


Figure 4.2: Recycling and re-processing scheme of the WPC in the present study.

(a) Part 1 (b) Part 2.

4.2 Characterization of PVC/WF Composites

Several mechanical, thermal and physical are commonly tested in plastics were conducted on the recycled WPC obtained from Part 1 and Part 2 as shown in **Figure 4.3**.

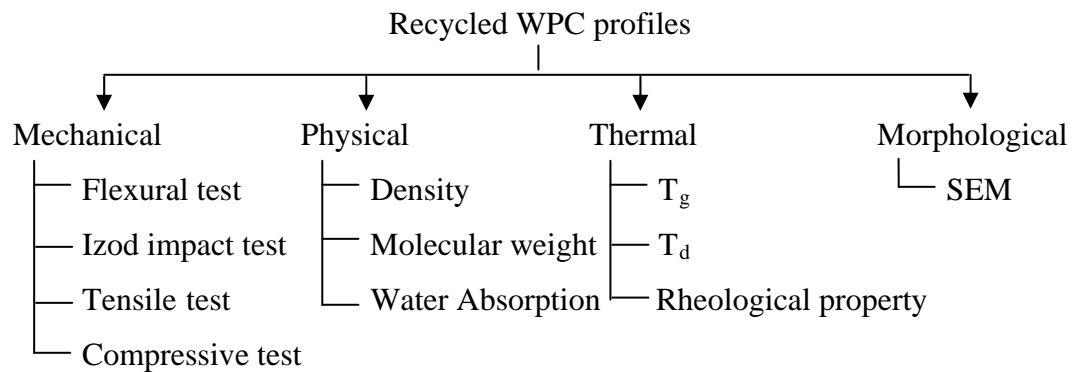


Figure 4.3: The characterization of recycled PVC/wood flour composites

4.2.1 Mechanical properties

4.2.1.1 Tensile test

Tensile tests yield normal tensile stress-strain relationships of the WPC. Specimens of recycled WPC were cut by a router to form a “dog bone” shape with a uniform thickness of 5mm. as shown in **Figure 4.4 (a)**. The dimension of each test specimen was 160 mm long, 12 mm wide and 5 mm in thickness, and a gage length of 115 mm. The WPC tensile tests for specimens prepared from freshfeed, scrap and their mixture were conducted according to ASTM D638-03 [29] by using a Universal Testing Machine (INSTRON Instrument, model 5567). The test was performed at a crosshead speed of 1.2 mm/min, pre-loading at 5 N. Only the results averaged from ten tested specimens were reported.

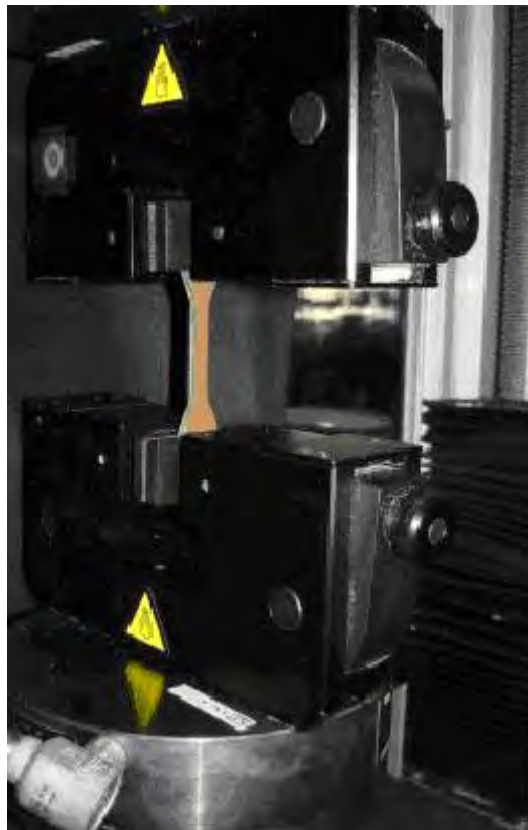
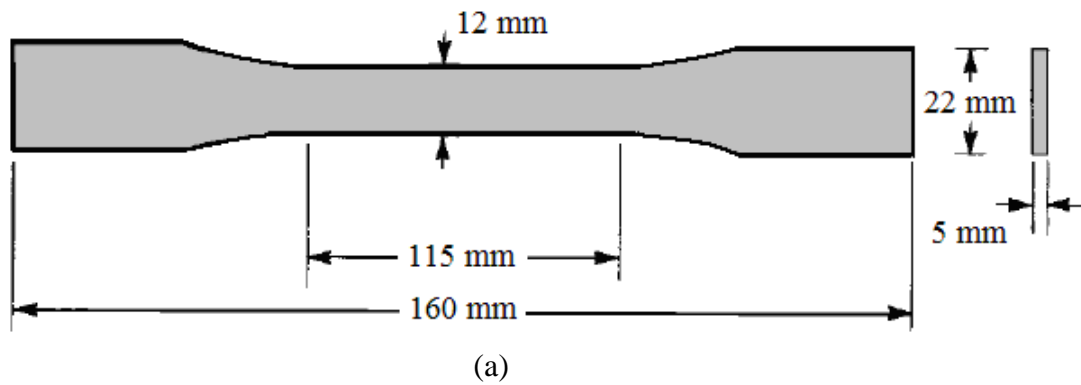
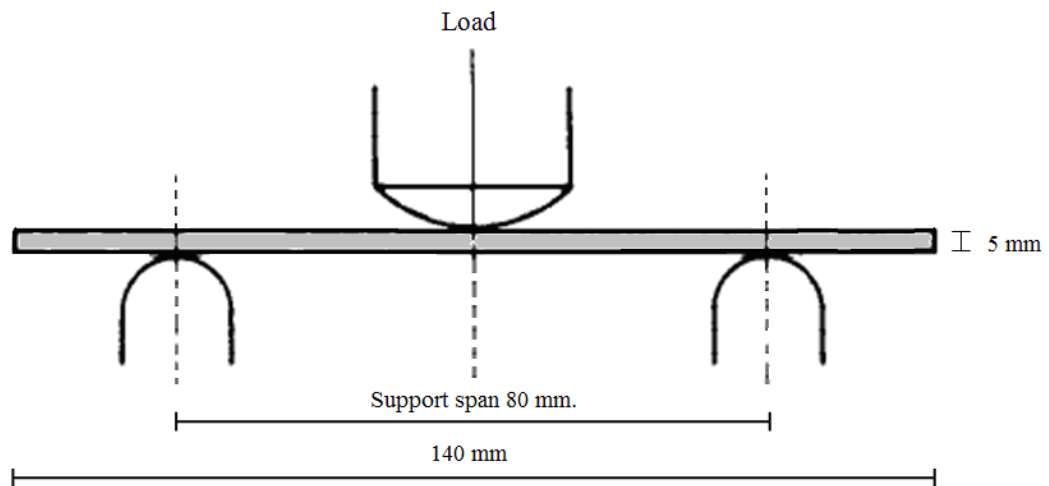


Figure 4.4: Tensile test: (a) Dimensions of tensile test specimen and (b) Tensile test.

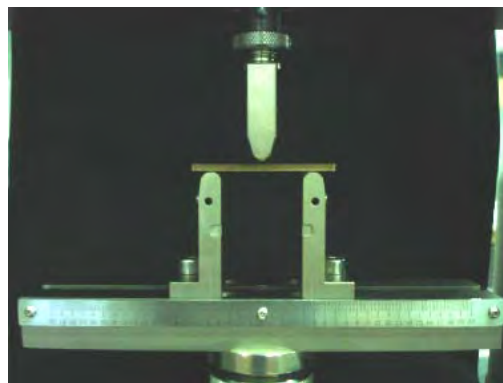
4.2.1.2 Flexural test

Wood-plastic composites are often used in applications where flexural properties are important. The flexural properties of the recycled wood-plastics composites were determined by using a mechanical testing machine (INSTRON Instrument, model 5567). A three-point loading test with a supporting

span of 80 mm was used . Loading was applied at the center of the rectangular beam of recycled WPC specimen, as shown in **Figure 4.5**, at a crosshead speed of 1.2 mm/min. The dimensions of the specimens were 140 mm long, 20 mm wide and 4 mm in thickness. The flexural properties were determined based on ASTM D790M-93. [30] Results reported were the average of ten tested recycled WPC samples.



(a)



(b)

Figure 4.5: (a) Dimensions of the flexural specimen and (b) Flexural test.

4.2.1.3 Compressive test

The compressive properties of the wood plastic composites were evaluated by using a Mechanical Testing Machine (INSTRON Instrument, model 5567) according to ASTM D695-02 [31] with the crosshead speed at 1.3 mm/min. As

shown in **Figure 4.6(a)**, the dimensions of the test specimen were 20 mm long, 20 mm wide and 5 mm in thickness. **Figure 4.6(b)** demonstrated the compressive test specimen placed between two parallel compressive plates, the upper plate was connected to the load cell. The compressive properties reported represented the average of the results obtained from testing ten recycled WPC samples.

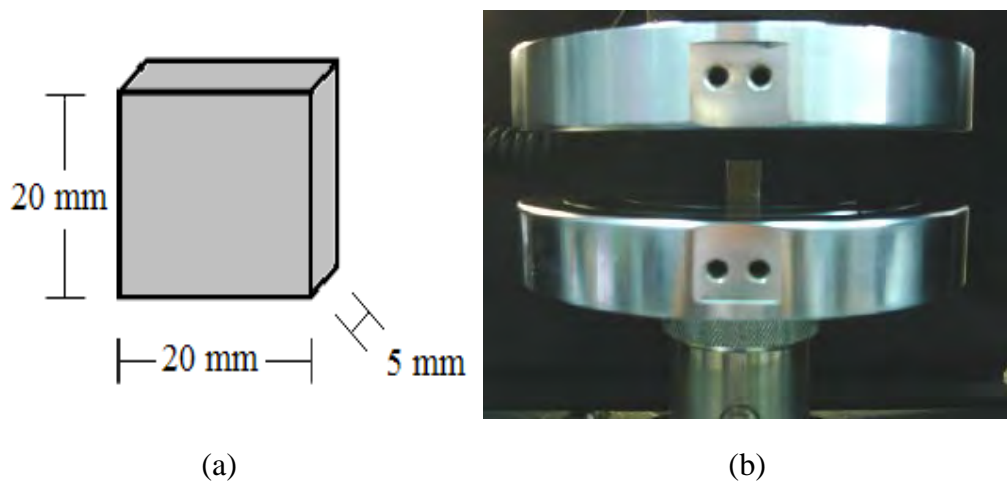


Figure 4.6: (a) Compressive test specimen and (b) The compressive test fixture.

4.2.1.4 Notched izod impact test

The impact energy was determined from the recycled WPC specimens having dimensions of 64 mm long, 12.7 mm wide and 5 mm in thickness, as shown in **Figure 4.7**. The tests were carried by using an Izod impact tester (Yasuda Impact Tester no. 258-PC) according to ASTM D256-06 [32] with a notch angle 45° at room temperature. Each recycled WPC sample was clamped in the sample holder. The dropped pendulum impacted the sample and then continued on and moved the gauge indicator. When high amount of energy was required to break the WPC samples, the pendulum did not continue very far past the sample impact point and the gauge reading was low. The energy absorbed from the impact was measured. The impact energy reported was the average of ten tested recycled WPC samples.

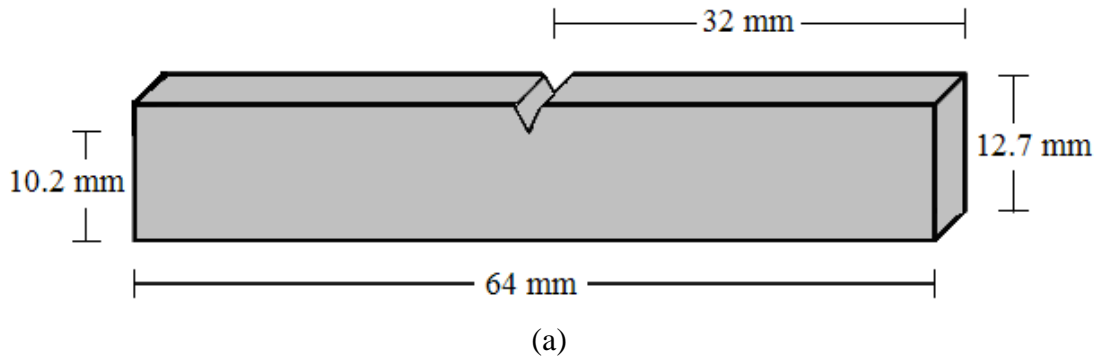
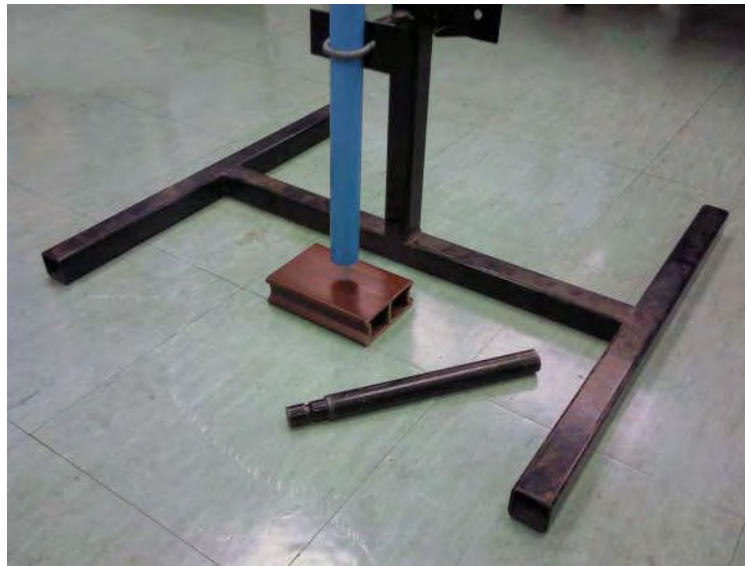


Figure 4.7: Notched Izod impact test (a) Notched Izod impact test specimen
(b) Izod impact tester

4.2.1.5 Drop-weight impact test

Impact energy can determine by the drop-weight impact testing machine, the recycled WPC specimens having dimensions of 15 cm long, 10 cm wide and 3 cm in thickness at room temperature. The shape of impactor is a cylinder of 12 mm diameter and 300 mm in long. The weight of the impactor was 1 kg. The specimen was fixed on supporting floor which has a cutout hole of 30 mm in diameter. The tests were carried by using an instrumented drop-weight impact testing

according to ASTM D 4226-00 as shown in **Figure 4.8**. The impact energy reported was the average of twenty tested recycled WPC samples.



4.8: Drop-weight Impact test

4.2.2 Thermal properties

The thermal transitions of most plastics and composites are relatively low compared to metal and ceramics. The thermal properties in terms of glass transition temperature (T_g) and degradation temperature (T_d) of the recycled PVC/WF composites were measured in the present study.

4.2.2.1 Glass transition temperature

Dynamic mechanical analysis (DMA) is applied to characterize the recycled PVC/WF composites. It is most useful for observing the viscoelastic nature of polymers. Two methods are currently used. One is the decay of free oscillations and the other is forced oscillation. Free oscillation techniques involve applying a force to a polymer and allowing it to oscillate after the force is removed. Forced oscillations involve the continued application of a force to the polymer sample. An oscillating force is applied to a sample and the resulting displacement of the sample is measured.

The test specimen is clamped between the movable and stationary fixtures, and then enclosed in the thermal chamber. Frequency, amplitude, and a temperature range appropriate for the material being tested are input. The elastic modulus (or storage modulus, E'), viscous modulus (or loss modulus, E'') and damping coefficient ($\tan \delta$) are determined as a function of temperature, frequency or time.

The dynamic mechanical analyzer (DMA, model DMA242) was used to investigate the dynamic mechanical properties of the wood-plastic composites. The dimensions of specimens are $10 \times 50 \times 2 \text{ mm}^3$. The test was performed in a bending mode with a constant frequency of 1 Hz. The temperature was scanned from 30°C to 130°C with a heating rate of $2^\circ\text{C}/\text{min}$ under nitrogen atmosphere. The storage modulus (E'), loss modulus (E''), and loss tangent or damping curve ($\tan \delta$) of the composites were then obtained.



Figure 4.8: Dynamic mechanical analysis

4.2.2.2 Degradation temperature

The thermal behavior of each recycled WPC in terms of the decomposition temperature (T_d) and the char yield of the recycled WPC were determined by using a Thermogravimetric analyzer (TGA, TA instruments, SDT Q600) as shown in **Figure 4.9**. About 10 mg of each formulation of the WPC powder was heated from 30°C to 900°C at a heating rate of $10^\circ\text{C}/\text{min}$ under nitrogen atmosphere. The gas flow rate of the purged nitrogen was 100 ml/min. The sample was progressively heated, the changes in the weight of the recycled WPC sample were recorded. The weight changes were associated with the volatilization or decomposition of components within the recycled WPC sample. The weight loss of

each recycled WPC and the freshfeed was measured as a function of temperature. The T_d ranges and the char of all recycled WPC in this work were recorded.



Figure 4.9: Thermogravimetric analysis

4.3.2.3 Rheology measurement

Torque rheometer is an important tool and is widely used in most laboratories working on polymer processing, which is used to predict processability performance before committing large amounts of time and materials [20]. In this work, a Brabender torque rheometer (Brabender, model 815606) was used to study the processability of the recycled WPC by determining the rheological behaviour during processing. In this work, a Brabender torque rheometer, with mixing chamber was used to measure the torque required for sample test weight of 40 g, the temperature was set to operate at 190 °C, and the screw speed was maintained at 50 rpm.

A torque rheometer was used to provide an information basis for developing quantitative indices of processability as a function of mixing time. The test conditions of the torque rheometry are more similar to the processing condition in extruders, where the structure of the melt changed with the influence of thermal stress and oxygen, inducing change of the apparent viscosity of the melt and the variation of driving force of the mixing rollers rotating at a constant speed [49]. The fundamental work in the area of torque rheometry has been based on using a mixer with roller-type mixing heads as a sensor, on the basic measuring instrument. The torque data can be

interpreted in terms of shear force and shear rate. The torque is related to the concept of shear stress and the speed of the rheometer is related to shear rate.



Figure 4.10: Brabender torque rheometer

4.2.3 Physical properties

Physical properties were characterized to detect any changes in the physical nature of the recycled WPC, they include density, water absorption behavior and average molecular weight.

4.2.3.1 Density

Density determination of the WPC and the recycled WPC was performed by applying Archimedes' principle, which stated that every solid body immersed in a fluid apparently lost its weight by an amount equal to that of the fluid it displaced. Each specimen of the WPC and the recycled WPC was firstly weighed in air, then it was weighed again after full immersion in water. **Equation (4.1)** was used to calculate the density of the WPC from the two measured weights.

$$\rho = \left[\frac{A}{(A - B)} \right] \times \rho_0 \quad (4.1)$$

where

ρ is the density of the solid (g/cm^3)

A is the weight of the solid in air (g)

B is the weight of the solid in the liquid (g)

ρ_0 is the density of the auxiliary liquid at the given temperature (g/cm^3)

4.2.3.2 Water absorption

The moisture and water absorption behavior of the WPC and the recycled WPC is very important, especially for those to be used outdoor. The tendency of a plastic material to absorb water is largely dependent on its chemical nature. Hydrocarbon polymer, such as PP, PE and PS, were highly resistant to water absorption. Polymers with high polar groups, especially those containing pendent oxygen, are susceptible to water absorption.

The water absorption behaviors of the PVC/WF WPC and the recycled WPC were investigated. The test was conducted according to conditions and procedure described in ASTM D-570. [33] Each WPC sample was weighed in air. Then it was immersed in water for two months and reweighed in air every 7 days. Only the average obtained from three weighted samples was reported. The amount of water absorption, expressed as percentage, was calculated according to **Equation (4.2)**.

$$\% \text{ Water Absorption} = \left[\frac{\text{Wet weight} - \text{Dry weight}}{\text{Dry weight}} \right] \times 100 \quad (4.2)$$

4.2.3.3 Average molecular weight

The molecular weight of the PVC matrix was measured by using Gel Permeation Chromatography (GPC) technique. A small quantity of the finely crushed WPC was dissolved in tetrahydrofuran (THF). The wood flour was separated from the PVC matrix by decantation. Smaller particles of wood flour were then

removed by filtration twice using filter paper (2 μ m) and PTFE membrane (0.45 μ m) respectively. PVC solution was prepared at a concentration of 0.1 % (w/v) by dissolving it in tetrahydrofuran (THF) and then filtered by using PTFE membrane at room temperature. Analysis of the molecular weight was performed at 30 °C on the GPC (Waters 150-CV) apparatus using three columns (Styragel® HT 0.5, 1 and 4) with refractive index detector (RID, Waters 2414). The calibration curve of the GPC column was obtained by using polystyrene standards calibration (MW 4,490-1,112,000). The sample solution of 100 μ l was injected in the chromatogram and the elution time of the specimen was obtained. The average-molecular weight and the molecular weight distribution of the PVC matrix in the WPC and the recycled WPC were determined by the difference of small and large molecules on special particles that line a tube through which the PVC solution was caused to flow on the substrate on which samples are placed. Molecules of PVC were separated by the differences in the absorptivity that was associated with molecular weight. After time, these differences could be seen and quantitatively measured.



Figure 4.11: Gel permeation chromatography (GPC)

4.2.4 Microscopic observation

A scanning electron microscope (SEM) was applied to characterize the PVC/Wood flour composites. The SEM micrographs were taken after the WPC and the recycled WPC had been cleavages cryogenically and coated with thin film of gold with 300 Å in thickness. The accelerating voltage for the SEM was at 15 kV. In the

current research, SEM (JEOL model JSM 6480LV) was used to characterize the WPC microscopic structure and the interfacial adhesion between the wood flour particles and the PVC matrix from the fractured topology of the PVC/ wood flour composites.



Figure 4.12: Scanning electron microscope (SEM).

CHAPTER V

RESULTS AND DISCUSSION

The influences of re-processing the WPC scrap, the mixture of the WPC scrap and the freshfeed as well as the effect of re-extrusion WPC up to seven cycles were investigated by studying their mechanical, thermal and physical properties as presented in this chapter.

5.1 Mechanical Properties

5.1.1 Mixture of WPC scrap and WPC freshfeed (Part I)

Figures 5.1 shows the changes in the flexural strength and the flexural modulus of the profiles produced from WPC freshfeed, WPC scrap and their mixture. The flexural strength and the flexural modulus of the profiles extruded from 20 and 30 wt% of scrap mixed with 80 and 70 wt% of WPC freshfeed were very close to those of the WPC extruded from freshfeed only. A significant drop, by 23% in the flexural strength and by 29% in the flexural modulus, was apparent when 40 wt% of scrap was mixed. Further increment of the WPC scrap to 60 and 80 wt% in the mixture evidently reduced the flexural strength and the flexural modulus to the level close to those extruded from the pure scrap.

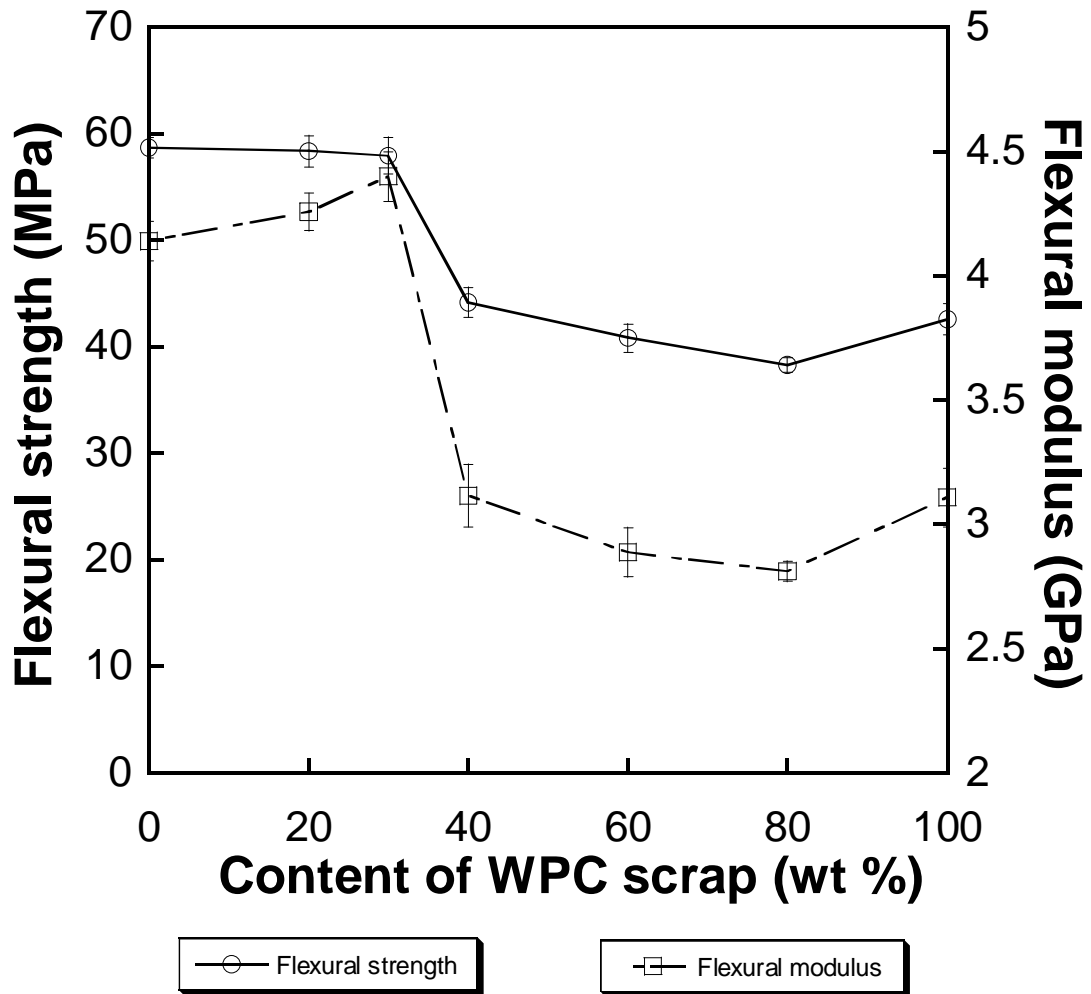


Figure 5.1: Flexural strength and flexural modulus of the WPC profiles extruded from freshfeed and those re-extruded from various mixtures of WPC scrap: freshfeed at 20:80, 30:70, 40:60, 60:40, 80:20 and 100:0.

Figures 5.2 demonstrate the changes in the tensile strength and the tensile modulus of the various mixture of WPC scrap and freshfeed. Profiles extruded from the mix ratio of the WPC scrap:WPC freshfeed at 30:70 showed an average tensile strength of 34.08 MPa and tensile modulus of 2.72 GPa. Noticable drop in both the tensile strength and the tensile modulus of the mixture of WPC scrap and freshfeed was observed when 40 wt% of scrap was mixed and reprocessed. A similar trend was evident in the compressive behavior of re-processed WPC. **Figures 5.3** demonstrate clearly that the compressive strength and the compressive modulus remained relatively unchanged when scrap was mixed by 20 and 30 wt%. The mixture of WPC scrap and its freshfeed at 30:70 showed an average compressive strength and a

compressive modulus of 59.5 MPa and 2.3 GPa respectively. However, the compressive strength and the compressive modulus were found to decrease when scrap was mixed at 40 wt%.

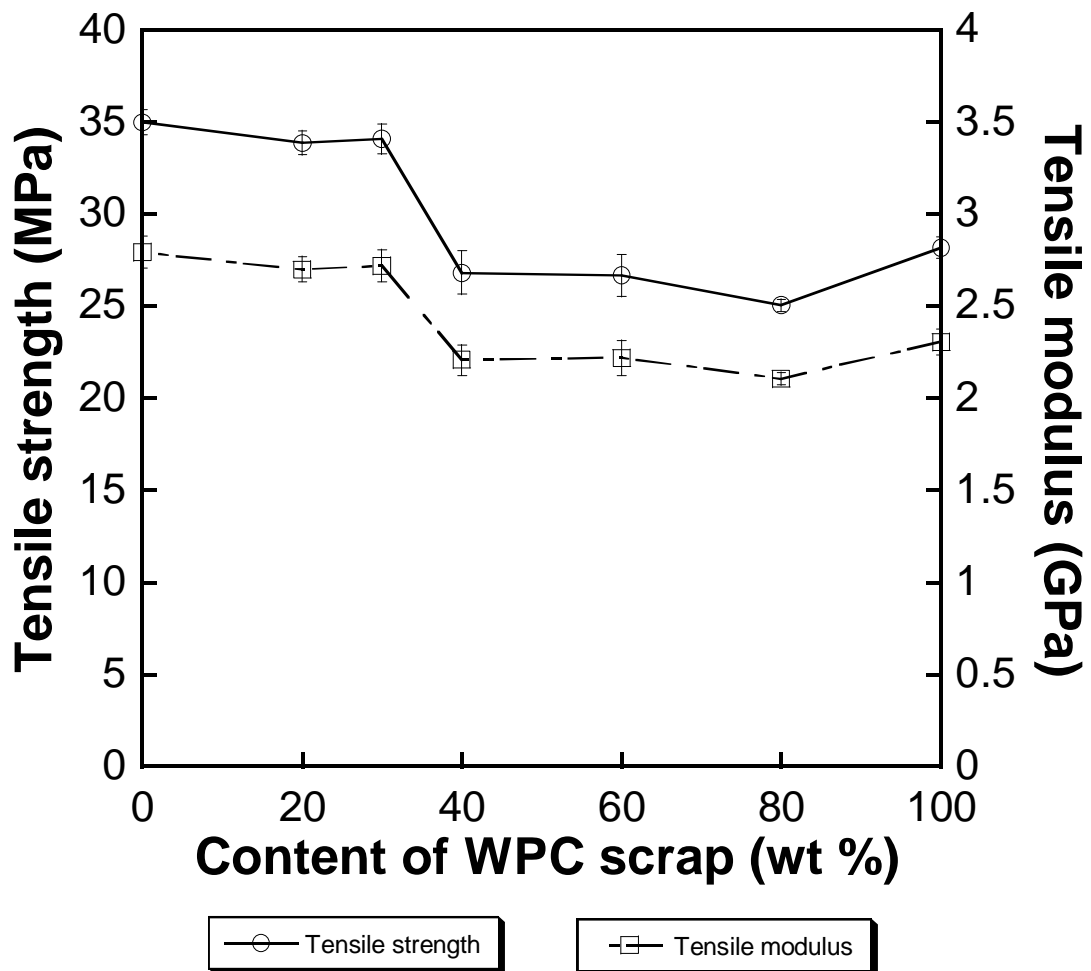


Figure 5.2: Tensile strength and tensile modulus of the WPC profiles extruded from freshfeed and those re-extruded from various mixtures of WPC scrap: freshfeed at 20:80, 30:70, 40:60, 60:40, 80:20 and 100:0.

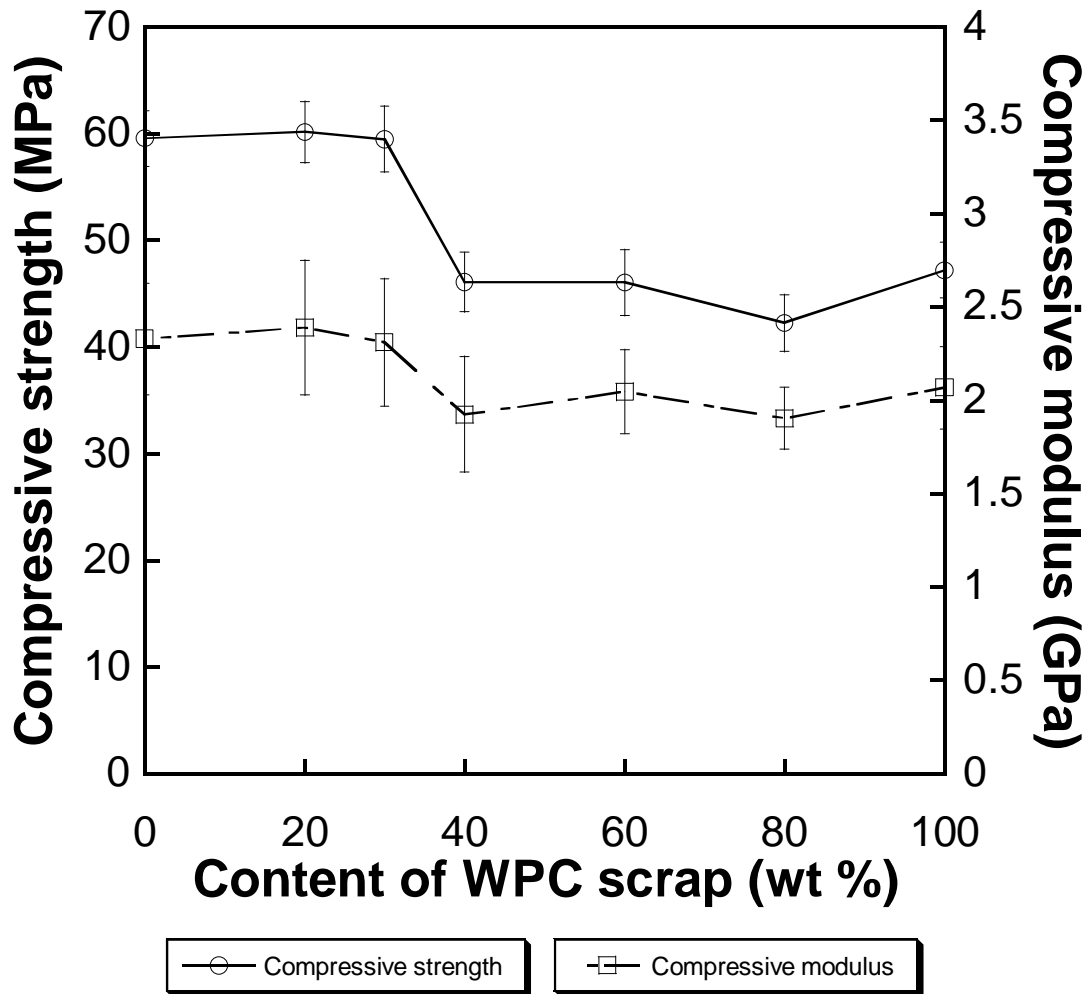


Figure 5.3: Compressive strength and compressive modulus of the WPC profiles extruded from freshfeed and those re-extruded from various mixtures of WPC scrap: freshfeed at 20:80, 30:70, 40:60, 60:40, 80:20 and 100:0.

The impact energy required to fracture all the WPCs prepared in Part 1 from freshfeed, scrap and their mixture is shown in **Figure 5.4**. The impact fracture energy required to fracture all the WPC profiles remained relatively constant and closed in value to that of the freshfeed.

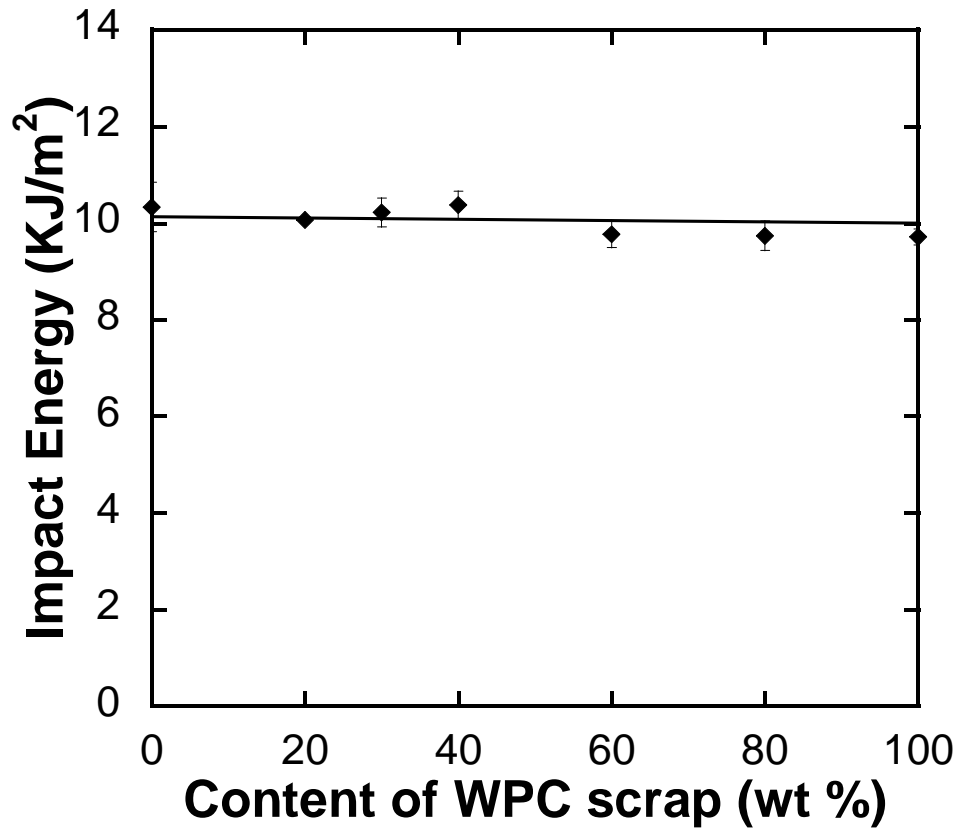


Figure 5.4: Impact energy of WPC profiles re-extruded from freshfeed, scrap, and their mixture upon the first pass of re-processing.

The results obtained from the flexural test, tensile test and compressive test on the various mixtures of WPC scrap and freshfeed indicated that the WPC scrap could be mixed with WPC freshfeed at 30 wt% without significantly altering the flexural, tensile, compressive and impact properties. They suggested that the 30:70 was the most appropriate scrap: freshfeed mix ratio in terms of mechanical properties and cost reduction for utilization of scrap in re-processing. Hence, this formulation ratio of WPC scrap:WPC freshfeed at 30:70 was selected to be further extruded repeatedly as WPC profiles in **Part Two**.

5.1.2 Effects of re-processing the mixture of WPC scrap and WPC freshfeed (Part II)

Figure 5.5 shows the changes in the flexural strength and the flexural modulus of the 30:70 mix formulation of WPC scrap:WPC freshfeed after having gone through each re-extrusion to a maximum of 7 passes. Both the flexural strength and the flexural modulus clearly decreased with the number of re-processing passes. A significant drop was observed, by 13% in the flexural strength and 23% in flexural modulus, at the second pass; beyond which the flexural strength and flexural modulus remained relatively constant until the sixth pass.

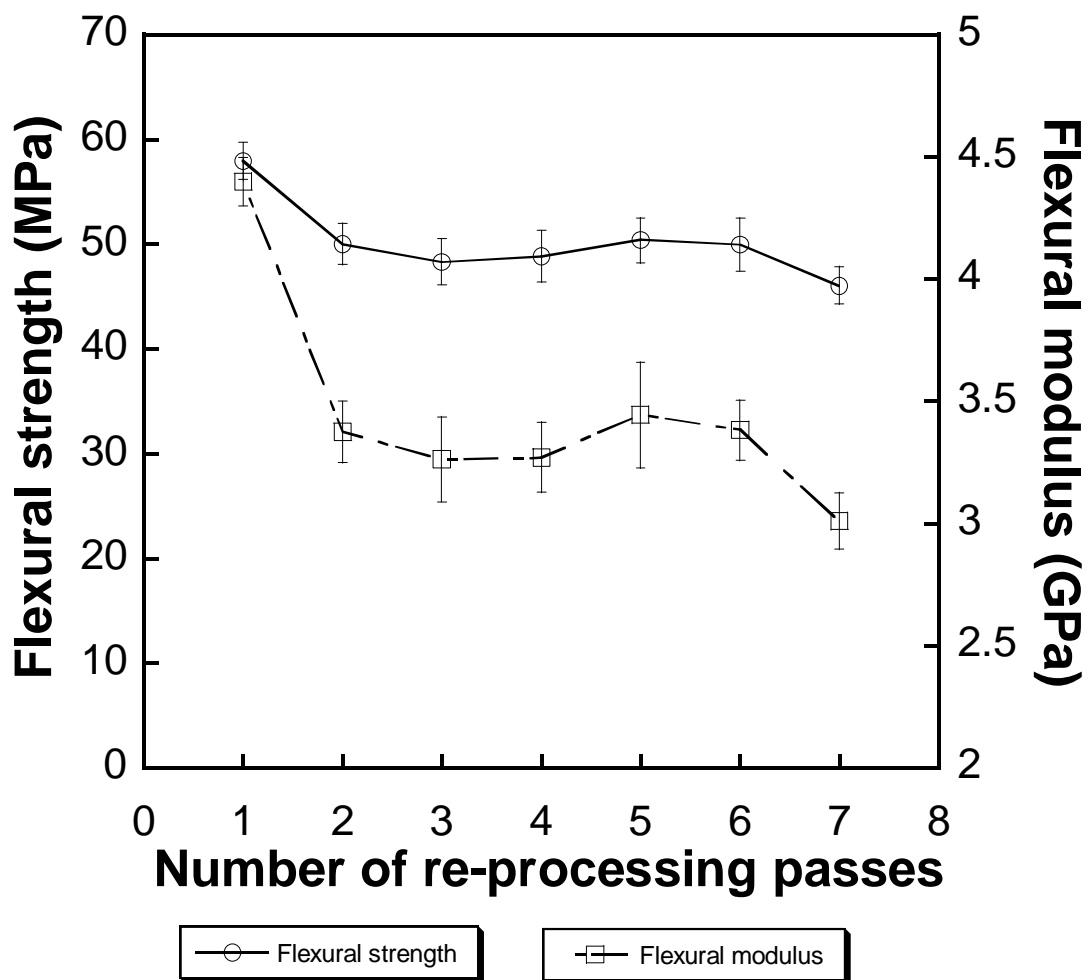


Figure 5.5: Flexural strength and flexural modulus of the WPC profiles produced from the mixture of WPC scrap:WPC freshfeed at 30:70 after each re-processing pass.

The changes in the tensile strength and the tensile modulus of the re-extruded WPC were found to be minute over the seven passes of re-extrusion, as is evident in **Figure 5.6**. The trend was such that the tensile strength and the tensile modulus of the 30:70 formulation WPC decreased with the increasing number of re-processing passes in a linear fashion. At the first pass, the WPC showed an average tensile strength of 34.08 MPa and a tensile modulus of 2.72 GPa, they were reduced to by a small fraction to 30.39 and 2.45 GPa, respectively after having been re-processed seven times.

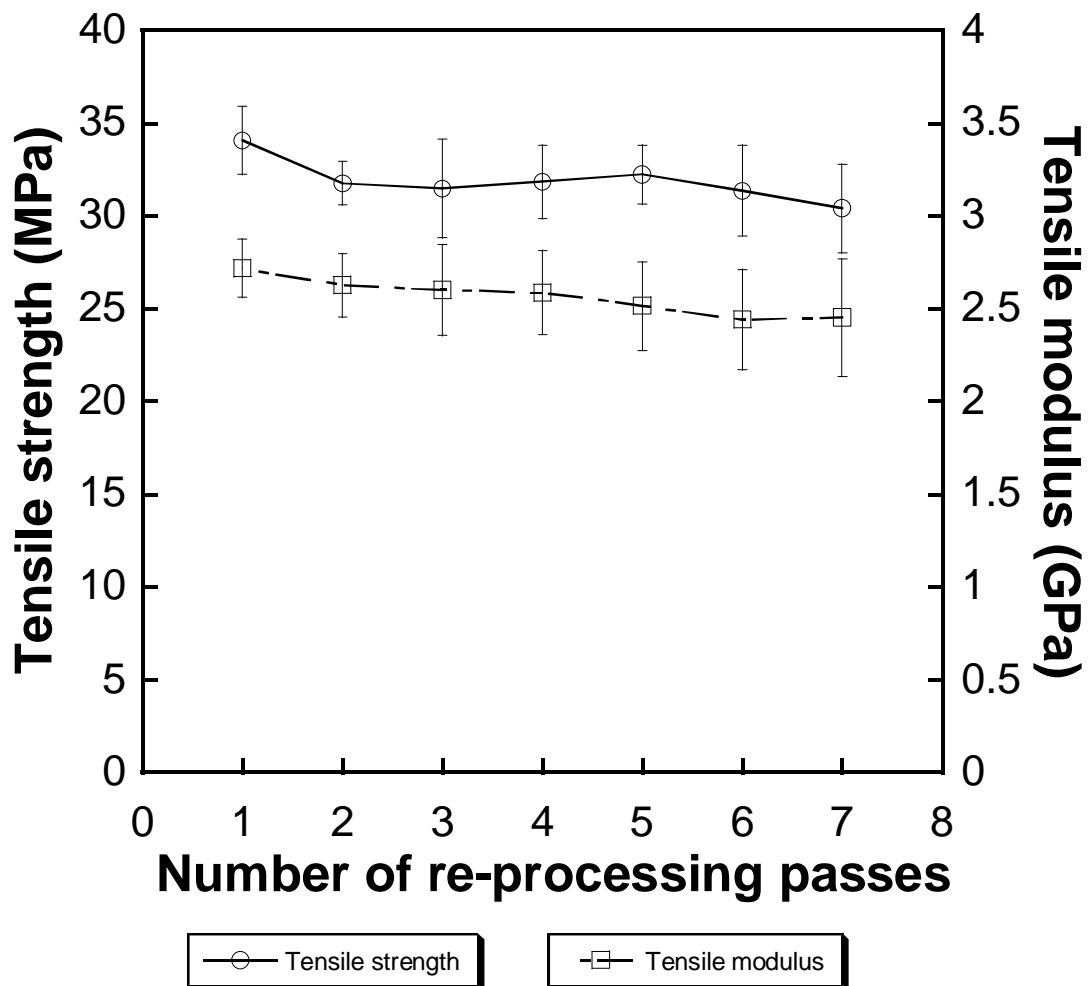


Figure 5.6: Tensile strength and tensile modulus of the WPC profiles produced from the mixture of WPC scrap:WPC freshfeed at 30:70 after each re-processing pass.

After having gone through re-extrusion for 7 passes, the WPCs with 30:70 formulation exhibited noticeable decrease in the compressive strength and the compressive modulus, with the number of recycling passes. This was depicted in **Figure 5.7**. The WPC at the first re-processing pass showed an average compressive strength and a modulus of 59.48 MPa and 2.31 GPa, they were reduced to 49.07 MPa and 2.02 GPa respectively after the second pass. Beyond the second re-extrusion pass, the compressive strength and the compressive modulus became relatively constant.

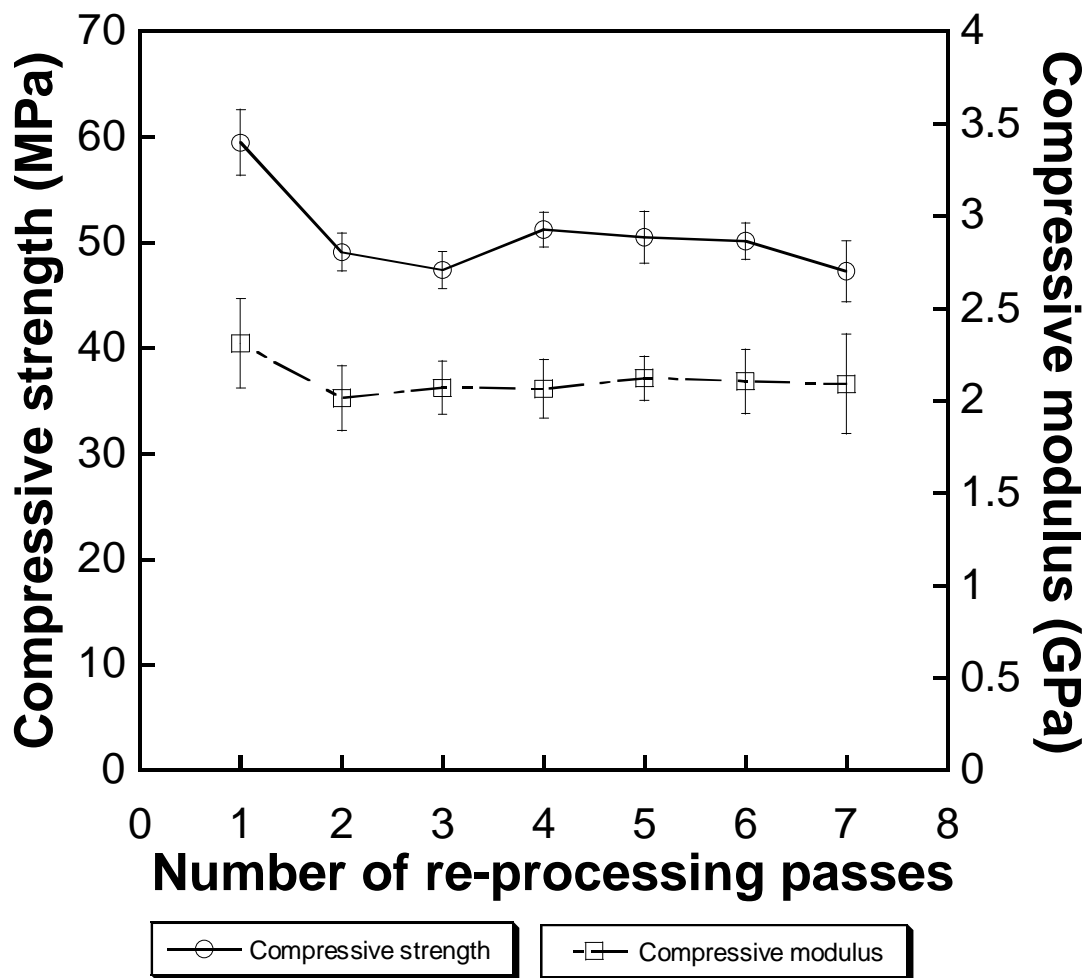


Figure 5.7: Compressive strength and compressive modulus of WPC profiles produced from the mixture of WPC scrap:WPC freshfeed at 30:70 after each re-processing pass.

The impact energy required to fracture the notch WPC re-extruded from the mixture of WPC scrap:WPC freshfeed at 30:70 was assessed by Izod impact

pendulum and plotted against the number of recycling passes in **Figure 5.8**. Despite the several passes of re-extrusion, the fracture energy of all the re-processed profiles was found to remain relatively constant. This was confirmed by a set of results on the impact energy of the same WPCs assessed by the weight dropping apparatus as shown in **Figure 5.9**. The mild deterioration of the mechanical properties and the impact energy upon each recycling pass suggested that close-loop recycling of the 30:70 of WPC scrap:WPC freshfeed formulation was highly probable for several re-processing passes.

Nevertheless, a comparison of the mechanical properties of the WPC profiles in Part I and Part II revealed that the WPC re-extruded only once from different weight ratio of the WPC scrap and its freshfeed possessed better properties than those undergone numerous re-processing passes. This was because the addition of WPC freshfeed contributed fresh wood fiber and longer chain length of PVC molecules in the matrix, both of which probably helped maintaining the mechanical properties of the WPC extruded from each mixture [49].

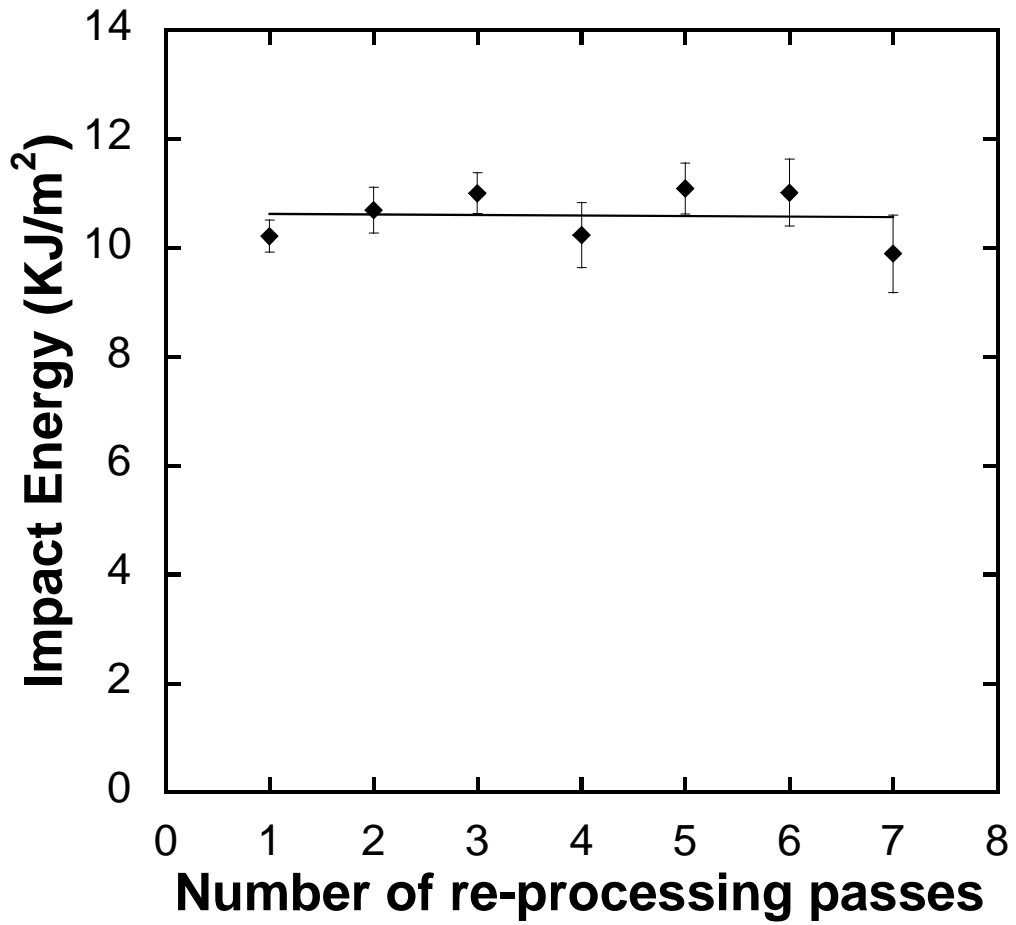


Figure 5.8: Impact energy obtained by notched izod impact test of the WPC profiles produced from the mixture of WPC scrap:WPC freshfeed at 30:70 plotted against each re-processing pass.

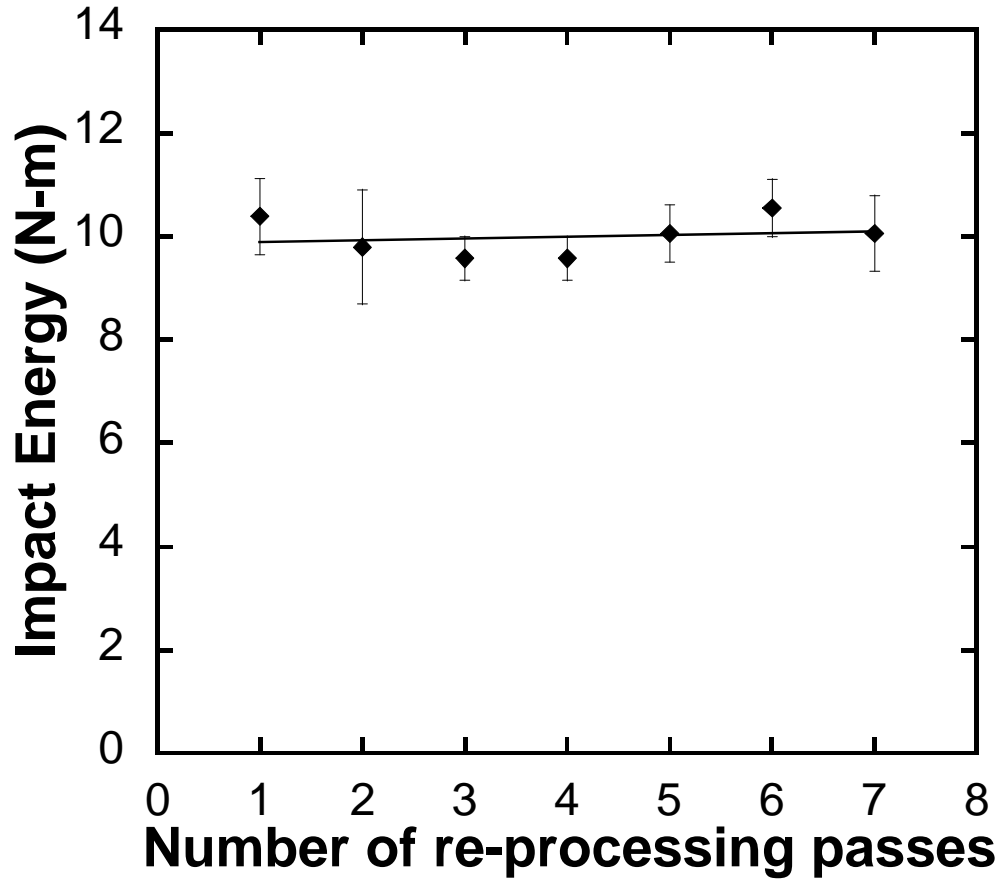


Figure 5.9: Impact energy obtained by the drop weight test of the WPC profiles produced from the mixture of WPC scrap:WPC freshfeed at 30:70 plot against each re-processing pass

5.2 Thermal Properties

5.2.1 Glass transition temperature (T_g)

Glass transition temperature (T_g) is one of the thermal properties indicating the temperature range for application of the WPC. The higher glass transition temperature means that the WPC can be used at higher temperature without being rubber-like. In this research, the T_g 's of WPC obtained from the loss tangent ($\tan \delta$) of dynamic mechanical analysis (DMA) are listed in **Table 5.1** and **Table 5.2**.

Table 5.1 compares the T_g 's of the WPC extruded from the various mixture of WPC scrap and freshfeed in Part I. It can be observed that the T_g 's of all the WPC were higher than that of the PVC compound at 91 °C. There is no significant difference between the T_g 's of the WPC extruded from the various mixtures of WPC scrap and freshfeed. Similarly, the T_g 's of the WPC of 30:70 formulation after having gone through re-extrusion for 7 passes, as shown in **Table 5.2**, were found to remain constant lying within the same narrow range from 97 to 98 °C as those re-extruded only once from the various mix of WPC scrap and freshfeed shown in **Table 5.1**

Table 5.1: Glass transition temperature of WPC of the various mixture of WPC scrap and freshfeed.

Sample (Scrap:Freshfeed)	Glass transition temperature (°C)
Freshfeed WPC	97
20:80	98
30:70	97
40:60	98
60:40	98
80:20	97
100% scrap WPC	97

Table 5.2 Glass transition temperature of WPC after re-processing for 7 passes

Sample	Glass transition temperature (°C)
30:70 after 1 passes	97
30:70 after 2 passes	98
30:70 after 3 passes	97
30:70 after 4 passes	97
30:70 after 5 passes	98
30:70 after 6 passes	97
30:70 after 7 passes	97

5.2.2 Degradation temperature

The TGA curve of neat PVC compound exhibited two ranges of decomposition. The first range at 260-320 °C associated with the decomposition of the PVC, releasing HCl at about 55 % of the weight loss. The second decomposition range at 360-500 °C corresponded to the thermal deterioration of the several additives and organics within the PVC, leaving a residue char weight of 17 % [50]. The TGA results for the WPC extruded with various mixtures of WPC scrap and freshfeed are shown in **Figure 5.10**, and those for WPCs of the 30:70 formulations after having gone through re-extrusion for 7 passes are shown in **Figure 5.11**.

In all the WPC, the first decomposition range at 260-320 °C was related with the decomposition of the PVC matrix in the WPC. The second degradation step below 310 °C could be attributed to the decomposition of individual within wood such as, hemicelluloses, lignin and extractives, while the third degradation step between 420 and 500 °C is due to the decomposition of cellulosic materials in the wood and 360-500 °C PVC compound were still prominent.

Figure 5.10 and **5.11** show that the char weight of the WPC remained relatively constant. The char weight of the WPC from freshfeed, WPC extruded from 30:70 formulation and WPC scrap were 22.1%, 23.4% and 23.7%, respectively.

The char weight of the WPC with 30:70 formulations after re-processing 1, 3, 5, 7 passes were 23.4 %, 22.4%, 21.2% and 23.6% respectively.

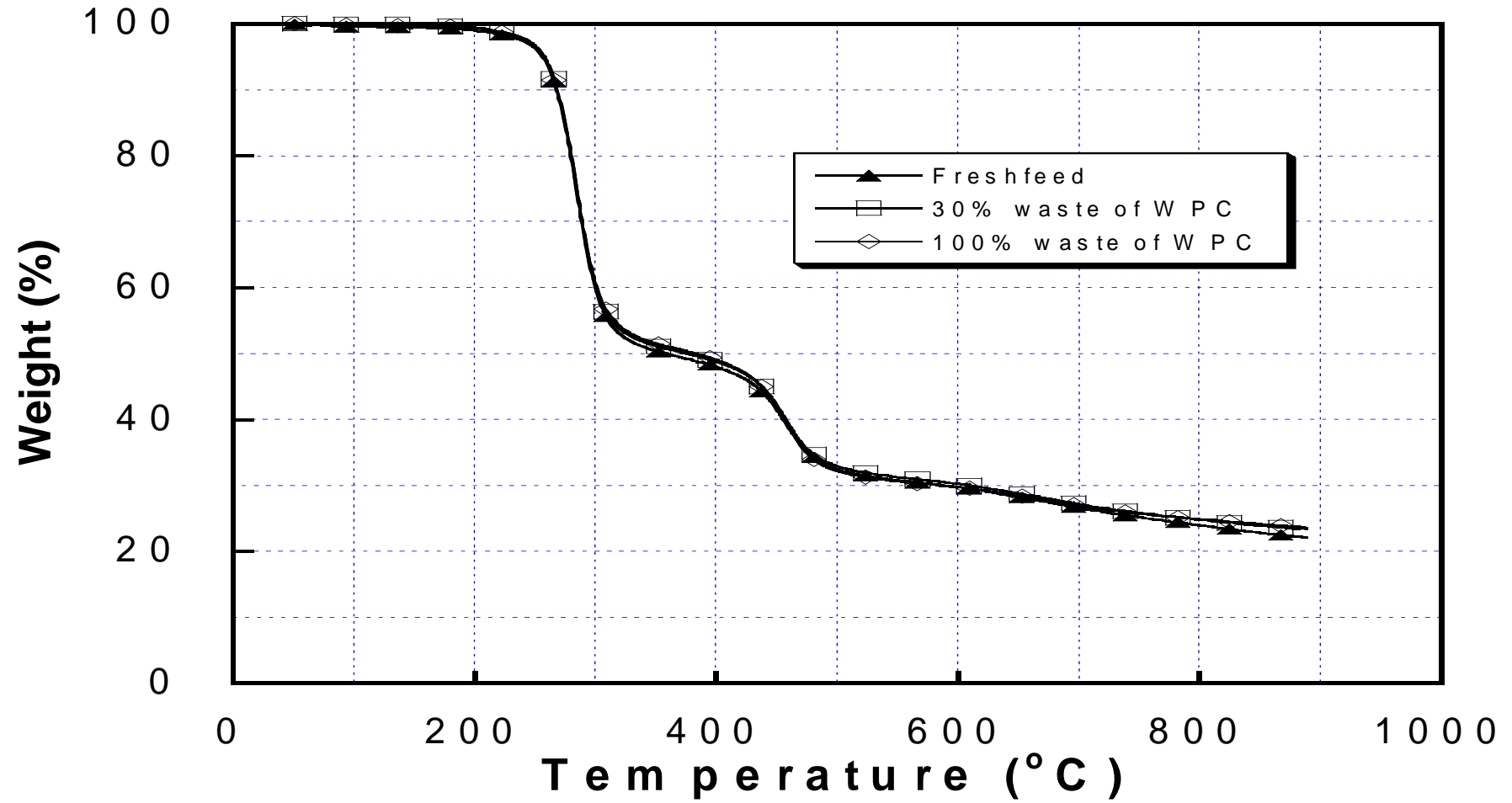


Figure 5.10: TGA thermograms of the WPC freshfeed, from 30:70 mixture WPC and WPC scrap.

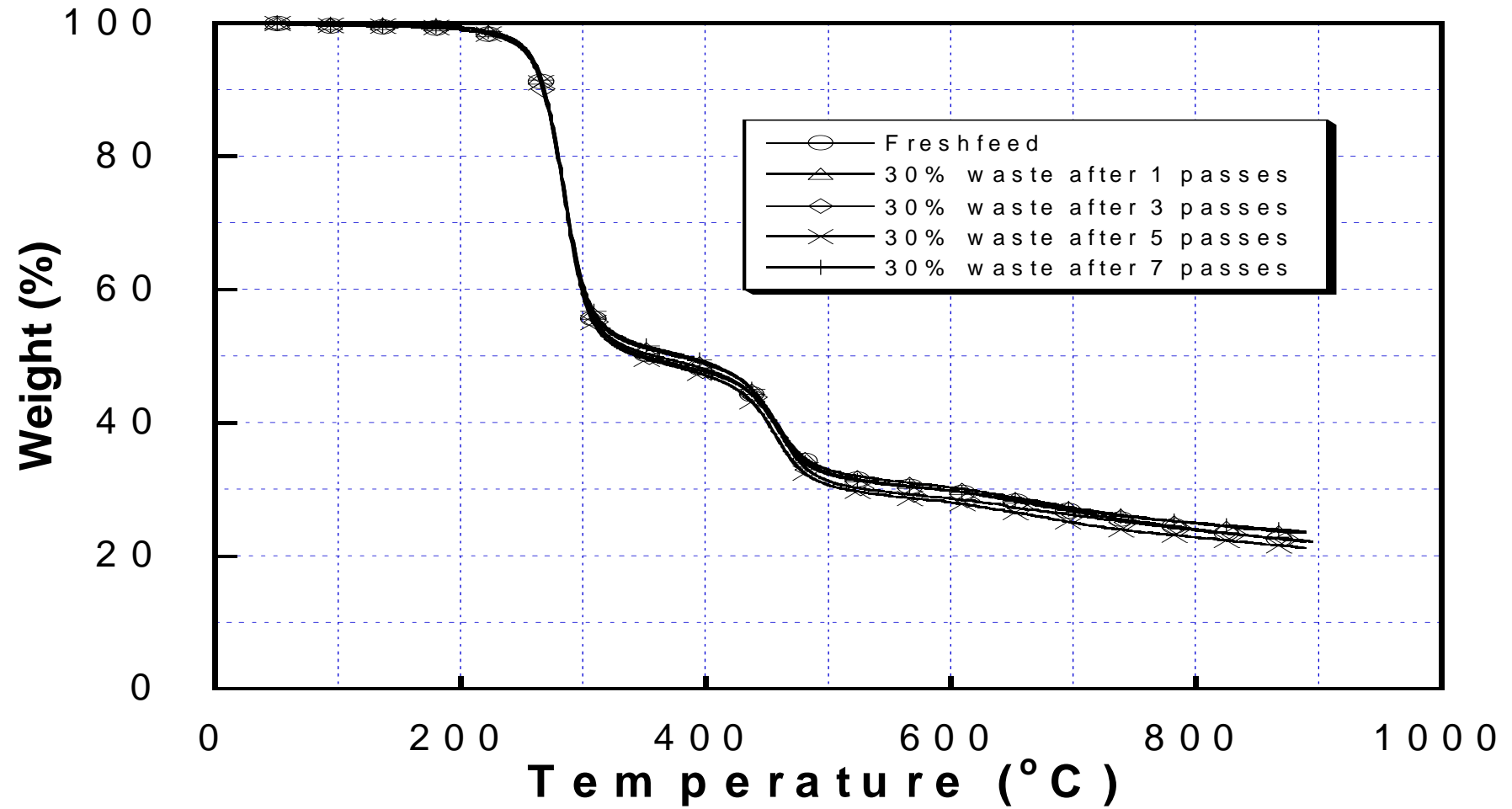


Figure 5.11: TGA thermograms of the WPC scrap:WPC freshfeed at 30:70 after each re-processing pass.

5.2.3 Rheological properties

Table 5.3: Rheological properties of WPC freshfeed, WPC scrap and the mixture of scrap: freshfeed at 30:70.

Type of WPC profiles	Fusion			End torque [Nm]
	Time [min]	Torque [Nm]	Temperature [°C]	
Freshfeed WPC (0:100)	1.20	30.50	182.60	21.00
30% Waste WPC (30:70)	1.20	29.00	182.40	22.30
100% Waste WPC (100:0)	1.15	29.40	183.30	21.90

Based on the fusion characteristics of recycled WPC, two characteristics were studied namely the fusion time and the torque at the equilibrium stage (end torque). The end torque was taken after 9 minutes when the torque had stabilized. From **Table 5.3**, the mixture of WPC scrap and WPC freshfeed showed no significant change in the fusion time, maximum torque of fusion and end torque. This suggested that one pass of extrusion of the recycled WPC on the industrial-scaled twin screw extruder did not effect on properties of the WPC scrap and the mixture of WPC scrap:WPC freshfeed at 30:70.

Table 5.4: Rheological properties of WPC freshfeed and mixtures of scrap: freshfeed at 30:70 after 1, 3, 5 and 7 passes of extrusion.

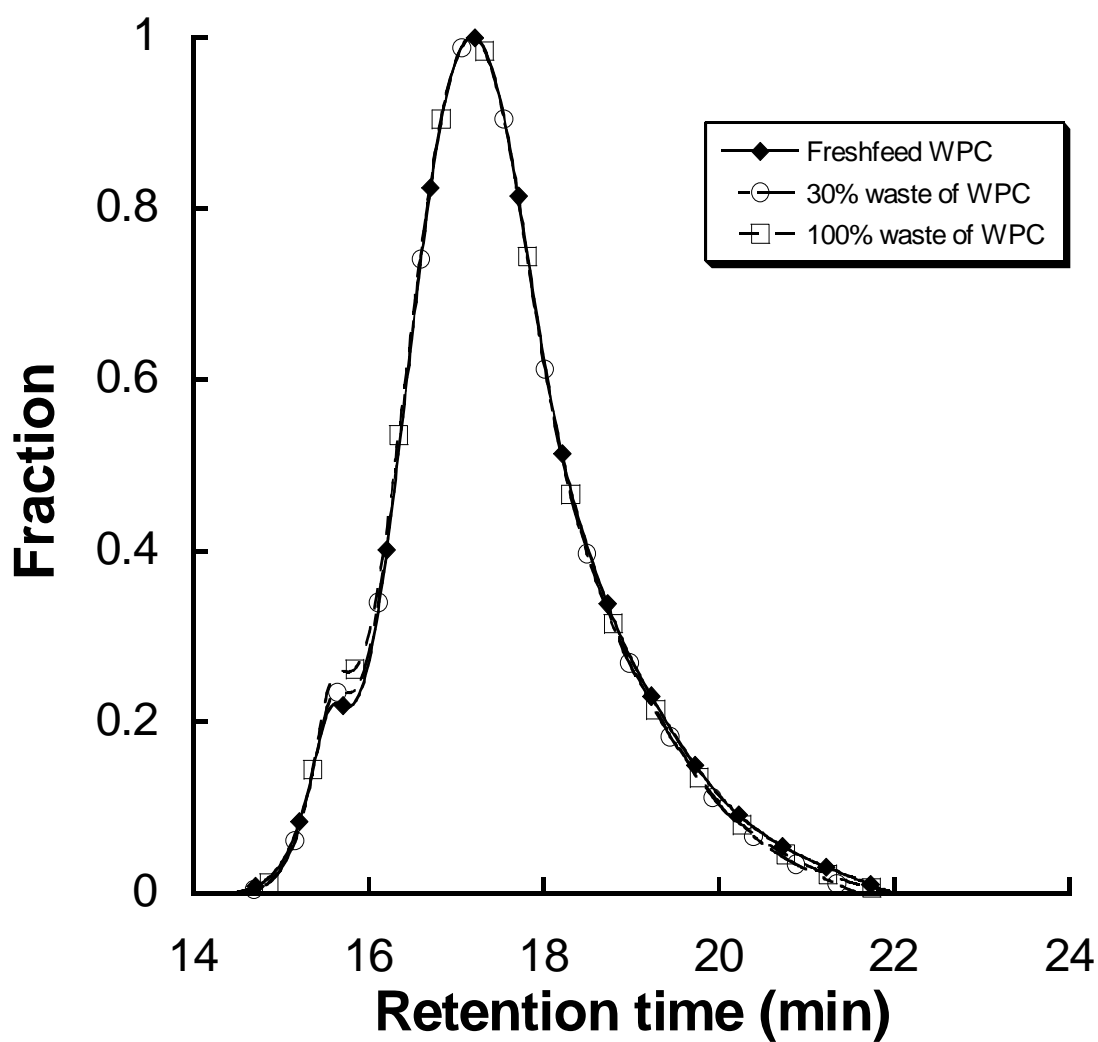
Number of re-processing passes	Fusion			End torque [Nm]
	Time [min]	Torque [Nm]	Temperature [°C]	
Freshfeed WPC	1.20	30.50	183.20	21.00
30:70 after 1 pass	1.20	29.00	182.40	22.30
30:70 after 3 pass	1.00	29.20	182.90	24.00
30:70 after 5 pass	1.20	29.00	182.00	24.20
30:70 after 7 pass	1.00	27.00	182.20	24.90

Table 5.4 demonstrates the rheological properties of the recycled WPC mixture of WPC scrap:WPC freshfeed at 30:70 after one, three, five and seven passes of recycling. The maximum torque of fusion decreased with the increasing re-processing while the end torque. The fact that the maximum torque of fusion was reduced when the WPC underwent further re-extrusion passes implied that chain scission of the PVC molecules must have taken place.

5.3 Physical Properties

5.3.1 Average molecular weight

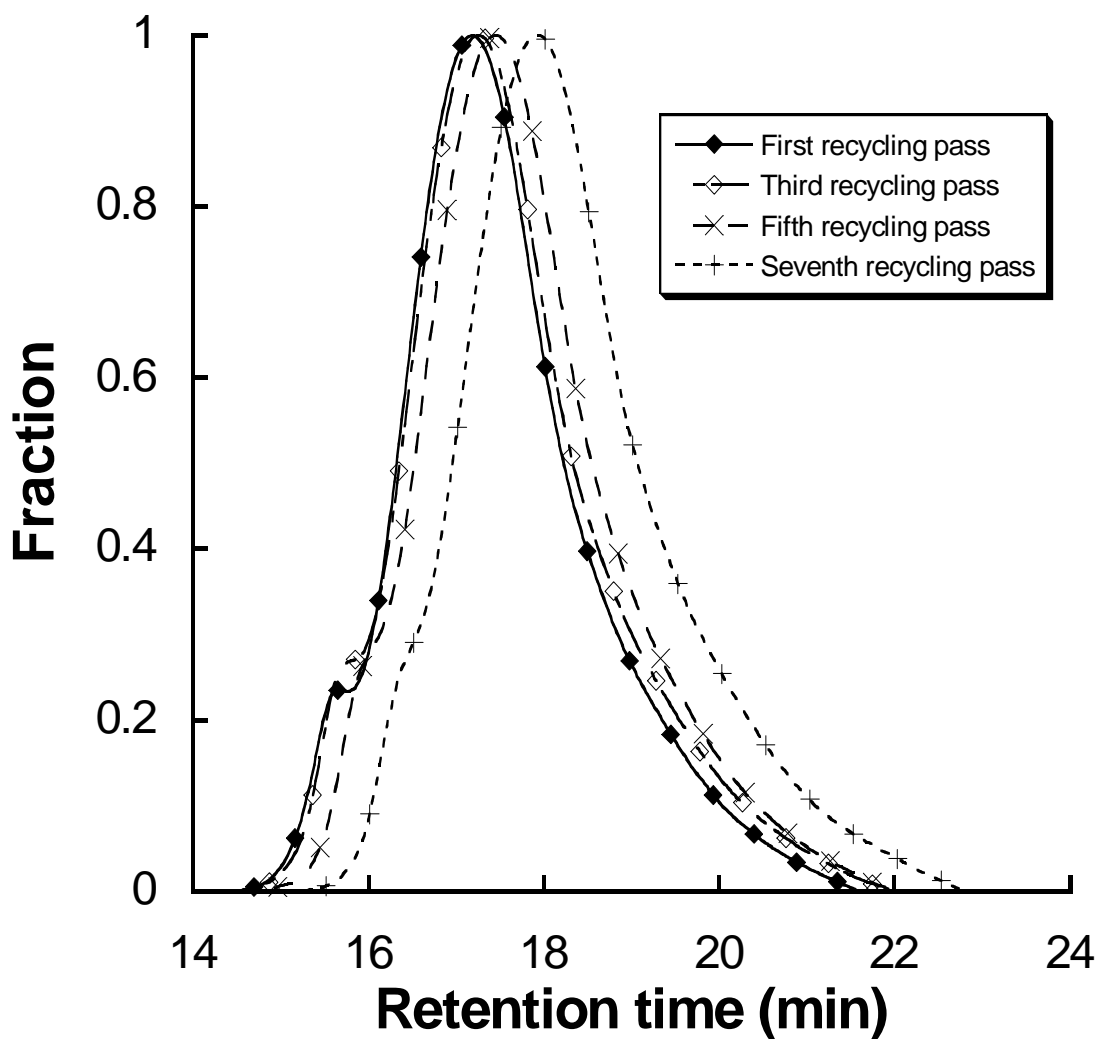
Figure 5.12 shows the molecular weight distribution of the WPC freshfeed which was almost identical with that of the scrap. Both the number-averaged molecular weight (\overline{M}_n) and the weight-averaged molecular weight (\overline{M}_w) analysed by gel permeation chromatography (GPC) showed no significant changes in the PVC chain length of the WPC freshfeed and the WPC scrap. The chart suggested that one pass of extrusion of the WPC profiles on the industrial-scaled twin screw extruder in the present study did not induce deterioration in the form of molecular chain scission to the PVC molecules.



Type of WPC profiles	Type of Molecular weight		PDI
	\bar{M}_n	\bar{M}_w	
Freshfeed WPC (0:100)	105,979	151,861	1.4329
30% Waste WPC (30:70)	111,150	154,190	1.3881
100% Waste WPC (100:0)	109,455	154,524	1.4118

Figure 5.12: Size exclusion chromatography elution charts of the dissolved PVC in THF for WPC freshfeed, WPC with 30:70 formulation of mix and WPC scrap.

The relative average molecular weights (\overline{M}_n and \overline{M}_w) and the molecular weight distributions (MWDs) of the WPC at 30:70 formulation after 1,3,5 and 7 re-processing passes were obtained by GPC and shown in **Figure. 5.13**. It can be seen that both the \overline{M}_n and the \overline{M}_w decreased with increasing re-processing passes. A gradual increase in the polydispersity index (PDI) was essentially due to the broadening of the GPC curve toward longer retention time (corresponding to lower molecular weights), as shown in **Figure. 5.13**. Moreover, a shoulder observed at the far side of the main distribution of the first and the third recycling pass gradually diminished and shifted to longer retention time. The disappearance of such shoulder implied that chain scission of the long PVC molecules must have taken place as a consequence of the shear stress introduced repeatedly on the PVC melt during re-processing. Both the \overline{M}_n and the \overline{M}_w were also found to decrease with progressive recycling. Their reduction was gradual and small over the initial recycling. After five passes of re-extrusion, the \overline{M}_n lowered by 14% while the \overline{M}_w did by only 11%. The relatively unchanged mechanical properties coupled with the minute drop in the average molecular weights verified that the mixture of WPC scrap to freshfeed at the ratio of 30:70 could be recycled close-looped type repeatedly up to five passes without critically affecting the performance of the recycled WPC profiles. The significant drop of the \overline{M}_n and the \overline{M}_w by 35% and 30% respectively upon recycling seven times induced the noticeable decline of the mechanical properties observe in Section 5.1.2.



Number of re-processing passes	Type of Molecular weight		PDI
	\bar{M}_n	\bar{M}_w	
30:70 after 1 passes	111,150	154,290	1.3981
30:70 after 3 passes	102,698	148,291	1.4439
30:70 after 5 passes	95,634	136,157	1.4237
30:70 after 7 passes	72,188	107,353	1.4871

Figure 5.13: Size exclusion chromatography elution charts of the dissolved PVC in THF for mixture of WPC scrap:WPC freshfeed at 30:70 after re-extrusion for one, three, five and seven passes.

5.3.2 Density

Figure 5.14 illustrates the density of the mixture of WPC scrap and its freshfeed. The density of all the WPC remained relatively constant and closed in value to that produced from the freshfeed. However, the density of the WPC composites tended to increase only minutely in a linear fashion with number of re-processing passes shown in **Figure 5.15**.

The density of the WPC extruded from the various mixtures of WPC scrap and its freshfeed and the WPC extruded from scrap: fresh at 30:70 formulation re-processed repeatedly within a very narrow range of 1.444 - 1.462 g/cm³. They were higher than those of natural wood which generally range from between 0.31 to 0.88 g/cm³ [51, 52].

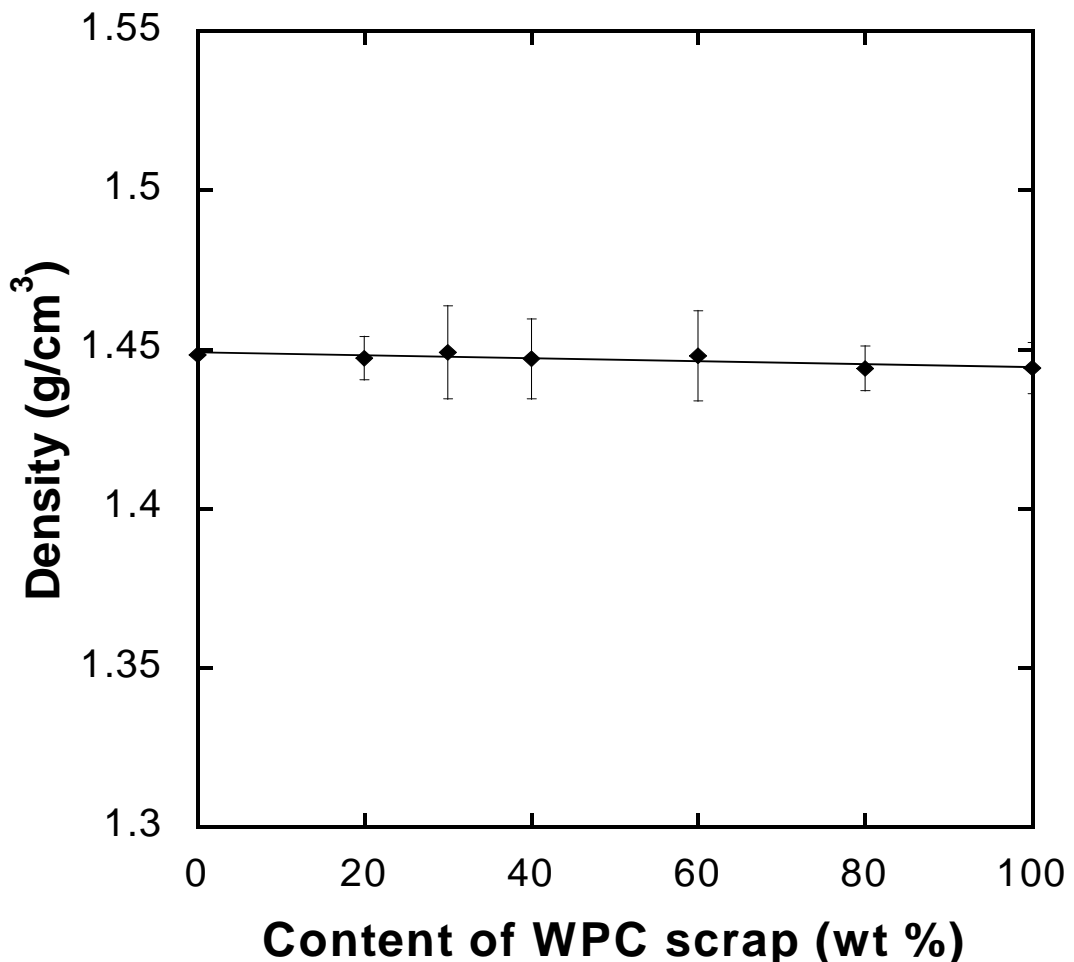


Figure 5.14: Density of WPC profiles extruded from freshfeed, scrap and their mixture after the first pass of re-processing.

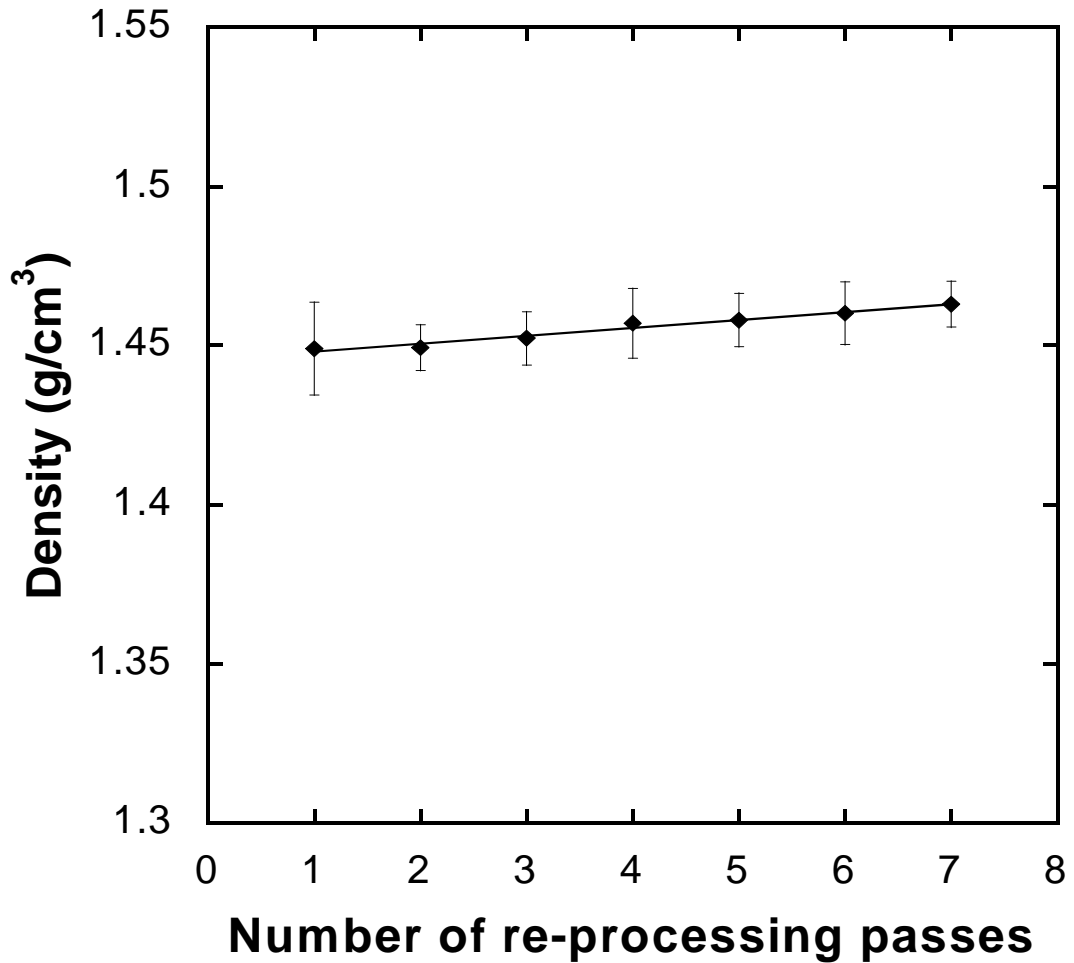


Figure 5.15: Density of WPC profiles produced from mixture of WPC scrap:WPC freshfeed at 30:70 after each re-processing pass.

5.3.3 Water absorption

Water had small effect on WPC composites despite prolonged immersion. It was absorbed by the hydrophilic wood fiber phase only since PVC was hydrophobic. **Figure 5.16** shows the water absorption behavior of the WPC prepared from freshfeed, scrap and their mixture. The plot indicated that the rate of water absorption was most rapid for all the WPC at the beginning to the 7th day. After that, the rate of water absorption decreased gradually. In the case re-processing of the 30:70 mixture of WPC scrap and its freshfeed, the amount of water absorbed was slight more in the WPC extruded from freshfeed than that from 100% WPC scrap.

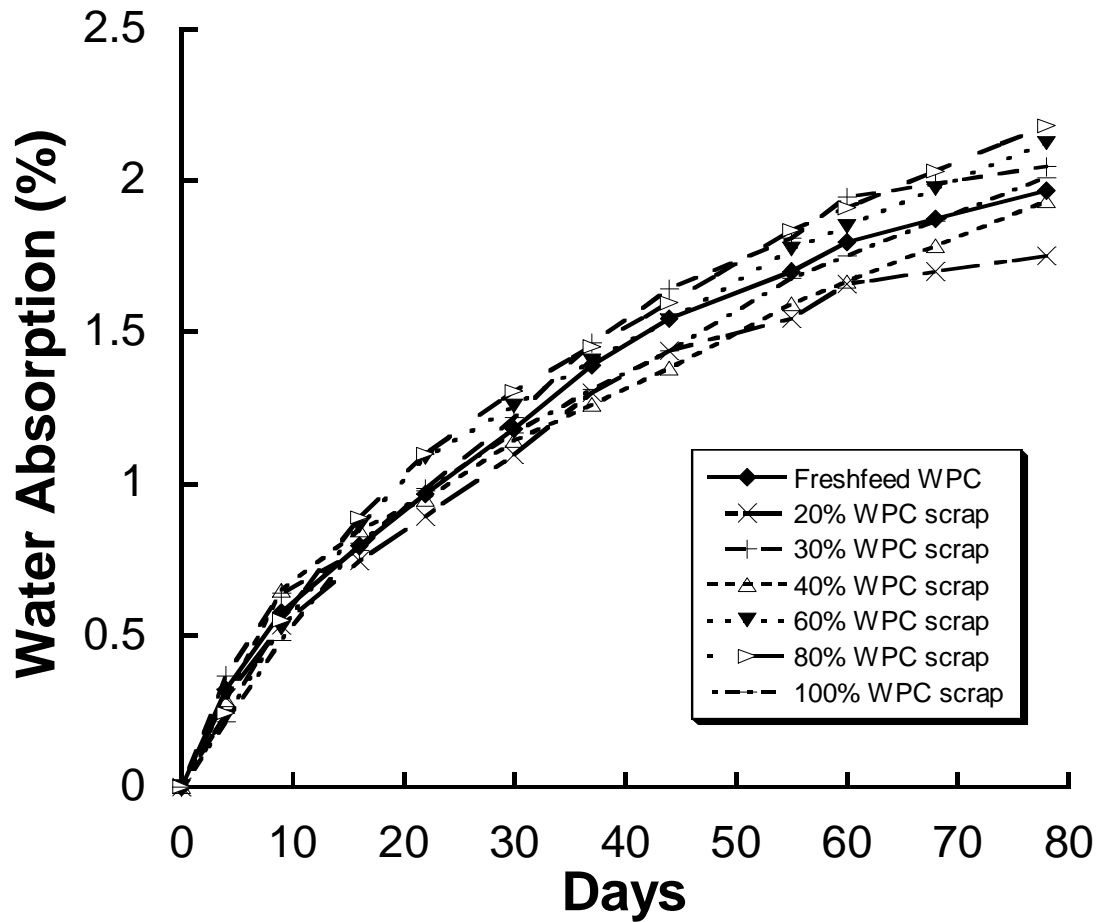


Figure 5.16: Water Absorption of WPC profiles re-extruded from freshfeed and their mixture after the first pass of reprocessing.

Figure 5.17 demonstrates the influences of the number of reprocessing passes on water absorption of the WPC composites for 70 days. The WPC composites had the high rate of water absorption at the start for about seven days. Then the rate of water absorption of the WPC reduced gradually. The WPC with seven recycling passes absorbed slightly greater amount of water than the ones with lesser passes.

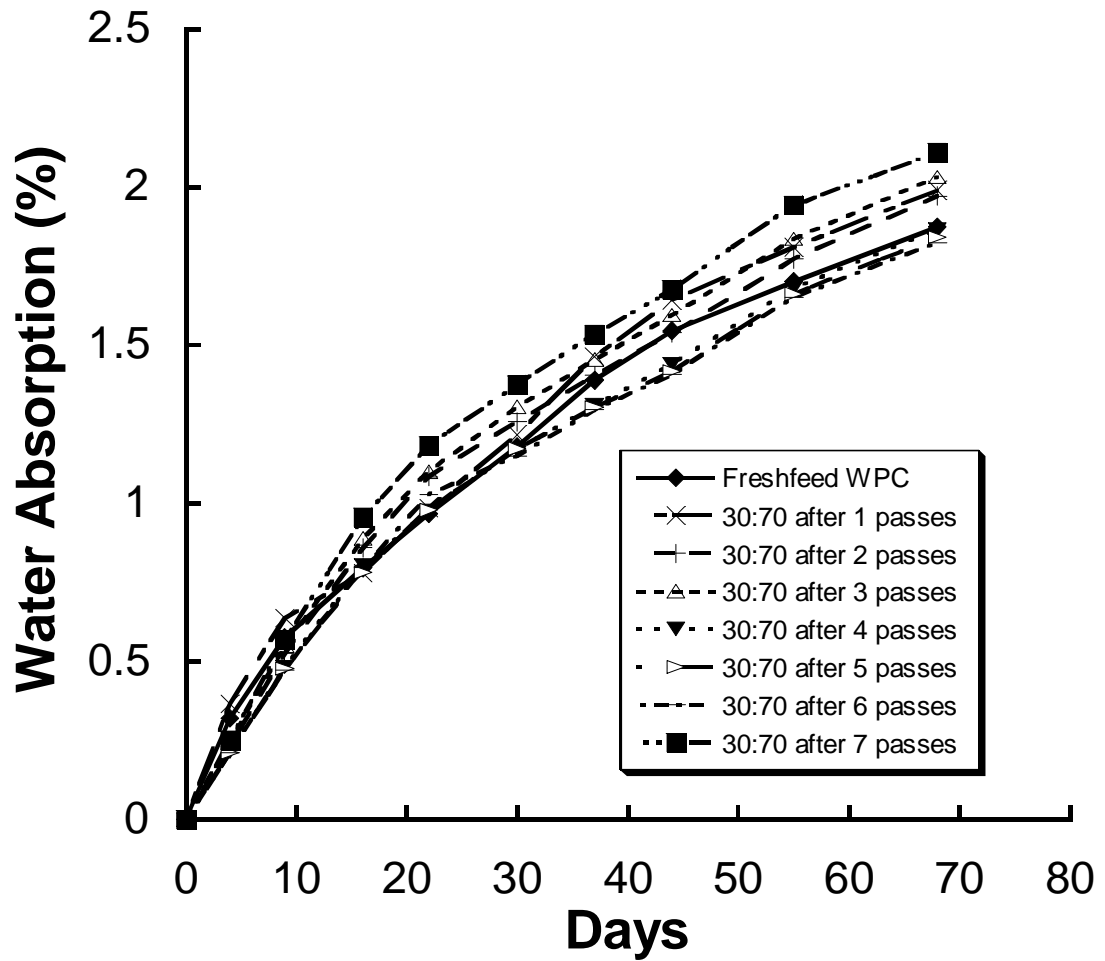


Figure 5.17: Water Absorption of WPC profiles produced from mixture of WPC scrap:WPC freshfeed at 30:70 after each re-processing pass.

5.3.4 Appearance

In terms of appearance, the recycled WPC extruded from various mixtures of WPC scrap and freshfeed are shown in **Figure 5.18**, and those for WPCs of the 30:70 formulations after having gone through re-extrusion for 7 passes are shown in **Figure 5.19**. In all the recycled WPC appeared lighter shade of brown induced by the pigment mixed initially with the freshfeed and have the appearance of wood.

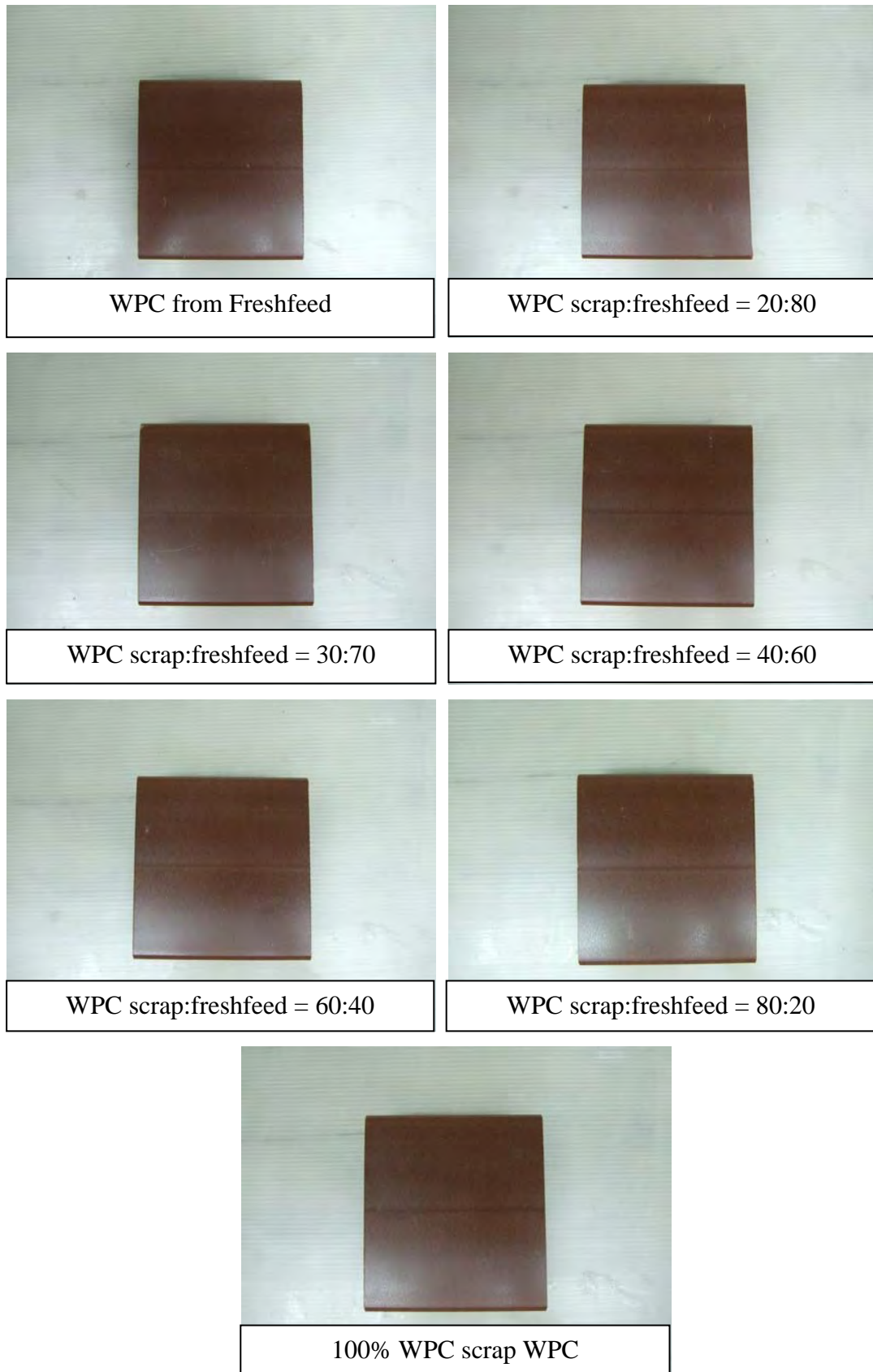


Figure 5.18: The appearance of the recycled WPC re-extruded from freshfeed, scrap, and their mixture upon the first pass of re-processing.

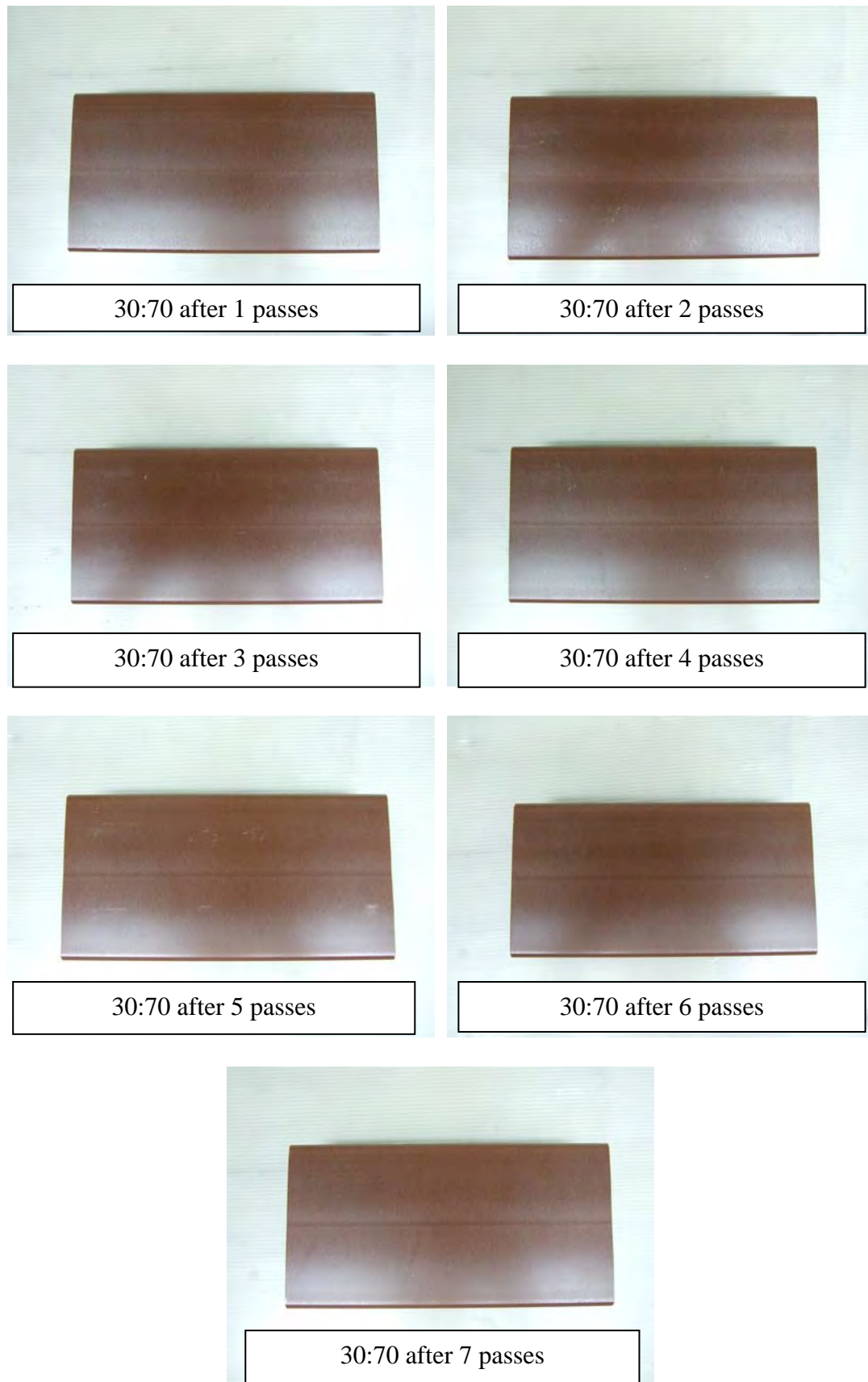
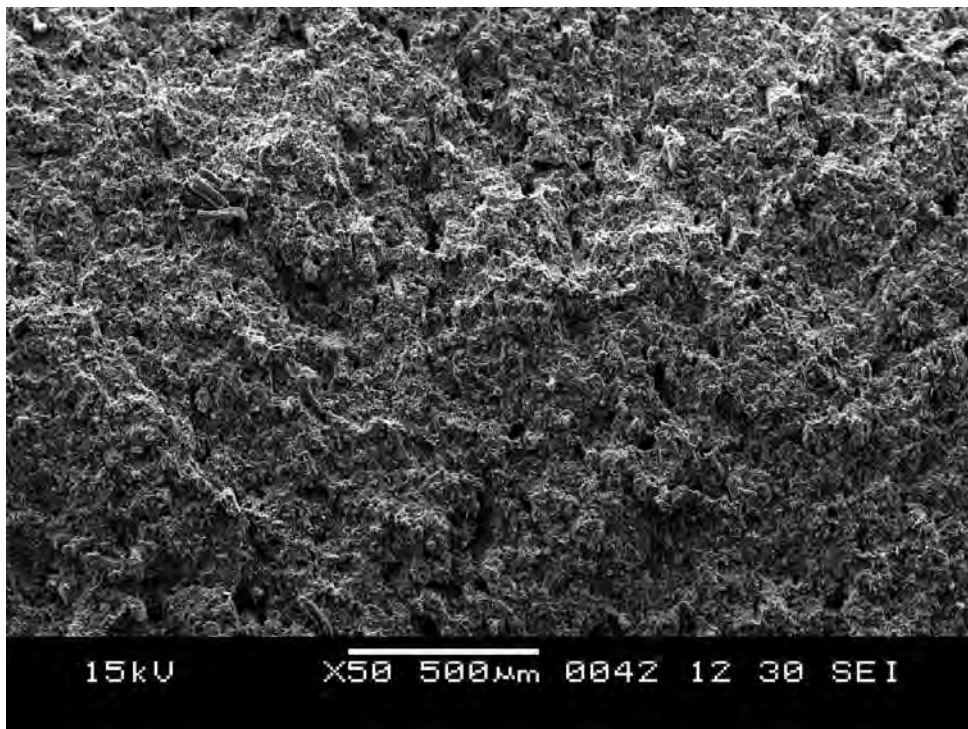


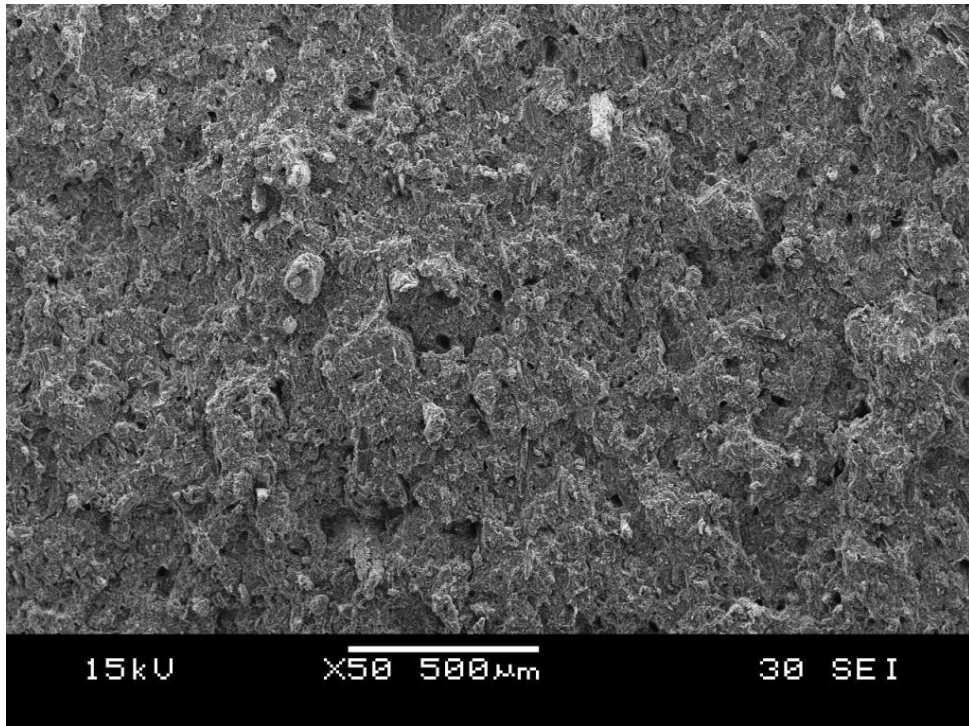
Figure 5.19: The appearance of the WPC profiles produced from the mixture of WPC scrap:WPC freshfeed at 30:70 after each re-processing pass.

5.4 Morphological Characterization

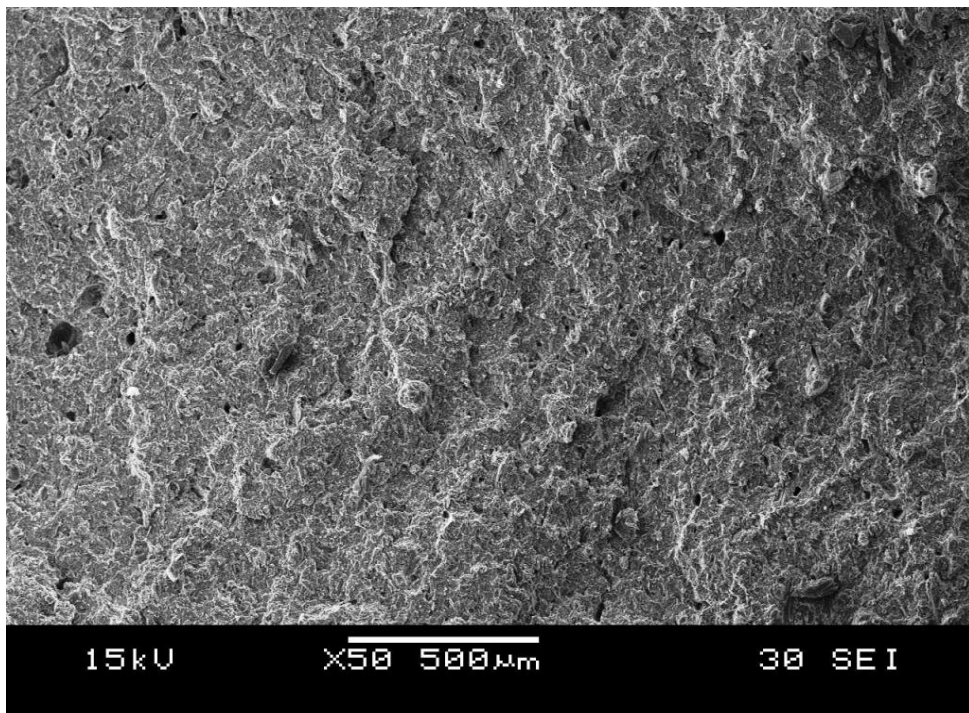
In this study, wood-plastic composites were cracked cryogenically for fracture surfaces observation. The morphology of the WPC scrap and its freshfeed pellets at different weight ratio are shown in **Figure 5.20** (a) - (g). The SEM micrographs of the WPC extruded from the various mix ratio of WPC scrap: freshfeed revealed no significant changes on the fracture surface morphology. **Figure 5.21** (aa) – (ff) showed that the fracture surfaces of the WPC after seven passes of recycling tend to be rather smooth. Molecular chain scission and possibly some thermal degradation of the PVC matrix in the WPC during the repeated recycling process led to a slightly more brittle fracture manner for the WPC with seven recycling passes.



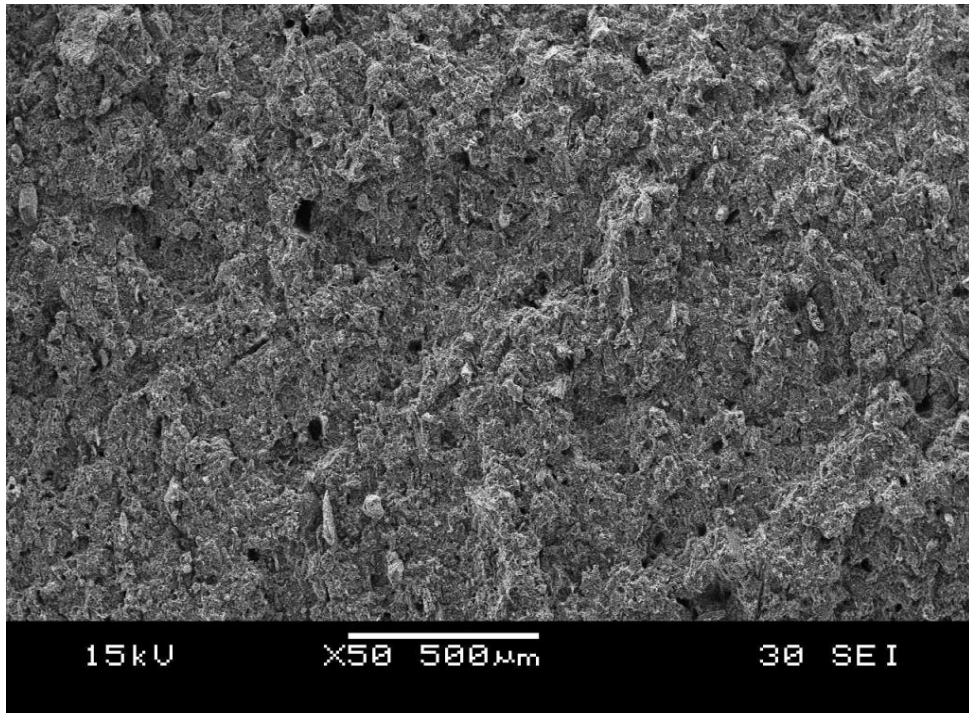
(a) WPC from freshfeed



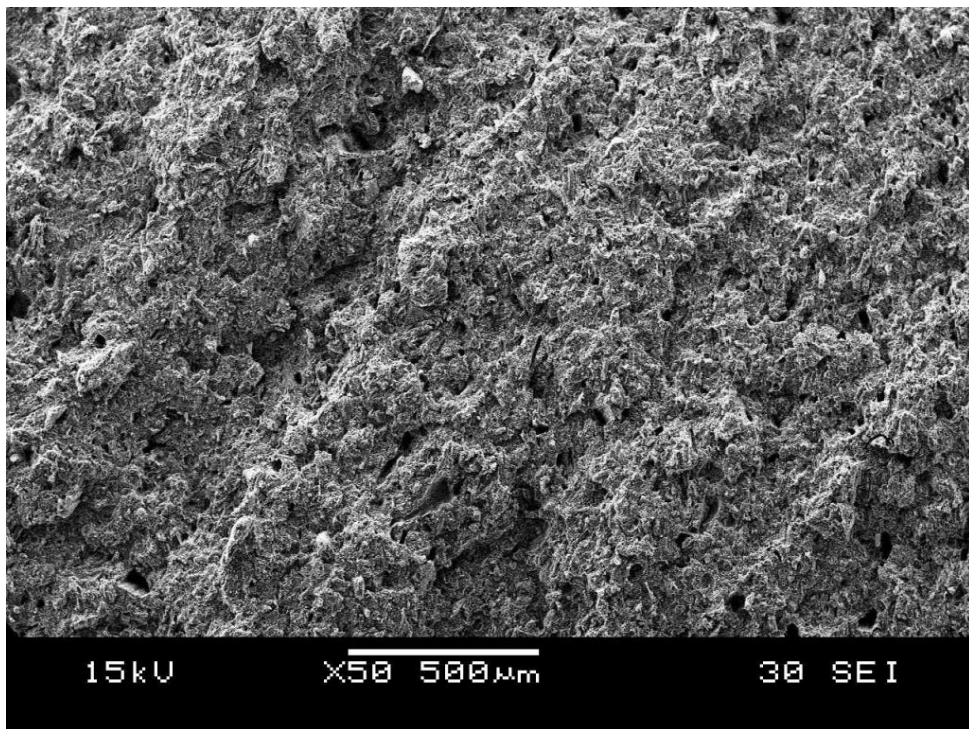
(b) WPC scrap:freshfeed = 20:80.



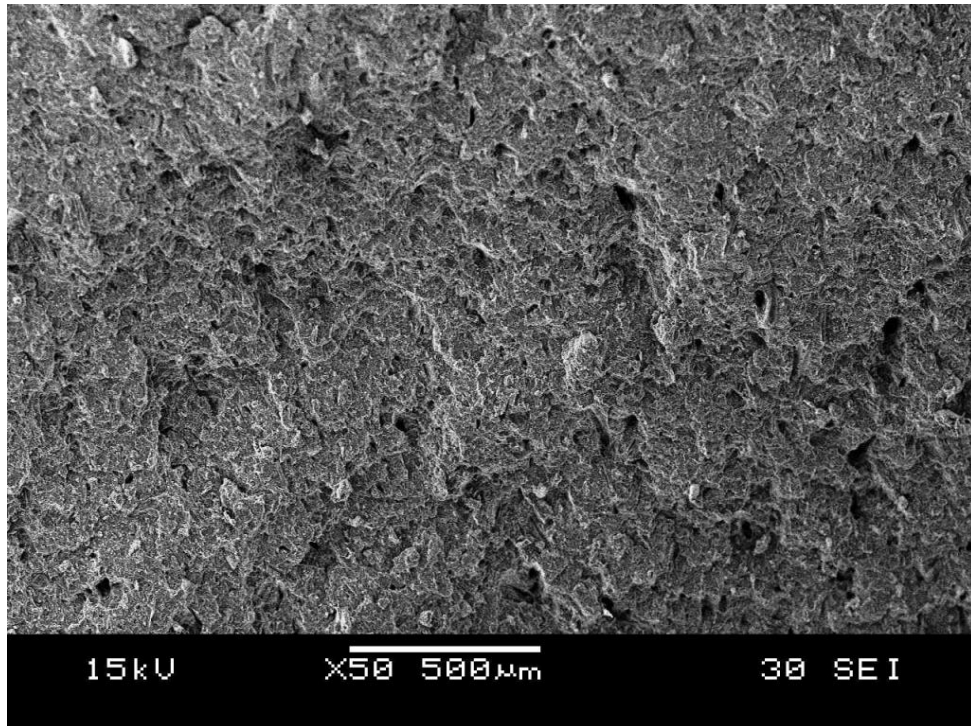
(c) WPC scrap:freshfeed = 30:70.



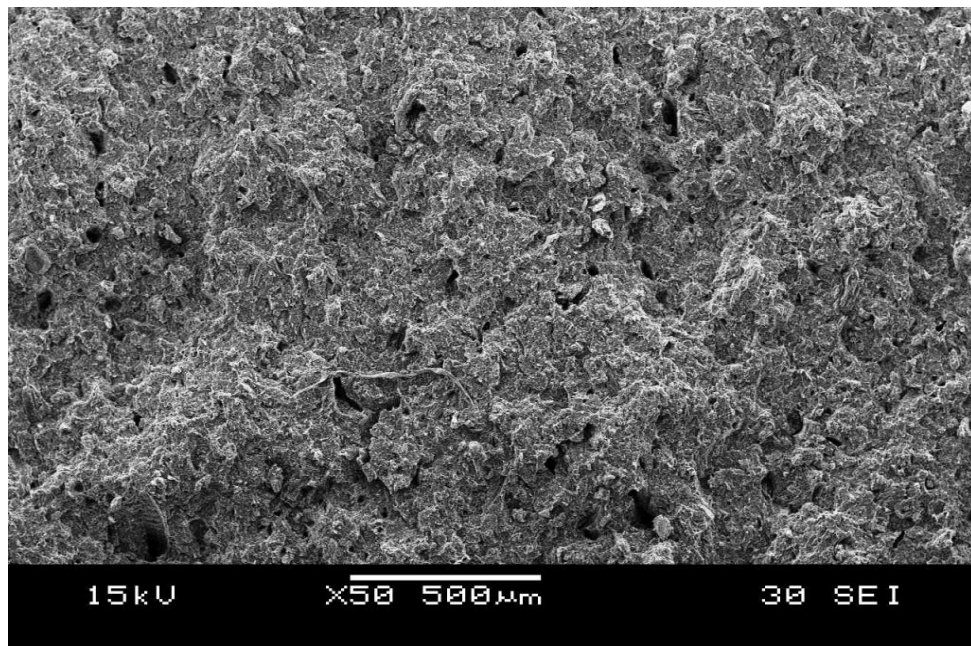
(d) WPC scrap:freshfeed = 40:60.



(e) WPC scrap:freshfeed = 60:40.

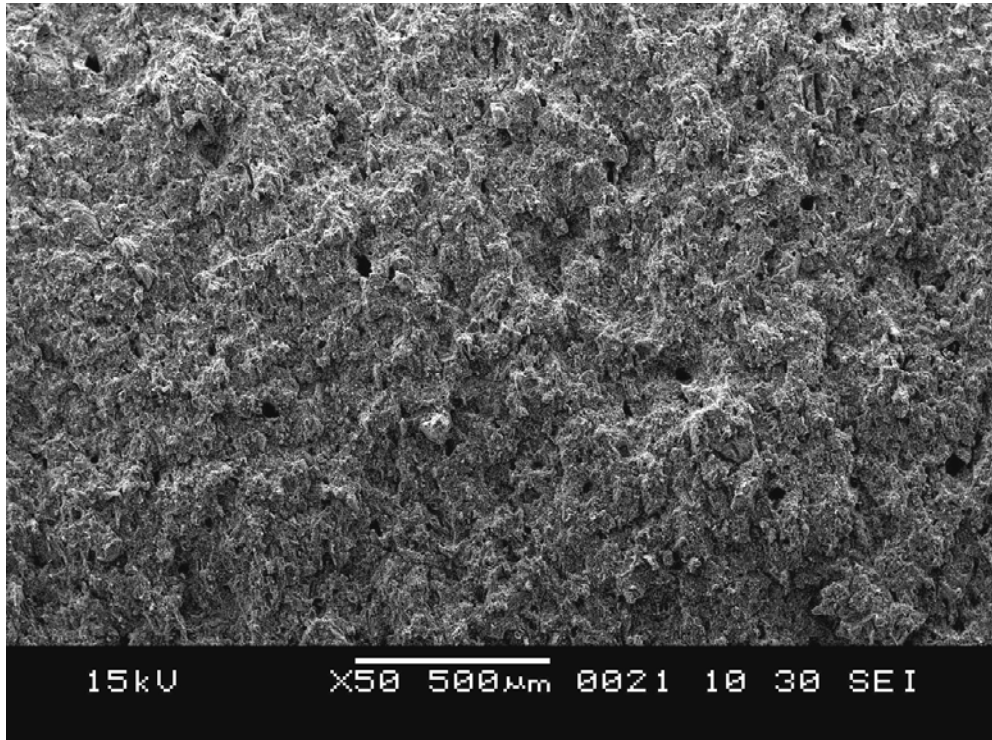


(f) WPC scrap:freshfeed = 80:20.

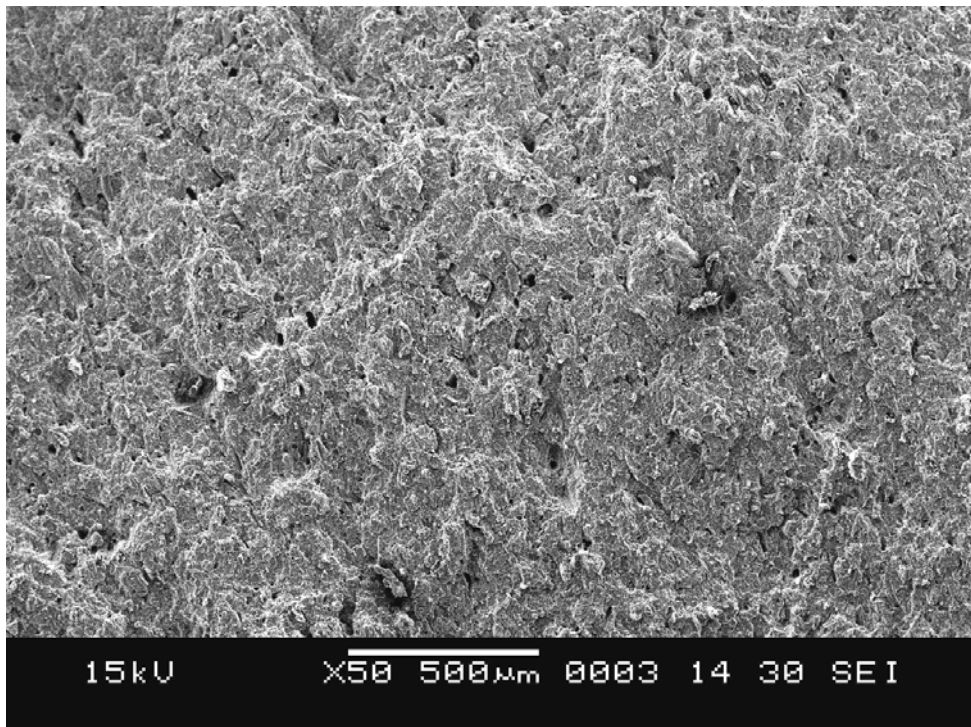


(g) 100% WPC scrap.

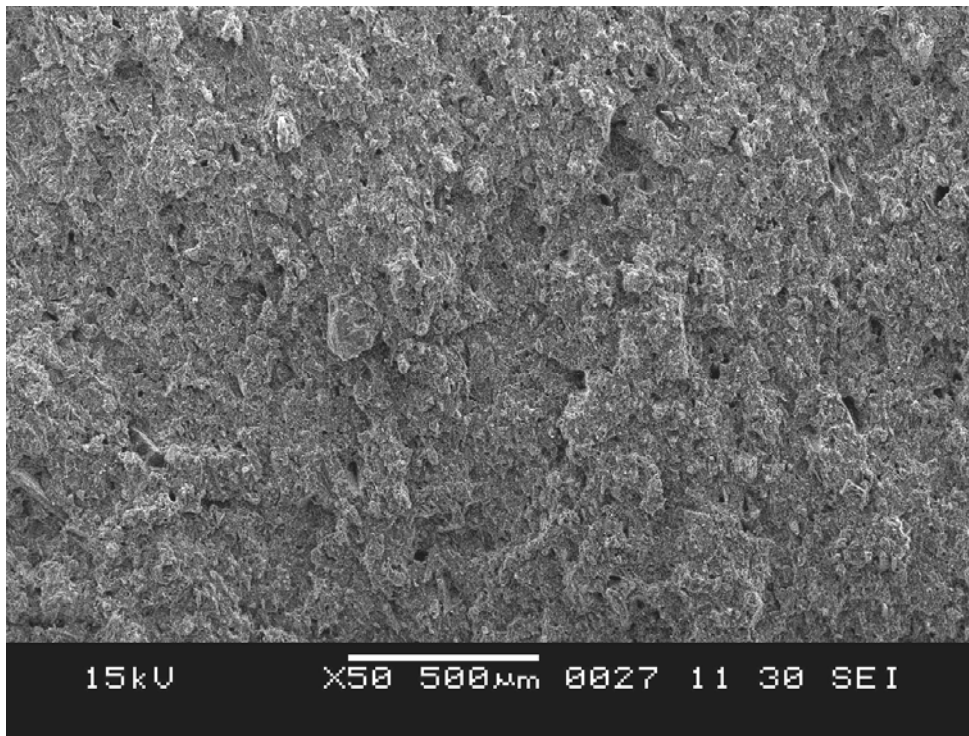
Figure 5.20: The SEM micrographs of the WPC re-extruded from freshfeed, scrap and their mixture after the first pass of re-processing. (a) freshfeed, (b) 20:80, (c) 30:70, (d) 40:60, (e) 60:40, (f) 80:20 and (g) 100% WPC scrap



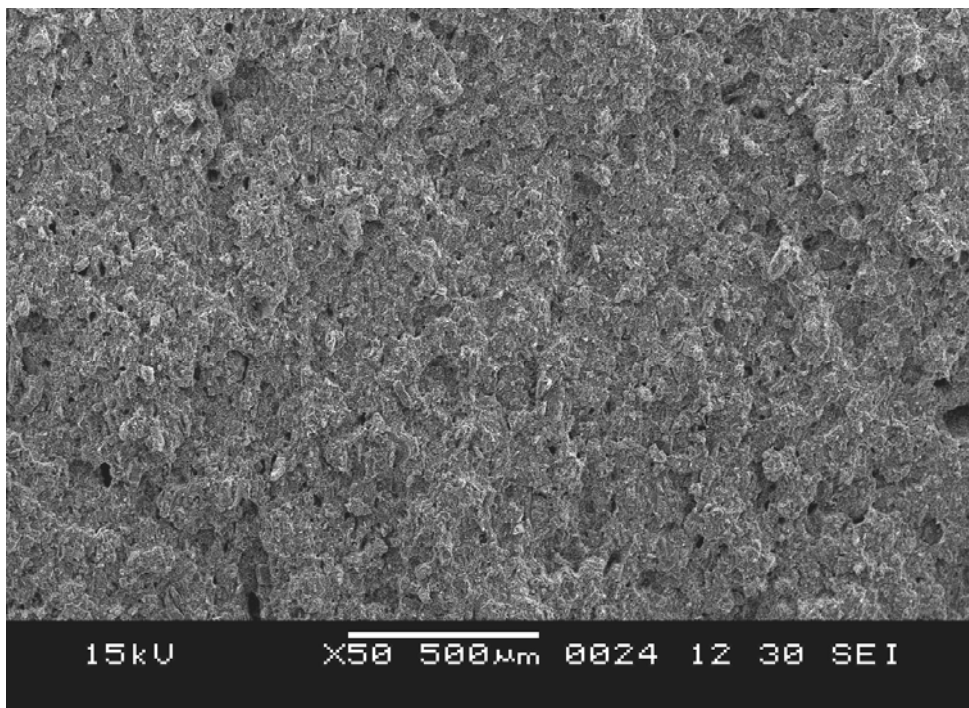
(aa) 30:70 after 1 passes.



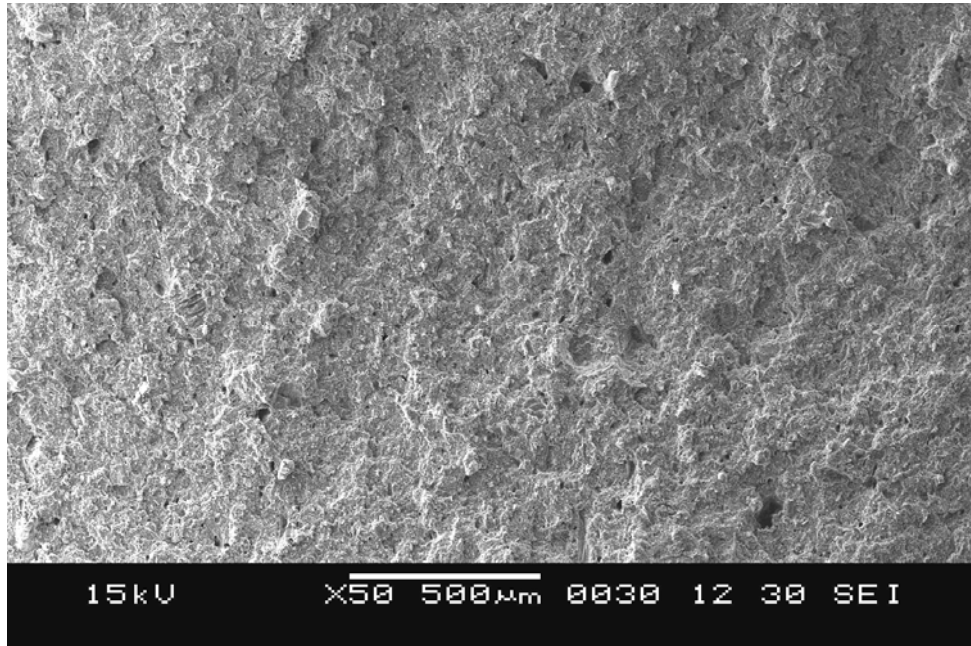
(bb) 30:70 after 2 passes.



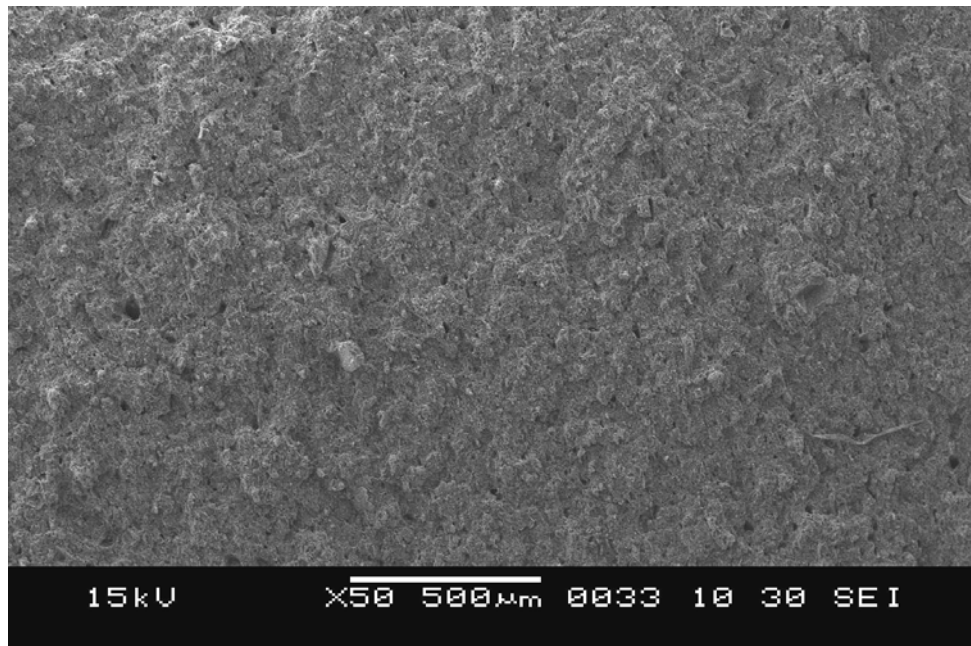
(cc) 30:70 after 3 passes.



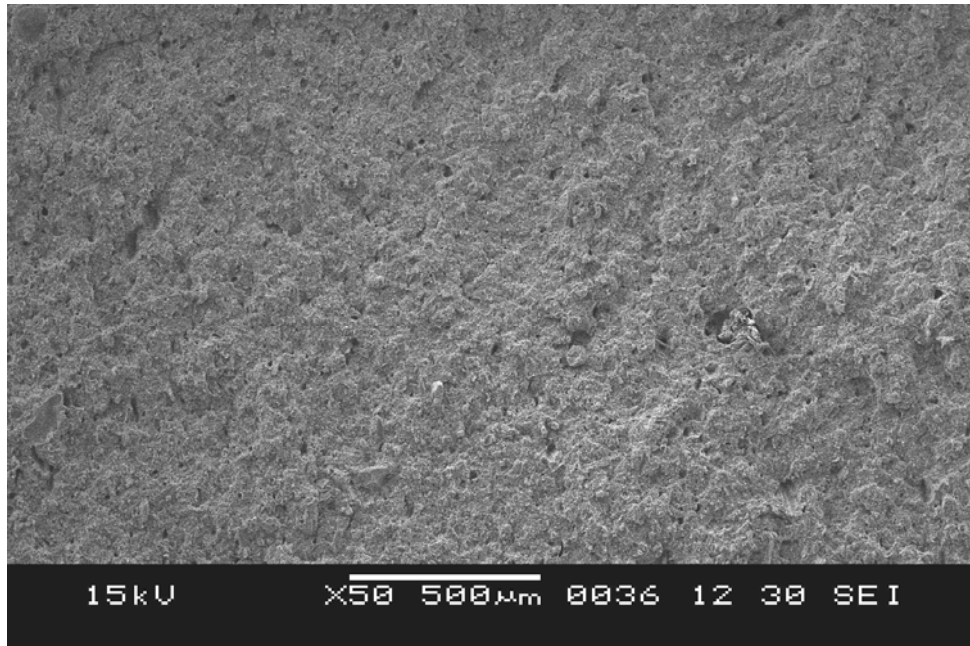
(dd) 30:70 after 4 passes.



(ee) 30:70 after 5 passes.



(ff) 30:70 after 6 passes.



(gg) 30:70 after 7 passes.

Figure 5.21: The SEM micrographs of the WPC from mixture of WPC scrap:WPC freshfeed at 30:70 after each re-processing pass (aa) first passes, (bb) second passes, (cc) third passes, (dd) fourth passes, (ee) fifth passes, (ff) sixth passes and (gg) seventh passes of recycled.

CHAPTER VI

CONCLUSIONS

6.1 Conclusions

- i) Appropriate mix ratio for close-loop recycling of WPC scrap:WPC freshfeed was 30:70. the mechanical, physical and thermal properties of the recycled WPC were very close to those extruded from freshfeed
- ii) The mixture of WPC scrap and WPC freshfeed at 30:70 ratio can be close-looply recycled repeatedly up to five passes without critically affecting the mechanical and thermal performances of the WPC.
- iii) Molecular chain scission of PVC molecules occurred only minutely over the first five passes of re-extrusion by an industrial scale twin screw extruder. PVC chain scission as a result of the shear stress during the repeated re-extrusion was prominent in the seventh pass of recycling.

6.2 Recommendations for Further Studies

- i) Improvement of the interfacial adhesion between the PVC matrix and the WF of the recycled WPC by application of a coupling agent is recommended. It is anticipated that the mechanical properties would be improved.
- ii) Investigation of the effect of environment such as water absorption or weather on the mechanical properties is recommended as a further study.

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APPENDIX

Appendix

Mechanical and Physical Characterizations

Mixture of WPC scrap and WPC freshfeed (Part I)

Appendix 1. Flexural properties of the recycling ratio of WPC

Sample	Flexural strength (MPa)	SD	Flexural modulus (GPa)	SD
Freshfeed WPC	58.69	0.96	4.14	0.08
20% waste of WPC	58.35	1.48	4.26	0.07
30% waste of WPC	57.94	1.74	4.40	0.10
40% waste of WPC	44.17	1.38	3.11	0.13
60% waste of WPC	40.81	1.30	2.89	0.10
80% waste of WPC	38.30	0.71	2.81	0.04
100% waste of WPC	42.55	1.45	3.11	0.12

Appendix 2. Tensile properties of the recycling ratio of WPC

Sample	Tensile strength (MPa)	SD	Tensile modulus (GPa)	SD
Freshfeed WPC	34.98	0.69	2.79	0.09
20% waste of WPC	33.86	0.65	2.70	0.07
30% waste of WPC	34.08	0.83	2.72	0.09
40% waste of WPC	26.80	1.18	2.21	0.08
60% waste of WPC	26.67	1.13	2.22	0.10
80% waste of WPC	25.05	0.33	2.10	0.03
100% waste of WPC	28.16	0.58	2.30	0.07

Appendix 3. Compressive properties of the recycling ratio of WPC

Sample	Compressive strength (MPa)	SD	Compressive modulus (GPa)	SD
Freshfeed WPC	59.56	2.61	2.33	0.30
20% waste of WPC	60.14	2.88	2.39	0.36
30% waste of WPC	59.48	3.12	2.31	0.34
40% waste of WPC	46.09	2.78	1.92	0.31
60% waste of WPC	46.06	3.07	2.05	0.23
80% waste of WPC	42.25	2.64	1.91	0.17
100% waste of WPC	47.20	2.61	2.07	0.22

Appendix 4. Notched Izod impact strength of the recycling ratio of WPC

Sample	Impact energy (Kj/m²)	SD
Freshfeed WPC	10.33	0.51
20% waste of WPC	10.06	0.06
30% waste of WPC	10.22	0.30
40% waste of WPC	10.37	0.29
60% waste of WPC	9.77	0.28
80% waste of WPC	9.74	0.31
100% waste of WPC	9.71	0.16

Appendix 5. Density of the recycling ratio of WPC

Sample	Density (g/cm³)	SD
Freshfeed WPC	1.4483	0.0005
20% waste of WPC	1.4472	0.0067
30% waste of WPC	1.4490	0.0146
40% waste of WPC	1.4470	0.0126
60% waste of WPC	1.4480	0.0142
80% waste of WPC	1.4440	0.0070
100% waste of WPC	1.4442	0.0080

Appendix 6. Water absorption of the recycling ratio of WPC

Sample	Water absorption (%)	SD
Freshfeed WPC	1.7982	0.02
20% waste of WPC	1.6594	0.03
30% waste of WPC	1.9448	0.02
40% waste of WPC	1.6669	0.02
60% waste of WPC	1.8496	0.03
80% waste of WPC	1.9112	0.04
100% waste of WPC	1.7510	0.02

Effects of re-processing the mixture of WPC scrap and WPC freshfeed (Part II)

Appendix 7. Flexural properties of the recycling passes of WPC

Sample	Flexural strength (MPa)	SD	Flexural modulus (GPa)	SD
30:70 after 1 passes recycled	57.94	1.74	4.40	0.10
30:70 after 2 passes recycled	50.02	1.98	3.37	0.13
30:70 after 3 passes recycled	48.31	2.20	3.26	0.17
30:70 after 4 passes recycled	48.86	2.47	3.27	0.14
30:70 after 5 passes recycled	50.37	2.16	3.44	0.22
30:70 after 6 passes recycled	49.96	2.54	3.38	0.12
30:70 after 7 passes recycled	46.03	1.80	3.01	0.12

Appendix 8. Tensile properties of the recycling passes of WPC

Sample	Tensile strength (MPa)	SD	Tensile modulus (GPa)	SD
30:70 after 1 passes recycled	34.08	1.83	2.72	0.16
30:70 after 2 passes recycled	31.75	1.18	2.63	0.17
30:70 after 3 passes recycled	31.46	2.65	2.60	0.25
30:70 after 4 passes recycled	31.82	2.00	2.59	0.23
30:70 after 5 passes recycled	32.22	1.59	2.51	0.24
30:70 after 6 passes recycled	31.36	2.44	2.44	0.27
30:70 after 7 passes recycled	30.40	2.39	2.45	0.31

Appendix 9. Compressive properties of the recycling passes of WPC

Sample	Compressive strength (MPa)	SD	Compressive modulus (GPa)	SD
30:70 after 1 passes recycled	59.48	3.12	2.31	0.24
30:70 after 2 passes recycled	49.07	1.81	2.02	0.18
30:70 after 3 passes recycled	47.40	1.70	2.07	0.14
30:70 after 4 passes recycled	51.21	1.63	2.07	0.16
30:70 after 5 passes recycled	50.50	2.46	2.12	0.12
30:70 after 6 passes recycled	50.13	1.74	2.11	0.18
30:70 after 7 passes recycled	47.27	2.88	2.09	0.27

Appendix 10. Notched Izod impact strength of the recycling passes of WPC.

Sample	Impact energy (Kj/m²)	SD
30:70 after 1 passes recycled	10.22	0.30
30:70 after 2 passes recycled	10.69	0.42
30:70 after 3 passes recycled	11.00	0.37
30:70 after 4 passes recycled	10.23	0.60
30:70 after 5 passes recycled	11.09	0.47
30:70 after 6 passes recycled	11.01	0.61
30:70 after 7 passes recycled	9.89	0.71

Appendix 11. Density of the recycling passes of WPC.

Sample	Density (g/cm³)	SD
30:70 after 1 passes recycled	1.4490	0.0146
30:70 after 2 passes recycled	1.4493	0.0072
30:70 after 3 passes recycled	1.4522	0.0084
30:70 after 4 passes recycled	1.4570	0.0110
30:70 after 5 passes recycled	1.4580	0.0084
30:70 after 6 passes recycled	1.4601	0.0099
30:70 after 7 passes recycled	1.4629	0.0072

Appendix 12. Water absorption of the recycling passes of WPC.

Sample	Water absorption (%)	SD
30:70 after 1 passes recycled	1.8099	0.06
30:70 after 2 passes recycled	1.7730	0.08
30:70 after 3 passes recycled	1.8362	0.07
30:70 after 4 passes recycled	1.6790	0.08
30:70 after 5 passes recycled	1.6618	0.09
30:70 after 6 passes recycled	1.6520	0.11
30:70 after 7 passes recycled	1.9440	0.10

VITA

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