

การพัฒนาสารประกอบแต่งจาก PVC เป็นวัสดุทดแทนไม้



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DEVELOPMENT OF WOOD-SUBSTITUTED COMPOSITES FROM PVC



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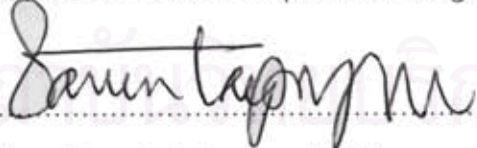
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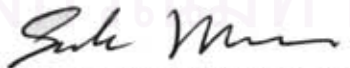
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100 หน้า.

วัสดุประกอบแต่งทดแทนไม้เป็นวัสดุที่ประกอบไปด้วยเทอร์โมพลาสติกทำหน้าที่เป็นเมตริกซ์ และเส้นใยธรรมชาติเป็นสารเติม วัสดุนี้นี้ได้รับการยอมรับเป็นอย่างมากเนื่องด้วยคุณสมบัติทางกลดี ผลิตง่าย มีลักษณะคล้ายกับไม้ธรรมชาติ เป็นมิตรกับสิ่งแวดล้อมและที่สำคัญเป็นการนำวัสดุเหลือทิ้งมา ทำให้เกิดประโยชน์ ในงานวิจัยครั้งนี้ใช้ผงไม้สามชนิดซึ่งหาได้ง่ายในประเทศไทยคือ ไม้ยางพารา, ไม้เบญจพรรณ, และ ไม้สักทอง เป็นสารเสริมแรงในพอลิไวนิลคลอไรด์โดยอนุภาคของผงไม้ที่มีขนาด <math><150, 150-180, 180-250, 250-450, \text{ และ } 600-850 \text{ ไมครอน}</math> และปริมาณผงไม้ขนาด 0, 20, 40, และ 60 phr ถูกศึกษาผลที่มีต่อคุณสมบัติทางกล, ทางความร้อน, การดูดซับน้ำ, และ ความเสื่อมสภาพต่อสภาวะอากาศของวัสดุคอมพอสิต ผลการทดลองพบว่าคุณสมบัติด้านความแข็งแรง (Strength Properties) ของวัสดุคอมพอสิต มีแนวโน้มลดลงเมื่อเพิ่มปริมาณผงไม้มากถึง 60 phr โดยลดลงประมาณ 14% จากค่าของพอลิไวนิลคลอไรด์ ในทางตรงกันข้ามค่ามอดูลัส (Modulus properties) ของวัสดุประกอบแต่งมีค่าเพิ่มขึ้นประมาณ 40% จากพอลิไวนิลคลอไรด์ซึ่งเป็นเมตริกซ์ การลดลงของคุณสมบัติทางความแข็งแรงเนื่องจากการเกิดจุดบกพร่องระหว่างอนุภาคผงไม้และพอลิไวนิลคลอไรด์ สำหรับผลของขนาดอนุภาคผงไม้ต่อวัสดุคอมพอสิตมีผลทำให้คุณสมบัติทางกลลดลงเมื่ออนุภาคไม้มีขนาดเพิ่มขึ้นถึง 600-850 ไมครอน นอกจากนี้ชนิดของผงไม้ไม่ส่งผลต่อคุณสมบัติทางกลและทางความร้อนต่อวัสดุประกอบแต่งทดแทนไม้จากพอลิไวนิลคลอไรด์ อย่างไรก็ตามชนิดของผงไม้จะมีผลต่อความหลากหลายของลักษณะสีผิวของชิ้นงาน ยิ่งไปกว่านั้นการศึกษาถึงผลที่เกิดจากการเสื่อมสภาพของชิ้นงานต่อสภาวะอากาศเป็นเวลา 1 เดือน ณ กรุงเทพมหานคร ให้ผลต่อสมบัติการดัดโค้ง (Flexural properties) ของวัสดุคอมพอสิตลดลงจากวัสดุคอมพอสิตที่ไม่ได้ผ่านการทดสอบทางการเสื่อมสภาพ โดยค่าความแข็งแรงลดลงประมาณ 5% และค่ามอดูลัสลดลงประมาณ 9% ที่ปริมาณผงไม้ขนาด 60 phr อีกทั้งยังส่งผลถึงลักษณะผิวชิ้นงานที่ขรุขระมากขึ้นและสีผิวที่ซีดจางลง หายที่สุดสูตรที่เหมาะสมสำหรับวัสดุประกอบแต่งเพื่อทดแทนไม้จากพอลิไวนิลคลอไรด์ คือ การใช้ปริมาณและขนาดของอนุภาคผงไม้ที่ 40 phr และ 180-250 และ 250-450 ไมครอน ตามลำดับ และใช้ได้สำหรับไม้ทุกชนิด

ภาควิชา.....วิศวกรรมเคมี.....

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KEY WORD: COMPOSITES / POLYVINYLCHLORIDE / WOODFLOUR

SILAWAN CHONSARANON: DEVELOPMENT OF WOOD-SUBSTITUTED COMPOSITES FROM PVC. THESIS ADVISOR: ASST. PROF. SARAWUT RIMDUSIT, Ph.D., THESIS COADVISOR: ASSOC. PROF. SIRIPORN DUMRONGSAKKUL, Ph.D., 100 pp.

Wood plastic composite is generally a combination of thermoplastic and natural filler. This kind of thermoplastic composites evolves as structural polymer composites, which have many beneficial properties such as low density and good mechanical properties, as well as good insulation and environmental resistance. In this study, three species of woodflours i.e. rubber wood, mixed wood, and teak wood, are used as a reinforcing agent in a polyvinylchloride matrix because these woods are abundant and widely grown in Thailand. The effects of particle loading (0-60 phr), particle sizes (<150 to 600-850 μm), and species of woodflours on mechanical properties, thermal properties, water absorption, and weathering aging behaviors of the obtained PVC wood composites are characterized. The results reveal that, the composite strengths decrease about 14% with increasing particle loading up to 60 phr but moduli increase about 40% of the unfilled PVC matrix. The decrease of strength of PVC wood composites is attributed to some interfacial defects between the woodflour and the PVC matrix. The effects of particle sizes on the mechanical properties of PVC wood composites are found to decrease with increasing the particle size up to 600-850 μm . In addition, the woodflour types have no significant effect on the mechanical and thermal properties of the PVC wood composites. However, the types of woodflours can cause the variation in color and texture of the obtained composites. Furthermore, the natural weathering after 1 month exposure in Bangkok, Thailand, results in the decrease in the composites flexural strength about 5% and the composites flexural modulus about 9% i.e. a particle loading of 60 phr. All the specimens also show a significant color fading. Finally, the suitable formulations of PVC wood composites are to use a woodflour loading of 40 phr at average particle size of 180-250 μm and 250-450 μm .

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CHAPTER I

INTRODUCTION

1.1 General Introduction

Wood is one of the oldest building materials. It has established itself as a major wood product in several countries, particularly for the production of furniture, furniture components and wood panels. The obvious drawbacks of natural wood products are high water uptake (30-200%), high flammability (Limiting Oxygen Index: LOI=21), termite irresistibility and liability to damage by attack with fungi (Simpson et al., 1999). The development of wood-substituted materials particularly based on wood-plastics composites is consequently of increasing interest in recent years since most shortcomings mentioned above can effectively be solved or minimized by an incorporation of polymeric materials to form wood-plastics composites. The wood-plastic product development is also one of the best measures to utilize waste wood materials. In the case of flammability problem, only limited number of polymers can be used to impart fire-resistant characteristics to wood composites especially when ease of processing and good mechanical performance have to be achieved. As a result, the choice of the polymer matrix used is crucial to the successful development of the wood composite products. Thermoplastic and thermosetting wood composites are the two major classes of wood plastics products (Jiang et al., 2004).

Typically, useful properties of conventional thermoplastic wood composites compared to thermosetting wood include high cost-competitiveness, ease of processing, and recyclability but generally rendering wood composite products with low fire resistance, low thermal stability (Li et al., 2004), and low compatibility with natural wood (Blendzki et al., 2004). These kinds of wood composites often render products of low load-bearing characteristics. Most previous works indicated the necessity of using either compatibilizers or coupling agents to enhance the mechanical properties of thermoplastic

wood. Moreover, the use of commodity thermoplastic matrices such as PE, PS, and PP, due to their inherently low LOI and low char yield, results in rather flammable wood composites which of major concern in some applications. Those drawbacks are, however, less significant in polyvinylchloride matrix due to the more compatible nature (more polarity) with natural wood as well as its inherent fire resistant characteristics. The polymer is thus highly attractive for wood composite matrix particularly for the construction or home decoration purposes.

The term wood-plastic composite (WPCs) refers to any composites that contain wood and thermosets or thermoplastics (Youngquist et al., 1999). The wood used in WPCs is most often in particulate form (e.g. woodflour, sawdust, etc.) or very short fibers, rather than longer individual wood fibers. Filler particles are often incorporated into a polymer to modify properties to meet performance requirements. For certain applications, high filler content are needed to achieve the desired property modification. Two groups of wood particle composites are commonly recognized i.e. based on size of wood components and methods of manufacture. The wood particles are mainly produced from residue of other primary wood manufacturing operations or from round wood. Slaps, edgings, trim ends, and whole logs are used to produce the particle.

Woodflour has several advantages compared to inorganic filler, including low cost per volume basis, low density, high specific strength, high specific modulus, nonabrasiveness that shows flexibility during the processing with no harm to the equipment, renewable nature and the utilization of waste materials. Polymers with particulate filler or short fiber reinforcements are enjoying a rapid increment in the volume and number of applications because of their good processability (Lioa et al., 1997). A wide variation of mechanical and physical properties can be developed through an appropriate compounding of polymer and fillers. There are, however, some adverse effects for example; addition of high modulus wood fiber to a plastic matrix usually results in increased brittleness (Xu et al., 2001). In other words, an increment of the stiffness and strength of wood fiber composites are obtained at an expense of their toughness and ultimate elongation (Balasuriya et al., 2001).

Wood plastic composites products have the look and workability of wood, require less maintenance, and offer lower life-cycle costs than wood. The use of WPCs by construction building has resulted in concern about the durability of these products when exposed to outdoor environments. The weathering and light stability of PVC wood composites are usually assessed by long-term outdoor exposure test. The influence of the combined action of water, temperature and radiation in the oxidative ageing was evaluated by means of a comparison may involve measurements of haze, transmission, lightness, color change, and/or physical such as impact strength and flexural strength. WPCs are detrimentally affected by the presence of moisture, primary because of the wood component. Wood is a naturally hydrophilic material. As a result, wood cell walls swell when penetrated by water, facilitating deeper light penetration and providing sites for further degradation (Hon, 2001). In addition, some water-soluble extractives are removed. In WPCs, swelling of the wood fiber compromises the wood/matrix interface and creates microcracks in the matrix (Rangaraj et al., 2001). The result is a change in appearance and deterioration of mechanical properties.



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1.2 Objectives of the Research

This research aims to generate the technical knowledge and formulations for producing the PVC Wood composites by combining PVC resin produced by Vinythai PCL and natural woodflours available locally as an alternative for natural wood i.e rubber wood, teak wood, and mix wood. The objectives are to:

1. To study the effects of particle size, particle loading, and types of woodflour on the properties of the resulting PVC wood composites.
2. To evaluate the effects of woodflour on texture of the PVC wood composites.

1.3 Scopes of the Research

1. Research materials preparation (PVC resins, woodflour)
2. Treatment of woodflour i.e. drying, grinding, sieving etc.
3. Preparation of PVC matrix and woodflour (rubber wood, teak wood and mixed wood) composites at the woodflour contents of 0, 20, 40 and 60 phr, and average particle sizes of <150, 150-180, 180-250, 250-450, and 600-850 μm .
4. Determination of optimal compositions of PVC wood composites including using two-roll mill and compression molder.
5. Investigation of the PVC wood products using two-roll mill and compression molder.
6. Characterizations of the PVC wood composites including thermal stability, impact properties, tensile and flexural behaviors, water absorption, weathering aging and other essential physical properties as wood-substituted materials.
7. Research discussion, conclusions
8. Report preparation

CHAPTER II

THEORY

2.1 Composites Material

Composite material can be defined as a macroscopic combination of two or more distinct materials, having a recognizable interface between them. However, because composites are usually used for their structural properties, the definition can be restricted to include only those materials that contain reinforcement (such as fibers or particles) supported by a binder (matrix) material. The constituents can be organic, inorganic or metallic (synthetic and naturally occurring) in form of the particles, rods, fibers, plates, foams, etc. Compared with homogeneous materials, these additional variables often provide greater latitude in optimizing, for a given application, such physically uncorrelated parameters as strength, density, electrical properties, and cost. Further, the composite may be the only effective vehicle for exploiting the unique properties of certain special material (Reinhart et al., 1989).

2.2 Wood Composite Matrices

A role of a matrix in a fiber-reinforced composite is (1) to transfer stresses between the fibers, (2) to provide a barrier against an adverse environment, and (3) to protect the surface of the fibers from mechanical abrasion. The matrix plays a minor role in the tensile load-carrying capacity of a composite structure. However, selection of a matrix has a major influence on the interlaminar shear as well as on in-plane shear properties of the composite material. The interlaminar shear strength is an important design consideration for structures under bending loads, whereas the in-plane shear strength is important for structures under torsional loads. The matrix provides lateral support against the possibility of fiber buckling under compression loading, thus influencing to some extent the compressive strength of the composite

material. The interaction between fibers and matrix is also essential in designing damage-tolerant structure. Finally, the processability and defects in a composite material depend strongly on the physical and thermal characteristics, such as viscosity, melting point, and curing temperature of the matrix. Additional functions of a matrix are to keep the fibers in place in the structure, to help distribute of transfer load, to protect the filaments, both in the structure and before fabrication, to control the electrical and chemical properties, and to carry interlaminar shear. Other desirable properties of a matrix, depending on the purpose of the structure, are (i) to minimize moisture absorption, (ii) to wet and bond to fiber, (iii) to penetrate completely and eliminate voids during the compacting and curing processes, (iv) to have strength at elevated temperature (depending on the application), (v) to have low shrinkage, (vi) to have dimensional stability, with reasonable strength, modulus, and elongation.

Poly(vinyl chloride) as Wood Composite Matrix

The utilization of poly(vinyl chloride) as a wood composite matrix has been reported to experience a dramatic increase in use recently (Jiang et al., 2004). Typically the composites are used to produce window/door profiles, decking, railing, and siding. The PVC compounding based on conical counterrotating intermeshing twin-screw extruders is demonstrated to be one effective way to get well dispersed wood composite products. To obtain the enhanced PVC wood performance, heat stabilizers, processing aids, impact modifiers, lubricants, and pigments are also incorporated in the materials. The use of several types of natural fibers as well as the use of natural fiber with mica or glass fiber to form hybrid reinforcements is proved to enhance the mechanical properties of the final products. Furthermore, PVC wood has been proven to exhibit superior ultraviolet light resistance and weathering dimensional stabilities to those of natural wood besides its inherent fire-resistant characteristics with an limiting oxygen index (LOI) as high as 50 (LOI > 26 is classified as self-extinguishable).

2.3 Polymer Matrix

2.3.1 Poly(vinyl chloride) (PVC)

Polyvinyl chloride, PVC, is a thermoplastic material that has the following chemical formula: $\text{CH}_2=\text{CHCl}$.

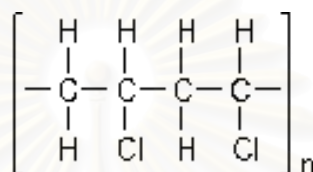


Figure 2.1: Poly(vinyl chloride) (PVC)

(<http://www.lentech.com>).

The molecular weight of PVC ranges molecular weight (M_w) 40000 – 480000 and molecular number (M_n) 20000 – 92000 whereas molecular weight distribution (M_w/M_n) of commercial PVC ranges between 1.9 and 5.2.

After the First World War, there were a boom in new forms of plastics due to the improvements in the chemical technology sector, including "polystyrene (PS)" and "polyvinyl chloride (PVC)", developed by the I.G. Farben company of Germany. Nowadays, PVC is commonly used in construction sector, for example in window frames and shutters, pipe cabling and coating, etc. Vinyl is also used in gramophone records, and that is why we use the term vinyl records to refer to them. PVC can be used for tons of other applications from industrial ware and widely used in the healthcare sector, car spare parts, toys factory, food packaging, raingear, etc. PVC can be clear or colored, rigid or flexible, depending on the added compounds and final application that needs to be achieved; For example there exists different PVC grades such as coast or blown film, high impact, wire and cable grade, thermoforming, injection molding, rotational molding.

The basic raw materials for PVC are derived from salt and oil. Chlorine is manufactured by the electrolysis of sodium chloride, salt. The electrolysis of salt

water produces chlorine and then combined with ethylene that has been obtained from oil. The resulting element is ethylene dichloride, which is converted at very high temperatures to vinyl chloride monomer. These monomer molecules are polymerized forming polyvinyl chloride resin.

2.3.2 Properties of PVC

Commercial PVC is generally produced by addition polymerization. It may be produced by a variety of techniques such as suspension, emulsion, micro-suspension and bulk. PVC, as normally prepared, is a white granular material, ranging in particle size from 5-400 microns and with apparent bulk densities of 0.5-0.8 g/cm³. Emulsion type of PVC is usually smaller in particle size. This type of PVC also is called dispersion type. Dispersion type PVC is ground to further decrease particle size which is called PVC paste type. PVC paste particles are irregular in shape and have relatively high surface area. Both dispersion and paste types of PVC are suitable for plastisol applications.

Table 2.1: The properties of rigid and plasticized PVC

Property	Rigid PVC	Plasticized PVC
Density range	1.3 – 1.4 g/cc	1.1 – 1.7 g/cc
Specific heat	0.25 cal/g °C	-
Sag temperature	78°C	-
Milling temperature	160°C	140 - 150°C
Coeff. Of linear ther. Experiment	5x10 ⁻⁵ °C	-
Dielectric constant 30°C 60cps	3.7	5.85
Power factor 30°C 60cps	1.25%	-
Heat distortion at 30 Kg	80°C	-
Thermal conductivity	3.94x10 ⁻⁴ °C	-
Vicat softening point	-	58°C
Shore Hardness (D)	-	47
Tear resistance	-	8500 kg/m

Uncompounded PVC is tough, brittle and has relatively poor heat stability compared to other thermoplastic materials and thus is never used without some modification. Providing that the plasticizer level is low, most PVC compounds burn slowly and tend to be self-extinguishing. Its high flash ignition temperature is another advantage. The physical properties of the compounded material are a function of both the resin and the compounding conditions. Plasticized PVC is the one of the most versatile plastic materials as PVC properties shown in Table 1 and Table 2. Compounding gives a very wide range of applications to PVC which approaches to that of rubbers and engineering plastics. Moreover, PVC compounds are significant fire retardant properties, weathering resistance, excellent clarity and good flexural strength. However, the limitations of PVC processing are that, it degrades at elevation temperatures, can be corrosive to processing equipments, has relatively higher density than other plastics and susceptible to solvent.

Table 2.2: Mechanical properties of PVC

Property	Rigid PVC	Plasticized PVC
Tensile strength (MPa)	34 – 62	10 – 24
Elongation (%)	2 – 40	200 – 450
Modulus of Elastic (MPa)	5520	7
Izod Impact strength(kJ/m ²)	21-1067	Varies
Compressive strength (MPa)	54 – 88	6 – 12
Rockwell Hardness	M 70	-

2.3.3 Suspension PVC Resin (<http://www.vinythai.co.th>)

Suspension PVC resins are PVC homo-polymer resins which can be used in such conversion techniques as extrusion, calendaring and injection. PVC resins are chosen from various molecular weights, presented by the different K-value that related to mechanical properties. An exemplification, Saimvic 258 RB is PVC suspension type that shows characteristics in Table 3.

Moreover, Siamvic 258RB is a low K-value resin that is suitable for rigid PVC processing due to a low K-value resin is very low dust during transportation and pneumatic filling of silos, regular feed of extruders as dry blend, easy processing even at low temperature, good thermal stability, good initial color, no tendency to stick on hot metal surface and, very low level of fish-eyes.

Table 2.3: Characteristics of suspension PVC resin (258 RB)

Characteristics	Units	Values (*)	Standards
Viscosity index	ml/g	82	ISO 1628-2
K-value (cyclohexanone)	-	58	DIN 53726
Polymerization degree	-	680	JIS K6721
Bulk density-compaction	kg/l	0.56	ISO 1068
Volatile matter	%	≤ 0.3	ISO 1269
Particle size (sieve analysis)	micron	120-150	ISO 1624

(*) In the case of certain characteristics the values given in this data sheet are means based on a large number of individual measurements distributed around the means in a range corresponding to the normal manufacture and measurement tolerances. These values should not be considered as specifications.

2.3.4 PVC Additives

1. Processing aids

Processing aids are chemicals which assist the melt processing of PVC. Processing aids do not reduce melt viscosity but they improve the elastic behavior of the melt. These additives are characterized by a high level of compatibility with PVC and much higher molecular weights than PVC, therefore these long chains tend to bind together the PVC melt structure thus increasing the extensibility of the melt. Examples of processing aid are methacrylate/acrylate copolymer, poly 2-methyl styrene, poly neopentelene terephalate, poly alkylene carbonate and, etc. Use levels can vary widely, depending on the particle conditions the range is 0.8 to 20 phr, but most common range is 1-3 phr.

2. Lubricants

Lubricants are added to PVC compounds to facilitate processing and to permit control of processing rate. They prevent sticking and may also function as antistatic agents. Lubricant cannot be categorized as external or internal since it is likely that act in both ways depending on their concentration and the processing condition applied. Internal lubricants are chemical which lubricate the flow of PVC molecules within the melt. Wax and wax derivatives, glycerly ester of fatty acids, long chain esters such as cetyl plamitate are most commonly used. Concentration depends on the number of lubricants used to get the desired balance of behavior. Total concentrations rarely exceed 4 phr. The normal concentration of internal/external lubricants is between 0.2-2.0 phr.

3. Stabilizers

Stabilizers are used to inhibit the degradation caused by heat or light. PVC is thermally unstable and degrades rapidly at normal processing conditions. It may also be degraded slowly by light. Typical examples are, metallic soaps such as

cadmium, barium, and zinc stearates which also act as lubricants. These chemicals simply react with HCl and are able to reduce further degradation. Calcium and zinc stabilizers are low in toxicity and they are often prepared in the form of dispersion in epoxidized oil. Barium and cadmium stabilizers are mainly solid products which are liquid contain phosphite groups. Organotin compounds such as sulphur containing thio-tin compounds, octyl-tin compounds also act as partial plasticizers and improve melt flow properties. Carboxylate derivatives sulphur containing organotin carboxylate-mercaptides most organo-tin stabilizers could be used in transparent applications. Octyl-tin compounds are regarded as non toxic. Trivasic lead sulphate is the stabilizer which also provides opacity and gives white pigmentation. It is cheap and popular stabilizer but presents toxicity problems. Stabilizers are added in the concentration range of <1 to 3 phr.

4. Fillers

Fillers are added to PVC compounds to mainly reduce the cost. Additional technical improvements are also desirable such as better opacity, electrical properties, UV resistance, thermal properties, reduced plate out and sticking. The fillers could be used up to about 60 phr and higher concentrations are sometimes used where the application permits. Evaluations of the effect of various fillers on the physical properties of the compound are complicated and depend on the other ingredient of the compounds.

2.4 Filler

Natural Fibers and Their Chemical Compositions

(Blendzki and Gassan, 1999)

Natural fibers or fillers such as wood, cellulose and jute are renewable materials with very attractive mechanical properties. For instance, cellulose fibers with moduli up to 40 GPa can be separated from wood by a chemical pulping process.

A growing awareness of environmental problems and the importance of energy conservation have made such renewable reinforcing materials of great importance.

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.

Cellulose is the essential component of all plant-fibres. The cell walls of large numbers of plants consist of the same substance, to which he gave the name cellulose. It is generally accepted that cellulose is a linear condensation polymer consisting of D-anhydroglucopyranose units joined together by 1,4- β -D-glucan. The pyranose rings are in the 4C_1 conformation, which means that the $-CH_2OH$ and $-OH$ groups, as well as the glycosidic bonds, are equatorial with respect to the mean planes of the rings. One of cellulose's most characteristic properties is the presence of both crystalline and amorphous regions within its microfibrils. According to the so-called *fringe micellar theory*, a single cellulose chain may run through several crystalline regions as well as through amorphous zones in between. Cellulose, as the skeletal substance, contributes its high tensile strength to the complex of wood structure. Generally, the fibers contain 60-80 % of cellulose. However, hardwoods contain more cellulose than do softwoods.

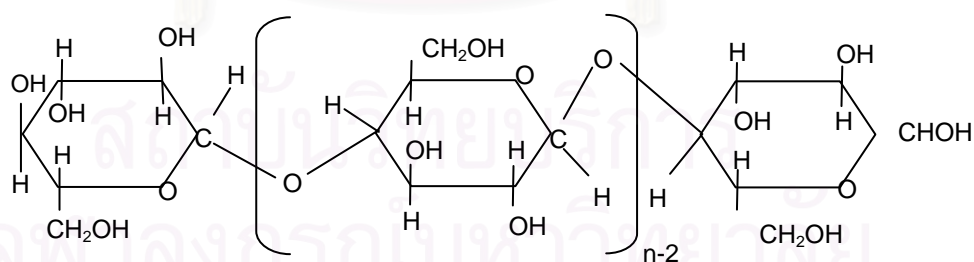


Figure 2.2 : Structure of cellulose contains 1,4- β -D-glucan

(<http://www.fibersource.com>)

Hemicellulose is not a form of cellulose at all and the name is an unfortunate one. They comprise a group of polysaccharides (excluding pectin) that remains associated with the cellulose after lignin has been removed. The constituents of

hemicellulose differ from plant to plant. The function of hemicellulose is less clear, there are existing some possibility that they serve as a temporary matrix before lignification. The hemicellulose differs from cellulose in three important aspects. In the first place they contain several different sugar units whereas cellulose contains only 1,4- β -D-glucopyranose units. Secondly they exhibit a considerable degree of chain branching, whereas cellulose is a strictly linear polymer. Thirdly, the degree of polymerization of native cellulose is ten to one hundred times higher than that of hemicellulose. Hemicellulose is responsible for the biodegradation, moisture absorption, and thermal degradation of the fiber as it shows least resistance. Generally, wood contains the hemicellulose about of 5 to 20 %.

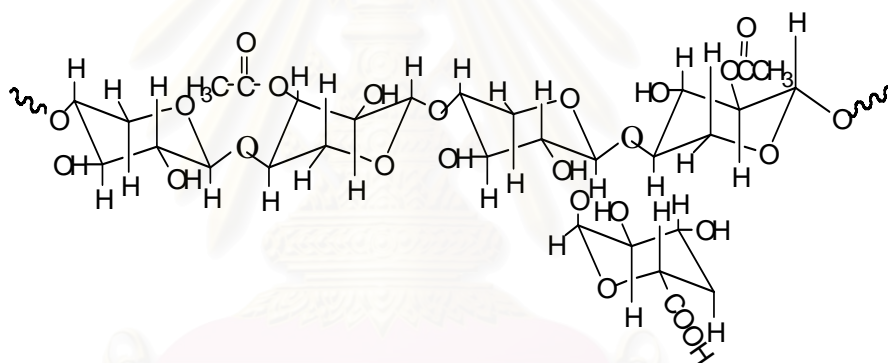


Figure 2.3 : Structure of hemicellulose

(<http://www.fibersource.com>)

Lignins are complex hydrocarbon polymers with both aliphatic and aromatic constituents. Their chief monomer units are various ring-substituted phenyl-propanes linked together by C-O-C and C-C linkages. However, these links are still not fully understood. Structural details differ from one source to another. The mechanical properties are lower than those of cellulose. The lignin is thermally stable but is responsible for the UV degradation. Composition of lignin in wood contains about of 2 to 15 %.

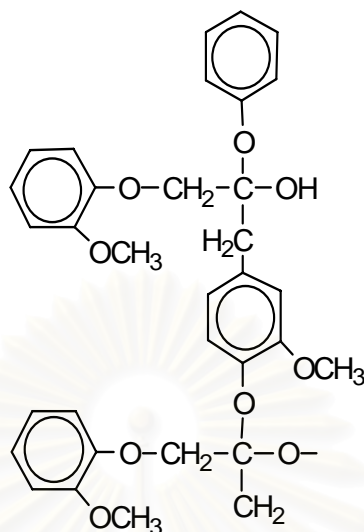


Figure 2.4 : Structure of lignin

(<http://www.fibersource.com>)

Pectin is a collective name for heteropolysaccharides, which consists essentially of polygalacturon acid. Pectin is soluble in water only after a partial neutralization with alkali or ammonium hydroxide.

Waxes make up the part of the fibres, which can be extracted with organic solutions. These waxy materials consist of different types of alcohols, which are insoluble in water as well as in several acids.

Table 2.4: Properties of *Hevea brasiliensis* (<http://www.mnflooring.com>)

Properties	Value
Density (g/cm ³)	0.56-0.64
Tangential Shrinkage Coefficient (%)	1.2
Radical Shrinkage Coefficient (%)	0.8
Hardness (N)	4350
Static Bending, N/mm at 12% MC	66
Modulus of elasticity, N/mm at 12% MC	9700

In Thailand, several kinds of wood or natural fibers are available and some are highly attractive as reinforcing filler as well as an extender for wood composites. Natural fiber sources such as sawdust of rubber wood, rice hull, flax, jute, etc., have been reported to be used in the formulation of wood composite compounds. Those fiber sources will be evaluated systematically for the processability and composite properties when compounded with PVC matrix.

2.5 Theory of the Action of Filler and Reinforcements

An interface is a dominant factor in the fracture toughness properties of composite materials and in their responses to aqueous and corrosive environments. Composite materials with weak interfaces have relatively low strength and stiffness but high resistance to fracture whereas materials with strong interfaces have strength and stiffness but are very brittle. In a simple system, bonding at an interface is due to adhesion between a fiber and a matrix. The action of an active filler can be attributed to several mechanisms. Some fillers form chemical bond with the material to be reinforced, such as carbon black produces crosslinking in elastomers by means of radical reaction. Other fillers act mainly through the volume they take up. The chain molecules of the polymer to be reinforced cannot assume all the conformational positions that are basically possible. Moreover, it can be assumed that in certain zones around the filler particles the polymeric phase differs in structures and properties from the polymer matrix. The polymer segments attached to filler surfaces by primary or secondary valence bonds, in turn, cause a certain immobilization of adjacent segments and, in some circumstances, permitting an orientation of the polymer matrix. An increase in glass transition temperature observed in filled polymers, resulting from the restricted mobility in the filler/polymer boundary zone, can be regarded as due to reduced conformation in this theoretical concept.

The zone adjacent to filler surface, whose structure might appear to be ordered, causes a stiffening of the material as a whole. Lower deformability and higher strength are also attributed to this composite nature. Uniform distribution of fillers is especially important, so that maximum polymer chains can be bound to the

free filler surface. The free surface energy and the polarity of the bond between the filler and the matrix are, thus, significant factors in this regard.

Another mode of action of active fillers arises from the fact that when the polymer molecules are subjected to mechanical stress with energy absorption mechanisms, they can slide off the filler surface. Impact energy can, therefore, be more uniformly distributed and impact strength increases as, for example, in the unplasticized PVC/calcium carbonate system. In some cases, microcraze, that destroys the impact energy at the filler/polymer interface, is also facilitated (Gachter and Muller, 1987).

2.6 Theories of Adhesion (Hall, 1981)

Adhesion can be attributed to five main mechanisms, which can occur at the interface either in isolation or in combination to produce the bond.

2.6.1 Adsorption and Wetting

When two electrically neutral surfaces are brought sufficiently close together, as illustrated in Fig.2.5(a), there is a physical attraction which is best understood by considering the wetting of solid surfaces by liquids, as shown in Fig.2.5(b), and by using the Young's equation.

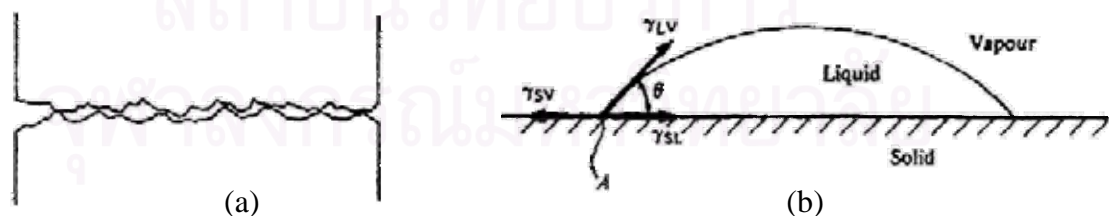


Figure 2.5: (a) Isolated contact points leading to weak adhesion between two rigid rough surfaces, (b) Surface tensions (γ) for a liquid drop on a solid surface (Hall, 1981).

Work of adhesion, W_{SL} , as shown by the Yong's equation

$$W_{SL} = \gamma_{LV}(1 + \cos\theta) = \gamma_{SV} + \gamma_{LV} - \gamma_{SL} \quad (1)$$

Where:

θ is the contact angle

γ_{SV} is the surface tensions of solid-vapor interfaces

γ_{SL} is the surface tensions of solid-liquid interfaces

γ_{LV} is the surface tensions of liquid-vapor interfaces

2.6.2 Mechanical Adhesion

Some bonding may occur purely by the mechanical interlocking of two surfaces as illustrated in Fig.2.6(a). The strength in shear may be very significant and depends on the degree of roughness.

2.6.3 Electrostatic Attraction

Forces of attraction occur between two surfaces when one surface carries a net positive charge and the other surface a net negative charge as in the case of acid-base interactions and ionic bonding and coupling agents laid down on the surface of the filler, as shown in Fig.2.6(b).

2.6.4 Chemical Bonding

A chemical bond is formed between a chemical grouping on the fiber surface and a compatible chemical group in the matrix as seen in Fig.2.6(c) and 2.6(d), respectively. The strength of the bond depends on the number and type of bonds and interface failure must involve bond breakage.

2.6.5 Interdiffusion

It is possible to form a bond between two polymer surfaces by the diffusion of polymer molecules on one surface into the molecular network of the other surface, as shown in Fig. 2.6 (e) and 2.6(f), respectively. The bond strength will depend on the amount of molecular entanglement and the number of molecules involved.

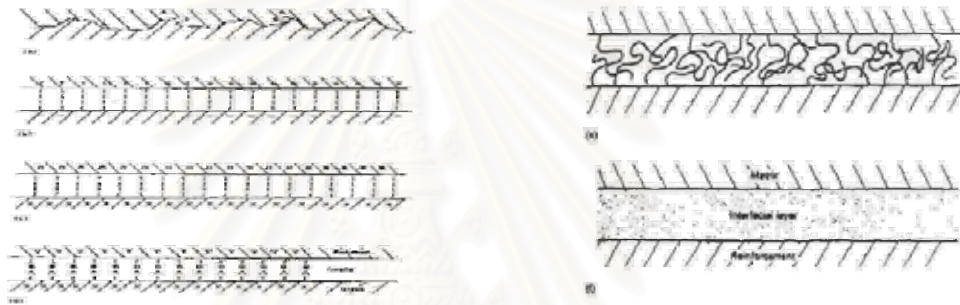


Figure 2.6: Mechanism of interfacial adhesion. Mechanical bond (a), Bond formed by electrostatic attraction (b), Chemical bond (c) and (d), Bond formed by molecular entanglement following interdiffusion (e) and Bond formed by interfacial layer by interdiffusion (f) (Hall, 1981).

2.7 Weathering Aging

The ability of a material to resist deterioration of its electrical, mechanical, and optical properties caused by exposure to light, heat, and water can be very significant for many applications. Wood-plastic lumber is being promoted as a low maintenance, high durability product. However, the use of wood-plastic composites by construction industry has resulted in concern about the durability of these products when exposed to outdoor environments. Ultraviolet (UV) durability is of particular concern. When exposed to natural weathering, wood-plastic composites experience color change and a loss in mechanical properties which results in changes in both the polymer matrix and the wood component.

2.7.1 Weathering of Wood

Weathering is the general term used to describe the degradation of materials exposed outdoors. This degradation occurs on the surface of all organic materials, including wood. The process occurs through photo-oxidation of the surface catalyzed by ultraviolet (UV) radiation in sunlight, and it is augmented by other processes such as washing by rain, changes in temperature, changes in moisture content, and abrasion by windblown particles. The weathering process can take many forms depending on the exposed material; in general, the process begins with a color change, followed by slow erosion (loss of material) from the surface. The surface initially develops slight checking; with some materials, deep cracks may ultimately develop. Weathering is dependent on the chemical makeup of the affected material. Because the surface of a material may be composed of many different chemicals, not all materials on the surface may erode at the same rate.

The surface of wood consists of four types of organic materials: cellulose, hemicellulose, lignin, and extractives. Each of these materials is affected by the weathering process in a different way. The extractives (that is, the material in the wood that gives each species its distinctive color) undergo changes upon exposure to sunlight and lighten or darken in color. With some wood species, this color change can take place within minutes of exposure. Changes in the color of the surface are accompanied by other changes that affect the wettability and surface chemistry of the wood. From 20% to 30% of the wood surface is composed of lignin, a polymeric substance that is the adhesive that holds wood celluloses together. Because lignin is affected by photodegradation more than are celluloses, lignin degrades and cellulose fibers remain loosely attached to the wood surface. Further weathering causes fibers to be lost from the surface (a process called erosion); but this process is so slow that on the average only about 6 mm (1/4 in.) of wood is lost in a century (Fig. 2.7). This erosion rate is slower for most hardwoods and faster for certain softwoods. Other factors like growth rate, degree of exposure, grain orientation, temperature, and wetting and drying cycles are important in determining the rate of erosion. Water-soluble colored extractives occur naturally in the heartwood of such species as

western redcedar, cypress, and redwood. These substances give the heartwood of some species their attractive color, water repellency, and natural decay resistance. However, discoloration of paint may occur when the extractives are dissolved and leached from the wood by water. The water carries the extractives to the painted surface, and then evaporates, leaving the extractives as a yellow to reddish brown stain on the paint. The water that gets behind the paint and causes moisture blisters also causes migration of extractives.

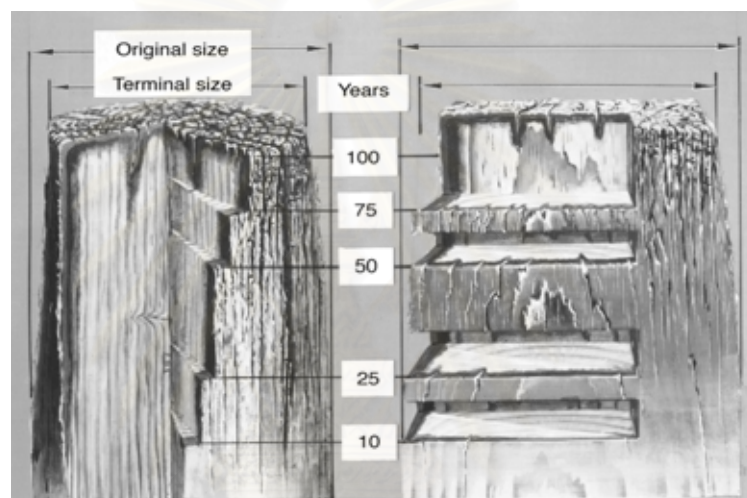


Figure 2.7: Artist's rendition of weathering process of round and square timbers. As cutaway shows, interior wood below surface is relatively unchanged (Reinhart et al., 1989).

2.7.2 Weathering of Poly(vinyl chloride) (Titow, 1984)

Weathering may be broadly defined as the overall changes in a material, brought about by outdoor exposure. In PVC the change is the combined effect of several factors, interacting and affecting the material in a highly complicated manner: thus from the technical standpoint the weathering of PVC is a composite phenomenon, complex in both its mechanism and nature. Some of the principal factors instrumental in this process are listed in Table 5, with indications of their main effects on PVC. However, it is a cardinal characteristic of weathering that the factors never operate singly in actual service conditions, but co-act and interact in many

ways: moreover their individual roles, and their contributions to the overall results, can vary considerably depending upon the place and conditions of exposure; seasonal variations at one and the same site also affect the progress of weathering.

Table 2.5: Some Factors Instrumental in the Natural Weathering of PVC, and Example of Their Main Effects (Titow, 1984).

Factor (environmental agent)	Typical action on PVC material	Main observable effects ^a	Remarks
Sunlight	Degradation of PVC polymer by UV component of the radiation Fading of colourants	1, 2, 3, 4, 5, 6, 7, 8	Direct sunlight is also a source of heat and hence can promote temperature effects
Temperature	Mainly effects of heat and temperature fluctuation, including: (i) Heat degradation (ii) Exudation and volatilization of components (iii) physical disruption by local and general stresses caused by temperature changes	1, 2, 3, 4, 5, 6, 8	Temperature effects can promote and enhance those of other agents
Water (rain, snow, hail, vapour, condensate)	(i) Mechanical erosion of surface (ii) Leaching out of components (iii) Mechanical disruption (iv) Chemical effects of pollutants dissolved in rain	1, 3 1, 8, 9 1, 5, 7, 8 1, 2, 4, 9	Effects aggravates by the action of other factors
Air	Oxidation of reaction sites in PVC polymer and some other components	1, 2, 4, 5, 6	Effects promoted and enhance by those of UV radiation
Atmospheric pollutants (in vapour, liquid and solid particle form)	(i) Leaching out of components (ii) Chemical reactions with the PVC polymer and possibly other components of the material (iii) Surface erosion	1, 2, 3, 4, 5, 6, 8, 9, 10	Effects can be accelerated by heat and sunlight

^a

1. dulling, marring and pitting of surface
2. cracking (in severe or advance cases)
3. stiffening
4. discoloration (yellow and darkening)
5. reduced strength

6. reduced extensibility
7. surface buckling or rippling
8. distortion
9. development of microporosity
10. environment stress cracking

2.7.3 Photochemical Degradation of PVC (Titow, 1984)

All the main external manifestations associated with thermal degradation of PVC appear also as a result of photolysis (i.e. dehydrochlorination, development of colour, and deterioration of properties). The situation can be further complicated where-as is often the case in service-exposure to light is associated with weathering: this introduces a number of further destructive factors (Table 5).

The dissociation energy of a carbon of carbon-chlorine bond is about 77kcal/mol: this corresponds to the energy of light of wavelength 375 nm. In theory, therefore, light of this wavelength (in the near-UV part of the spectrum) should be able to cause photodegradation of PVC. In practice the process requires higher photo energies (UV light of shorter wavelengths): it is known that the UV band between about 290 and 315 nm is mainly responsible for the photochemical degradation of plastics, including PVC. However, the mechanisms of this process in PVC, alone or part of weathering, are less well understood than those of thermal degradation: the main features indicated by available evidence may be briefly summarized as follows.

The first phase of photolysis of PVC in air appear to be photooxidation, proceeding through a free-radical mechanism and at a general rate proportional to the intensity of radiation (in the appropriate UV region) with the formation of hydroperoxide, keto, and aldehyde groups which undergo further light-induced reactions and breakdown: the presence of these hydrophilic groups is thought to play a part in the disruptive effects of atmospheric water in weathering of PVC materials. Dehydrochlorination also occurs at an early stage, but usually proceeds more slowly than in typical thermal degradation conjugated double bond sequences are formed but these are liable to be disrupted early in the process by reaction with oxygen (probably resulting in the formation of carbonyl groups, themselves light-absorbent and thus capable of accelerating the photolysis), which would counteract the formation of colour. Chain scission and cross-linked also take place. Exudation of plasticizers on weathering of PVC has been attributed to their reduced affinity for the cross-linked structures. Prior degradation by heat processing, can enhance and accelerate

photodegradation: this should be borne in mind in the formation and processing of PVC compositions for outdoor use. Impurities and other adventitious additives can also have an effect in this direction.



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CHAPTER III

LITERATURE REVIEW

Stokke et al. (2003) studied fundamental aspects of wood as a component of thermoplastic composites. They suggested that an important physical characteristic of wood affecting its interaction with thermoplastic polymers in composites is its hygroscopicity. Dry wood particles at ambient temperature and relative humidity contain from 5% to 12% moisture content (oven dry weight basis). Wood particles used in wood plastic composites need to be dried to 0 to 2% moisture content (“bone dry”) to process adequately with thermoplastic polymers. Dry wood particles are limited in thermal processing to a temperature of about 225°C before thermal decomposition occurs. Most of the common thermoplastics (polyethylene, polypropylene, polystyrene, and PVC) are easily processed with wood because the melt temperatures are below the thermal decomposition temperature of wood.

Mechanical Properties

One of the primary benefits of adding wood particles to thermoplastics is the increases in stiffness (modulus of elasticity) and strength (modulus of rupture) of the resulting composites. Depending on the wood loading, the wood plastic composite stiffness can be increased 3 to 4 times while the strength can be increased twofold. Impact resistance of thermoplastic composites is reduced depending on the type, amount, and treatment of wood fiber added to the composite.

Structure of fiber/plastic composites

Commercial formulations of PVC for extrusion contain resin, stabilizers, wetting agents and lubricants for ease of processing. The PVC additives also appear to have the added benefit of improved interaction with the wood particles during

extrusion, as evidenced by Figure 3.1. The PVC compound penetrates into the wood cell lumen. The filling of voids in the wood cell structure by the PVC can help impart better physical and mechanical properties to the composite.

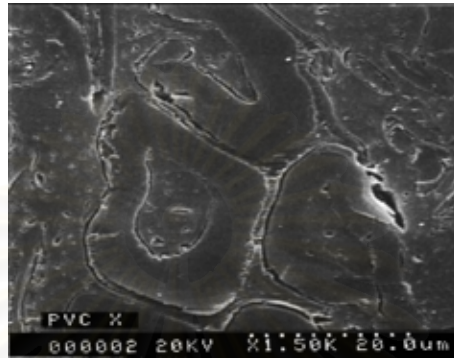


Figure 3.1: Scanning electron micrograph of PVC-Ponderosa pine composite (Stokke et al. 2003).

Sombatsompop et al. (2003) studied the effect of wood sawdust content on rheological and structural changes, and thermo-mechanical properties of PVC / sawdust composites. The results revealed that the tensile (Fig. 3.2), impact (Fig. 3.3), and flexural (Fig. 3.4) properties of the PVC/sawdust composites considerably decreased with up to 16.7 wt% sawdust content before leveling off for higher sawdust loadings. Thermal degradations of PVC in PVC/sawdust composites were evidenced by a decrease in decomposition temperature (Fig. 3.5). The glass transition temperature was found to improve with sawdust content. The overall results in this work suggest that the properties of PVC/sawdust composites were strongly influenced by sawdust content up to 16.7 wt%. Beyond this value the effect of sawdust content on the properties was comparatively small.

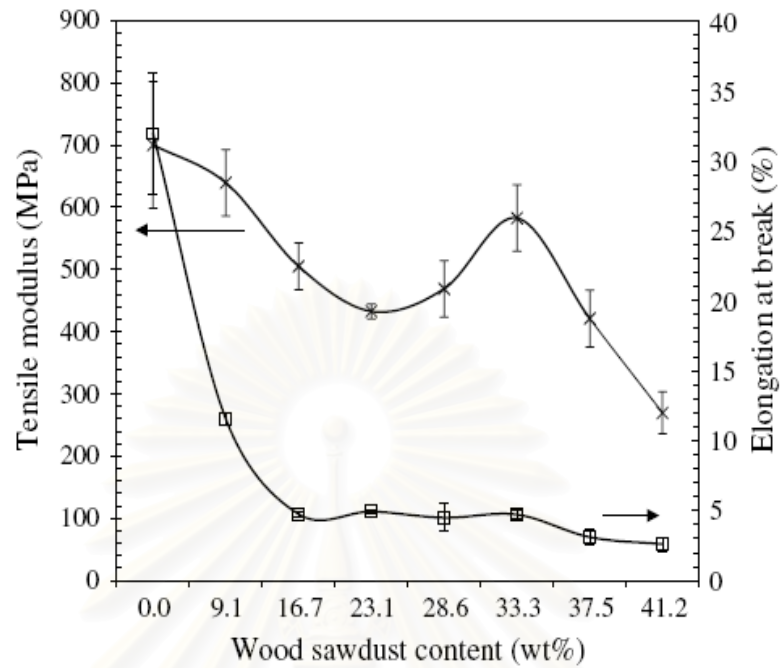


Figure 3.2: Plots of tensile modulus and elongation at break of PVC/sawdust composites (Sombatsompop et al., 2003).

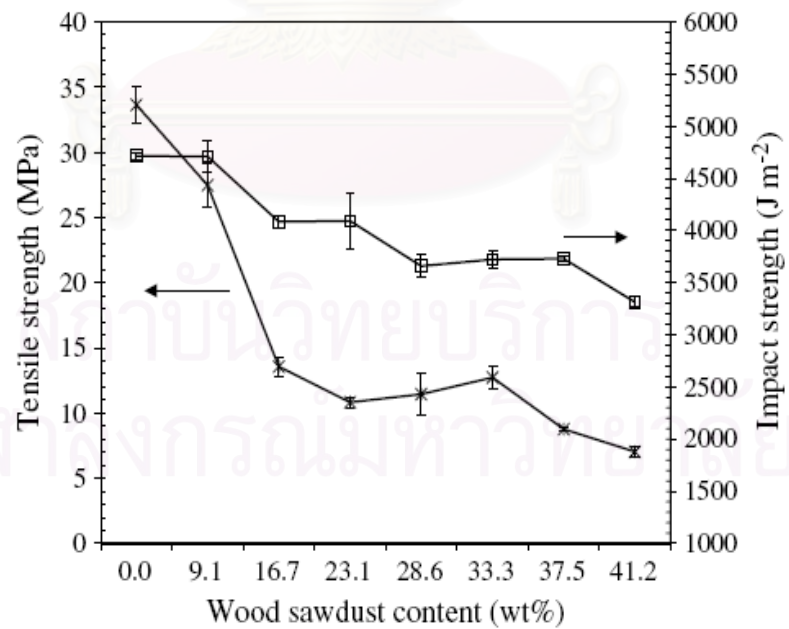


Figure 3.3: Variations of tensile and impact strength as a function of sawdust content (Sombatsompop et al., 2003).

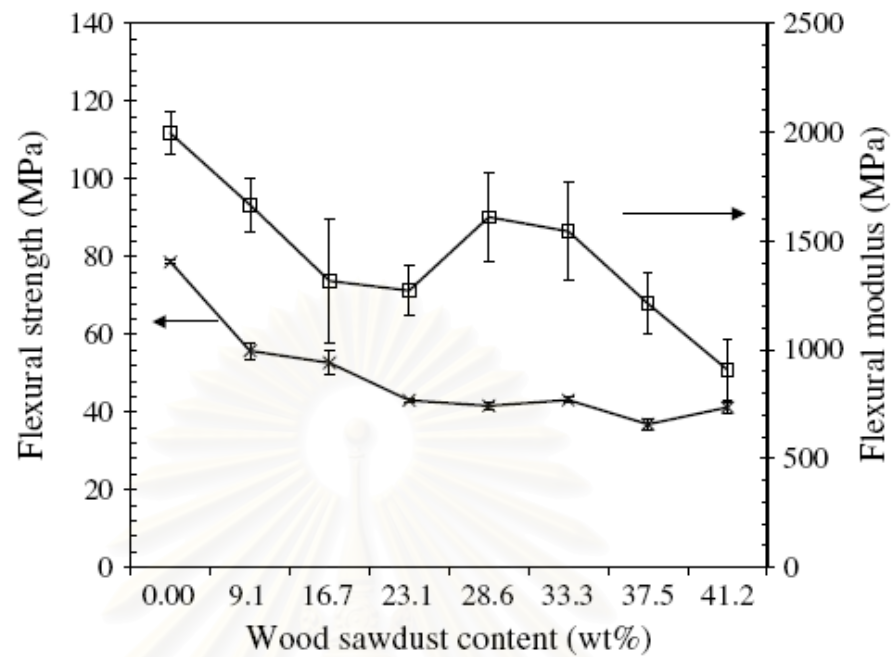


Figure 3.4: Flexural modulus and flexural strength of sawdust filled PVC for varying sawdust concentrations (Sombatsompop et al., 2003).

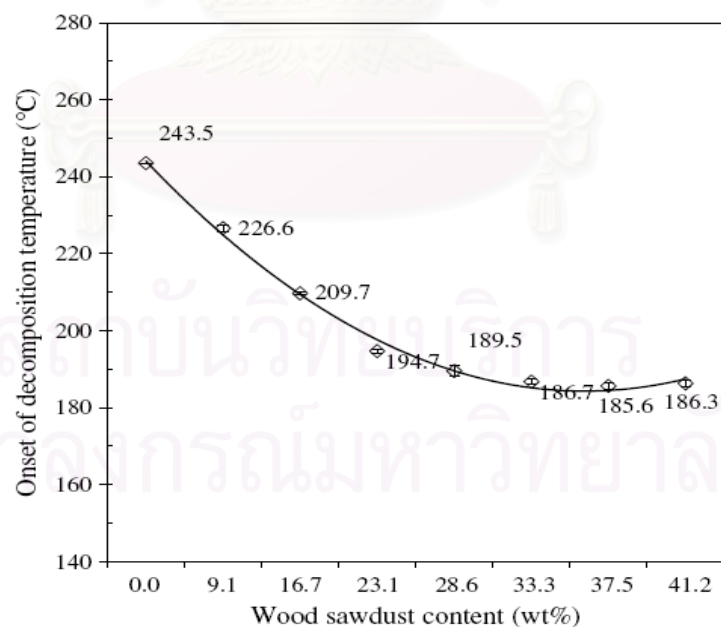


Figure 3.5: Variations in the onset of decomposition temperature with sawdust content (Sombatsompop et al., 2003).

Chetanachan et al. (2001) studied PVC wood, which includes PVC foam and PVC/wood flour composite, shows improved performance over wood in the following properties: termite resistance, weathering aging, less moisture absorption, and ease of installation. Termite resistance was evaluated by Wood Testing, it can be seen that termites totally damage standard rubber wood, which has poor termite resistance, after 4 months but do not damage PVC wood products within a 6-month period. Color change indicated that PVC wood undergoes less color change after aging for 12 months. The dimensional stability of PVC/wood flour after aging for 12 months is much better than that of wood. PVC wood has lower moisture absorption than three types of wood. PVC /wood flour gives a higher maximum load than either teak or rubber wood. PVC/wood flour shows a higher load, which indicates longer service life after installation than teak or rubber wood.

Sanadi et al. (1999) studies effects of particle size and interface on the dynamic and mechanical behavior of wood flour composites. Particle size and interparticular distance can influence the morphology and therefore the mechanical response of the system. Table 3.1 shows the mechanical properties of the uncoupled wood-flour (WF35 and WF235) and coupled (WF35 MAPP and WF235 MAPP) blends. The results reveal that the tensile properties of the 235-mesh composites were about 10 percent higher and unnotched Izod impact strength was about 70 percent higher than the 35-mesh composites. Because the surfaces of the two wood flours can be different from each other. As the wood flour size is reduced, new surfaces are exposed that will have different surface chemical structures. This could have some effect on the polymer morphology of the composites. Apart from the size differences that lead to different stress concentrations, the possibility exists that the interfacial strength is higher and/or the morphology of the interphase region between the WF235 particles and the matrix is tougher than the WF35 interphase. The difference in particle size and interparticular distances could result in different molecular morphologies that influence the composite behavior.

When comparing the two sizes of coupled wood flour, the tensile properties of the WF235 mesh were greater than the WF35 mesh, with the differences being about

29 percent and 19 percent for strength and modulus, respectively. Two factors need to be considered to explain the above results. First, the two sizes will have different surface properties since different regions of the cell walls are likely to be exposed. Second, the interparticular area will be smaller for the WF235 composites, leading to different morphologies of the matrix phase in the system.

Table 3.1: Mechanical properties of woodflour-polypropylene blends: effect of particle size (Sanadi et al., 1999).

Composite type	Tensile strength (MPa)	Tensile modulus (GPa)	Flexural strength (MPa)	Flexural modulus (GPa)	Izod unnotched (J/m)
40%wt WF35	21.8	3.2	38.7	2.7	54
40%wt WF235	24.3	3.5	41.4	2.9	91
40%wt WF35 MAPP	27.0	3.1	47.3	2.8	54
40%wt WF235 MAPP	34.8	3.7	56.0	3.2	81

Ichazo et al. (2001) studied the water absorption of polypropylene/wood flour composites. The concentration of WF in the composite for a constant particle size increases with increasing the water absorption. When the amount of WF is increased in the composite, its polar character increases and hence the water retention increases. The variation of water absorption with the particle size of the filler at 40% load is possible to observe that the water absorption is always slightly superior in larger particle size composites. This is a contradiction, because a larger surface area of particles should be expected at lower particle sizes and consequently, a higher availability of OH groups coming from cellulose that could absorb water. This discrepancy could be attributed to the general higher tendency of agglomeration of particles of small size.

Stark et al. (1997) studies effects of species, wood flour derived from species, ponderosa pine, loblolly pine, maple, and oak, was used as filler for polypropylene at 20, 30, 40, 50, and 60%wt of wood flour. This represents both softwoods (the pines) and hardwoods (the oaks and maples) from different regions of the United States. The hardwoods exhibited slightly higher values for flexural and tensile strength and modulus, higher heat deflection temperature, and lower percentage tensile elongation than did the softwoods. But specific gravity was species independent.

Table 3.2: Summary of performance of several species of wood flour-filled polypropylene at difference weight percentage (Stark et al., 1997).

Material	Izod impact Notched (J/m)	Tensile strength (MPa)	Elongation at Break (%)	Tensile modulus (GPa)	Flexural strength (MPa)	Flexural modulus (GPa)	Heat deflection (°C)
PND-20	15.4	26.5	5.65	1.99	41.6	1.89	69
PND-30	19	24.6	3.12	3.24	43.1	2.58	76
PND-40	20.8	25.5	2.29	3.71	44.2	3.22	85
PND-50	20.5	23.0	1.67	4.25	41.8	3.66	89
PND-60	21.1	20.1	1.44	4.56	38.8	4.04	91
LBL-20	12.4	24.9	4.83	2.14	40.6	1.71	64
LBL-30	12.7	23.7	3.45	2.53	41.2	2.28	72
LBL-40	13.7	21.4	1.76	3.28	39.3	2.84	78
LBL-50	13.8	19.7	1.32	3.97	37.1	3.40	79
LBL-60	10.4	17.8	0.92	4.32	34.1	3.81	77
MPL-20	12.8	27.9	4.11	2.87	46.2	2.00	69
MPL-30	15	27.1	3.26	3.33	46.5	2.47	88
MPL-40	16.5	25.6	2.03	4.72	4.4	3.23	104
MPL-50	17.7	24.0	1.44	5.20	42.1	4.16	111
MPL-60	17.9	19.9	1.13	4.77	38.0	4.35	110
OAK-20	14.6	27.2	4.66	2.48	44.1	1.83	74
OAK-30	17.5	25.7	2.43	3.81	45.9	2.87	98
OAK-40	18.6	25.2	2.09	4.19	44.8	3.39	100
OAK-50	20.9	23.4	1.51	4.79	42.8	3.99	112
OAK-60	18.8	19.8	1.20	5.05	38.1	4.60	114
PP	15	28.5	10.44	1.31	34.7	1.03	55

^aPND, ponderosa pine; LBL, loblolly pine; MPL, maple; OAK, oak; PP, unfilled polypropylene. Number refers to the weight percentage of filler in the composite.

Green et al. (2001) studied flexural properties of structural lumber products after long-term exposure to 150°F and 75% relative humidity. This paper presents results on permanent loss in flexural properties for solid-sawn and structural composites 2- by 4-ft. lumber, heated in air at 150°F and 75% relative humidity (RH), for periods of up to 6 years. The prolonged exposure to temperatures above 150°F should be avoided; when such exposures do occur, reductions in allowable properties should be made for both the permanent and immediate effects of temperature. Furthermore, permanent effects should be based on the cumulative time that the members will be exposed to temperature level over 150°F during the life of the structure and the strength losses associated with these levels. For temperature species, wood is composed of about 40-50 % cellulose, 20-35% lignin, and 12-35% hemicelluloses, plus extractives. When heated, these components are relatively stable up to about 212°F and up to 48 hours of heating. Chemical acid hydrolysis is the most typical degradation mechanism with the hemicelluloses being more sensitive to thermal degradation than cellulose or lignin. Because the hemicelluloses are composed of shorter chains of molecules and have a more branched structure, they are generally easier to hydrolyze by acids than is cellulose. Of the hemicelluloses, arabinose and galactose have been found to be especially sensitive to thermal degradation. As wood degrades, acetyl groups being lost from the chemical structure combine with available water to form acetic acid. This acid acts as a catalyst to further speed the rate of degradation.

Falk et al. (2000) studied effects of weathering on color loss of natural fiber thermoplastic composites. The results show data on the color loss of the natural fiber-plastic specimens were analyzed. A statistical evaluation of the test results for each formulation indicated that the variability in color fade was low. The effect of UV exposure on specimens containing wood flour, the PP-based composites faded at a more rapid rate than the HDPE-based composites and that the specimens with higher wood flour content faded somewhat more than the specimens with lower wood flour content. The color loss on the fibers in wood-thermoplastic composites was caused by the UV exposure.

Stark et al. (2002) studied photostabilization of wood flour filled HDPE composites. Ultraviolet exposure can lead to photodegradation, resulting in a change in appearance and/or mechanical properties after 250, 500, 1000, and 2000 hours of accelerated weathering. The photodegradation of polyolefins originates from excited polymer-oxygen complexes and is caused mainly by the presence of catalyst residues, hydroperoxide groups, carbonyl groups, and double bonds introduced during polymer manufacturing. Even in the absence of a significant amount of water absorption, small amounts of these impurities can be sufficient to induce degradation of the polymer. Degradation of polymers due to photooxidation has such undesirable effects as loss of strength, stiffness, and surface quality. Slowing down or stopping the reactions that are responsible for degradation is necessary for UV stabilization. Similarly, wood undergoes photodegradation. The individual components of wood; cellulose, hemicelluloses, lignin, and extractives, are all susceptible to photodegradation. Research has shown that the weathering of wood is a process that is confined to the surface of the wood, involving photo-induced breakdown of lignin to water-soluble reaction products. This leads to the generation of chromophoric functional groups such as carbonyls, carboxylic acids, quinones, hydroperoxy radicals, etc. Coating the surface of wood often disrupts the degradation process.

CHAPTER IV

EXPERIMENTAL

4.1. Materials

Polymeric matrix used in this study is polyvinylchloride grade 258RB obtained from Vinythai Public Company Limited., Thailand. It has K value of 58 and bulk density-compaction of 0.56 kg/l. The additives are Kane Ace 20 (Processing aids), SAK-WP08-NP (Heat stabilisers), Loixol G16 (Internal lubricant), and Calcium stearate (External lubricant) from Vinythai Public Company Limited., Thailand. The fillers used are rubber woodflour, mixed woodflour, and teak woodflour, having average particle sizes of <150, 150-180, 180-250, 250-450, and 600-850 μm . It is a waste material from a saw-mill factory in Thailand.

4.2 Equipments

- 4.2.1 Ball Mills
- 4.2.2 Sieve Shaker
- 4.2.3 Vacuum Oven
- 4.2.4 Two-Roll Mill with rolls diameter: 200mm and 3-Zone electric heating (Scientific, Labtech Engineering)
- 4.2.5 Compression Molder
- 4.2.6 Universal Testing Machine
- 4.2.7 Impact Tester
- 4.2.8 Dynamic Mechanical Analyzer (DMA)
- 4.2.9 Thermogravimetric Analyzer (TGA)
- 4.2.10 Scanning Electron Microscopy (SEM)
- 4.2.11 Vicat and HDT Tester
- 4.2.12 Ultra Scan Pro

4.3 Methodology

4.3.1 Preparation of Polymer Matrix and Additives

The polymer matrix and four additives for this research are polyvinylchloride (PVC), Kane Ace 20, SAK-WP08-NP, Loxiol G16, and Calcium stearate, respectively.

Table 4.1: Formula for PVC matrix.

Ingredient	Concentration (phr *)
PVC (Siamvic 258 RB)	100
Stabilizer (SAK-WP-08-NP)	4
Processing aid (Kane Ace PA 20)	6
External lubricant (Loxiol G16)	2
Internal lubricant (Calcium stearate)	0.5

(* phr = part per hundred parts of PVC resin)

PVC was blended with the additives in hot and cold mixer as followed:

Hot Mixing Steps

- Adding PVC resin and solid additives to the mixer.
- Mixing at low speed (400 rpm).
- Mixing at high speed (1200 rpm) until the temperature reaches 70°C.
- Adding Loxiol G16, mixing and heating up to 30°C.
- Discharging to a cold mixing.

Cold Mixing Steps

- Mixing until the temperature is decreased to 30°C.
- Discharging to a container.

4.3.2 Preparation of Woodflour

The woodflours for this research are rubber wood (*Hevea brasilienes*), mixed wood (*Xylia xylocarpa*), and teak wood (*Tectona grandis*). They were selected for this work because of their availability and these woods are widely grown in Thailand. The wood sawdust was crushed using a ball mill apparatus to reduce their average particle sizes to <150, 150-180, 180-250, 250-450, and 600-850 μm . The density of the woodflour determined using a gas pycnometer is 1.49 g/cm^3 (Jubslip et al., 2005). The woodflour was dried in an oven at 105°C for 24 hours to constant weight before use.

4.3.3 Preparation of Wood Composites

The PVC matrix of 337.5 g was blended with dried woodflour at 60, 120, 180 g as specified. The mass ratios of the woodflour contents with respect to pure PVC resin were 20 phr, 40 phr, and 60 phr, respectively. The obtained compound was then mixed in two-roll mill with 0.3 mm gap at 175°C for 5 min. The obtained compound was then compression-molded at the temperature of 170°C and pressure of about 1 MPa for 4 min. The dimension of the compression molded sheet is $200 \times 200 \times 4 \text{ mm}^3$ (W×L×D). The composite was then cut into test pieces for further mechanical properties evaluations.

4.4 Wood Composites Characterization

4.4.1 Mechanical Characterization

4.4.1.1 Flexural Property Measurement

Flexural modulus and flexural strength of the composites specimens were measured by a universal testing machine (Instron Instrument, model 5567) according to ASTM D790. Three-point bending test was carried out at room temperature at the crosshead speed of 1.2 mm/min with the support span of 64 mm.

The dimension of the specimen was $3.2 \times 12.7 \times 64 \text{ mm}^3$. Five specimens from each composites composition were examined and the average values were reported.

4.4.1.2 Tensile Property Measurement

Tensile modulus and tensile strength of the specimens were obtained by a universal testing machine (Instron Instrument, model 5567) according to ASTM D638. The test specimens are a dumbbell shape with a uniform thickness. They were tested using a crosshead speed of 5 mm/min with the pre-load of 100 N giving a straight tensile force. The tensile modulus defined as the ratio of stress to strain was determined from the initial slope of the stress-strain curve whereas the tensile strength is the ultimate stress. Five specimens from each composite were tested with the average values reported.

4.4.1.3 Izod Impact Property Measurement

Izod Impact strength of the specimens was obtained by Impact Tester (Yasuda) according to ASTM D256. Izod specimen is notched to prevent deformation of the specimen upon impact. Notched Izod Impact is a single point test from a swinging pendulum. The sample is clamped into the pendulum impact test fixture with the notched side facing the striking edge of the pendulum. The pendulum is released and allowed to strike through the specimen. ASTM impact energy is expressed in J/m. The test result is typically the average of 10 specimens. The dimension of the standard specimen was $64 \times 12.7 \times 3.2 \text{ mm}^3$. The dept under notch of the specimen was 10.2 mm.

4.4.2 Thermal Characterization

4.4.2.1 Dynamic Mechanical Analysis (DMA)

Thermal characteristic of composite specimen was examined using a dynamic mechanical analyzer (NETZSH, model. DMA242). The shape of the test

sample was rectangular with 55 mm length, 10mm width, and 2 mm thickness. The dual cantilever mode of deformation was used under a test temperature range from 30°C to 140°C with a heating rate of 3°C/min. The test amplitude and frequency were 30µm and 1Hz, respectively. The cooling process was achieved through liquid nitrogen. The glass transition temperature was obtained from the peak of the loss modulus.

4.4.2.2 Thermogravimetric Analysis (TGA)

Thermal decomposition behaviors of each specimen such as degradation temperature and weight loss the sample were determined by using a Perkin Elmer's TG/DTA thermogravimetric analyzer model SII Diamond. The experiment was performed under nitrogen purging with a constant flow of 100 ml/min. Sample mass of about 10 mg was heated using a linear heating rate of 20°C/min from 50°C to 1000°C. The degradation temperature (T_d) of the specimens was reported at 5 % weight loss.

4.4.2.3 Vicat Softening Temperature

The vicat softening temperature is the temperature at which a flat-ended needle penetrates the specimen to the depth of 1 mm under a specific load. The temperature reflects the point of softening to be expected when a material is used in an elevated temperature application. A test specimen was placed in the testing apparatus so that the penetrating needle rested on its surface at least 1 mm from the edge. A load of 10 N was applied to the specimen. The specimen was then lowered into a silicon bath at 23°C. The bath was raised at a rate of 50°C per hour until the needle penetrated 1 mm according to ASTM D1525.

4.4.2.4 Heat Distortion Temperature

Heat distortion temperature is defined as the temperature at which a standard test bar deflects a specified distance under a load. It is used to determine

short-term heat resistance. It distinguishes between materials that are able to sustain light loads at high temperatures and those that lose their rigidity over a narrow temperature range. The specimen was placed under the deflection measuring device. The specimen was then lowered into a silicone oil bath where the temperature was raised at 120°C per hour until they deflected 0.25 mm for ASTM D648 edgewise.

4.4.3 Interfacial Interaction

The interfacial interaction or the adhesion or between woodflour filler and polyvinylchloride polymer matrix was examined using SEM micrographs. The micrographs were obtained using Scanning Electron Microscope (Model JSM-5800LV, Jeol), at an acceleration voltage of 15 kV. The fracture surface of each specimen was coated with thin gold film, of which the thickness was between 10 to 20 nm, prior to obtaining the micrograph.

4.4.4 Water Absorption

Water absorption is used to determine the amount of water absorbed under specified conditions according to ASTM D570. The data sheds light on the performance of the materials in water or humid environments. For the water absorption test, the specimens are emerged in water at agreed upon conditions, often 23°C for 105 days. Specimens are removed, patted dry with tissue paper, and weighed. Water absorption is expressed as increase in weight percent. Percent Water Absorption = $[(\text{Wet weight} - \text{Dry weight}) / \text{Dry weight}] \times 100$. The dimension of the specimen was $3.2 \times 12.7 \times 64 \text{ mm}^3$.

4.4.5 Outdoor Weathering

Weathering conducted in accordance with this practice is used to evaluate the stability of plastic materials when they are exposed outdoors. The relative durability of plastics in outdoor use can be very differences depending on the location of the exposure because of differences in ultraviolet (UV) radiation, time of wetness,

temperature, pollutants, and other factors. It cannot be assumed, therefore, that results from one exposure in a single location will be useful for determining relative durability in a different location. The specimens to be tested were exposed in October of 2007 to outdoor weathering in Bangkok, in accordance with ASTM D1435, at an angle of 0° horizontal, and were removed periodically to perform the flexural properties and lightness index analyses every 5 days. The dimension of the specimens was 100×200×4 mm³. The lightness index of the specimens was obtained by ultra scan pro to measure color using the CIELAB color system. CIELAB is a three-dimensional color space measuring the lightness (L*) of the specimens. L* has range between 0 and 100 (black and white, respectively). An increasing in L* means the specimens are lighting. Color was measured for three replicate specimens.



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CHAPTER V

RESULTS AND DISCUSSION

The effects of particle loadings, particle sizes, and types of woodflour of PVC wood composites on the mechanical, thermal, morphological, weathering aging properties, and surface texture were studied. The wood composites were prepared by two-roll milling at 170°C for 5 min and compression molding at 170°C, 1 MPa for 4 min. The PVC wood composites were tested for the following properties i.e. tension, flexural, impact, and weathering aging properties. The microstructure of the fracture surface under flexural of the composites was observed by a scanning electron microscope. Thermal properties and water absorption of the wood composite samples were also investigated.

5.1 Effects of Particle Loadings of Woodflour Filled PVC Wood Composites.

5.1.1 Effects of Woodflour Loadings on Mechanical Properties of PVC Wood Composites.

5.1.1.1 Tensile Properties.

Tensile properties of the neat PVC and PVC wood composites were studied by using a universal testing machine. The effects of woodflour contents ranging from 0-60 phr on tensile properties of PVC wood composites are shown in Figure 5.1. The woodflour average particle size used in this figure is 150-180 μm and the type of woodflour used is mixed wood. Figure 5.1 shows the effect of particle loadings on tensile modulus and tensile strength of the PVC wood composites. The result was also summarized in Table 5.1. From the figure, tensile strength of the PVC wood composites was found to decrease with increasing woodflour content. Similar

behavior was also found by Sombatsompop et al. The tensile strength of the neat PVC was determined to be 46.2 MPa and systematically decreased with the woodflour content to approximately 39.6 MPa at 60 phr of the woodflour content. The slight decrease in the strength, e.g. as observed in PP/wood sawdust composites, is possibly caused by a number of reasons including: (i) poor dispersion of the fiber in the matrix due to interfiber hydrogen-bonding or increase in fiber loading, (ii) moisture pick-up causing the reduction in physical bonding between the filler and the matrix or acting as a lubricant between the two phases, and (iii) increase of interfacial defects and debondings (Sombatsompop et al., 2005). On the other hand, the tensile modulus of PVC wood composites was found to increase with increasing the woodflour content. The modulus of the neat PVC used in this experiment was approximately 2.45 GPa and the value was increased to about 4.54 GPa at the maximum woodflour content of 60 phr. The rigid woodflour particles were able to reinforce the PVC matrix with about 85% improvement in the modulus value. The similar improvement was reported in the systems of PP/woodflour (Sombatsompop et al., 2005). This is due to the fact that the woodflour is more rigid than the polyvinylchloride matrix used (Sombatsompop et al., 2003).

The elongation at break of the PVC wood composites as a function of the woodflour content is shown in Figure 5.2. The elongation at break of PVC wood composites was found to decrease considerably with the presence of woodflour at any level. The elongation at break of the PVC matrix used in this experiment was approximately 54% and the value was decreased to about 2.1% at the woodflour content of 20 phr. Beyond 20 phr, the elongation at break slightly decreased further to 1.2% at the maximum content of 60 phr. The considerable decrease of the elongation at break of our PVC wood composites with the presence of woodflour and the slight decrease further with increasing the woodflour content was probably caused by the presence of the rather rigid woodflour filler in the PVC matrix and the rather low interfacial interaction between the PVC matrix and woodflour as discussed in the composites' tensile strength. This low interfacial adhesion typically leads to the debonding and the initiation of void formation or cracks at the composite interface thus the low elongation at break of the specimens. This behavior was also

observed in the systems of PP-woodflour composites (Sombatsompop et al., 2005).

5.1.1.2 Flexural Properties.

The effects of particle loading of woodflour ranging from 0-60 phr on flexural properties of PVC wood composites are shown in Figure 5.3. The woodflour particle size used in this figure is 150-180 μm . The type of woodflour used in Figure 5.3 was mixed wood. The results were also summarized in Table 5.2. From this Figure, the flexural strength of the PVC wood composites was found to decrease with increasing the woodflour content. The flexural strength of the unfilled PVC was determined to be 74.3 MPa and decreased to approximately 55.6 MPa with an addition of 60 phr of the woodflour. The effect of woodflour content on the flexural strength of the composites tended to be similar to that of tensile strength as discussed earlier. The reasons of decrease in strength of PVC wood composites included moisture pick-up in woodflour, poor dispersion of the woodflour in PVC matrix, and increases of interfacial defects or debonding between polymer and woodflour (Sombatsompop et al., 2005). However, the flexural modulus of the PVC wood composites as a function of woodflour content was found to slightly increase with increasing the woodflour content. The modulus of the PVC matrix used in this experiment was approximately 2.8 GPa and the value was increased to about 4.4 GPa at the maximum woodflour content of 60 phr. The woodflour particles, having greater rigidity, are able to reinforce the PVC matrix with about 57% improvement in the modulus value. As expected, the addition of the woodflour has a similar effect on enhancement of both the flexural modulus and the tensile modulus of PVC wood composites due to the reasons described previously. The similar improvement was also reported in the systems of PP wood (Sombatsompop et al., 2005). The PVC wood composites were stiffened by filled woodflour in PVC matrix because filler is more rigid than the PVC matrix.

5.1.1.3 Impact Behaviors.

Figure 5.4 shows the effect of woodflour content ranging from 0 to 60 phr on Izod impact strength of the PVC wood composites. The woodflour particle size used in this experiment was 150-180 μm . The type of woodflour used in Figure 5.4 was mixed wood. Impact properties of the neat PVC and PVC wood composites were studied by Yasuda Impact Tester. The result was also summarized in Table 5.3. In this figure, the Izod impact strength of the PVC wood composites was found to decrease with increasing the woodflour content. The notch acts as a crack initiator; therefore, the notched Izod impact test is a measure of crack propagation. The Izod impact strength of the unfilled PVC was determined to be 8.8 kJ/m^2 and systematically decreased with the woodflour content to approximately 4.6 kJ/m^2 at the 60 phr of the woodflour content. The similar decrease was report by system of PP/wood of Sombatsompop et al. (Sombatsompop et al., 2005). The decrease in impact strength was probably due to the dilution effect. That was because, as the woodflour content was increased, the ductile portion (PVC matrix) automatically reduced, thus decreasing the composites toughness (Sombatsompop et al., 2003). The decreased of impact strength of natural fiber/polymer composites was usually derived from debonding and friction effects, especially for the composites when no fiber treatments were applied. It can be seen that the presence of woodflour greatly reduced the toughness of the PVC wood composites with an increase in the composite hardness. The results on Izod impact strength of PVC wood composites corresponded to these on tensile and flexural strengths. The same reasons of strength involve increases of interfacial defects and poor dispersion between PVC and woodflour.

5.1.2 Effects of Woodflour Loadings on Thermal Properties of PVC Wood Composites.

5.1.2.1 Effects of Woodflour Loadings on Glass Transition Temperature of PVC Wood Composites.

Glass transition temperature (T_g) of the neat PVC and PVC wood composites can be precisely determined by dynamic mechanical analyzer (DMA). The T_g of PVC wood composites at different filler contents (i.e. 0-60 phr) is shown in Figure 5.5. The result was also summarized in Table 5.4. The woodflour particle size used in this experiment was $<150 \mu\text{m}$. The type of woodflour used in Figure 5.5 was mixed wood. The glass transition temperature of PVC wood composites was determined from the peak of loss modulus in this figure. As shown in this figure, the glass transition temperature of neat PVC was about 86°C and the value tended to slightly increase while adding more content of woodflour. However, the significant effect of woodflour on glass transition temperature of PVC was not observed possibly due to a limit range of woodflour contents. The similar behavior was found by Sombatsompop et al. The slight enhancement in T_g due to the presence of woodflour in the PVC matrix might imply that the adding of woodflour has an effect on motion of molecular chains of PVC matrix. The PVC chains become more difficult to mobilize due to impediment of a more rigid woodflour particle. Additionally, the increase in T_g with adding woodflour may due to hydrogen bonds and the polar-polar interactions between the PVC matrix and those hydroxyl groups on the woodflour molecules as discussed in the work of Sombatsompop et al. (Sombatsompop et al., 2003).

5.1.2.2 Effects of Woodflour Loadings on Degradation Temperature of PVC Wood Composites.

Thermal stability of PVC wood composites is one of important parameters for processing and service condition of materials. The manufacture of such composites sometimes requires high temperature for mixing the fiber and the matrix

which can cause thermal degradation of the material, thus can generate undesirable effects on the final properties. The onset of decomposition temperature (T_d) of PVC wood composites as a function of different particle loading ranging from 0-60 phr is shown in Figure 5.6. The woodflour particle size used in this figure was $<150\ \mu\text{m}$ and the type of woodflour used was mixed wood. In the TGA thermograms, the neat PVC and PVC wood composites showed two main degradation steps in the range of 350-420°C and 420-500°C. The obtained thermogram is consistent with the observation by Marongiu et al. Their work presented an initial attempt to describe PVC thermal degradation through semi-detailed and lumped kinetic models. They reported that, in the first step, dehydrochlorination of PVC molecules would occur to form HCl and polyene (C=C) structures. In the second step, Cl had been released from the melt, followed by the rearrangement of the polyene molecules and through cyclization and cross-linking reactions, formed alkyl aromatic hydrocarbons and char residues (Marongiu et al., 2003). The corresponding degradation temperature at 5% weight loss is also listed in Table 5.4. This table reveals that woodflour contents have some negative effects on the degradation temperature of PVC wood composites. The degradation temperature of the unfilled PVC was determined to be 273°C and systematically decreased with the woodflour content to approximately 257°C at 60 phr of the woodflour. This behavior was also reported by e.g. Sombatsompop et al. The authors studied the effect of wood sawdust content on rheological and structure changes, as well as thermomechanical properties of PVC/sawdust composites. It was observed that the decomposition temperature of PVC slightly decreased with the amount of the woodflour. The decrease in the thermal stability with the increase of woodflour may be associated with a reformation of hydrogen bonds between the woodflour and PVC molecules due to hydroxyl groups in the wood structure to form a large number of hydrogen bonds between the macromolecules of the wood and the polymer (Sombatsompop et al., 2003). With the presence of moisture, these bonds might be destroyed and new hydrogen bonds with water molecules were formed during the processing. This reformation of hydrogen bonds between water molecules and woodflour was relatively large and was accelerated by the effect of heat during the TGA experiment, so that Cl atoms could be more easily liberated from the PVC molecules and form HCl gas, thus reducing the decomposition temperature. The

decrease in T_d can also be substantiated by an increase in polyene sequences (C=C) with increasing moisture and woodflour contents (Sombatsompop et al., 2004).

5.1.2.3 Effects of Woodflour Loadings on Vicat Softening and Heat Distortion Temperature of PVC Wood Composites.

The vicat softening temperature and heat distortion temperature are a useful measure of specimens' resistance to distortion under a give load at elevated temperature. These properties of a given plastic material are applied in many aspects of product design, engineering, and manufacture of products using thermoplastic components. However, the vicat softening temperature and HDT tests are a short-term test and should not be used alone for product design. Other factors such as the time of exposure to elevated temperature, the rate of temperature increase, and the part geometry all affect the performance. The effect of woodflour contents ranging from 0-60 phr on vicat softening temperature and heat distortion temperature (HDT) of PVC wood composites are shown in Figure 5.7. The woodflour particle size used in this figure was 150-180 μm and the type of woodflour was mixed wood. Both characteristic temperatures are also summarized in Table 5.4. From this figure, the vicat softening temperature and HDT of PVC wood composites were found to increase with increasing woodflour content. As shown in this figure, the vicat softening temperature of the neat PVC was determined to be about 79°C and the value was slightly increased to about 84°C at the woodflour content of 60 phr. Whereas the heat distortion temperature of the neat PVC was determined to be 71°C and also slightly increased with the woodflour content to approximately 74°C at the 60 phr of the woodflour. The addition of woodflour in the PVC matrix tended to enhance the vicat softening temperature and HDT of the polymer. These trends had also been observed in other wood composites such as that reported by Kuan et al. With added woodflour, it seems that the enhanced rigidity of PVC wood composites, which affected on vicat softening temperature and HDT. In addition, the woodflour has an effect on mobility of PVC chains due to obstruction of woodflour, and thus increased the vicat softening temperature and HDT. The similar improvement was also noticed in the glass transition temperature.

5.1.3 Effects of Woodflour Loadings on Water Absorption Properties of PVC Wood Composites.

The amount of water was absorbed by a composite material when immersed in water for a stipulated period of time. The ratio of the weight of water was absorbed by a material, to the weight of the dry material. All organic polymeric materials will absorb moisture to some extent resulting in swelling, dissolving, leaching, plasticizing and/or hydrolyzing, events which can result in discoloration, embitterment, loss of mechanical and electrical properties, lower resistance to heat and weathering and stress cracking (Gauthier et al., 1998). The effect of woodflour contents ranging from 0-60 phr on water absorption of PVC wood composites is shown in Figure 5.8. The woodflour particle size used in this figure was 150-180 μm . The type of woodflour used in Figure 5.8 was mixed wood. The water absorption experiment was performed up to specimen for 105 days. From this figure, PVC matrix absorbed water about 0.3% and increased with the woodflour content to approximately 5% at 60 phr of woodflour content. The results show that the water absorption of PVC wood composites was found to increase with increasing woodflour content. Similar behavior was reported by Blendzki et al. They studied influence of wood fiber treatment of PP composites on the moisture absorption. Water absorption by PVC wood composites was limited to sorption mainly by woodflour since the water uptake by PVC was only about 0.3% as seen in Figure 5.8. There are three main regions where the adsorbed water in the composites can reside: the lumen, the cell wall and the gaps between woodflour and PVC matrix. With an increase of woodflour content, the gaps between the two phases would increase as a result of the poorer dispersion of woodflour. Consequently, the degree of moisture absorption of the composite could significantly further increase. To develop composites with better mechanical properties and to decrease their moisture uptake, it is necessary to make the woodflour hydrophobic possibly by suitable treatment (Blendzki et al., 2005).

5.1.4 Effects of Woodflour Loadings on Surface Texture Properties of PVC Wood Composites.

Wood plastic composite (WPC) products have the look and workability of wood. For these reason, WPC products have gained steady by builders and consumers. In addition, the specific demands of customers also focused on the surface appearance such as a variety of surface texture and color of WPC for several applications. The surface texture of PVC wood composites at various particle loadings of woodflour is exhibited in Figure 5.9. The woodflour particle size used in this experiment was 600-850 μm of rubber wood. The surface texture of PVC wood composites was visualized and recorded using a digital camera. From the figure, different color and surface appearance of our PVC wood composites can be obtained by the variation in the woodflour contents. When the woodflour was added into the neat PVC matrix which is white and opaque in nature, the color of the obtained PVC wood composites became light brown to darker brown with the increase of the woodflour contents (i.e. 0 phr up to 60 phr).

5.2 Effects of Particle Sizes of Woodflour Filled PVC Wood Composites.

5.2.1 Effects of Woodflour Sizes on Mechanical Properties of PVC Wood Composites.

The effect of average particle sizes of <150, 150-180, 180-250, 250-450, and 600-850 μm on mechanical properties (i.e. tensile strength, tensile modulus, flexural strength, flexural modulus, and impact strength) of PVC wood composites is shown in Table 5.5. The woodflour content was 60 phr and the type of woodflour used in Table 5.5 was rubber wood. From this table, the mechanical properties of PVC wood composites were found to increasing with decreasing particle sizes of woodflour. Apart from the size differences that lead to different stress concentrations, the possibility exists that the interfacial strength is higher and the morphology of the

interphase region between the small particle size of 75 μm and the PVC matrix is tougher than the large particle size of 725 μm interphase (Sannadi et al., 1999). This behavior may attribute to the reinforcing effect of the woodflour particle on the PVC matrix. The added woodflour in to polar polymer can increase the mechanical properties of composites. Woodflour possesses a lot of hydroxyl group on its surface, that forming some hydrogen bonding with hydroxyl group and polar of PVC matrix. Hence, the compatibility between woodflour and PVC was enhanced by the hydrogen bonding; the surface of woodflour transfer was also improved by the physical bonding between filler and matrix (Kuan et al., 2006). It also implies that certain level of interfacial interaction exists between the filler and the matrix as both woodflour and PVC matrix exhibit some polarity in nature. As the wood flour size is reduced, new surfaces are exposed that will have different surface chemical structures. This could have some effect on the polymer morphology of the composites. The difference in particle size and interparticular distances could result in different molecular morphologies that influence the composite behavior (Sannadi et al., 1999).

5.2.2 Effects of Woodflour Sizes on Thermal Properties of PVC Wood Composites.

Thermal properties of PVC wood composites at difference particle sizes of woodflour are shown in Table 5.6. The woodflour content used in this experiment was 60 phr. The type of woodflour used in Table 5.6 was mixed wood. From Table 5.6, the glass transition temperature of PVC wood composites in average particle sizes of <150 to 600-850 μm of woodflour is ranging from 88°C to 90°C. The degradation temperature at 5 % weight loss of the PVC wood composites in average particle sizes of <150 to 600-850 μm of woodflour is ranging from 256 to 262°C under nitrogen atmosphere comparing with the value of 273°C of the neat PVC. The vicat softening temperature of PVC wood composites is about 85°C and heat distortion temperature of PVC wood composites is about 73°C of large woodflour size (i.e. 725 μm). It can be seen that the thermal properties of PVC wood composites slightly increased with decreasing particle size of woodflour. The increase was similar to the effect of particle loading. The amounts of surface area of wood particle were associated with hydroxyl

groups of woodflour which have effect on hydrogen bonds and polar-polar interactions between the PVC matrix and woodflour molecules. The small woodflour sizes have more surface areas than large woodflour sizes which have fewer amounts of hydroxyl groups. Therefore, the differences particle sizes of woodflour have effects on thermal properties of PVC wood composites.

5.2.3 Effects of Woodflour Sizes on Water Absorption Properties of PVC Wood Composites.

Woodflour contains numerous hydrogen groups (-OH), which are available for interaction with water molecules by hydrogen bonding. Woodflour interacts with water not only at the surface but also bulk. Water absorption by PVC wood composites is limited to sorption mainly woodflour since the water uptake PVC was only 0.3% for 105 days. The effect of particle size of woodflour ranging from <150 to 600-850 μm on water absorption of PVC wood composites is exhibited in Figure 5.11. The water was absorbed by specimen for 105 days. The water absorption of the large woodflour size in PVC wood composites was determined to be 7.14 % and systematically decreased with the small woodflour size in PVC wood composites content to approximately 5.42 % at 60 phr of the woodflour content. The results show that increase particle sizes of woodflour are being made to increase water absorption of PVC wood composites. The same trend reported by Ichazo et al. It is possible to observe that the water absorption is always slightly superior in larger particle size (i.e. 600-850 μm). This is contradiction, because a larger surface area of particles should be expected of lower particle sizes (i.e. <150 μm) and consequently, a higher availability of OH groups coming from cellulose that could absorb water. This discrepant could be attributed to the general higher tendency of PVC matrix around small particle size of woodflour which can absorb less water than water absorption of large particle size of woodflour. The PVC matrix was water absorbent barrier of PVC wood composites since water uptake PVC matrix was only 0.3%.

5.2.4 Effects of Woodflour Sizes on Interfacial Interaction of PVC Wood Composites.

Adhesion between the interface of woodflour fillers and the PVC matrix is one of the main factors determining most properties of their polymer composites. The influence of particle size of woodflour has been shown to influence the physical and mechanical properties of the wood composites such as their strength and Young's modulus. In practice, the interfacial interaction can be qualitatively examined using a scanning electron micrograph (SEM). SEM micrographs of fracture surface of our PVC wood composites with 20 phr of mixed woodflour are exhibited in Figure 5.12. The micrographs were also used to examine the presence of voids or degree of dispersion of the woodflour in the PVC wood composites. From Figure 5.12a, the SEM micrograph of the PVC wood composites reveals the relatively good dispersion of the woodflour (particle size of $<150\ \mu\text{m}$). In this figure, the PVC matrix was held fairly homogeneously with the wood particles with low level of the observable voids. On the other hand, the micrograph of the composite filled with 600-850 μm woodflour (Figure 5.12b) exhibits some distinct interfaces between the woodflour filler and the PVC matrix. From this figure, some particles had been pulled out off the matrix and a few had fractured. The observed phenomenon suggested that weak interfacial interactions between PVC matrix and woodflour particles caused a decrease in the properties of PVC wood composites.

5.2.5 Effects of Woodflour Sizes on Surface Texture of PVC Wood Composites.

The effect of average particle size of <150 , 150-180, 180-250, 250-450, and 600-850 μm on surface texture of PVC wood composites is shown in Figure 5.13. The woodflour content used in this figure was 60 phr and the type of woodflour used was mixed wood. Woodflour is added to PVC matrix generally to improved customer appeal such as workability, vary color, and surface texture to be like natural wood. Particle size of woodflour has an effect on texture surface of PVC wood composites. Small particle size ($<150\ \mu\text{m}$ and 600-850 μm) in PVC wood composites has the look

of plasticlike appearance but good mechanical-physic properties. PVC wood composites which were made from small woodflour size were relatively smooth and non-texture. On the other hand, the addition of large particle size (600-850 μm) of woodflour was provided good surface texture of PVC wood composites with poor strength and elasticity. This particle size of PVC wood composites has an effect on surface texture which has the look of wood grain in the specimens.

5.3 Effects of Types of Woodflour Filled PVC Wood Composites.

All wood is composed of cellulose, lignin, hemicelluloses, and minor amounts (5% to 10%) of extraneous materials contained in a cellular structure. Variations in the characteristics and volume of these components and differences in cellular structure make woods heavy or light, stiff or flexible, and hard or soft. The properties of a single species are relatively constant within limits; therefore, selection of wood by species alone may sometimes be adequate. However, to use wood to its best advantage and most effectively in engineering applications, specific characteristics or physical properties must be considered (Simpson and Tenwolde, 1999). The types of wood in this research were rubber wood, mixed wood, and teak wood.

Rubber wood (*hevea brasiliensis*) has proven to be very versatile in its use in furniture manufacturing and the wood-based panels industry. The natural color of rubber wood is one of the principal reasons for its popularity. It has good overall woodworking and machining qualities for sawing, boring, turning, nailing and gluing. It also takes finishes and stains well. Its strength and mechanical properties are comparable to traditional timbers used for furniture making and woodworking. Modulus of elasticity of rubber wood is 9.24 GPa (<http://www.108wood.com>).

The wood of *Xylia xylocarpa* is very heavy and dimensionally stable as indicated by a low to moderate rate of shrinkage. The dark red-brown, hard and durable wood is termite resistant and can be used for heavy constructions, furniture, boats, railway sleepers, carving and household implements. The wood properties of planted *Xylia xylocarpa* were almost comparable to a number of more mature local

popular heavy hardwood species, indicating its suitability for heavy construction uses. The modulus of elasticity (MOE) was 12.9 GPa (Josue, 2004).

Teak wood (*Tectona grandis*) occurs in commercial quantities in India, Burma, Thailand, Laos, Cambodia, North and South Vietnam, and the East Indies. The heartwood varies from yellow–brown to dark golden–brown and eventually turns a rich brown upon exposure to air. Teak wood has a coarse uneven texture (ring porous), is usually straight grained, and has a distinctly oily feel. The heartwood has excellent dimensional stability and a very high degree of natural durability. Teak is generally worked with moderate ease with hand and machine tools. Teak is one of the most valuable woods, but its use is limited by scarcity and high cost. Teak is currently used in the construction of boats, furniture, flooring, decorative objects, and decorative veneer. The mechanical properties of teak wood have modulus of elasticity of 9.4 GPa (Josue, 2004).

5.3.1 Effects of Woodflour Types on Mechanical Properties of PVC Wood Composites.

The mechanical properties of PVC wood composites using various types of woodflour including tensile strength, tensile modulus, elongation at break, flexural strength, flexural modulus, and impact strength were examined in this report. The particle size and loading used were fixed at 150-180 μm and 60 phr. Three types of woodflour listed in Table 5.7 were rubber wood, mixed wood, and teak wood. For all the woodflour types, tensile strength, elongation at break, flexural strength, and impact strength were observed to decrease with increasing woodflour contents, while tensile modulus and flexural modulus were found to increase with the filler loading. The results revealed that the mechanical properties of PVC wood composites using three different woodflour types were similar. The same behavior was also found by Stark et al. possibly because the three woodflour used in this research is hardwood which has similar mechanical properties thus shows the same effect on mechanical properties of the obtained PVC wood composites. Additionally, the morphological properties of PVC wood composites with different woodflour types, which are shown

in Figure 5.14, revealed a similarity in dispersion and interfacial interaction of the woodflour filler in the PVC matrix (Stark et al., 1997).

5.3.2 Effects of Woodflour Types on Thermal Properties of PVC Wood Composites.

The HDT represented one of the major thermal properties of PVC wood composites for the identification of their engineering service temperature. The effect of different wood types in the PVC wood composites on their HDT is listed in Table 5.8. The particle size used in Table 5.8 was 150-180 μm and particle loading was 60 phr. From this table, the HDT of PVC wood composites using rubber wood, mixed wood, and teak wood were 85°C, 84°C, and 84°C, respectively. Using three different wood types in our work, HDT of the PVC wood composites was found to be similar. Other thermal properties of PVC wood composites at different types of woodflour also showed similar trends as the HDT results. Interfacial interaction between PVC matrix and woodflour, and morphology of PVC wood composites were very similar for different woodflour types. They can be seen in the SEM micrographs in Figure 5.14 using woodflour size of <150 μm and at the loading of 20 phr.

5.3.3 Effects of Woodflour Types on Water Absorption Properties of PVC Wood Composites.

The natural origin of woodflour and its structure involves particularities that will affect the preparation and properties of its composites (Gauthier et al., 1998). The first problem of wood is water absorption that has effect on the mechanical properties of the composites. Therefore, this experiment studied the effect of woodflour types on water absorption of PVC wood composites in order to select suitable types of wood with low water uptake for PVC wood composites fabrication. The water absorption of PVC wood composites as a function of woodflour types is shown in Table 5.9. The water absorption experiment was performed up to 105 days. From the table, the water absorption of PVC wood composites was found to be slightly different with different types of woodflour. The water absorption of PVC

wood composites based on rubber wood, mixed wood, and teak wood were determined to be 6.97%, 5.42%, and 4.83%, respectively using the same particle size of 600-850 μm . The result suggested that the use of teak woodflour to make PVC wood composites rendered lower absorbed water than rubber woodflour and mixed woodflour. This may be due to the fact that teak wood has natural oils that can protect water absorption into wood. Teak wood suitable for use in exposed locations, where it is durable even when not treated with oil or varnish (Simpson and Tenwolde, 1999).

5.3.4 Effects of Woodflour Types on Surface Texture of PVC Wood Composites.

From the previous sections, the types of woodflour were found to exhibit negligible effect on the mechanical and thermal properties of the PVC wood but can influence the color and surface appearance of the obtained PVC wood composites as well as their water absorption characteristics. The effect of wood types on texture surface and color of the obtained PVC wood composites is shown in Figure 5.15 at woodflour contents of 60 phr and average particle size of 600-850 μm . Table 5.10 shows the lightness values of the PVC matrix and its wood composites from the three types of woodflour. The lightest color of the composites was obtained when using rubber wood and the darkest color using mixed wood. The natural color of rubber wood is initially pale cream to yellowish brown, but after kiln drying and exposure to light, the wood turned darker with brownish and pinkish tints. The sapwood and heartwood appeared to show similar changes and cannot be easily separated. The vertically running vessels give the grain texture a coarse appearance (<http://www.carbinetmarkeonline.com>). Naturally, teak wood exhibits a wide range of colors when fresh cut. The natural color of teak wood is dark golden-yellow, turning brown and dark brown with age. Sapwood is white to pale yellowish brown, narrow to medium wide. The color and the markings of the wood vary considerably with localities. Teak wood from Thailand is golden brown in color, and giving the timber an extremely handsome appearance (<http://www.woodltd.com>). Finally the natural color of mixed wood is uniform reddish-brown with a few markings or faintly veined with darker lines. The wood turns to dark reddish-brown with aging. Sapwood

is pale reddish-white and narrows (<http://www.biz-supply.com>). Those discrepancies thus yielded the PVC wood composites of different colors and texture.

5.4 Characteristics of PVC Wood Composites on Weathering Aging Properties.

5.4.1 Effect of Woodflour Loadings, Woodflour Sizes, and Woodflour Types on Decrease in Flexural Properties of PVC Wood Composites after Outdoor Exposure.

Part of the reason for the rapid growth that WPCs lumber is promoted as a low maintenance and high durability product. However, its use by the construction industry has results in about exterior durability. WPCs exposed to natural weathering may experience color change, which affects their aesthetic appeal, as well as mechanical properties loss, which limited their performance (Stark and Matuana, 2006). Change in mechanical properties of materials after weathering can be due to combination of factors, such as composites surface oxidation, change in chain of matrix, and interfacial degradation caused by radiation, temperature, oxygen, moisture absorption, and contaminates. Table 5.11 lists percentage of decrease in flexural properties as function woodflour content (i.e. 0-60 phr), woodflour sizes, and woodflour types after 720 h (1 month) weathering. The woodflour particle sizes used in this experiment were <150, 150-180, 180-250, 250-450, and 600-850 μm . The types of woodflour used in table 5.11 were rubber wood, mixed wood, and teak wood. The specimens were exposed to natural weathering in Bangkok during October to November 2007. The results show that the natural weathering of either the PVC matrix or its wood composites provided a decrease in their flexural strength and modulus. The same behavior was also found by Stark et al. The decrease in flexural strength and modulus of the neat PVC were determined to be 2% and 10%, respectively and systematically increased with the woodflour content to approximately 7% of decrease in flexural strength and 20% of decrease in flexural modulus at 60 phr and 600-850 μm of the woodflour. These decreases in flexural

properties were observed in HDPE/wood sawdust composites exposed to xenon-arc radiation. Exposure can result in the changes in both the polymer matrix and the wood component. The exposed to water degrades mechanical properties of WPCs, mainly due to swelling particles cause microcracks in the matrix, causing a decrease in modulus, and reduces efficiency of stress transfer from fiber to matrix, causing a decrease in the strength (Stark and Matuana, 2006). Additionally, woodflour undergoes photodegradation which primarily occurs in lignin at the wood surface and results in cellulose-rich surface. As wood undergoes Photodegradation, the cellulose-rich oxidized surface becomes more wettable (Kalnins et al., 2002). Real et al. studied four difference types of TiO₂ pigmented PVC formulations, containing CaCO₃ and various additives, which designed for outdoor application which were submitted to artificial accelerated aging in xenon light, without and with spray of water, and to natural exposed in Libon. The influence of the combined action of water, temperature and radiation in the oxidation aging was evaluated by means of molecular analysis, using infrared spectroscopy, and in terms of evolution of the aesthetic properties, using scanning spectroscopy and colorimetry. They reported that the results from infrared and ultraviolet-visible spectrometry showed that there is good similarity between artificial aging with cyclic water spray and natural weathering of such PVC materials. During weathering, the photo-oxidative aging results from the combined action of atmospheric agents in the PVC matrix, certainly with important participation of the rain (Reinhart and Clements, 1989). In addition, the chain scission may be an important mode of PVC degradation when the increase temperature which is elevated by heat of natural weathering. The chain scission occurs during exposure, as evidenced by the growth of polyene group and Cl atoms, they expected that hydrogen chloride would increase. These effects contribute to the general deterioration in properties.

5.4.2 Effect of Woodflour Loadings, Woodflour Sizes, and Woodflour Types on Change in Lightness (ΔL^*) of PVC Wood Composites after Outdoor Exposure.

Ultraviolet (UV) had effects on unfilled PVC and PVC wood composites by changing color of specimens. PVC wood composites have been tested for color fade after natural weathering aging. The weathering aging was tested by outdoor weathering in Bangkok. The specimens were exposed to natural weathering for one month during October to November 2007. Particle loading of woodflour in this experiment was ranged from 0-60 phr. The woodflour average particle sizes used in this experiment were <150, 150-180, 180-250, 250-450, and 600-850 μm . And the types of woodflour used were rubber wood, mixed wood, and teak wood. The easy way to determine the evolution of the color of the specimen after weathering process consists of graphical representation of the lightness index. The evolution of parameter L^* is alternative representation of the lightness index. The results show a significant change in lightness (L^*) of the neat PVC and the PVC wood composites. The effect of woodflour content of PVC wood composites on change in lightness is shown in Figure 5.16. The change in lightness of PVC wood composites was found to decrease with increasing woodflour content. The results show that the change in lightness of the neat PVC was determined to be -2.99% and systematically decreased with the woodflour content to approximately 40.54% at 60 phr of woodflour content. The lightness index of neat PVC was decreased from initial lightness index by weathering due to the color of PVC matrix was yellow after exposure. The color of the PVC matrix changes that were observed during the initial stages of natural weathering, consisted of brief initial whitening, followed by a recovery of the initial appearance and subsequent yellowing (Real et al., 2005). The yellowing of the neat PVC results from the photo-oxidation of conjugated double bonds ($\text{C}=\text{C}$) formed during the weathering of the polymer. Moreover, PVC wood composites are detrimentally affected by UV radiation and the presence of moisture, primary because of wood component. Wood also undergoes photodegradation. Degradation primary occurs in lignin at the wood surface and results in cellulose-rich surface. As wood undergoes photodegradation, the color of wood was faded due to lignin in the wood was damage

(Kalnins et al., 2002). Therefore, the discoloration of PVC wood composites occurred at woodflour filler of the composites after natural weathering.

The effect of particle sizes of woodflour on the change in lightness of PVC wood composites is shown in Figure 5.17. As a result, the change in lightness of PVC wood composites was increased with decreasing of woodflour sizes. From this Figure, the change in lightness of PVC wood composites at particle size of $<150\ \mu\text{m}$ and $600\text{-}850\ \mu\text{m}$ was determined to be 46% μm and 32%, respectively after weathering for 1 month. Because the small woodflour size ($<150\ \mu\text{m}$) have more surface areas of exposure than the large woodflour size ($600\text{-}850\ \mu\text{m}$). The change in lightness of PVC wood composites is affected by UV radiation due to the specimens have primarily wood component. The surfaces of woodflour also undergo photodegradation which the woodflour was loosen color after weathering. Therefore, PVC wood composites were higher faded by UV radiation at the small woodflour size.

Further, Figure 5.18 shows the effect of wood types on change in lightness of the PVC wood composites. The woodflour particle size and particle loading used in this experiment was $600\text{-}850\ \mu\text{m}$ 60 phr, respectively. The types of woodflour used in this figure were rubber wood, mixed wood, and teak wood. In this Figure, The change in lightness of PVC wood composites with adding rubber wood, teak wood, and mixed wood was determined to be 37.20, 34.17, and 31.82, respectively. The change in lightness of PVC wood composites was found to most fading with adding rubber wood after weathering for 1 month. As a result, PVC wood composites were generally more light brown with adding rubber wood than teak wood and mixed. In other hand, PVC wood composites with adding mixed wood was less changed color due to the specimen was more dark brown than rubber wood and teak wood. Therefore, the initial color of PVC wood composites has effect on color appearance of the specimens after natural weathering.

5.4.3 Effect of Woodflour Sizes on Change in Surface of PVC

Wood Composites after Outdoor Exposure.

SEM micrographs of the surface of PVC wood composites after 30 days of exposure at woodflour content of 60 phr, particle size of 75 μm and 725 μm and woodflour type of mixed wood were shown in Figure 5.19. The SEM micrographs of Figure 5.19a and Figure 5.19c reveals that the surface of PVC wood composites at <150 μm and 600-850 μm before exposure and Figure 5.19b and Figure 5.19d show surface cracking of PVC wood composites after exposure at <150 μm and 600-850 μm of woodflour, respectively. In PVC wood composites, swelling and shrinking of wood particles after absorbing and desorbing moisture apparently resulted in voids at the PVC wood composites interface (Stark et al., 2004). Surface cracking and destruction of interfacial properties continued as weathering time increased. From Figure 5.19b, SEM micrograph show smaller microcracks in surface of PVC wood composites than Figure 5.19d. Because the large wood flour size of PVC wood composites more absorbed water the small woodflour size as followed by Figure 5.11. The wood is a naturally hydrophilic material. As a result, wood cell walls swell when penetrated by water, facilitating deeper light penetration and providing sites for further degradation (Hon, 2001). In addition, some water-soluble extractives are removed. In PVC wood composites, swelling of the woodflour compromises the wood/matrix interface and creates microcracks in the matrix (Rangaraj and Smith, 2000). The result is a change in appearance and deterioration of mechanical properties.

Table 5.1: Tensile Properties of PVC Wood Composites at Various Woodflour Contents.

Woodflour Content (phr)	Tensile Strength (MPa)	Tensile Modulus (GPa)	Elongation at Break (%)
0	46.2 ± 1.1	2.45 ± 0.03	54.0 ± 0.7
20	42.8 ± 0.8	3.15 ± 0.06	2.1 ± 0.1
40	40.2 ± 1.1	3.38 ± 0.14	1.4 ± 0.1
60	39.9 ± 0.5	4.54 ± 0.18	1.2 ± 0.1

Table 5.2: Flexural Properties of PVC Wood Composites at Various Woodflour Contents.

Woodflour Content (phr)	Flexural Strength (MPa)	Flexural Modulus (GPa)
0	74.3 ± 0.8	2.79 ± 0.11
20	73.2 ± 0.2	3.68 ± 0.08
40	56.5 ± 1.8	4.02 ± 0.15
60	55.6 ± 1.9	4.35 ± 0.24

Table 5.3: Izod Impact Strength of PVC Wood Composites at Various Woodflour Contents.

Woodflour Content (phr)	Izod Impact Strength (kJ/m ²)
0	8.3 ± 1.6
20	5.4 ± 0.2
40	5.2 ± 0.5
60	4.6 ± 0.2

Table 5.4: Thermal Properties of PVC Wood Composites at Various Woodflour Contents.

Woodflour Content (phr)	Glass transition Temperature (°C)	Degradation Temperature (°C)	Vicat Softening Temperature (°C)	Heat Distortion Temperature (°C)
0	86	273	79	71
20	87	261	80	73
40	89	258	82	73
60	88	256	84	74

Table 5.5: The Mechanical Properties of PVC Wood Composites at Various Particle Sizes of Woodflour.

Particle Size (μm)	Tensile Strength (MPa)	Tensile Modulus (GPa)	Flexural Strength (MPa)	Flexural Modulus (GPa)	Izod Impact Strength (kJ/m^2)
<150	40.7 ± 1.5	4.23 ± 0.08	50.1 ± 1.0	3.91 ± 0.09	4.9 ± 0.1
150-180	38.8 ± 0.5	4.15 ± 0.04	47.6 ± 0.3	3.79 ± 0.06	4.6 ± 0.2
180-250	34.7 ± 0.5	3.59 ± 0.09	45.2 ± 2.9	3.71 ± 0.32	4.6 ± 0.4
250-450	31.4 ± 0.8	3.19 ± 0.07	43.4 ± 0.4	3.61 ± 0.01	3.9 ± 0.6
600-850	30.4 ± 0.9	2.95 ± 0.08	39.2 ± 2.7	3.55 ± 0.03	3.7 ± 0.4

Table 5.6: Thermal Properties of PVC Wood Composites at Various Particle Sizes of Woodflour.

Particle Size (μm)	Glass transition Temperature ($^{\circ}\text{C}$)	Degradation Temperature ($^{\circ}\text{C}$)	Vicat Softening Temperature ($^{\circ}\text{C}$)	Heat Distortion Temperature ($^{\circ}\text{C}$)
<150	88	257	85	73
150-180	90	258	84	74
180-250	90	262	84	74
250-450	90	260	83	75
600-850	90	263	85	73

Table 5.7: Tensile Modulus of PVC Wood Composites at Various Woodflour Types.

Specimen	Tensile Modulus (GPa)
Rubber wood/PVC	4.15 ± 0.04
Mixed wood/PVC	4.54 ± 0.18
Teak wood/PVC	4.45 ± 0.08

Table 5.8: Heat Distortion Temperature of PVC Wood Composites at Various Woodflour Types.

Specimen	Heat distortion Temperature (°C)
Rubber wood/PVC	75
Mixed wood/PVC	74
Teak wood/PVC	74

Table 5.9: Water Absorption of PVC Wood Composites at Various Woodflour Types.

Specimen	Water Absorption (%)
Rubber wood/PVC	6.97
Mixed wood/PVC	5.42
Teak wood/PVC	4.83

Table 5.10: The Lightness of PVC Wood Composites at Various Woodflour Types.

Specimen	Lightness
PVC matrix	86.4
Rubber wood/PVC	43.7
Teak wood/PVC	30.2
Mixed wood/PVC	19.8

Table 5.11: Decrease in Flexural Strength of PVC Wood Composites after Weathering.

Woodflour Size/Content ($\mu\text{m}/\text{phr}$)	Flexural Strength Before Weathering (MPa)	Flexural Strength After Weathering (MPa)	Decrease in Flexural Strength (%)
0	79.7 ± 2.5	78.3 ± 1.7	1.86
<150/20	71.9 ± 2.3	69.2 ± 1.8	3.69
<150/40	68.2 ± 1.7	65.3 ± 1.5	4.29
<150/60	47.7 ± 1.5	44.6 ± 1.4	6.52
150-180/20	77.9 ± 1.4	75.6 ± 1.9	3.09
150-180/40	53.2 ± 1.4	50.5 ± 2.5	5.17
150-180/60	69.7 ± 1.7	66.2 ± 1.6	4.96
180-250/20	74.3 ± 1.9	71.3 ± 2.1	4.09
180-250/40	61.3 ± 2.4	57.9 ± 2.0	5.51
180-250/60	75.7 ± 2.2	71.1 ± 2.7	6.15
250-450/20	69.9 ± 1.9	65.9 ± 1.4	5.77
250-450/40	60.3 ± 1.3	55.6 ± 1.2	7.78
250-450/60	48.2 ± 1.2	43.1 ± 1.1	10.63
600-850/20	65.6 ± 1.8	62.4 ± 1.6	4.92
600-850/40	58.5 ± 1.5	53.4 ± 1.9	8.67
600-850/60	71.9 ± 2.3	66.7 ± 1.6	7.17

Table 5.12: Decrease in Flexural Modulus of PVC Wood Composites after Weathering.

Woodflour Size/Content ($\mu\text{m}/\text{phr}$)	Flexural Modulus Before Weathering (MPa)	Flexural Modulus After Weathering (MPa)	Decrease in Flexural Modulus (%)
0	2.68 ± 0.13	2.42 ± 0.07	9.70
<150/20	3.63 ± 0.15	3.30 ± 0.19	9.09
<150/40	4.59 ± 0.07	4.22 ± 0.38	8.06
<150/60	4.09 ± 0.13	3.63 ± 0.09	11.25
150-180/20	3.85 ± 0.12	3.47 ± 0.24	9.87
150-180/40	3.45 ± 0.07	2.98 ± 0.12	13.62
150-180/60	5.40 ± 0.36	4.90 ± 0.49	9.26
180-250/20	3.88 ± 0.07	3.54 ± 0.16	8.76
180-250/40	4.20 ± 0.18	3.78 ± 0.19	10.00
180-250/60	6.45 ± 0.23	5.96 ± 0.16	7.59
250-450/20	3.50 ± 0.12	3.19 ± 0.13	8.86
250-450/40	4.15 ± 0.17	3.67 ± 0.20	11.57
250-450/60	4.44 ± 0.16	3.91 ± 0.12	11.94
600-850/20	3.33 ± 0.08	2.86 ± 0.08	14.11
600-850/40	3.63 ± 0.31	3.11 ± 0.14	14.33
600-850/60	3.33 ± 0.28	2.67 ± 0.35	19.82

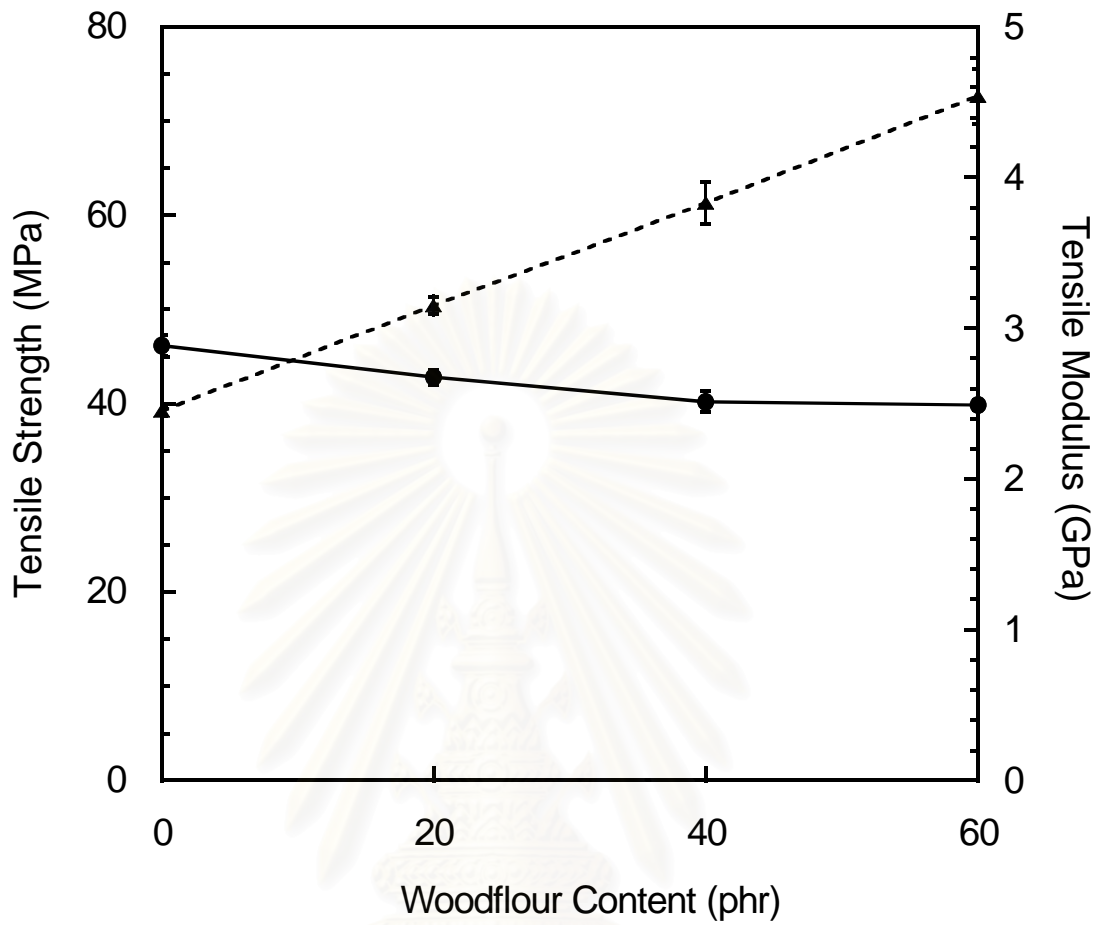


Figure 5.1: The tensile properties of PVC wood composites (mixed woodflour, particle size: 150-180 μm) at various woodflour contents: (●) tensile strength, (▲) tensile modulus.

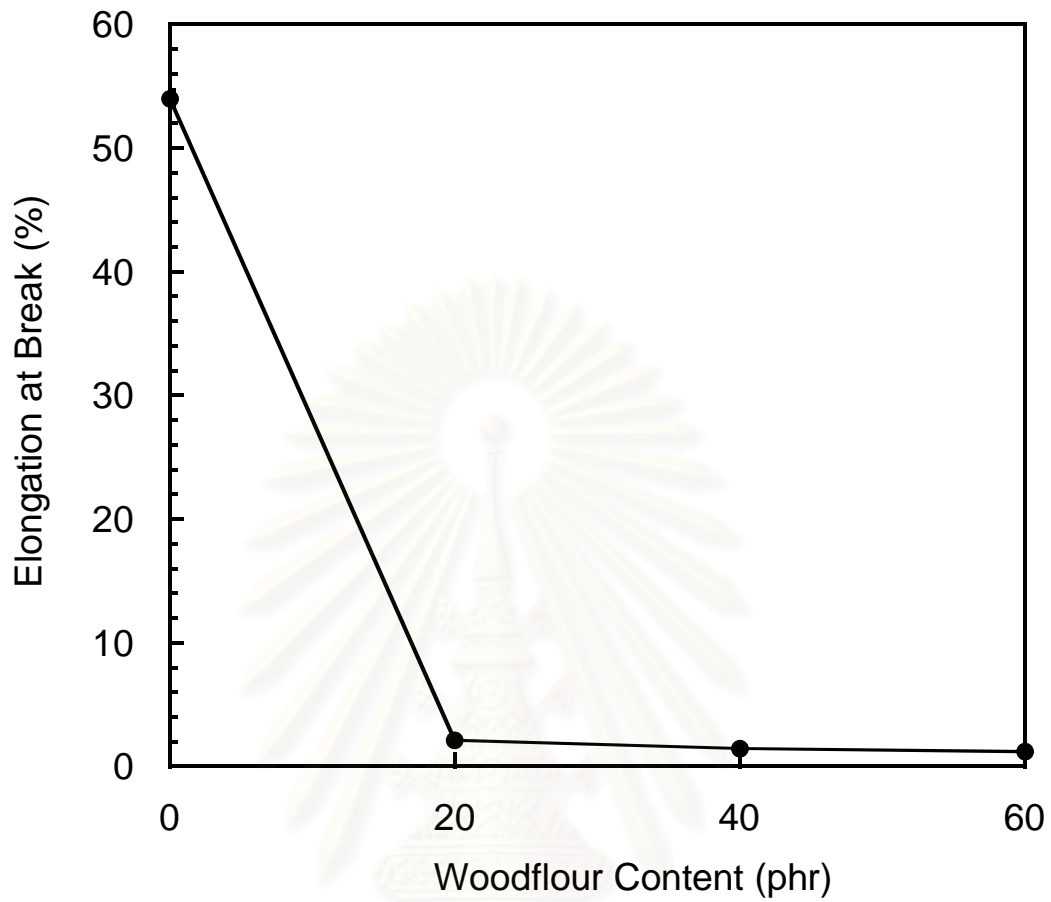


Figure 5.2: The elongation at break of PVC wood composites (mixed woodflour, particle size: 150-180 μm) at various woodflour contents.

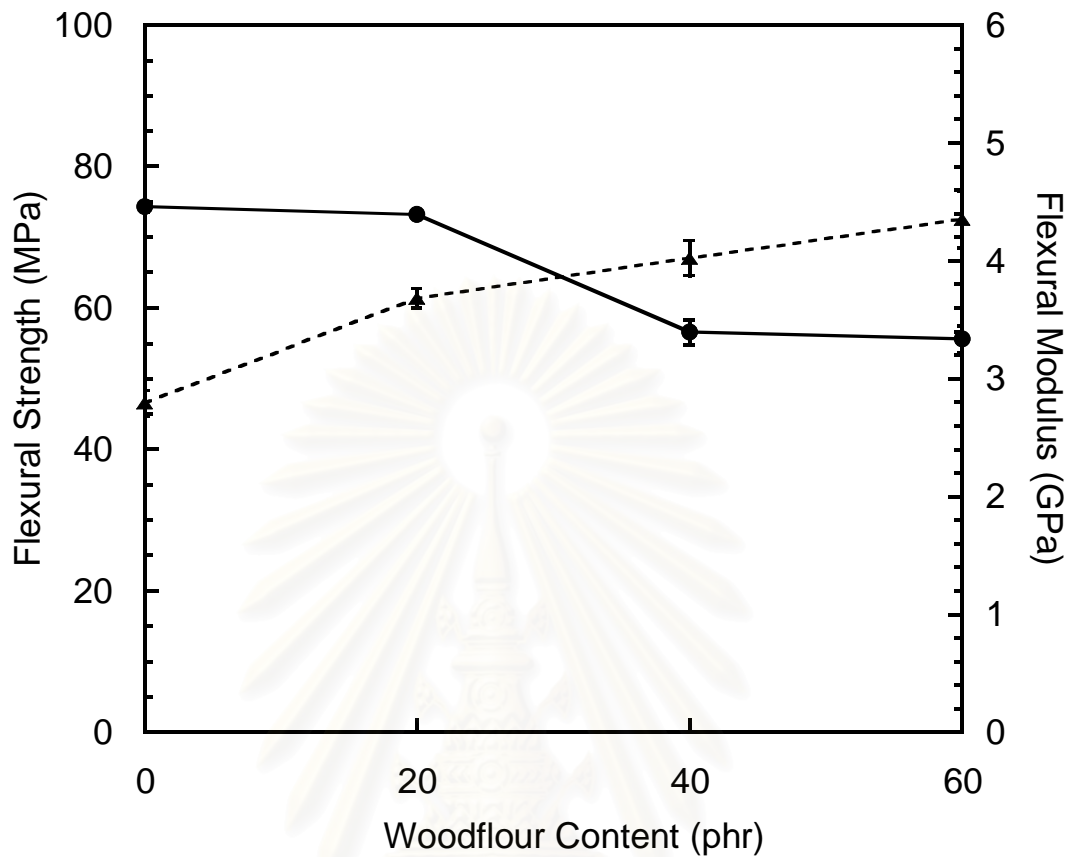


Figure 5.3: The flexural properties of PVC wood composites (mixed woodflour, particle size: 150-180 μm) at various woodflour contents: (●) flexural strength, (▲) flexural modulus.

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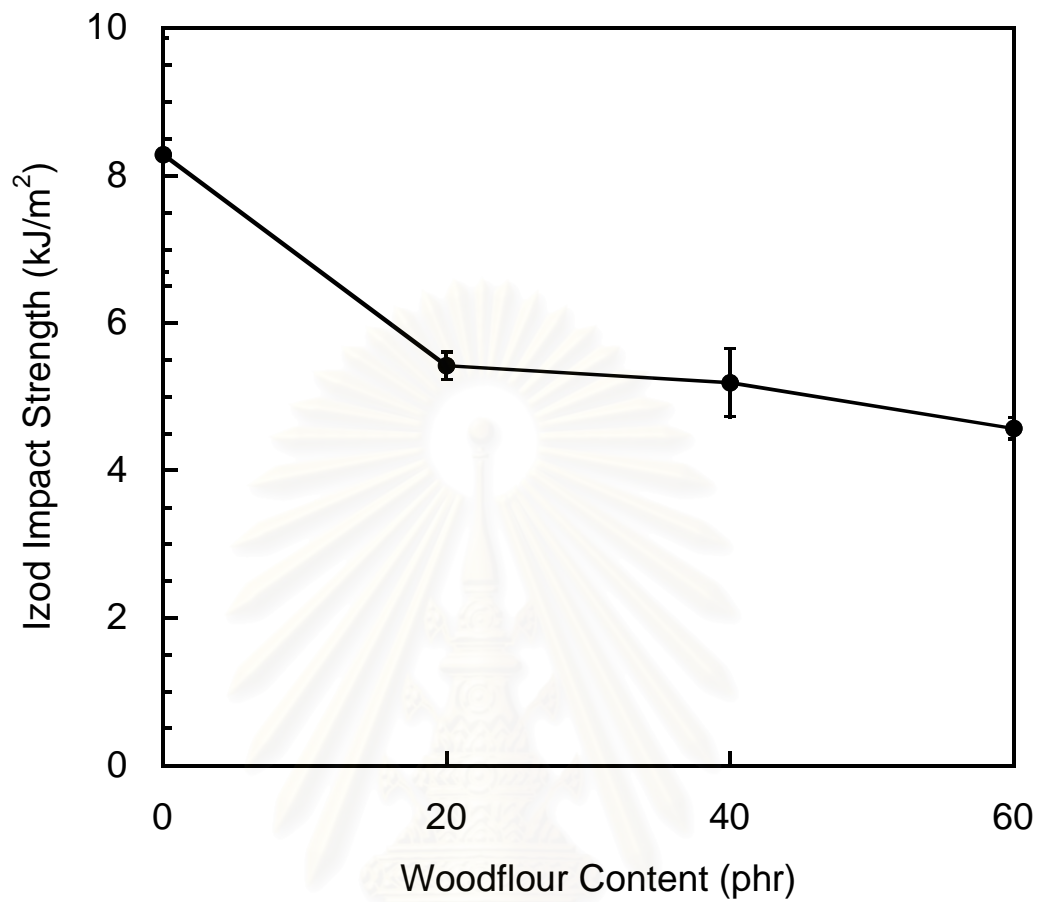


Figure 5.4: The Izod impact strength of PVC wood composites (mixed woodflour, particle size: 150-180 μm) at various woodflour contents.

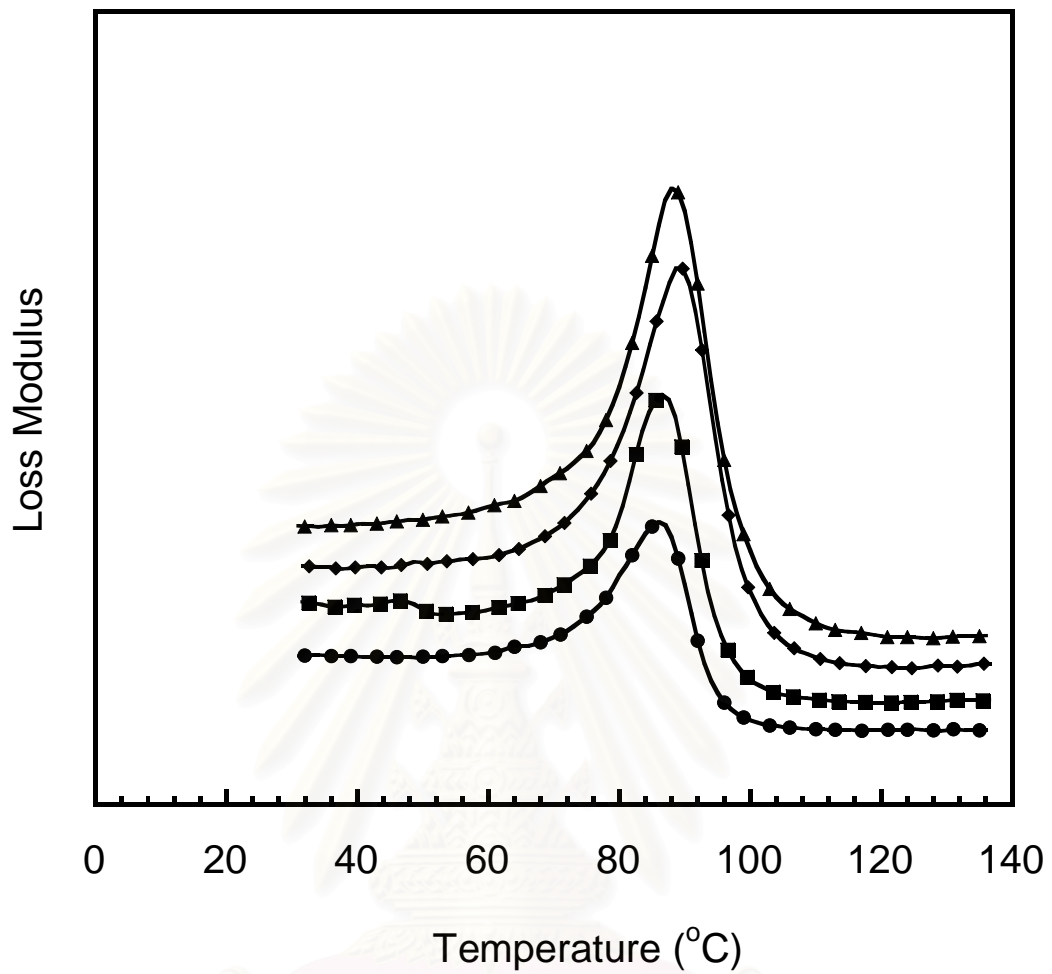


Figure 5.5: Loss modulus of PVC wood composites (mixed woodflour, particle size: <math>< 150 \mu\text{m}</math>) at various woodflour contents: (●) 0 phr, (■) 20 phr, (◆) 40 phr, (▲) 60 phr.

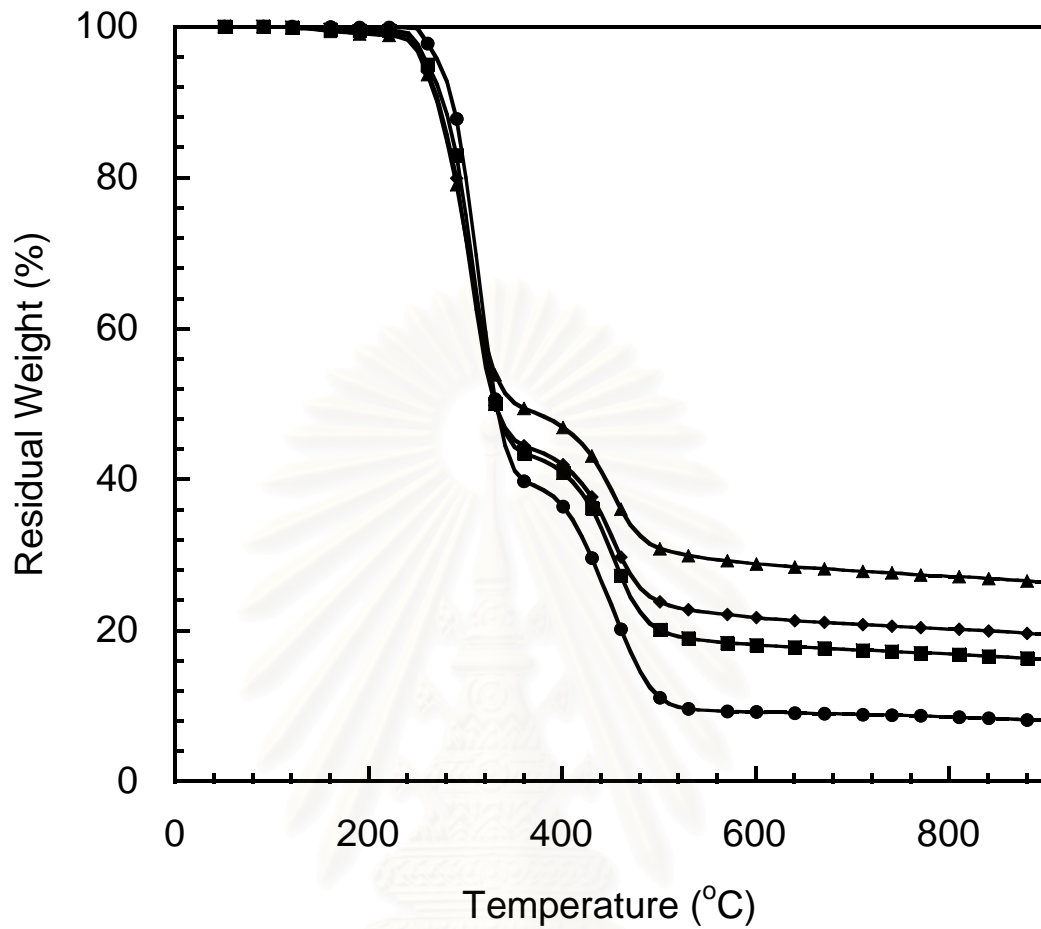


Figure 5.6: TGA thermograms of PVC wood composites (mixed woodflour, particle size: $< 150 \mu\text{m}$) at various woodflour contents: (●) 0 phr, (■) 20 phr, (◆) 40 phr, (▲) 60 phr.

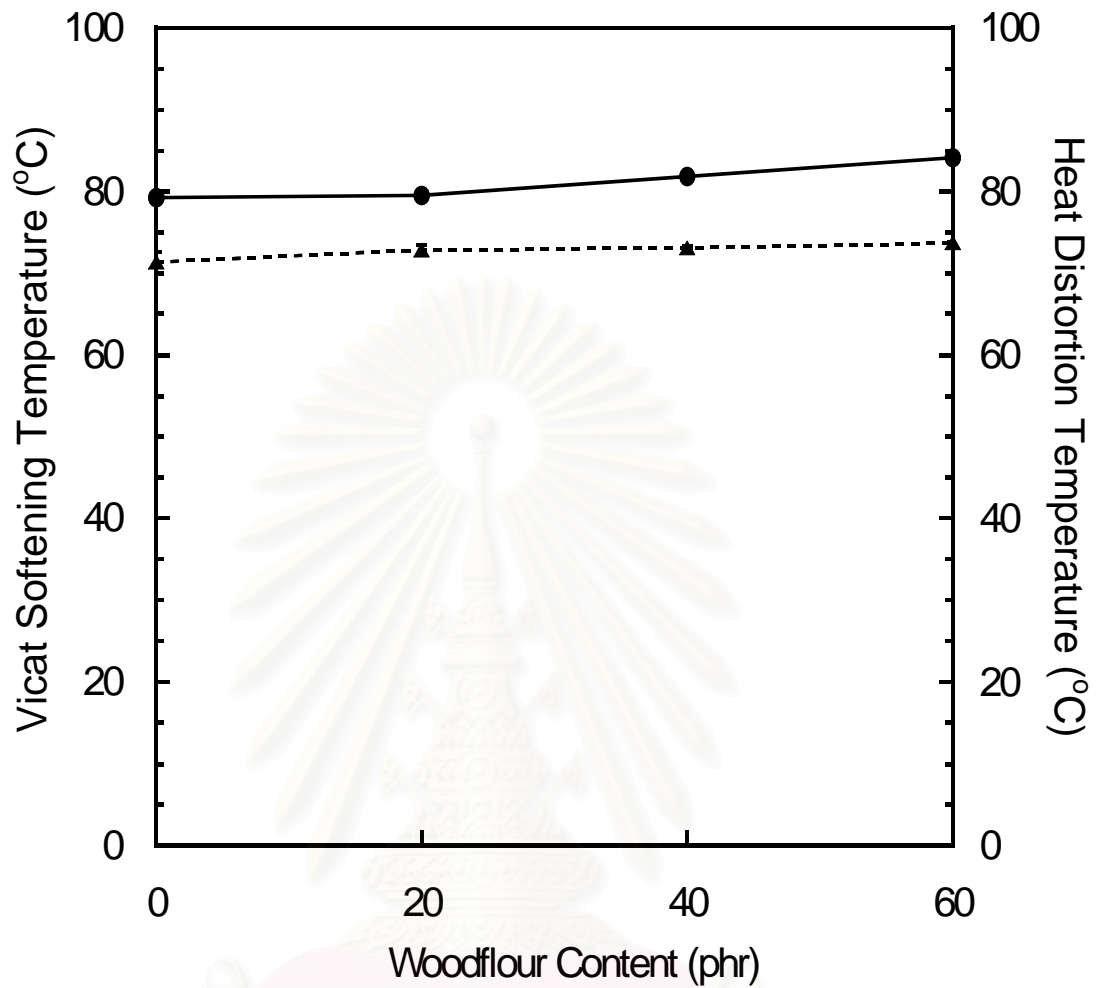


Figure 5.7: The vicat softening and heat distortion temperature of PVC wood composites (mixed woodflour, particle size: 150-180 μm) at various woodflour contents: (●) vicat softening temperature, (▲) heat distortion temperature.

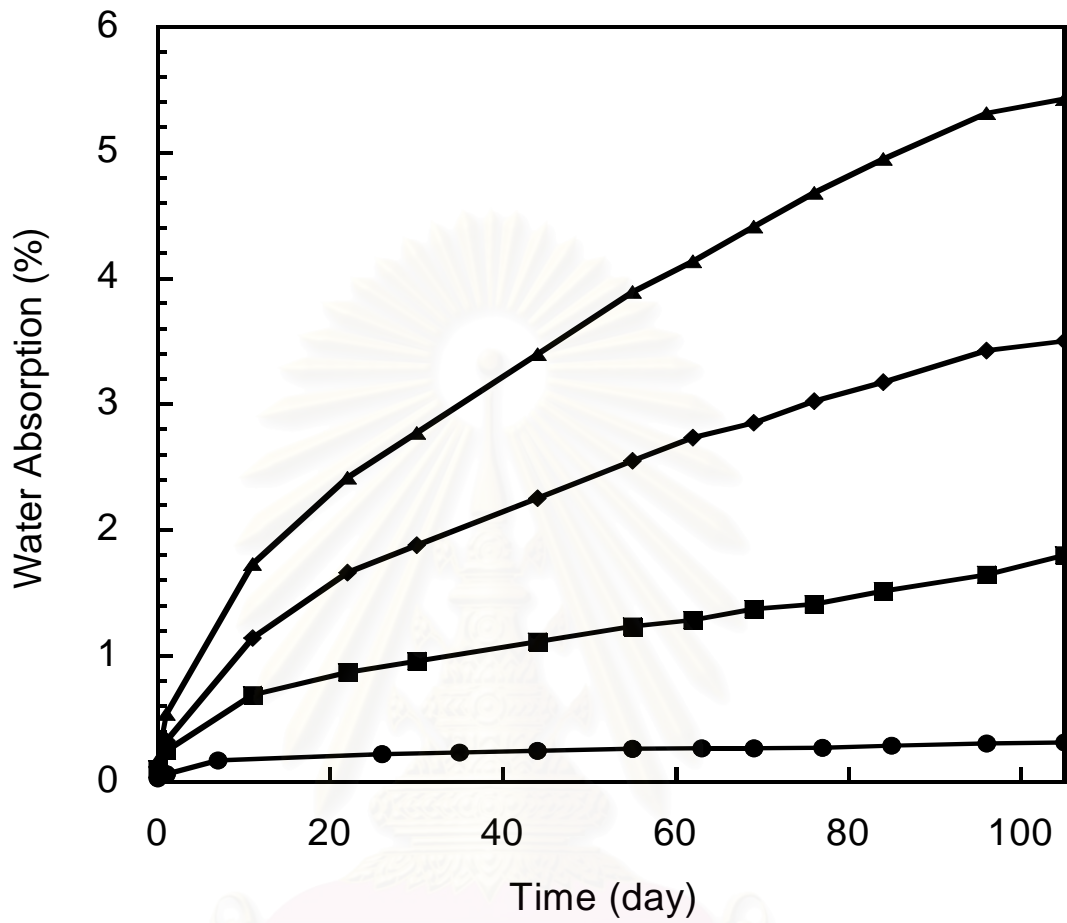


Figure 5.8: Water absorption of PVC wood composites (mixed woodflour, particle size: 150-180 μm) at various woodflour contents: (●) 0 phr, (■) 20 phr, (◆) 40 phr, (▲) 60 phr.

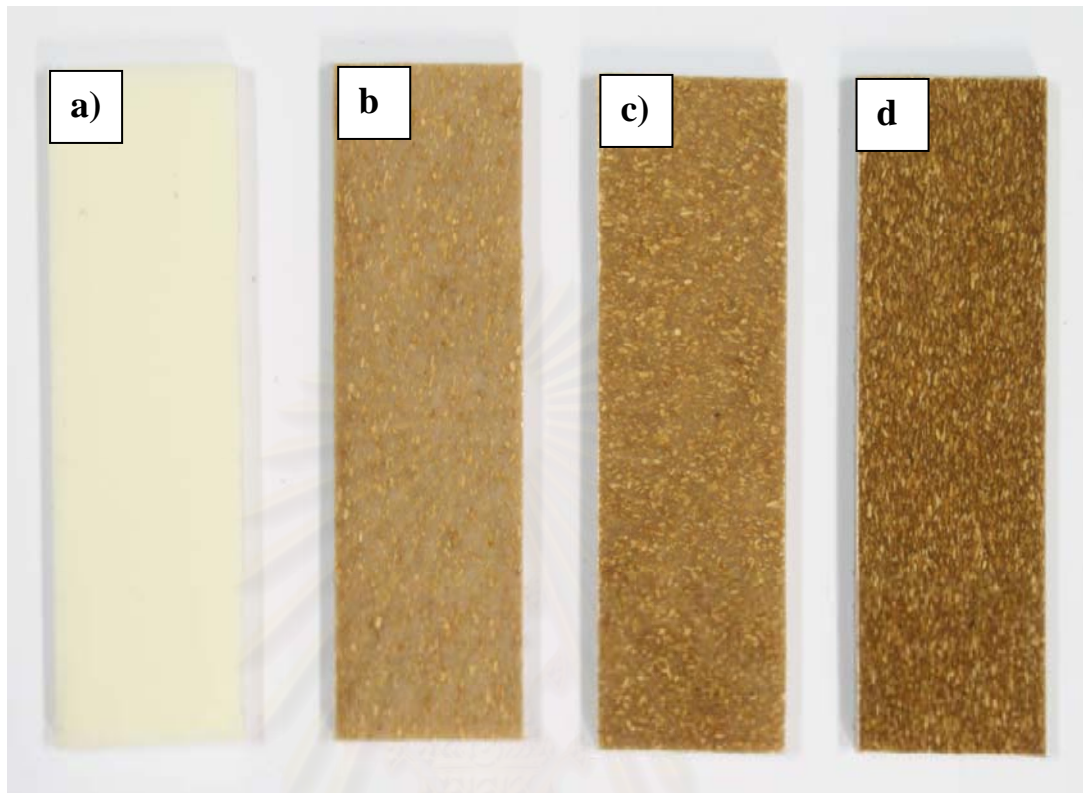


Figure 5.9: The texture surface of PVC wood composites (rubber woodflour, particle size: 600-850 μm) at various woodflour contents: (a) 0 phr, (b) 20 phr, (c) 40 phr, (d) 60 phr.

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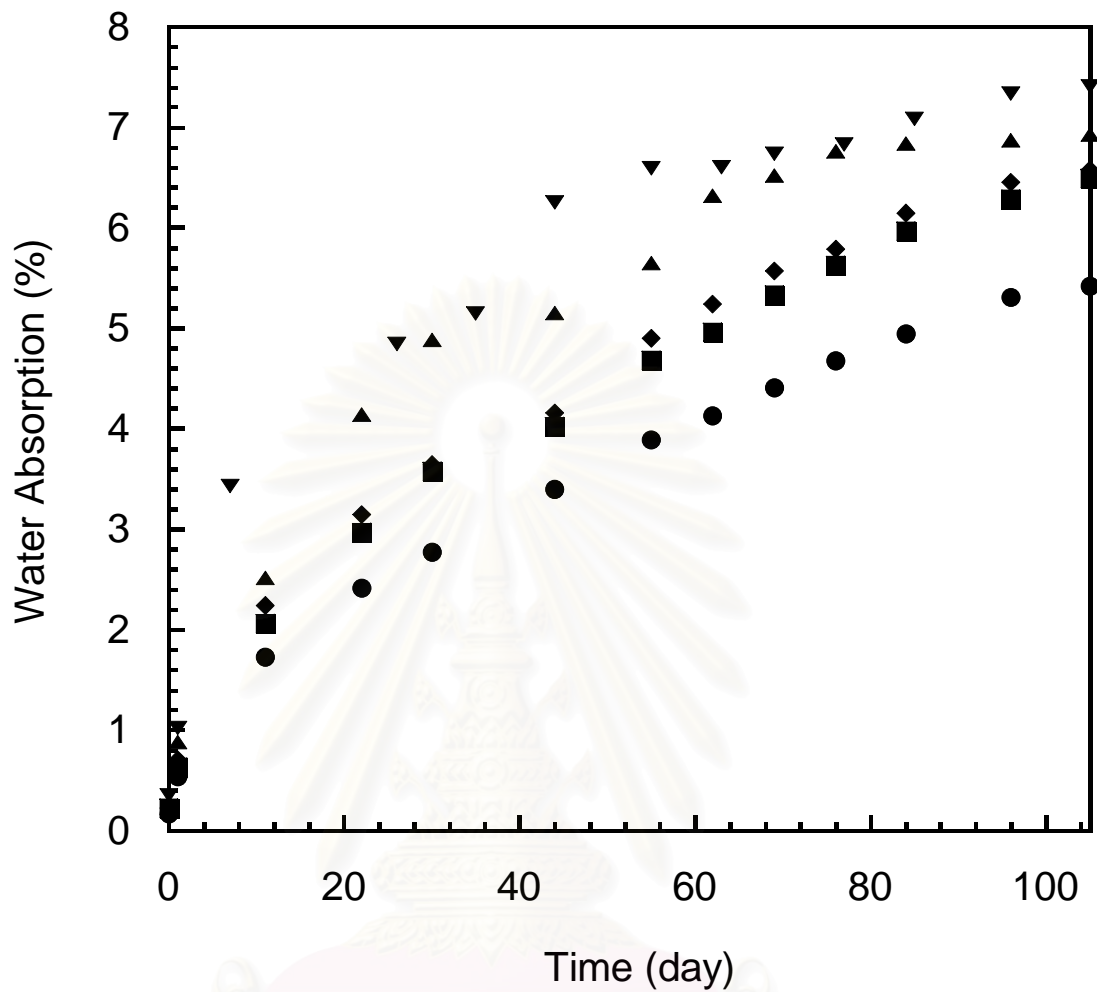


Figure 5.10: Water absorption of PVC wood composites (rubber woodflour, particle loading: 60 phr) at various particle sizes of woodflour: (●) $<150\ \mu\text{m}$, (■) $150\text{-}180\ \mu\text{m}$, (◆) $180\text{-}250\ \mu\text{m}$, (▲) $250\text{-}450\ \mu\text{m}$, (▼) $600\text{-}850\ \mu\text{m}$.

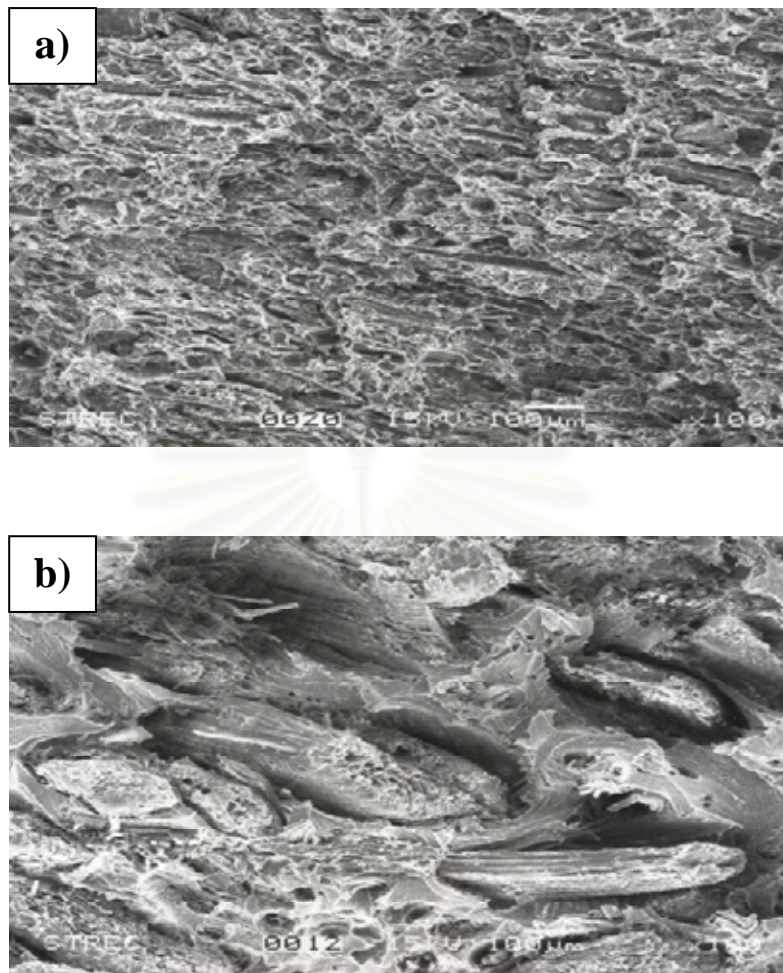


Figure 5.11: SEM micrographs on fracture surface of PVC wood composites (mixed woodflour, particle loading: 20 phr) at various particle sizes of woodflour: (a) $< 150 \mu\text{m}$, (b) 600-850 μm .

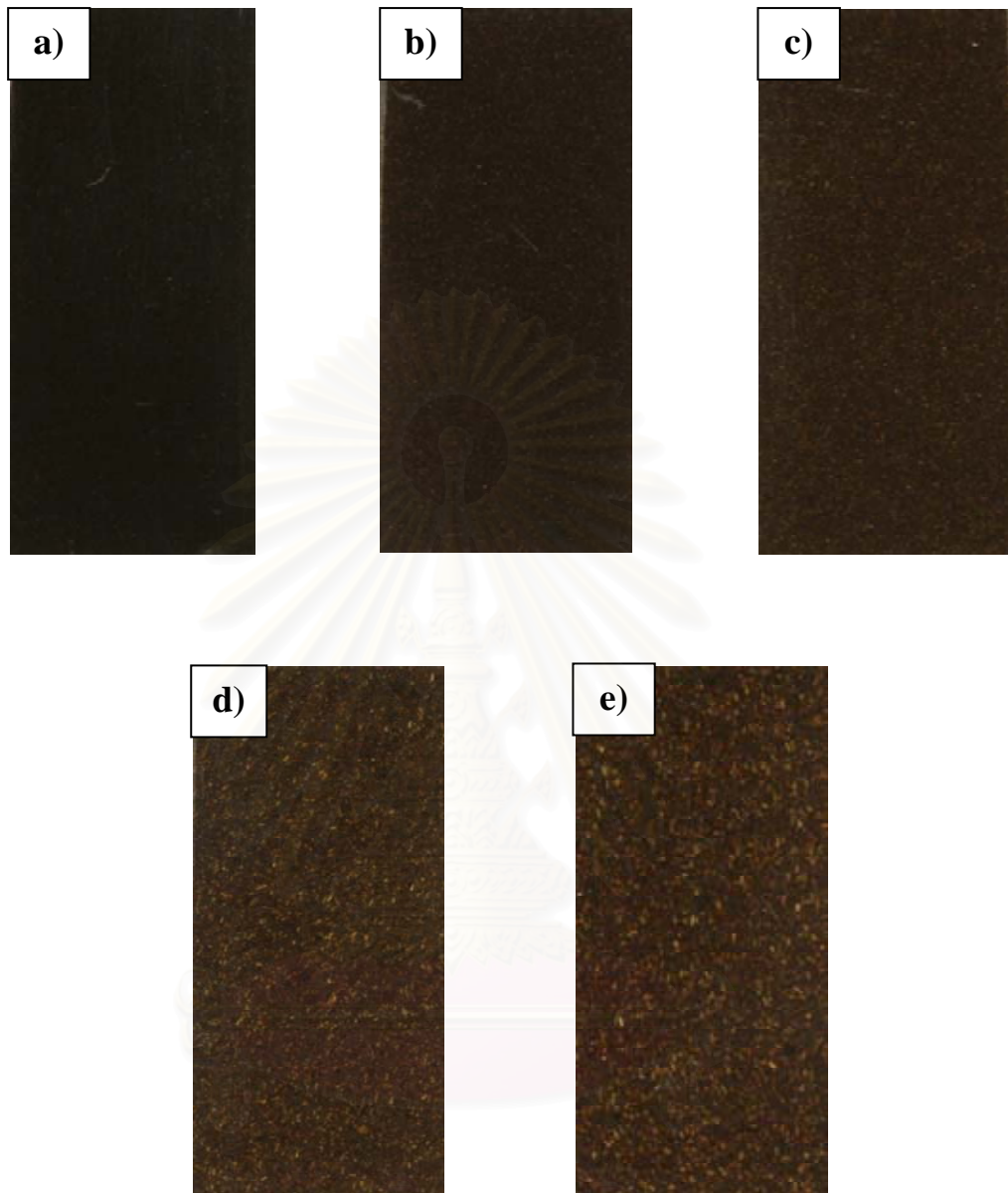


Figure 5.12: The texture surface of PVC wood composites (mixed woodflour, particle loading: 60 phr) at various particle sizes of woodflour: (a) $<150 \mu\text{m}$, (b) $150\text{-}180 \mu\text{m}$, (c) $180\text{-}250 \mu\text{m}$, (d) $250\text{-}450 \mu\text{m}$, (e) $600\text{-}850 \mu\text{m}$.

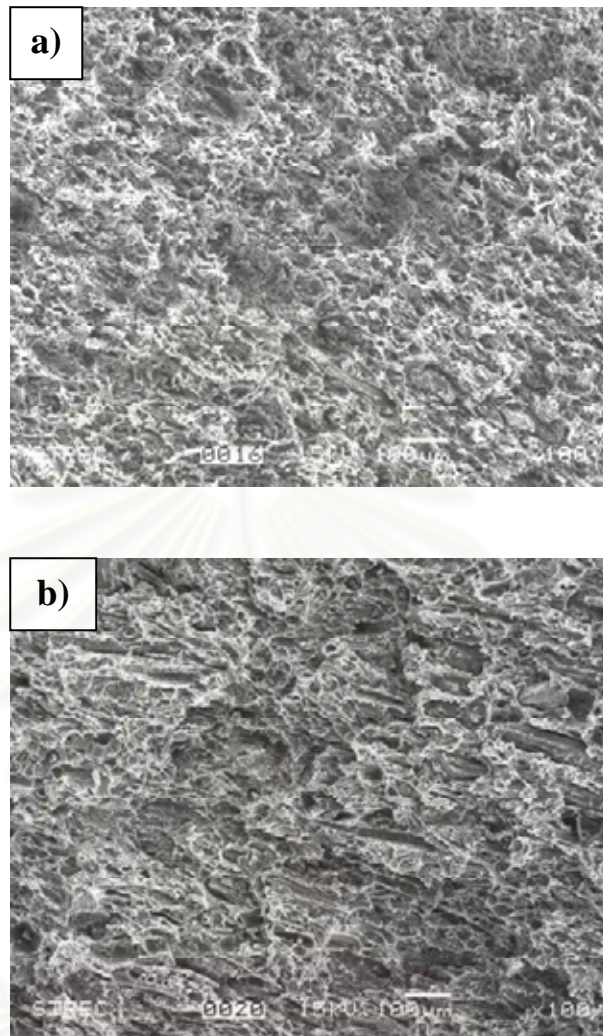


Figure 5.13: SEM micrographs on fracture surface of PVC wood composites (particle loading: 20 phr, particle size: < 150 μm) at various woodflour types: (a) rubber wood, (b) mixed wood.

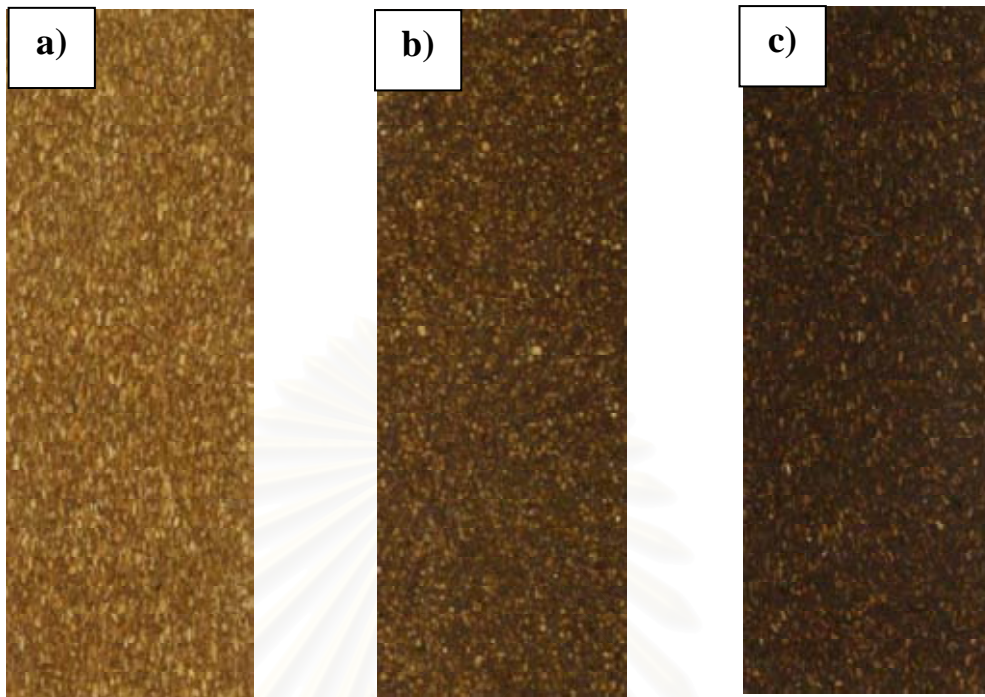


Figure 5.14: The texture surface of PVC wood composites (particle loading: 60 phr, particle size: 600-850 μm) at various woodflour types: (a) rubber wood, (b) teak wood, (c) mixed wood.

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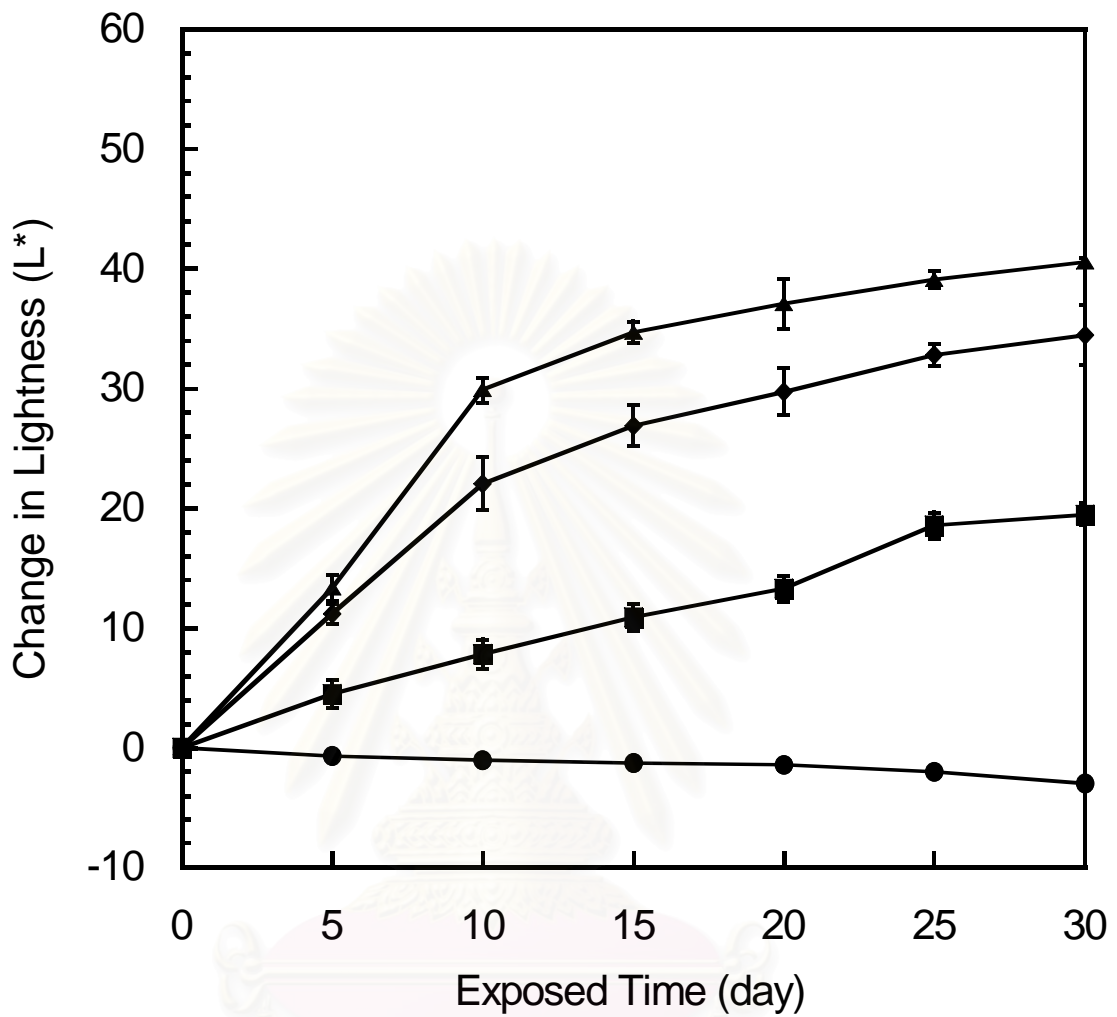


Figure 5.15: The change in lightness of PVC wood composites (mixed woodflour, particle size: 150-180 μm) at various woodflour contents: (●) 0 phr, (■) 20 phr, (◆) 40 phr, (▲) 60 phr.

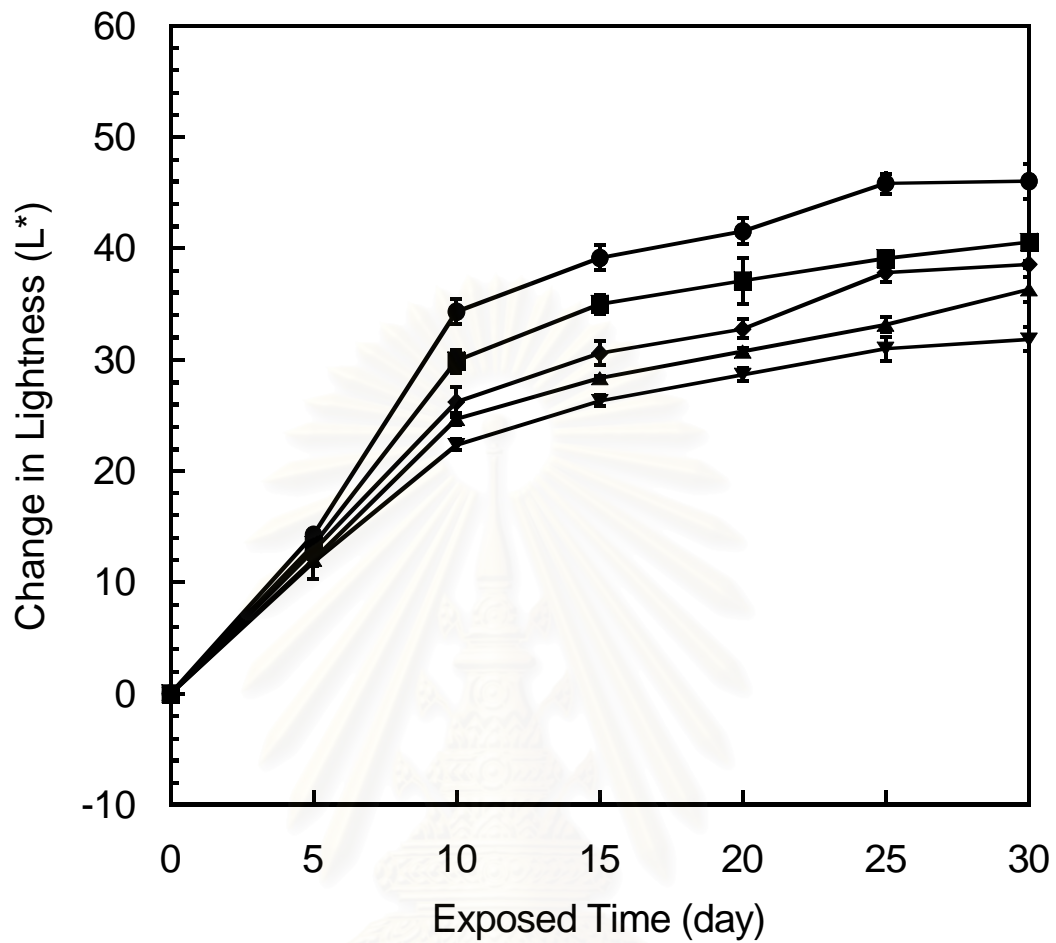


Figure 5.16: The change in lightness of PVC wood composites (mixed woodflour, particle loading: 60 phr) using various particle sizes of woodflour: (●) $<150\ \mu\text{m}$, (■) $150-180\ \mu\text{m}$, (◆) $180-250\ \mu\text{m}$, (▲) $250-450\ \mu\text{m}$, (▼) $600-850\ \mu\text{m}$.

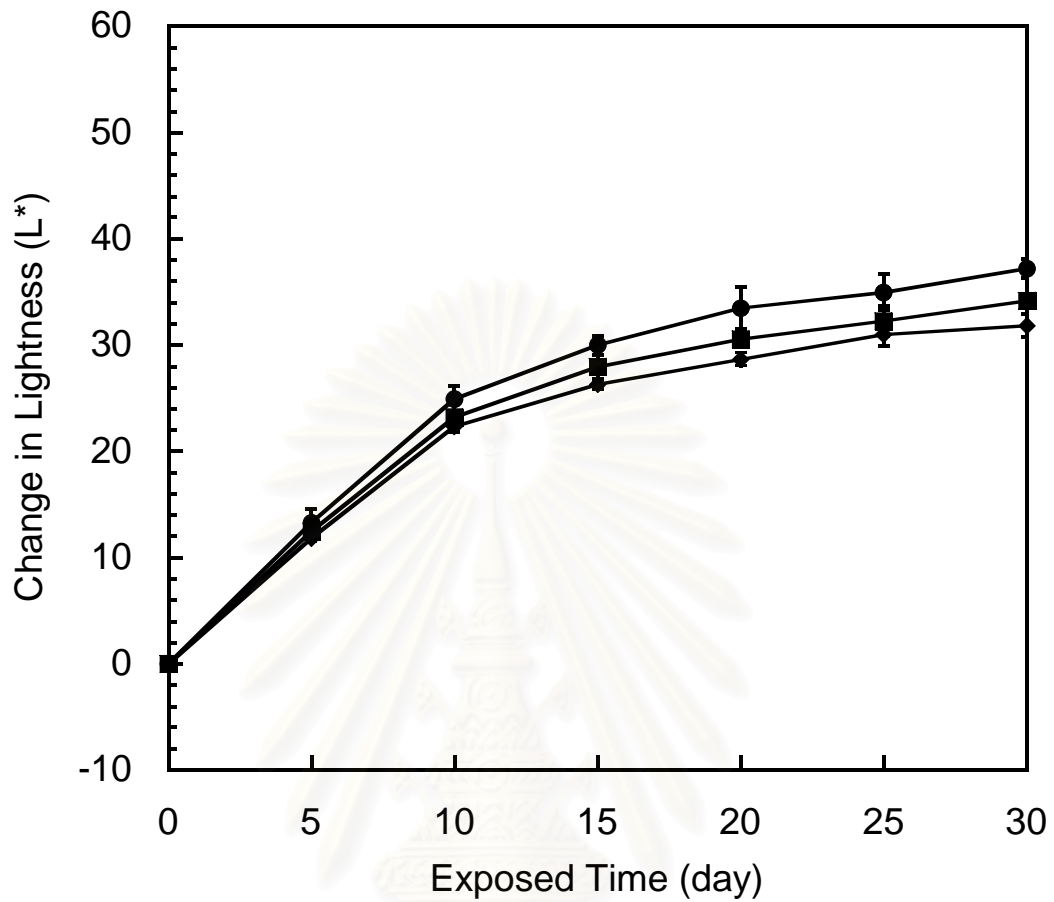


Figure 5.17: The change in lightness of PVC wood composites (particle size: 150-180 μm , particle loading: 60 phr) using various type of woodflour: (●) rubber wood, (■) teak wood, (◆) mixed wood.

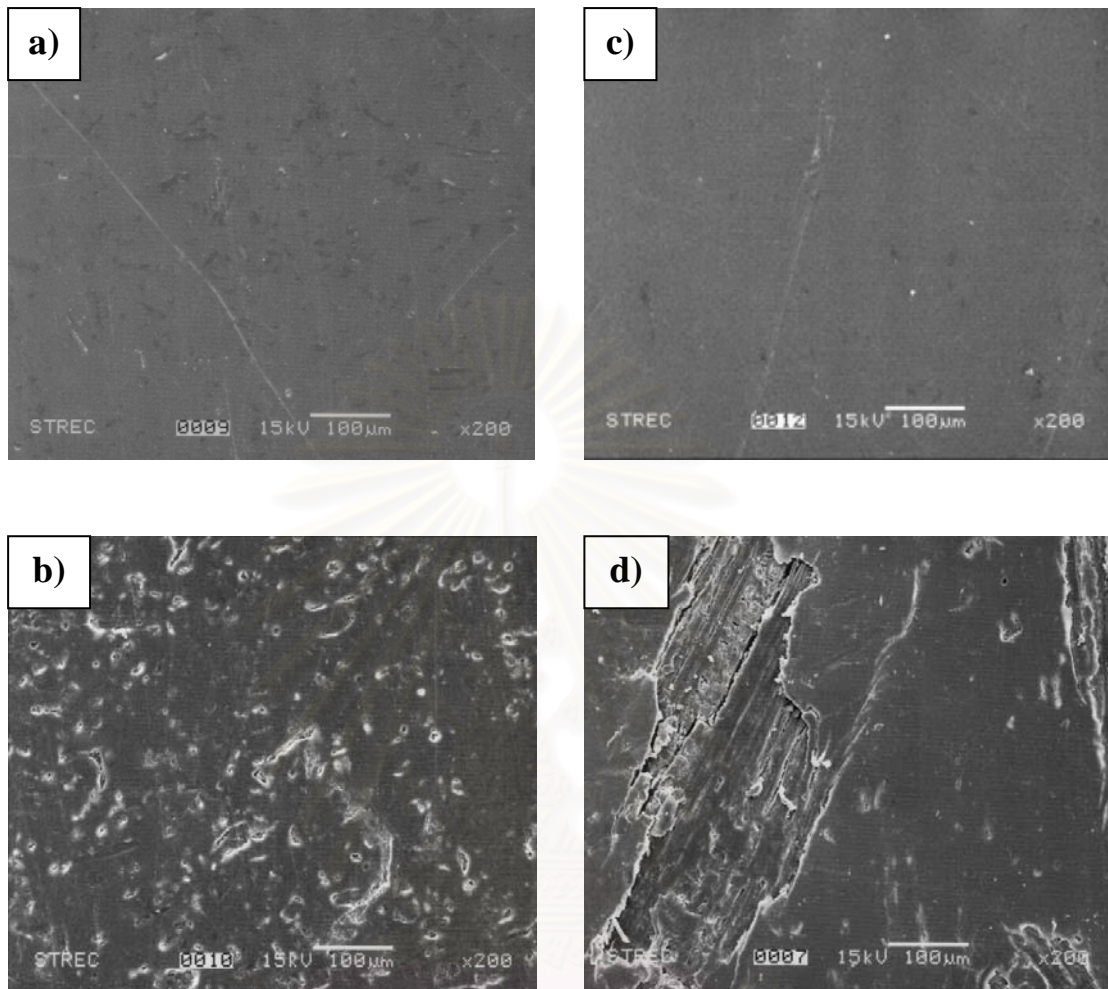


Figure 5.18: SEM micrographs on surface of PVC wood composites using various particle sizes of mixed woodflour: (a) <150 μm before exposure, (b) <150 μm after exposure, (c) $600\text{--}850$ μm before exposure, (d) $600\text{--}850$ μm after exposure.

CHAPTER VI

CONCLUSIONS

In this study, the effects of particle loadings, particle sizes, types of woodflour on mechanical properties (i.e. tensile strength, tensile modulus, elongation at break, flexural strength, flexural modulus, and impact strength), thermal properties (i.e. glass transition temperature, degradation temperature, vicat softening temperature, heat distortion temperature), water absorption, surface texture, and weathering aging of PVC wood composites were examined. The wood composites were prepared by two-roll milling at 170°C for 5 min and compression molding at 170°C, 1 MPa for 4 min. The woodflour contents were ranging from 0-60 phr with the particle sizes of woodflour of <150, 150-180, 180-250, 250-450, and 600-850 μm . The types of woodflour used are rubber wood, mixed wood, and teak wood.

As the woodflour particle loading increased, the tensile strength, elongation at break, flexural strength, and Izod impact strength of the obtained PVC wood composites decreased. On the other hand, the tensile modulus, and flexural modulus of the composites systematically increased. In addition, the mechanical properties of the PVC wood trended to decrease with the woodflour particles size. Furthermore, thermal properties of PVC wood were only marginally changed with particle loadings, particle sizes, and types of woodflour. The water absorption of PVC wood composites was found to increase with increasing the woodflour content. Moreover, the increase in particle sizes of woodflour increased the water absorption of PVC wood composites. Finally, the types of woodflour showed no significant effect on mechanical and thermal properties of the obtained composites but can influence the color and surface texture of the PVC wood composites.

In the 1 month natural weathering experiment, the flexural properties of the composites were found to decrease for about 4.69% at mixed woodflour, 165 μm , and 60 phr of woodflour. In addition, the change in lightness (ΔL^*) increased with increasing the woodflour contents and decreasing the particle sizes. An addition of

rubber wood of PVC wood composites showed the most fading in color and the mixed wood showed the least color change.



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Micrograph courtesy of Dr. Michael Wolcott, Washington State University.

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APPENDICES

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Appendix A

Mechanical Characterizations

Appendix A-1 Tensile properties of neat PVC and PVC/ rubber woodflour composites.

Woodflour Size /Content (microns/phr)	Tensile strength (MPa)	Tensile modulus (GPa)	Elongation at break (%)
0	46.2± 1.1	2.45 ± 0.03	54.0 ± 0.7
<150/20	44.0 ± 0.6	2.88 ± 0.03	2.6 ± 0.8
<150/40	41.3 ± 0.9	3.31 ± 0.05	1.7 ± 0.0
<150/60	40.7 ± 1.5	4.23 ± 0.08	1.3 ± 0.1
150-180/20	42.9 ± 0.3	2.82 ± 0.05	2.4 ± 0.5
150-180/40	40.4 ± 0.5	3.21 ± 0.04	1.5 ± 0.0
150-180/60	38.8 ± 0.5	4.15 ± 0.04	1.1 ± 0.1
180-250/20	40.6 ± 0.5	2.78 ± 0.03	2.5 ± 0.5
180-250/40	36.5 ± 1.2	3.15 ± 0.11	1.7 ± 0.2
180-250/60	34.7 ± 0.5	3.59 ± 0.09	1.1 ± 0.1
250-450/20	39.3 ± 1.0	2.74 ± 0.09	1.8 ± 0.1
250-450/40	35.3 ± 1.2	3.04 ± 0.07	1.7 ± 0.1
250-450/60	31.4 ± 0.8	3.19 ± 0.07	1.1 ± 0.1
600-850/20	36.7 ± 1.1	2.67 ± 0.06	2.0 ± 0.1
600-850/40	33.2 ± 2.0	2.89 ± 0.12	1.5 ± 0.1
600-850/60	30.4 ± 0.9	2.95 ± 0.08	1.2 ± 0.1

Appendix A-2 Flexural and Izod impact properties of neat PVC and PVC/ rubber woodflour composites.

Woodflour Size /Content (micros/phr)	Flexural strength (MPa)	Flexural modulus (GPa)	Impact strength (kJ/m ²)
0	74.3 ± 0.8	2.79 ± 0.11	8.3 ± 1.6
<150/20	72.4 ± 1.0	3.42 ± 0.23	5.4 ± 0.2
<150/40	54.7 ± 0.8	3.83 ± 0.23	5.2 ± 0.2
<150/60	50.1 ± 1.0	3.91±0.09	4.9 ± 0.8
150-180/20	71.8 ± 1.8	3.33 ± 0.07	5.3 ± 0.2
150-180/40	52.3 ± 1.2	3.78 ± 0.08	5.0 ± 0.1
150-180/60	47.6 ± 0.3	3.79 ± 0.06	4.6 ± 0.2
180-250/20	70.40 ± 1.0	3.19 ± 0.19	5.3 ± 0.6
180-250/40	52.1± 1.6	3.65 ± 0.18	4.8 ± 0.1
180-250/60	45.2 ± 2.9	3.710 ± 0.32	4.6 ± 0.4
250-450/20	69.8 ± 0.8	3.10 ± 0.04	5.0 ± 0.3
250-450/40	50.2 ± 1.0	3.59 ± 0.13	4.5 ± 0.5
250-450/60	43.4 ± 0.4	3.61 ± 0.01	3.9 ± 0.6
600-850/20	69.4 ± 5.2	2.96 ± 0.03	4.9 ± 0.2
600-850/40	46.8 ± 1.2	3.38 ± 0.13	4.0 ± 0.2
600-850/60	39.2 ± 2.7	3.55 ± 0.03	3.7 ± 0.4

Appendix A-3 Tensile properties of neat PVC and PVC/ mixed woodflour composites.

Woodflour Size /Content (microns/phr)	Tensile strength (MPa)	Tensile modulus (GPa)	Elongation at break (%)
<150/20	41.5 ± 0.7	3.03 ± 0.07	2.0 ± 0.2
<150/40	38.2 ± 0.7	3.61 ± 0.05	1.4 ± 0.1
<150/60	37.2 ± 1.4	3.97 ± 0.05	1.1 ± 0.1
150-180/20	42.8 ± 0.8	3.15 ± 0.06	2.1 ± 0.1
150-180/40	40.2 ± 1.1	3.83 ± 0.14	1.4 ± 0.1
150-180/60	39.9 ± 0.5	4.54 ± 0.18	1.2 ± 0.1
180-250/20	40.6 ± 0.6	2.82 ± 0.05	1.9 ± 0.2
180-250/40	36.2 ± 1.8	3.51 ± 0.01	1.1 ± 0.1
180-250/60	34.8 ± 1.1	3.86 ± 0.14	1.1 ± 0.1
250-450/20	40.1 ± 0.7	2.68 ± 0.04	1.8 ± 0.1
250-450/40	34.6 ± 0.7	2.89 ± 0.09	1.4 ± 0.1
250-450/60	27.4 ± 1.1	3.09 ± 0.11	1.2 ± 0.0
600-850/20	38.6 ± 0.4	2.64 ± 0.03	1.7 ± 0.0
600-850/40	29.9 ± 1.3	2.76 ± 0.06	1.3 ± 0.1
600-850/60	25.2 ± 0.2	2.85 ± 0.25	1.1 ± 0.1

Appendix A-4 Flexural and Izod impact properties of neat PVC and PVC/ mixed woodflour composites.

Woodflour Size /Content (microns/phr)	Flexural strength (MPa)	Flexural modulus (GPa)	Impact strength (kJ/m ²)
<150/20	72.6 ± 1.0	3.42 ± 0.18	5.3 ± 0.6
<150/40	54.7 ± 0.8	3.92 ± 0.15	5.0 ± 0.4
<150/60	53.4 ± 1.7	4.13 ± 0.17	4.2 ± 0.5
150-180/20	73.2 ± 0.2	3.68 ± 0.08	5.4 ± 0.2
150-180/40	56.5 ± 1.8	4.02 ± 0.15	5.2 ± 0.5
150-180/60	55.6 ± 1.9	4.35 ± 0.24	4.6 ± 0.2
180-250/20	72.2 ± 3.0	3.35 ± 0.23	5.2 ± 0.2
180-250/40	53.1 ± 0.8	3.49 ± 0.07	4.5 ± 0.6
180-250/60	48.4 ± 1.4	3.99 ± 0.24	4.1 ± 0.2
250-450/20	70.1 ± 2.9	3.22 ± 0.25	4.9 ± 0.6
250-450/40	52.7 ± 1.5	3.38 ± 0.05	4.4 ± 0.4
250-450/60	47.8 ± 1.3	3.54 ± 0.09	4.0 ± 0.5
600-850/20	68.7 ± 3.0	3.07 ± 0.27	4.8 ± 0.1
600-850/40	49.8 ± 4.1	3.23 ± 0.15	4.1 ± 0.4
600-850/60	45.1 ± 4.6	3.32 ± 0.29	3.6 ± 0.2

Appendix A-5 Tensile properties of neat PVC and PVC/ teak woodflour composites.

Woodflour Size /Content (microns/phr)	Tensile strength (MPa)	Tensile modulus (GPa)	Elongation at break (%)
<150/20	40.9 ± 1.2	2.76 ± 0.04	1.6 ± 0.1
<150/40	39.5 ± 1.3	3.82 ± 0.03	1.2 ± 0.1
<150/60	35.3 ± 0.6	4.28 ± 0.09	1.3 ± 0.0
150-180/20	42.0 ± 1.1	3.13 ± 0.06	2.0 ± 0.2
150-180/40	40.8 ± 0.9	3.86 ± 0.14	1.3 ± 0.2
150-180/60	38.8 ± 1.4	4.45 ± 0.08	1.0 ± 0.1
180-250/20	39.2 ± 0.5	2.74 ± 0.03	1.8 ± 0.1
180-250/40	38.8 ± 0.3	3.53 ± 0.05	1.4 ± 0.1
180-250/60	31.4 ± 0.4	3.62 ± 0.09	1.1 ± 0.1
250-450/20	38.9 ± 0.5	2.68 ± 0.04	1.6 ± 0.1
250-450/40	33.5 ± 1.0	3.23 ± 0.16	1.4 ± 0.1
250-450/60	30.0 ± 1.0	3.45 ± 0.09	1.1 ± 0.1
600-850/20	37.3 ± 1.2	2.56 ± 0.02	1.7 ± 0.1
600-850/40	32.3 ± 0.7	3.07 ± 0.09	1.2 ± 0.1
600-850/60	27.8 ± 0.8	3.32 ± 0.10	1.1 ± 0.1

Appendix A-6 Flexural and Izod impact properties of neat PVC and PVC/ teak woodflour composites.

Woodflour Size /Content (microns/phr)	Flexural strength (MPa)	Flexural modulus (GPa)	Impact strength (kJ/m ²)
<150/20	73.7 ± 1.7	3.25 ± 0.11	5.4 ± 0.1
<150/40	58.4 ± 1.9	3.83 ± 0.08	5.2 ± 0.1
<150/60	54.4 ± 0.9	4.29 ± 0.19	4.1 ± 0.2
150-180/20	73.9 ± 2.5	3.30 ± 0.09	5.5 ± 0.2
150-180/40	61.5 ± 2.6	3.99 ± 0.11	5.3 ± 0.1
150-180/60	55.1 ± 0.6	4.53 ± 0.12	4.50 ± 0.2
180-250/20	73.2 ± 2.0	3.11 ± 0.12	5.3 ± 0.2
180-250/40	52.6 ± 1.7	3.72 ± 0.20	5.1 ± 0.5
180-250/60	49.4 ± 1.2	3.87 ± 0.16	3.9 ± 0.2
250-450/20	71.3 ± 1.2	3.09 ± 0.05	5.30 ± 0.5
250-450/40	50.1 ± 2.4	3.39 ± 0.33	4.5 ± 0.8
250-450/60	48.3 ± 1.7	3.55 ± 0.21	3.8 ± 0.3
600-850/20	67.8 ± 1.7	2.91 ± 0.01	5.1 ± 0.3
600-850/40	49.7 ± 2.4	3.16 ± 0.07	4.2 ± 0.7
600-850/60	43.5 ± 1.5	3.41 ± 0.06	3.2 ± 0.3

Appendix B

Water Absorption Percentages

Water absorption percentages of the polymer matrix and its wood composites were used to determine the amount of water absorbed under specified conditions according to ASTM D570. Percent water absorption was determined according to the following equation:

$$\text{Water absorption (\%)} = \frac{(W_w - W_d)}{W_d} \times 100$$

Where:

W_w is weight of specimen being wet

W_d is weight of specimen being dried

Appendix B-1 Water absorption percentages of neat PVC and PVC/ mixed woodflour composites.

Woodflour Size /Content (microns/phr)	Weight before absorbed	Weight after absorbed	Water absorption (%)
0	9.2658	9.2945	0.31
<150/20	9.1427	9.3070	1.80
<150/40	9.2778	9.6026	3.50
<150/60	9.3955	9.9048	5.42
150-180/60	9.6058	10.2294	6.49
180-250/60	9.5702	10.1994	6.57
250-450/60	9.4701	10.1259	6.93
600-850/60	9.3715	10.0660	7.41

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