

**STUDY ON FABRICATION OF ARTIFICIAL WOOD FROM
POLY(VINYL CHLORIDE) MIXED WITH NATURAL RUBBER FIBER**

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บทคัดย่อ

งานวิจัยนี้ศึกษาแนวทางการผลิตไม้เทียมจากวัสดุประกอบระหว่างพอลิไวนิลคลอไรด์กับเส้นใยไม้ยางพารา วัตถุประสงค์ที่ใช้ คือ พอลิไวนิลคลอไรด์ เส้นใยไม้ยางพารา สเตบิลไอเซอร์ และพลาสติกไซเซอร์ วัตถุประสงค์ทั้งหมดถูกผสมเบื้องต้นในเครื่องผสมความเร็วสูง ด้วยความเร็ว 4×10^3 รอบต่อนาที จากนั้นผสมแบบหลอมเหลวในเครื่องอัดรีดแบบเกลียวหนอนเดี่ยว โดยใช้อุณหภูมิที่ส่วนป้อนวัสดุ (Feed zone) ส่วนการอัด (Compression zone) และ ส่วนมาตรวัด (Metering zone) 150, 160 และ 170 องศาเซลเซียส ตามลำดับ การเพิ่มปริมาณเส้นใยไม้ยางพาราในวัสดุประกอบทำให้ ค่าความแข็งแรงดึง (Tensile strength) มอดุลัสที่ 3 เปอร์เซ็นต์ (Modulus at 3% strain) ค่าความแข็งแรงกระแทก (Impact strength) ความแข็งแรงโค้งงอ (Flexural strength) และมอดุลัสโค้งงอ (Flexural modulus) สูงขึ้น และจะลดลงเมื่อเกินขีดความสามารถในการเข้ากันระหว่างพอลิไวนิลคลอไรด์เมตริกซ์กับเส้นใยไม้ยางพารา สำหรับค่าความแข็งกด (Hardness) จะเพิ่มขึ้นเมื่อปริมาณเส้นใยไม้ยางพาราเพิ่มขึ้น จากการวิเคราะห์สมบัติทางความร้อนพบว่า อุณหภูมิในการสลายตัวของวัสดุประกอบที่มีเส้นใยไม้ยางพาราในปริมาณ 10, 20, 30, 40 และ 50 phr อยู่ในช่วงใกล้เคียงกัน คือ 280-400 องศาเซลเซียส แสดงว่าปริมาณเส้นใยไม้ยางพาราไม่มีผลกระทบต่ออุณหภูมิการสลายตัวของวัสดุประกอบ จากเทอร์โมแกรมจากเครื่องดีพีเฟอร์เรนเชียลสแกนนิ่งแคลอริมิเตอร์พบว่า อุณหภูมิหลอมเหลวผลึก (Melting temperature: T_m)

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จะลดลงเมื่อปริมาณเส้นใยไม้ยางพาราเพิ่มขึ้น และจากการวิเคราะห์สมบัติทางความร้อนเชิงกลพบว่า Heat distortion temperature (HDT) เพิ่มขึ้นตามปริมาณการเพิ่มขึ้นของเส้นใยไม้ยางพารา ส่วนอุณหภูมิเปลี่ยนสถานะคล้ายแก้ว (Glass transition temperature: T_g) เพิ่มขึ้นจนถึงองค์ประกอบที่มีปริมาณเส้นใยไม้ยางพารา 30 phr จากนั้นจะลดลง การดูดซับน้ำของวัสดุประกอบเพิ่มมากขึ้นตามปริมาณของเส้นใยไม้ยางพาราเนื่องจากความสามารถในการดูดซับน้ำของเส้นใย ค่าความถ่วงจำเพาะของวัสดุประกอบต่างกันเล็กน้อยเมื่อปริมาณเส้นใยไม้ยางพาราเพิ่มขึ้น

การเพิ่มปริมาณพลาสติกไซเซออร์ คือ DOP ทำให้ค่าความแข็งแรงดึง มอดุลัสที่ 3 เปอร์เซนต์ ความแข็งแรงโค้งงอ มอดุลัสโค้งงอ และความแข็งแรงกดลดลง ในขณะที่ค่าความแข็งแรงกระแทกเพิ่มขึ้นเล็กน้อย จากการวิเคราะห์สมบัติทางความร้อนและความร้อนเชิงกลพบว่า อุณหภูมิในการสลายตัวของวัสดุประกอบที่ค่า DOP 5, 10, 15 และ 20 phr อยู่ในวงใกล้เคียงกัน คือ 220-370 องศาเซลเซียส แสดงว่าปริมาณ DOP ไม่มีผลต่ออุณหภูมิในการสลายตัวของวัสดุประกอบ ส่วนอุณหภูมิลอมเหลวผลึก HDT อุณหภูมิเปลี่ยนสถานะคล้ายแก้ว และค่าความถ่วงจำเพาะของวัสดุประกอบลดลงเมื่อปริมาณ DOP เพิ่มขึ้น ในงานวิจัยนี้วัสดุประกอบที่มีคุณสมบัติเชิงกลดีและสามารถขึ้นรูปเป็นแผ่นได้ง่ายโดยใช้เครื่องอัดรูปแบบเกลียวหมุนเดี่ยวประกอบด้วยพอลิไวนิลคลอไรด์ เส้นใยยางพารา สเตบิลไอเซออร์ และพลาสติกไซเซออร์ เท่ากับ 100, 30, 15 และ 10 phr ตามลำดับ

Thesis Title Study on Fabrication of Artificial Wood from Poly(vinyl chloride) Mixed with Natural Rubber Fiber

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ABSTRACT

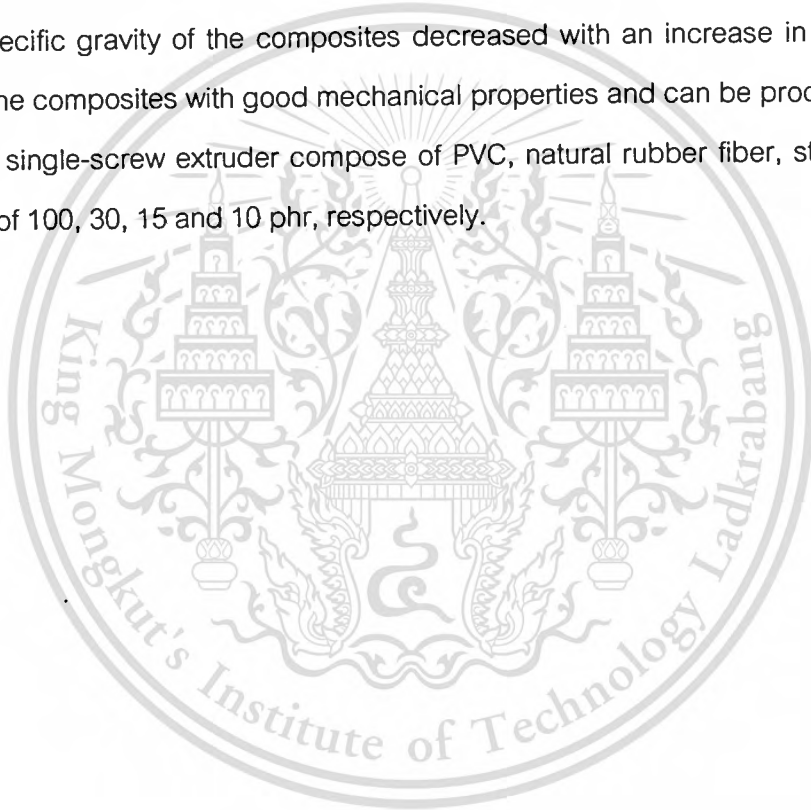
This research is a study on fabrication of artificial wood from poly(vinyl chloride) mixed with natural rubber fiber. Raw materials, i.e., PVC, natural rubber fiber, a stabilizer (monohydrous tribasic lead sulphate and normal lead stearate), and a plastizer (dioctyl phthalate: DOP) were compounded with different fiber content and DOP, and premixed in a high-speed mixer at a speed of 4×10^3 rpm; subsequently, blended in a single-screw extruder. The temperatures of the feed zone, compression zone, and metering zone of the extruder were 150, 160, and 170 °C, respectively. With an increase in fiber content, tensile strength, modulus at 3% strain, impact strength, flexural strength and flexural modulus increased up to the limit of compatibility between PVC matrix and the fibers then decreased. Hardness increased with the fiber content. From thermogravimetric analysis, the decomposition temperatures of the composites with fiber contents of 10, 20, 30, 40 and 50 phr were in the same range of 280-400 °C. It indicated that fiber content had no effect on the decomposition temperature of the composites. From the differential scanning calorimetry thermogram, the melting temperature (T_m) decreased when the fiber content increased, but heat distortion

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temperature (HDT) increased. The glass transition temperature (T_g) increased with the fiber content to 30 phr then decreased with an excess of the fibers. The water absorption of the composites increased because of the hydrophilic fibers. The specific gravity of the composites slightly changed with the fiber content.

In case of the effect of DOP, tensile strength, modulus at 3% strain, flexural strength, flexural modulus and hardness decreased with DOP; however, the impact strength slightly increased with DOP. The TGA thermogram indicated that the decomposition temperatures of the composites with different DOP content were in the same range of 220-370 °C showing that there was no effect of DOP. The T_m , HDT, T_g and the specific gravity of the composites decreased with an increase in DOP. From this work, the composites with good mechanical properties and can be processed into a sheet by a single-screw extruder compose of PVC, natural rubber fiber, stabilizer, and plasticizer of 100, 30, 15 and 10 phr, respectively.



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CHAPTER 1

INTRODUCTION

1.1 Rationale

There have been efforts at various places in the world to preserve forests, however, the speed of deforestation far exceeds the growth of trees. It is an undeniable fact that the destruction of forests on a global scale is progressing everyday. Many species of animals are becoming extinct due to the destruction of green forests.

The cultivation of the natural rubber wood in Thailand has the second most area after Indonesia. It is greatly cultivated in the east and the south of the country. The natural rubber wood has been economically grown for latex products in Thailand. The natural rubber wood is transformed into furniture, ready-made products, e.g., officeware, toys, kitchenware, construction materials, parquet flooring, etc; and the fibers for plywood and particle board materials. Figure 1.1 shows the products made from the natural rubber wood.

Basically, there are high demand in many wooden articles to serve mankind lifestyle. An artificial wood is an option to replace a natural wood. A method to fabricate the artificial wood is a composite between plastics and reinforcing agents. Figure 1.2 shows the examples of the composite products, which can be cut, fastened, and finished just like the natural wood. The natural wood has an attractive texture and strength. However, it cannot resist to the weather, water, rottenness and termites, in contrast to plastics, which possess a chemical and moisture resistances, dimensional stability and outdoor uses.

This research involves a study on fabrication of the artificial wood from a natural rubber wood (*Hevea brasiliensis*) fiber-poly(vinyl chloride) composites. The fibers used as the reinforcing filler, whereas poly(vinyl chloride), PVC, is a matrix in the composites.

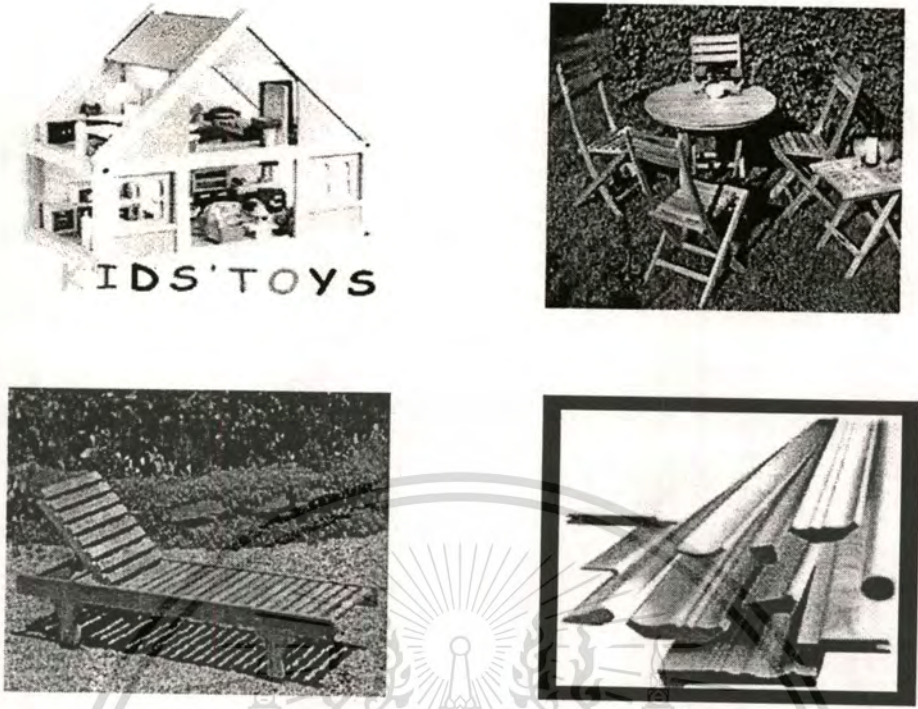


Figure 1.1 The natural rubber wood products [1]

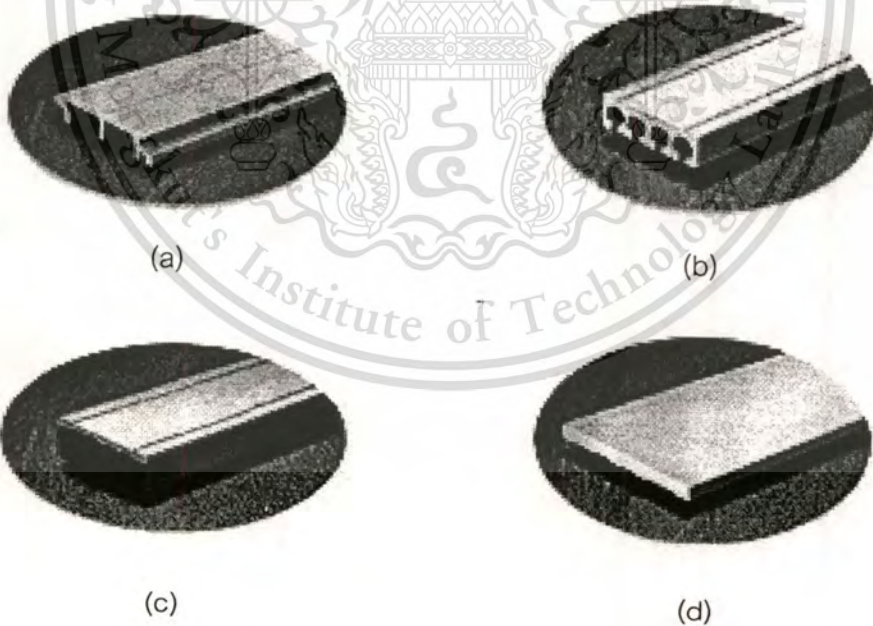


Figure 1.2 Products of composite materials [2]

(a) Tongue-and-groove planks

(b) 2 X 6 planks

(c) Deck covers

(d) Trimming products

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1.2 Objectives

The objectives of this research are:

1. To fabricate the artificial wood from natural rubber fiber and PVC.
2. To construct the artificial wood sheet from the extrusion process.

1.3 Hypothesis

1 The compatibility between the fibers and PVC without the modification, in accordance with their polarity.

2. The enhancement of the composite properties corresponding to the reinforcing fibers.

1.4 Scopes of the study

The scopes of the study are:

1. To review the literatures.
2. To find formulas between natural rubber fiber, PVC and the additives (stabilizer and plasticizer) for high property composites.
3. To find the temperature and time for composite fabrication.
4. To study the effects of the fiber content and plastizer content on the properties of the composites.
5. To extrude an artificial wood sheet.

CHAPTER 2

THEORY

2.1 Definition of composites [3]

The word "composites" implies that it is a material composed of two or more distinct components. Naturally occurring composites are bone, bamboo, feathers, natural fibers, and wood. Bone is composites of protein (collagen) and mineral (apatite). Bamboo is a cellulose reinforced by silica. This combination makes bamboo a hard material with high impact strength. The wood cellulose cell structure and the fibers are bound together with lignin, a natural polymeric substance. Wood cells are natural composites structures within themselves.

The word composites have evolved over a period of years. Some consider it a system or process of combining two or more reinforcing materials in a matrix binder. Others consider it a new material having characteristics derived from its processing and from its microstructure.

The properties of composites depend critically on the microstructure and properties of interface or interphase between fibers and matrix. The word "interphase" refers to a region (see Figure 2.1) where the fibers and matrix phases are chemically and/or mechanically combined or otherwise indistinct. The interphase may be a diffusion zone, a nucleation zone, a chemical reaction zone, a thin layer of fiber coating, or any combination of the above. An "interface" is a boundary demarcating distinct phase such as fibers, matrix, coating layer, or interphase [4].

2.2 Fibrous composites

There are three major classifications of composites: (1) fibrous, (2) laminar, and (3) particulate but most of the major developments in recent times have been in the area of fibrous reinforcement as well as this work so more details will be discussed on fibrous composites. The underlying philosophy in the design of fiber composite materials is to find or to make a fiber material of high elastic modulus and strength, and preferably low

density, and then to arrange the fibers in suitable manner to give useful engineering properties to the final products [5].

Reinforcing fibers in single-layer composites may be short or long compared to its overall dimensions. Composites with long fibers are called continuous-fiber-reinforced composites. Those with short fibers are called discontinuous-fiber-reinforced composites. A further distinction is that the latter can be considered to be one in which the fiber length affects the properties of the composites. In the continuous-fiber-reinforced composites it may be assumed that the load is directly applied to the fibers and the fibers in the direction of load are the principal load-carrying constituent. Thus the principal purpose of a matrix is not to be a load-carrying constituent but essentially to bind the fibers together and protect them. The failure mode of the continuous-fiber-reinforced composites is also generally controlled by the fibers.

The orientation of short or discontinuous fibers cannot be easily controlled in a composite material. In most cases the fibers are assumed to be randomly oriented in the composites. However, in the injection molding of a fiber-reinforced polymer, the considerable orientation can occur in the flow direction (Figure 2.2) [6].

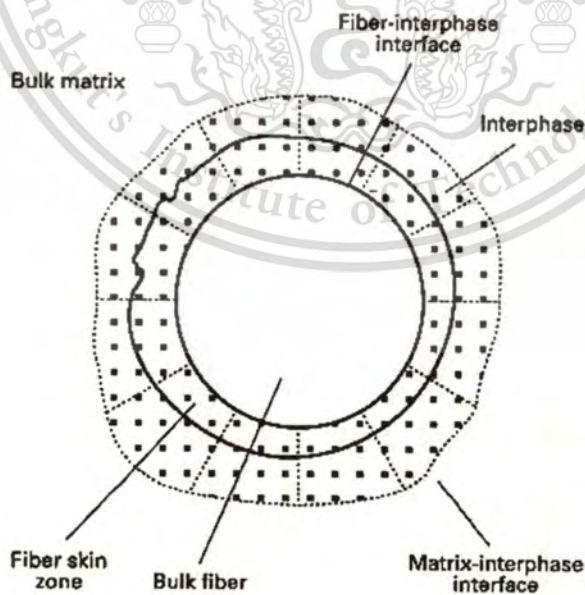


Figure 2.1 Interface and interphase [4]

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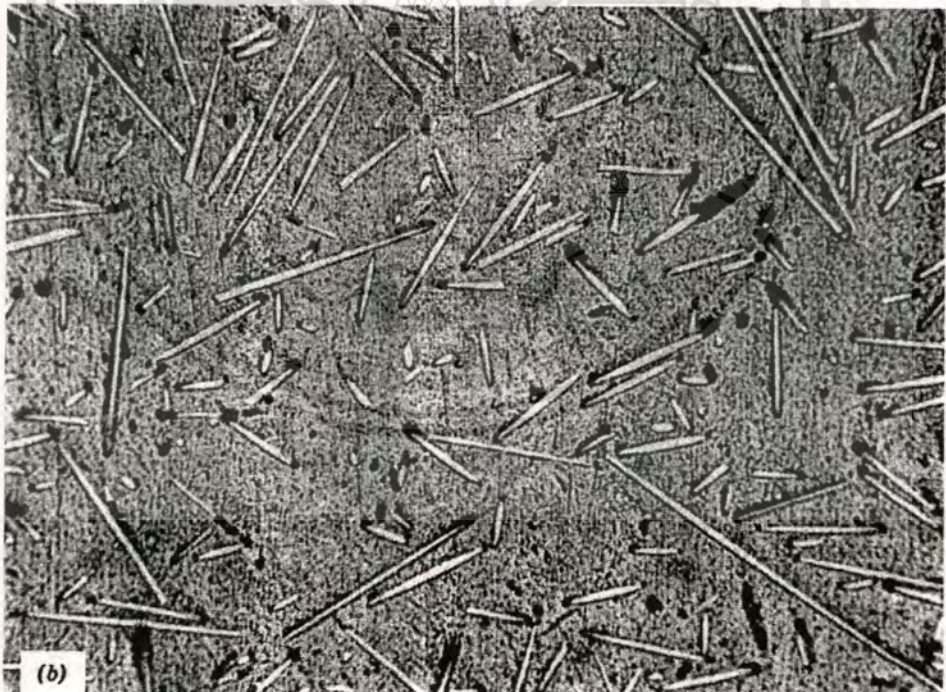
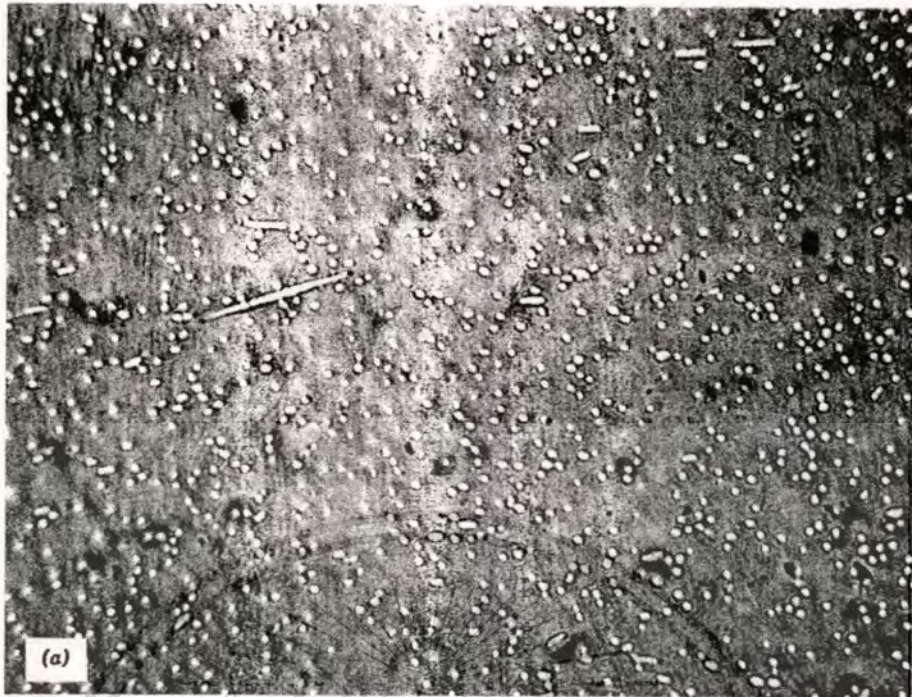


Figure 2.2 Injection-molded glass-fiber-reinforced nylon : (a) section of part where fibers have preferred orientation: (b) section of part where fibers have random orientation [6]

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Figure 2.3 shows several examples of the possible composite reinforcement forms. Composites can differ in the amount of fibers, fiber types, fiber length, fiber orientation, and possibly fiber hybridization [4].

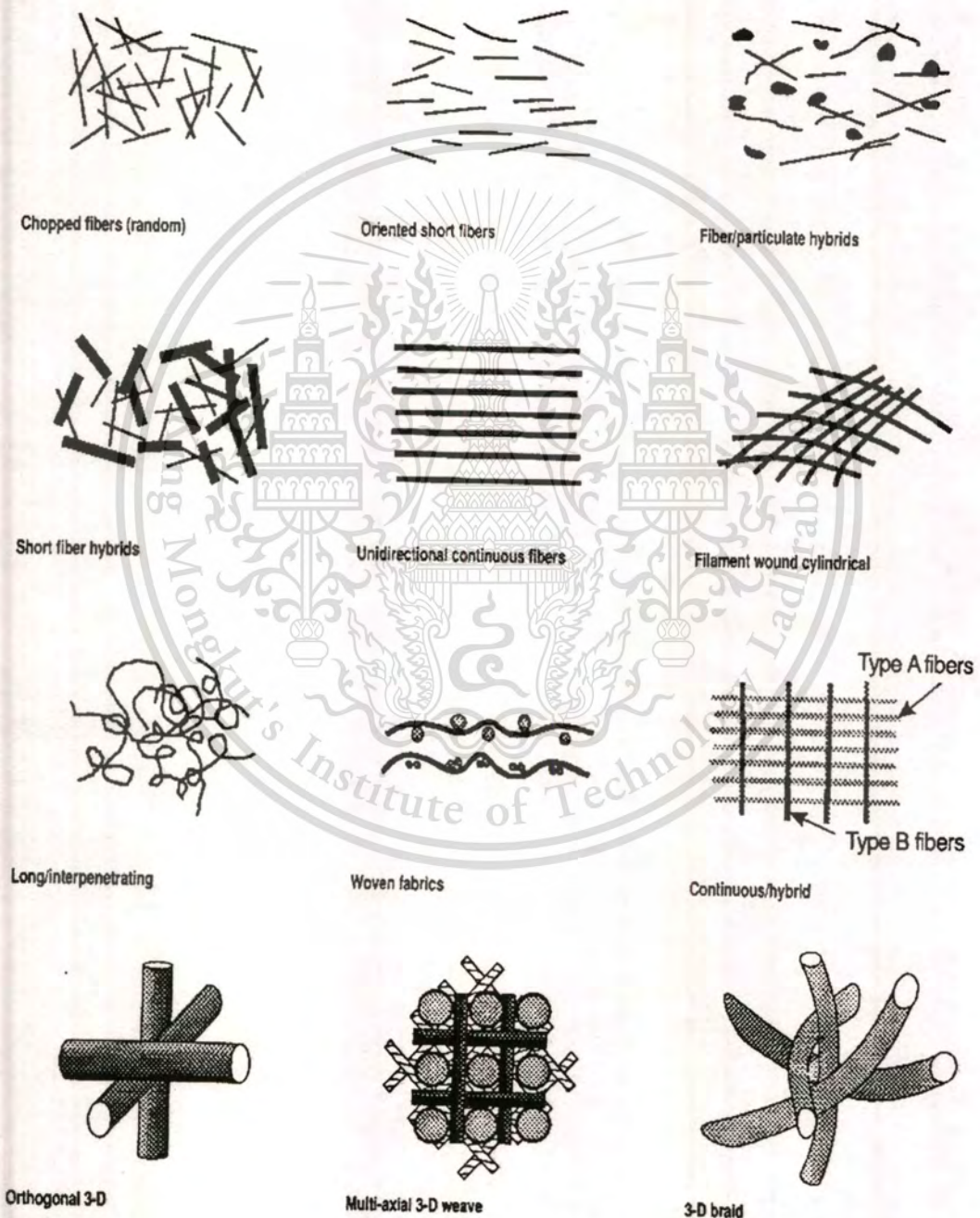


Figure 2.3 Examples of reinforcement forms [4]

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The fibers using in the fibrous composites are natural fibers and synthetic fibers. Both of them have various advantages and disadvantages to be described below.

2.2.1 Natural fibers [5]

The natural fibers such as cotton, silk, wool, jute, hemp and sisal are widely used for textiles, twine and rope throughout the world. These fibers are of course animal or plant products. The latter are essentially micro-composites consisting of cellulose fibers in an amorphous matrix of lignin and hemicellulose.

2.2.2 Synthetic fibers [7]

The typical synthetic fibers used as reinforcement materials are :

2.2.2.1 Glass fibers

Glass fibers are the most common of all reinforcing fibers for polymeric or plastic matrix composites.

The principal advantages of glass fibers are low cost, high tensile strength, high chemical resistance, and excellent insulating properties. Its disadvantages are low tensile modulus, relatively high specific gravity among the commercial fibers, sensitivity to abrasion with handling resulting in low tensile strength, relatively low fatigue resistance, and high hardness.

The principal part in all glass fibers is silica (SiO_2). Other oxides such as B_2O_3 and Al_2O_3 are added to modify the network structure of SiO_2 as well as to improve its workability.

2.2.2.2 Carbon fibers (Graphite fibers)

Carbon fibers contain a blend of amorphous carbon and graphitic carbon. Their high tensile modulus results from the graphitic form, in which the carbon atoms are arranged in crystallographically parallel planes of the regular hexagons.

2.2.2.3 Kevlar 49 fibers

The repeating unit in molecules of Kevlar 49 contains an amide group, which is also found in nylons, and an aromatic ring. The aromatic ring gives a higher chain stiffness (modulus) as well as better chemical and thermal stabilities over other commercial organic fibers, such as nylons.

2.2.2.4 Boron fibers

Boron fibers are manufactured by chemical vapor deposition (CVD) of boron onto a heated substrate either a tungsten wire or a carbon monofilament.

2.2.2.5 Ceramic fibers

Silicon carbide (SiC) and aluminum oxide (Al_2O_3) fibers are examples of ceramic fibers notable for their high-temperature applications in metal and ceramic matrix composites.

2.3 Matrices

The matrix is the material that gives body and grips or holds the reinforcements of the composites, and is usually of lower strength than the reinforcement. The matrix must be capable of being forced around the reinforcement during some stage in the manufacture of the composites [3].

The role of the matrix in a fiber-reinforced composites 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 [7].

There are a number of matrix materials available, including carbon, ceramic, glass, metal, and polymer [3].

2.3.1 Carbon matrix

A carbon matrix is used as rocket nozzles, ablative shields for reentry vehicles, clutch and brake pads in aircraft because carbon and graphite have a high heat capacity per unit weight.

2.3.2 Ceramic matrix

Ceramic materials are crystalline and have similar properties to glass. The matrix is brittle even with attempts at annealing. Sintered and cast pieces shrink during firing. Carbon, ceramic, metal, and glass fibers are used in ceramic matrices.

2.3.3 Glass matrix

Strength at high service temperatures is the most unique attribute of glass and ceramic matrices. Heat-resistant parts for engines, exhausts, and electrical components are their primary applications.

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2.3.4 Metal matrix

For demanding high temperature usage in oxidizing environments, metal matrices of iron, nickel, tungsten, titanium, aluminum, and magnesium are used in place of polymers. The low-density metals have long been a favorable choice in aircraft and aerospace designs.

2.3.5 Polymer matrix

A polymer is defined as a long-chain molecule containing one or more repeating units of atoms, joined together by strong covalent bonds. A polymeric material is a collection of a large number of polymer molecules of similar chemical structure but not equal length [7].

Polymers are selected because they are easily processed and offered good mechanical and dielectric properties. Although polymers have lower softening points than metals, they are low-density materials. It is because of the relatively low processing temperatures and production techniques that many organic reinforcements may be used. Natural and synthetic fibers including silk, cotton, wool, cellulose, polyester, polyamide, acrylic, and olefins are used [3].

2.4 Polymeric composites [8]

The role of the matrix, reinforcement, and interface in composites are well defined. The matrix is responsible for transferring the load from the matrix to the reinforcement, for distributing the stress among the reinforcement elements, for protecting the reinforcement from environmental attack, and for positioning the reinforcing materials. Meanwhile, the task of the reinforcement is to carry the load, due to its higher stiffness and strength compared with that of the matrix. The interface, for two dimensions, or the interphase, for three dimensions, is a negligible or finite thin layer with its own properties, and its role is stress transfer from the matrix to the reinforcement.

The blended polymer, usually presented as a fine dispersion in the matrix, can take on many different roles: flame retardant, processing aid, surface finishing, impact modifier, adhesion promoter between the matrix and reinforcement, and so on. Nevertheless, in the majority of cases, blending is aimed at improving the toughness of the composites, or more exactly, to achieve the desired balance between stiffness and

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toughness. It is widely assumed that stiffness and strength characteristics are related to the reinforcement, and the toughness is related to the matrix. However, this presumption generally does not hold. The toughness-enhancing mechanisms are quite complex, and both matrix deformation and fiber-related failure events are involved.

Blending of the matrix polymer for thermoplastic composite applications is done when the matrix tends to undergo brittle fracture and /or exhibits notch sensitivity. Other goals of blending are to improve the heat distortion temperature (HDT) and to reduce some environmental effects, e.g., water uptake. Blending may also be the right tool for cost-reduction purposes. The mechanical performance of the reinforced thermoplastic blends is affected by the following factors (Figure 2.4):

1. matrix blend composition and morphology
2. type and amount of the reinforcement
3. interface or interphase between matrix and reinforcement
4. processing methods and conditions, and
5. testing conditions.

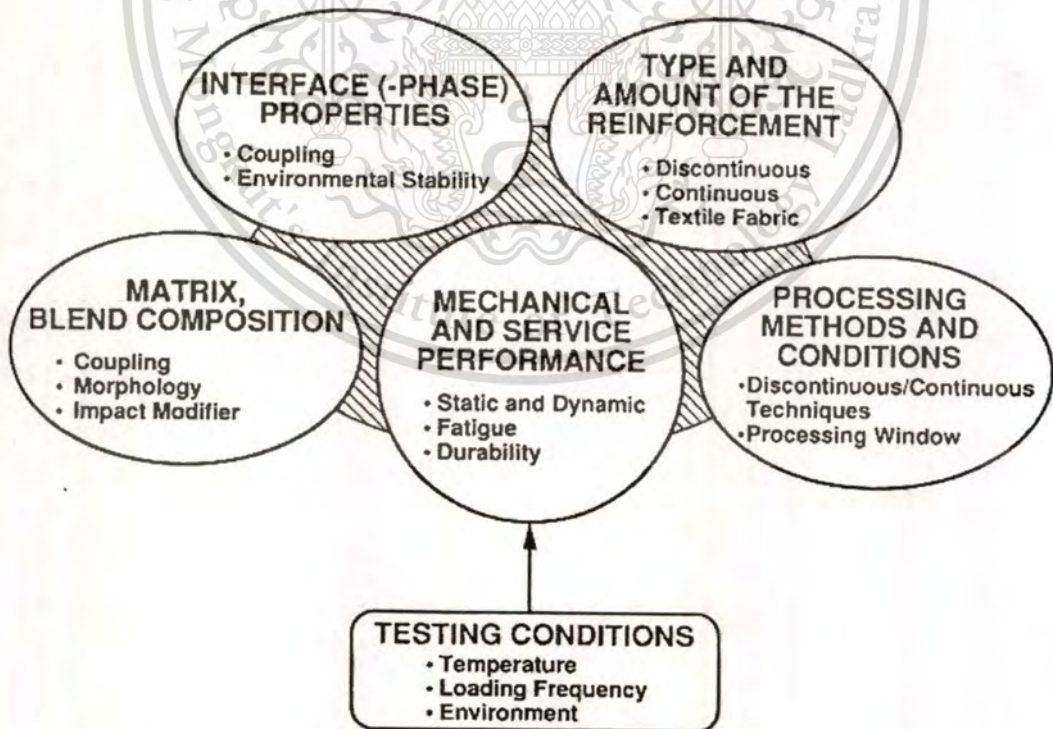


Figure 2.4 Factors affecting the mechanical performance of the reinforced polymer blends [8]

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The toughness of discontinuous fiber-reinforced composites can hardly be predicted, especially for composites with fiber structuring and polymer blend matrices. This is due to the fact that the fiber-related energy dissipation mechanisms (i.e., debonding, pullout, fracture, see Figure 2.5) may trigger or hamper the matrix-related ones. Figure 2.6 shows the interaction between fiber- and matrix-related failure mechanisms: matrix crazing is terminated by fiber-pullout-induced shear yielding, the overall outcome of which is toughness improvement. On the other hand, the impact strength may also be reduced, since the ductility (i.e., the maximum strain value) is substantially reduced by the reinforcement.

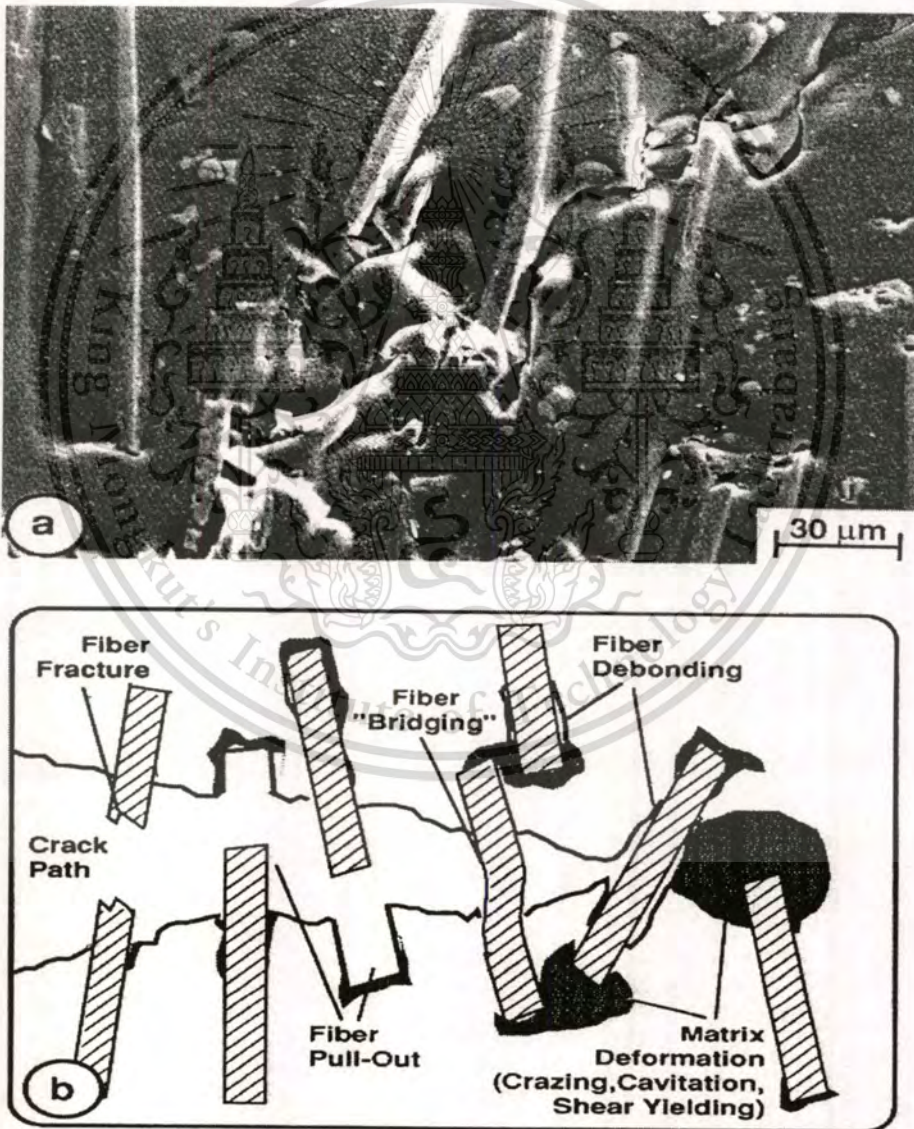


Figure 2.5 Individual failure mechanisms (a) in reality and (b) schematically for

This material discontinuous fiber-reinforced polymeric composites [8] for commercial use.

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Figure 2.6 Fiber pullout in a rubber-toughness PA-6 composites [8]

2.5 Lignocellulosic composites [9]

Any substance that contains both cellulose and lignin is a lignocellulosic material. Lignocellulosic materials include wood, agricultural crops like jute or kenaf, agricultural residues, such as bagasse or corn stalks, grasses, and other plant substances. In general, what is true for wood is also true for other lignocellulosics even though they may differ in chemical composition and matrix morphology. Wood is anisotropic. It may contain sapwood, heartwood, latewood, earlywood, juvenile wood, and abnormal reaction wood. In the wood industry, the terms composites and reconstituted wood are usually used to describe any wood product that is "glued" together. The composite products in the wood industry range from fiberboard to laminated beams and structural components.

The objective of composite developments is to produce a product with performance characteristics that combine the positive attributes of each constituent component. Like other lignocellulosic materials, wood is strong, lightweight, abundant, nonhazardous, and relatively inexpensive. Any lignocellulosic material can be chemically modified to enhance properties such as dimensional stability and biodeterioration resistance. This gives incentives to produce a variety of value-added

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products from different raw materials for the improvements in cost or performance, or both.

2.6 Derivatization of wood in lignocellulosic composites [9]

Wood as an industrial material enjoys many technological advantages. It has high specific strength and stiffness, it is relatively tough, it requires relatively little input of energy to convert itself to useful structural and decorative materials, and is increasingly important in our environmentally aware society, it is renewable, with careful management, in perpetuity. However, there are of course some drawbacks to the use of wood in comparison with other materials.

1. It is water reactive and dimensionally unstable in response to moisture change.
2. It is biodegradable, especially when moist.
3. It is photosensitive. This is important where surface integrity is of significance, e.g., under clear surface finishes.
4. It supports combustion and, in particular, flaming combustion.

2.6.1 Wood cell wall chemistry [10]

The major chemical components in wood cell walls are cellulose, hemicellulose and lignin. Cellulose is present in the greatest amount accounting for between 40 to 55% of the total cell wall mass while the hemicellulose content makes up approximately 25 to 40% and lignin about 18 to 33% of the total. The function of cellulose microfibrils is to impart strength to the cell wall while the hemicelluloses exists as a matrix material and lignin is present as an encrusting substance. Crystalline cellulose is composed of unit cells which make up the crystalline core of the microfibrils. Chain end dislocations in cellulose, and the presence of non-crystalline (amorphous) hemicellulose and lignin point to the fact that the wood cell wall is not wholly crystalline. Lignin is completely amorphous. It affords considerable rigidity to the cell wall and because of its less hydrophilic properties; it also influences the swelling characteristics of wood.

2.6.2 Enhancement of dimensional stability

Wood is arranged as a natural fiber-reinforced composite material, cellulose filling the role of fiber and the hemicellulose and lignin together forming the matrix. All three are hydrophilic to a greater or lesser extent. Each has a relatively high abundance of functional groups able to form hydrogen bonds with water. In cellulose and hemicellulose, there are 2-3 -OH groups per monomer unit, together with two "acetal" oxygens. Such a structure must be exceptionally hydrophilic. The capacity for lignin to react with water is less since it has only about 1.2 free hydroxyls and 1 ether oxygen per monomer (phenyl propane) unit. On modification with appropriate reagents, the extent of swelling is greatly reduced. This reduction in swelling is brought about by a combination of several effects as follows,

1. Substitution of hydrophilic sites by less hydrophilic groups, e.g., esters for hydroxyl
2. Bulking of the cell wall with adducts attached chemical to the cell wall polymers
3. Shielding of sorptive sites by bulky adducts
4. Cross-linking between adjacent polymer chains, in the case of modification with difunctional reagents.

2.6.3 Biological, weathering, and fire protection

With the growing interest in enhancement of dimensional properties of composites by chemical modification, some studies have been carried out to assess the modifications on biological, weathering and fire protection. The research in this section indicated that reaction with isocyanates enhanced the resistance to decay and improvement the resistance to weathering degradation due to acetylation of aspen wood and aspen fiber boards. Alongside weathering, the important source of physical degradation of wood-based materials is fire. It has been shown that bonding of phosphate flame retardants to wood surfaces by chemical reaction leads to effective leach-resistant flame retardancy.

2.7 Composite applications [4]

The types of composites and composite design technologies adopted by different sectors of industry can be quite specific to the particular requirements and practices of that particular sector. Since weight reduction in a structural design is critical to the aerospace industry and usually low-volume production is involved, more expensive fibers and resins, long fabrication time, and less automated processing techniques can be tolerated. However, in consumer-oriented industries, for example automotive and sporting goods, high volume and high production rates are normally required. Automated fabrication, short processing time, and minimization of cost are vital to the success of these industries.

2.8 Natural rubber wood [11-12]

The genus *Hevea* is a member of the family Euphorbiaceae and comprises 10 species, of which the Para rubber tree, *H. brasiliensis*, is the only planted commercially. The most important product of the rubber tree is the latex and all efforts to improve the rubber tree have been from the point of obtaining higher yield of latex. Till recently, most of the wood from the felled trees was used as fuel. With the depletion of forests in many parts of tropical regions, leading to shortage of wood for many industrial and engineering uses, attention has been given to rubber wood as an alternative source of timber. Research and development activities on the industrial applications of rubber wood are only of recent origin. New developments indicate the possibility of wider use of rubber wood for a variety of purposes. Table 2.1 lists the products which can be made from rubber wood successfully in different countries.

Table 2.1 Industrial articles from rubber wood [12]

Apron sets	Garden sets	Plywood
Bedroom sets	Gift boxes	Pulp
Benches	Hardboards	Restaurant furniture
Bread boards	Ice buckets	Railings
Building components	Irradiated timber	Rocking chairs
Block boards	Kitchen cabinets	Salad bowls

Table 2.1 (continued)

Cabinets	Knife blocks	Screen partitions
Carving boards	Living room sets	Serving trays
Chairs	Lumber	Shelves
Chests	Magazine racks	Spice racks
Chopping boards	Molded hardboards	Steak plates
Cement boards	Moldings	Stools
Charcoal	Match splints	Suitcases
Dining sets	Match boxes	Tables
Doors	Packing cases	Tea trolleys
Drawing room sets	Pallets	Television cabinets
Drawer faces	Paneling	Toilet gears
Fiber boards	Paper	Toys
Folding chairs	Particle boards	Treated lumber
Fruit boards	Picture frames	Wine racks
Furniture	Parquet flooring	Wood racks

2.8.1 Chemical compositions of rubber wood

Rubber wood is a lignocellulosic material, which contains various amounts of cellulose, hemicellulose and lignin. Table 2.2 shows the major chemical composition of rubber wood.

Table 2.2 Relative proportions (vol %) of the major chemical composition of rubber wood [13]

Cellulose	Hemicellulose	Lignin
49.41	17.17	18.06

2.8.2 Physical and mechanical properties

Like most of the wood species, the dynamic properties of rubber wood, i.e., mechanical behavior under dynamic forces are higher than the static properties. In other words, under impact loads, rubber wood is capable of taking loads nearly twice

that under slowly applied loads. However, it may be noted that the static properties of rubber wood in dry condition are higher than those in green condition.

In some countries it is customary to explain the mechanical behavior of any species for a specific function or end use, in terms of the mechanical behavior of a popular species, widely used for a variety of purposes or for the same function and end use. In India teak is one such species and so the mechanical behavior of all species is compared to that of teak as 100. The comparative figures are known as 'suitability figures' or 'suitability indices' and the same are indicated for rubber wood in Table 2.3.

Table 2.3 Comparative suitability figures of rubber wood, taking teak as 100 [12]

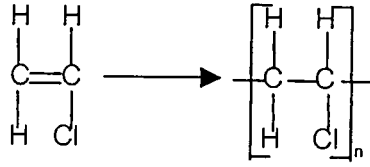
Parameters	Rating
Weight or heaviness	93
Strength as a beam	62
Stiffness as a beam	77
Suitability as a post	52
Shock resisting ability	75
Shear	92
Surface hardness	74
Splitting coefficient	75

From the table it may be seen that rubber wood is very near to the weight and shear properties of teak, and fairly comparable in other properties except in suitability as posts. However, rubber wood has to be used cautiously where compressive forces along the grain come into play. The suitability figures are derived by combining suitably the various properties in green and dry conditions that become important for the particular function or end-use. These figures serve only for comparison and not for any design or calculation of natural forces that come into action.

2.9 Poly(vinyl chloride)

Poly(vinyl chloride), PVC, a polymer prepared from vinyl chloride monomer (VCM), material is reserved for educational use only, not allowed for commercial use.

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where $n = 700-1500$, holds a unique position among all the polymers produced today. It is relatively inexpensive and is used in such a wide range of applications that its versatility is almost unlimited [14]. It can be utilized in rigid compounds or blended with plasticizers to produce flexible grades [15].

2.9.1 Commercial manufacturing processes

There are four commercial processes for the manufacture of PVC.

1. Suspension
2. Bulk
3. Emulsion
4. Microsuspension

The resins from the suspension and bulk processes are used for extruded pipe and profile. The emulsion and microsuspension processes produce resins which are used in plastisol applications, for example, coated fabric, roto-molding, and slush molding [16].

Most PVC is made via suspension polymerization. A small amount is made by mass polymerization or bulk polymerization. The organosols is produced by the emulsion process [15]. The major characteristics leading to specific usages are showed in Table 2.4.

Table 2.4 Major types of PVC resins [19]

Types	Properties	Usages
Mass	High purity and porosity, Excellent transparency and electrical properties	Film, Bottles, Containers, Cables, Pipes, etc.
Suspension	Overall good properties, High particle size	Pipe, Fittings, Leather cloth, Cables, Footware, Bottles, etc.

Table 2.4 (continued)

Types	Properties	Usages
Emulsion	Very low particle size, Low DOP absorption at room temperature, Not very pure	Leather cloth, Rotational & Slush molding, Coatings

2.9.2 Properties of PVC

Morphology plays a very important role in PVC processibility and its properties. In general, PVC can be regarded as an amorphous polymer with head-to-tail atactic structure. However, commercial PVC has about 8-10% crystallinity arising out of its small syndiotacticity [17].

Among the range of polymeric materials produced today PVC is unique because the bulky chloride atom imparts a strongly polar nature to the PVC polymer chain, and the essentially syndiotactic conformation of the repeat unit in the chain leads to a limited level of crystallinity. This results in good mechanical properties, particularly stiffness at low wall thickness, high melt viscosity at relatively low molecular mass, and the ability to maintain good mechanical properties even when highly plasticized [14].

The crystal structure of commercial PVC has been shown by both infrared and thermal analysis to melt over a wide temperature range, from about 105 to 210 °C. Crystals have a wide melting range because of different degrees of perfection (low perfection-low melting) or because the crystals are small and of varying size (small size-low melting). There is substantial evidence that PVC melts over a wide range for both reasons. It has also been shown that annealing PVC in the melting range 105 to 210 °C produces more perfect or larger crystals that subsequently melt somewhat above the annealing temperature. Either a melting and recrystallization or some other annealing process must be occurring at these temperatures [18].

In the rigid PVC case, it is generally processed at temperature between 160 and 200 °C. PVC is thus processed within its crystalline melting range and the presence of unmelted crystallinity at these temperatures can and does cause vestiges of the original particle structure to persist right through into the finished product [18].

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Commercial PVC has its glass transition temperature at 80 to 85 °C. As the PVC cools through this temperature, the molecular motions are reduced to a rate where the molecules can no longer change conformation rapidly enough to follow the equilibrium conformation that the drop in temperature would dictate [16].

PVC resin is self-extinguishing. In a fire, however, it produces hydrochloric acid and other toxic and corrosive chemicals [15].

Table 2.5 indicates the relation of PVC K-value, viscosity and molecular weight. Higher K-value increases inherent viscosity and the molecular weight. Increase in molecular weight results in stronger polymer with increasingly superior mechanical properties and also improved thermal stability. On the other hand processibility is better with lower molecular weight due to lower melt viscosity.

Table 2.5 Relation of PVC K-value, viscosity and molecular weight [17]

K-value	Inherent viscosity	No. ave. MW.	MW. distribution
1% Cyclohexanone	ASTM	g/mol	wt. ave./ no. ave. MW.
51	0.55	30,000	-
54	0.62	36,000	1.94
57	0.67	40,000	-
62	0.80	50,000	-
65	0.88	55,000	2.54
68	0.95	60,000	-
70	1.01	64,000	3.13
74	1.13	73,000	3.56
-	1.35	90,000	5.37

The various applications and processes associated with these end-products and the range of K-value are listed in Table 2.6.

Table 2.6 Applications and processing of PVC [17]

	Applications	Processing	K-value
	<u>Rigid</u>		
1.	Rigid transparent films	Calendering extrusion blown	57-60
	Bristle pack	Calendering	
	Bottles, Containers	Blow molding	
	Blow molding	Stretch blow molding	
2.	Rigid opaque bottles	Blow molding	57-62
	Containers	Blow molding	
	Pipe fittings	Injection molding	
3.	Rigid transparent films	Extrusion blown	65-69
4.	Rigid pipes, Profiles	Extrusion	65-69
	Conduits, etc.	Extrusion	
	<u>Flexible</u>		
1.	Leather cloth	Calendering	65-69
2.	Flexible films	Extrusion blown	65-69
3.	Cables	Extrusion	65-69
4.	Footwears	Injection molding	65-69

2.9.3 Compounding fundamentals [18]

PVC compounding essentially involves adding the components to the base PVC resins that will allow it be processed into a finished product with desired properties at the minimum cost. The 'families' of materials that will be chosen will probably fall into one of the following classifications:

1. Plasticizers
2. Stabilizers
3. Lubricants
4. Impact modifiers
5. Processing aid resins
6. Fillers
7. Colorants

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8. Miscellaneous, for example, antistatic agents, ultraviolet absorbers, antiblocking agents, smoke control agents, flame-retardants, fungicides, odorants.

Adding the proper type and amount of plasticizer, stabilizer, lubricant, pigment, and so on as outline above to the PVC resins results in a compounded mixture that can subsequently be processed on properly designed equipment into a satisfactory finished product.

2.9.4 Formulating rigid vinyl compounds [18]

Rigid PVC occupies a unique position in the field of major thermoplastics. It can generally be considered as a low-cost engineering thermoplastic providing excellent physical properties, excellent aging characteristic, rigidity, chemical resistance, and high impact strength. Rigid PVC is processed by the traditional means of extrusion, calendering, injection molding, blow molding, and compression molding.

The materials are as follows:

1. The basic PVC resins
2. A stabilizer system to prevent degradation during processing and use
3. Lubricants to facilitate processing
4. An impact modifier to enhance impact strength
5. A processing aid to facilitate fusion and provide ease of extrusion or molding
6. Pigments for proper color and to provide light stability, if required
7. Fillers to reduce cost or to improve impact or certain other properties
8. Plasticizers
9. Miscellaneous additives such as heat distortion improvers, light stabilizers, and antistatic agents

2.9.4.1 PVC resins

Almost all PVC resins used for rigid applications are suspension polymerized homopolymers. The physical properties are enhanced as molecular weight increases, but lower-molecular-weight resins provide higher flow and are generally easier to process. Compounds intended for extrusion are usually based on medium-molecular-weight PVC resins of which its inherent viscosity is 0.90 to 1.0. Injection molding or blow molding formulations will be based on resins with inherent viscosities of

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0.75 to 0.90. Resins with inherent viscosities as low as 0.5 are chosen for injection blow molding applications or for injection molding of intricately shaped products.

2.9.4.2 Stabilizers

PVC resins are inherently one of the most heat-sensitive polymers of the major commercial thermoplastic resins. Nevertheless, virtually all vinyl compounding and fabrication techniques require heating PVC resins. Furthermore, many fabricated vinyl articles are exposed to varying degrees of heat and aging during their normal service life. Consequently, all PVC compounders worldwide, independent of location, application, or processing technique, have a need for PVC heat stabilizers.

Heating unstabilized PVC above its fusion point initially gives rise to yellowing, followed quickly by gross discoloration, the evolution of hydrochloric acid, cross-linking, and ultimate charring to an infusible, unprocessable, corrosive black mass. To inhibit the degradation that unmodified PVC would undergo at processing temperatures, a stabilizer is added. Other purposes of the stabilizer are to react with any hydrogen chloride liberated and prevent the formation of color in the vinyl plastics as it is being processed.

A major group of stabilizers can be divided in five families: leads, organotins, mixed metal, antimony mercaptides, and stabilizer lubricant one-pack. The last two families are special classes. Stabilizers used in rigid compounding in the United States can be divided into six basic families:

1. Alkyltin mercaptides
2. Alkyltin carboxylates
3. Barium-cadmiums
4. Calcium-zincs
5. Leads
6. Antimony mercaptides

2.9.4.3 Lubricants

Lubricants used in rigid vinyl formulations generally are classified as being either internal or external in nature. The function of an internal lubricant is to facilitate the flow of the polymeric molecular network through the processing equipment.

The function of an external lubricant is to provide a barrier between the polymer melt and the processing equipment, promoting flow and inhibiting adhesion.

The proper type and amount of lubricant depend on all the other compounding ingredients and the specific processing techniques used. To a great extent, proper lubricant selection has been a trial-and-error proposition. The materials used for lubrication of rigid PVC formulations are metallic stearates and laurates, stearic acid, glyceryl mono- and diesters, fatty alcohols, paraffin waxes, stearamides, montan wax esters, low-molecular-weight polyethylenes, mineral oils, and organic stearates.

2.9.4.4 Impact modifiers

Choosing a suitable resin, stabilizer, and lubricant system will allow manufacturing of rigid vinyl products by most of the standard processing techniques. The product will be fairly brittle, however, to achieve better impact strength, certain rubberlike materials so called impact modifiers are often compounded into the formulation. Such materials generally have a limited degree of compatibility with PVC, forming a heterogeneous, two-phase system of vinyl and rubbers. The most widely used impact modifiers are ABS (acrylonitrile-butadiene-styrene) or MBS (methacrylate-butadiene-styrene) terpolymers. Impact properties can be moderately improved through the addition of ultrafine calcium carbonates with particle sizes in the range 1 to 2 μm .

2.9.4.5 Processing aids

Processing aids are designed to provide ease of processing while having a minimum adverse effect on final product properties. In calendaring they contribute to the development of a more uniform, free-flowing bank, and in extrusion they facilitate the achievement of a smooth extrudate. Three basic commercially available processing aids are the acrylics, the styrene-acrylonitrile copolymers, and the chlorinated polyethylene.

2.9.4.6 Pigments

Pigments chosen will be governed basically by the color required. One additional point to be recognized is that some pigments are based on metallic salts of lead, selenium, antimony, or copper. The metals in such pigments can react with the sulfide groups in tin mercaptide stabilizers, leading to internal sulfide

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staining. High concentrations of light-stable pigment (e.g., 10 phr TiO_2 , phr = parts per hundred parts of resin, by weight) markedly improve light stability and are often used in outdoor applications.

2.9.4.7 Fillers

The term "fillers" encompasses a very wide range of materials. For ideal filler, the characteristics should include the following:

1. Low cost
2. Availability
3. Low oil absorption
4. Good surface wetting and bonding
5. Good chemical resistance
6. High strength

2.9.4.8 Plasticizers

In processability of rigid PVC, plasticizers may be regarded as processing aids. They reduce melt viscosity and generally aid in producing a smooth end product.

Plasticizers for vinyl resins can be divided conveniently into the following seven chemical classes:

1. Phthalates — general purpose
2. Epoxides — stabilizers
3. Esters of aliphatic dibasic acids — low temperature
4. Phosphates — flame retardant
5. Polyesters — permanent
6. Extenders — low cost
7. Miscellaneous

2.10 Solid vinyl compound systems

There are three processes that must be completed to convert a PVC formulation into a finished product. It is possible that mixing, compounding, and shaping may be completed independently of one another, it is just as possible that two or all three of the steps may overlap.

2.10.1 Mixing

Synonymous with "dry blending," mixing is considered to be a process by which all the ingredients of a PVC compound are blended together mechanically to obtain a uniform distribution of the ingredients, including adequate plasticizer absorption, if it is a plasticized compound. Since mixing is normally a "batch" rather than a continuous process, any quantity samples drawn from the completed mixture or dry blend should contain the correct proportion of each component of the formula. Maximum distribution, absorption, or coating may not have occurred, but the mixing process needs to be carried on to the extent required for the particular succeeding process step [19].

Intensive dry mixers are used for dry-blending PVC resins with plasticizers and other additives. A typical mixer essentially consists of a high speed propeller-like impeller located at the bottom of a container. Heat generated during the blending cycle is continuously removed to stabilize the dry blend and to improve flow characteristics [17].

2.10.2 Compounding

Compounding is considered to be mixed and heated on a molecular scale until a continuous polymer matrix is formed, which is then said to be "fluxed." The fused mass may be processed directly into an end product or shaped into pellets and cooled [19]. This process is achievable by a mill or an extruder. The single-screw extruder functions as a mixing device, because it subjects materials to laminar-flow deformation.

2.10.3 Shaping

Shaping includes processes to form in a final product. These can include milling, extrusion, blow molding, injection molding, and calendaring of dry-blend mixtures and/or semifluxed or fully fluxed compounds into end products ready for sale, or the shaping of compounds into pelletized or diced form for later shaping into an end product [19].

2.11 Test methods

With the advent of science and technology, the concept of testing is an integral part of research and development, product design, and manufacturing. The following are some of the major reasons for testing:

1. To prove design concepts
2. To provide a basis for reliability
3. Safety
4. Protection against product liability suits
5. Quality control
6. To meet standards and specifications
7. To verify the manufacturing process
8. To evaluate competitors' products
9. To establish a history for new materials

2.11.1 Mechanical properties [20]

Among all the properties of plastic materials, the mechanical properties are often the most important property because virtually all service conditions and the majority of end-use applications involve some degrees of mechanical loading.

The basic understanding of stress-strain behavior of plastic materials is of utmost importance to design engineers. One such typical stress-strain (load-deformation) diagram is illustrated in Figure 2.7. For a better understanding of the stress-strain curve, it is necessary to define a few basic terms that are associated with the stress-strain diagram.

Stress. The force applied to produce deformation in a unit area of a test specimen. Stress is a ratio of applied load to the original cross-sectional area expressed in N/m^2 .

Strain. The ratio of the elongation to the gauge length of the test specimen, or simply stated, change in length per unit of the original length ($\Delta L/L$). It is expressed as a dimensionless ratio.

Elongation. The increase in the length of a test specimen produced by a tensile load.

Yield point. The first point on the stress-strain curve at which an increase in strain occurs without the increase in stress.

Yield strength. The stress at which a material exhibits a specified limiting deviation from the proportionality of stress to strain. Unless otherwise specified, this stress will be at the yield point.

Proportional limit. The greatest stress at which a material is capable of sustaining the applied load without any deviation from proportionality of stress to strain (Hooke's Law). This is expressed in N/m^2 .

Modulus of elasticity. The ratio of stress to corresponding strain below the proportional limit of a material. It is expressed in F/A , usually N/m^2 . This is also known as Young's modulus. A modulus is a measure of material's stiffness.

Ultimate strength. The maximum unit stress a material will withstand when subjected to an applied load in compression, tension, or shear. This is expressed in N/m^2 .

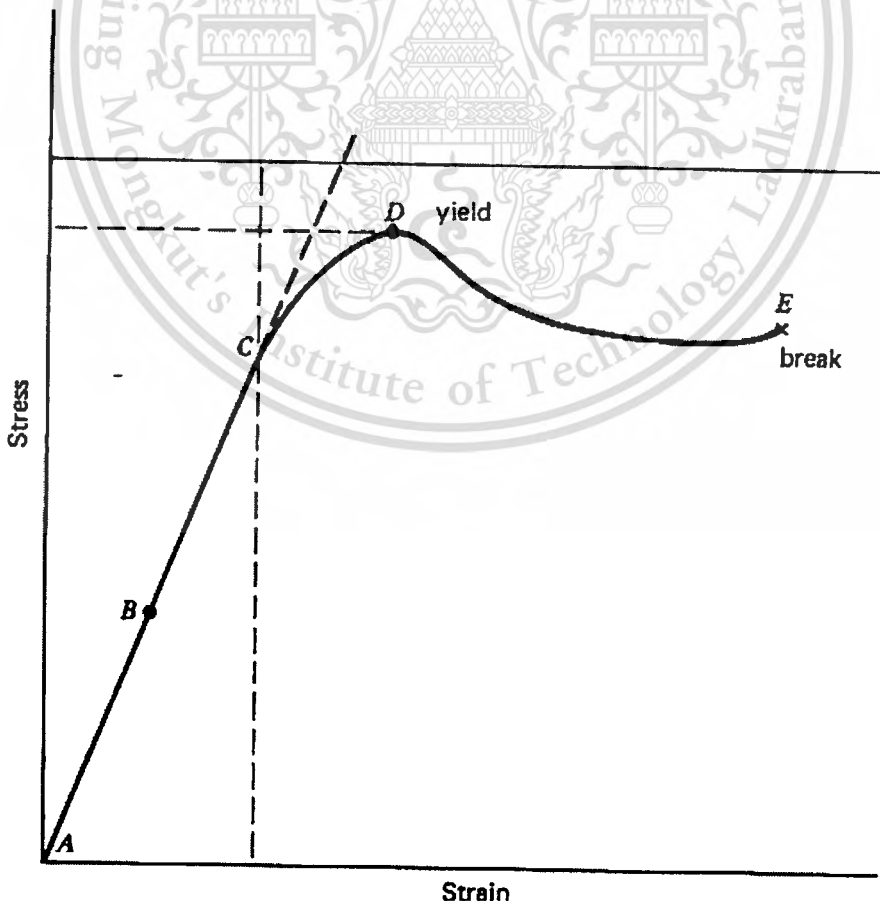


Figure 2.7 A typical stress-strain curve [20]

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Secant modulus. The ratio of the total stress to corresponding strain at any specific point on the stress-strain curve. It is also expressed in F/A or N/m²

2.11.1.1 Tensile test (ASTM D 638)

Tensile test, in a broad sense, is a measurement of the ability of a material to withstand forces that tend to pull it apart and to determine to what extent the material stretches before breaking. Figure 2.8 shows a commonly available tensile testing machine.

$$\text{Tensile strength (Pa)} = \frac{\text{Force (load) (N)}}{\text{Cross-section area (m}^2\text{)}}$$

$$\text{Tensile strength at yield (Pa)} = \frac{\text{Maximum load recorded (N)}}{\text{Cross-section area (m}^2\text{)}}$$

$$\text{Tensile strength at break (Pa)} = \frac{\text{Load recorded at break (N)}}{\text{Cross-section area (m}^2\text{)}}$$

$$\text{Tensile modulus (Pa)} = \frac{\text{Difference in stress}}{\text{Difference in corresponding strain}}$$

$$\text{Elongation at yield (mm.)} = \text{Strain (at yield)} \times \text{Original length}$$

$$\text{Percent elongation at yield} = \text{Elongation at yield} \times 100$$

$$\text{Elongation at break (mm.)} = \text{Strain (at break)} \times \text{Original length}$$

$$\text{Percent elongation at break} = \text{Elongation at break} \times 100$$

The advent of new microprocessor technology has virtually eliminated time-consuming manual calculations. Stress, elongation, modulus, energy, and statistical calculations are performed automatically and presented on a visual display or hard copy printout at the end of the test.

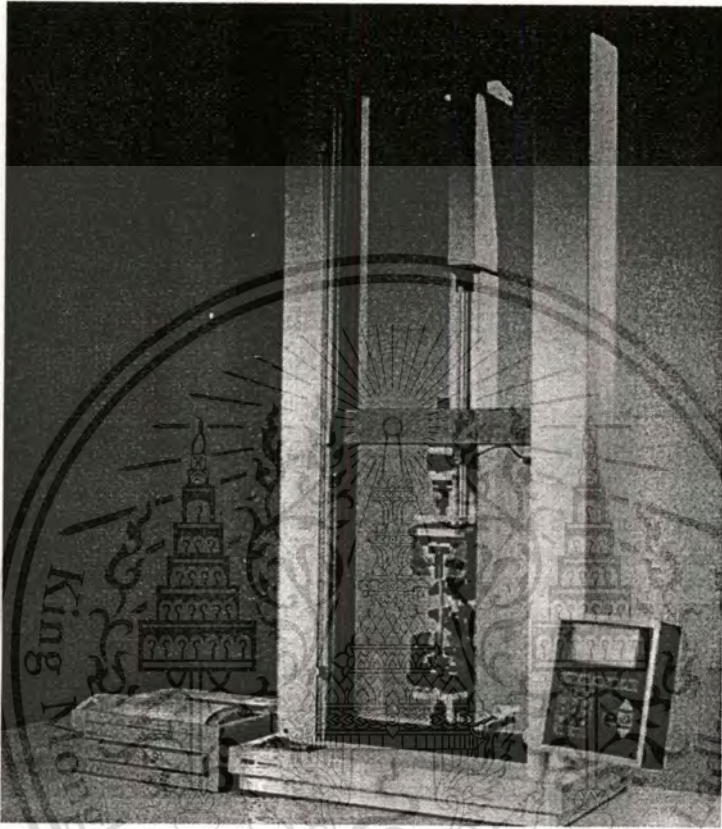


Figure 2.8 Tensile testing machine [20]

2.11.1.2 Impact test (ASTM D 256)

Impact properties of the polymeric materials are directly related to the overall toughness of the materials. Toughness is defined as the ability of the polymer to absorb applied energy. The area under the stress-strain curve is directly proportional to the toughness of a material. Impact energy is a measure of toughness. Impact resistance is the ability of a material to resist breaking under a shock loading or the ability to resist the fracture under stress applied at a high speed.

The objective of Izod-Charpy impact test is to measure the relative susceptibility of a standard test specimen to the pendulum-type impact load. The results are expressed in terms of kinetic energy consumed by the pendulum in

order to break the specimen. The energy required to break a standard specimen is actually the sum of energies needed to deform it, to initiate its fracture, and to propagate the fracture across it, and the energy expended in tossing the broken ends of the specimen. This is called the "toss factor." The energy lost through the friction and vibration of the apparatus is minimal for all practical purposes and usually neglected. A typical of machine is shown in Figure 2.9.

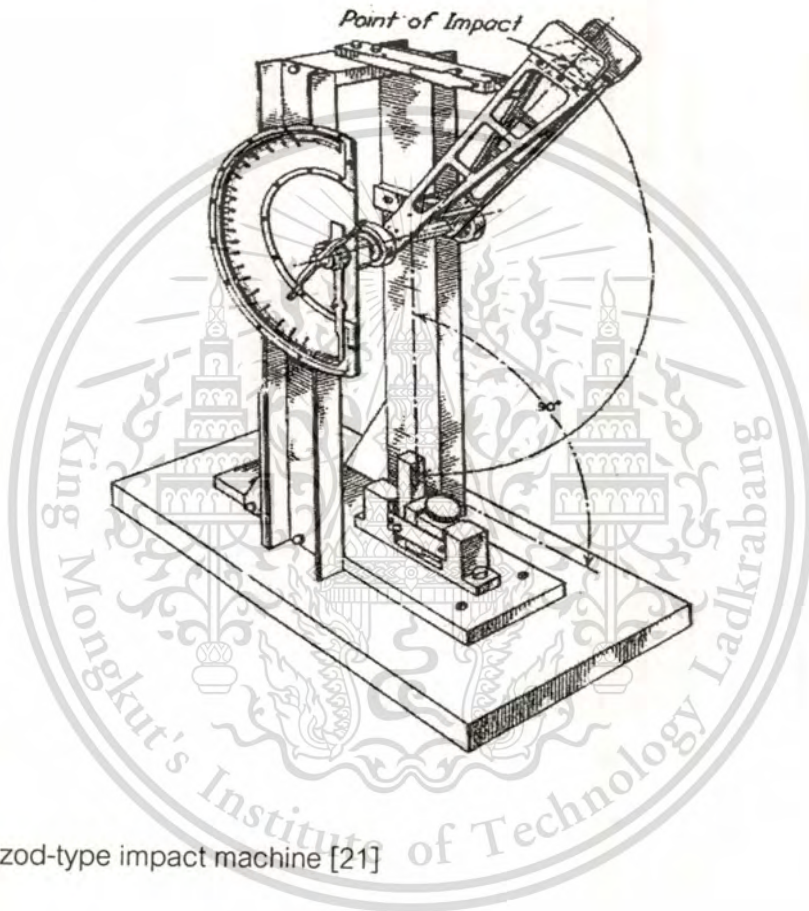


Figure 2.9 Izod-type impact machine [21]

2.11.1.3 Flexural test (ASTM D 790)

Flexural strength is the ability of the material to withstand bending forces applied perpendicular to its longitudinal axis. The stresses induced by the flexural load are a combination of compressive and tensile stress. This effect is illustrated in Figure 2.10. Flexural properties are reported and calculated in terms of the maximum stress and strain that occur at the outside surface of the test bar. Many polymers do not break under flexure even after a large deflection that makes determination of the ultimate flexural strength impractical for many polymers. In such

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cases, the common practice is to report flexural yield strength when the maximum strain in the outer fiber of the specimen has reached 5 percent. For polymeric materials that break easily under flexural load, the specimen is deflected until a rupture occurs in the outer fibers.

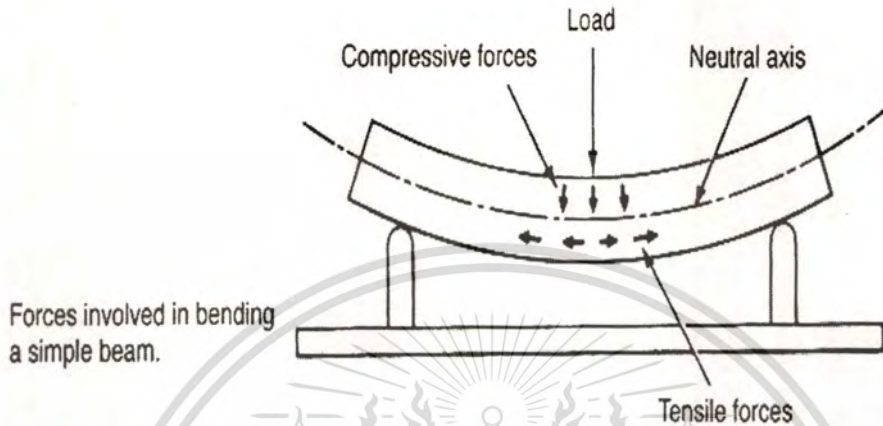


Figure 2.10 Forces involved in bending a simple beam [20]

There are two basic methods that cover the determination of flexural properties of plastics. Method 1, Figure 2.11, is a three-point loading system. This method is especially useful in determining flexural properties for quality control and specification purposes. Method 2, Figure 2.12, is a four-point loading system. This method is very useful in testing materials that do not fail at the point of maximum stress under a three-point loading system.

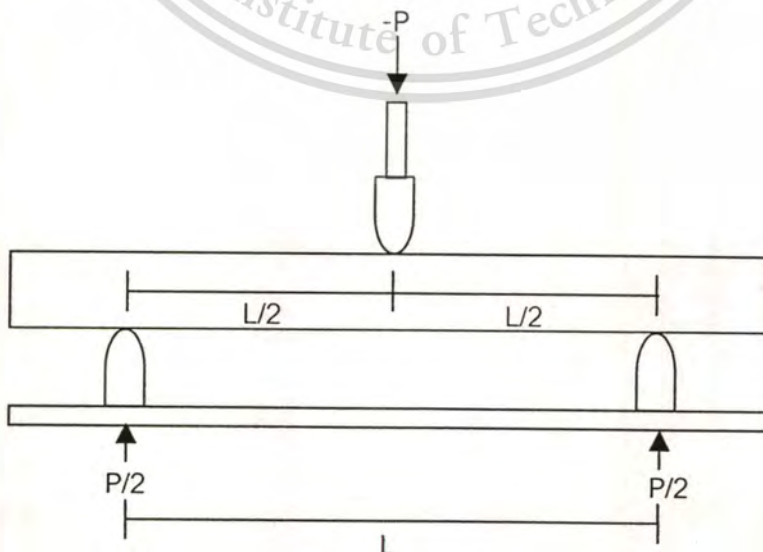


Figure 2.11 Schematic of specimen arrangement for flexural testing (Method 1) [22]

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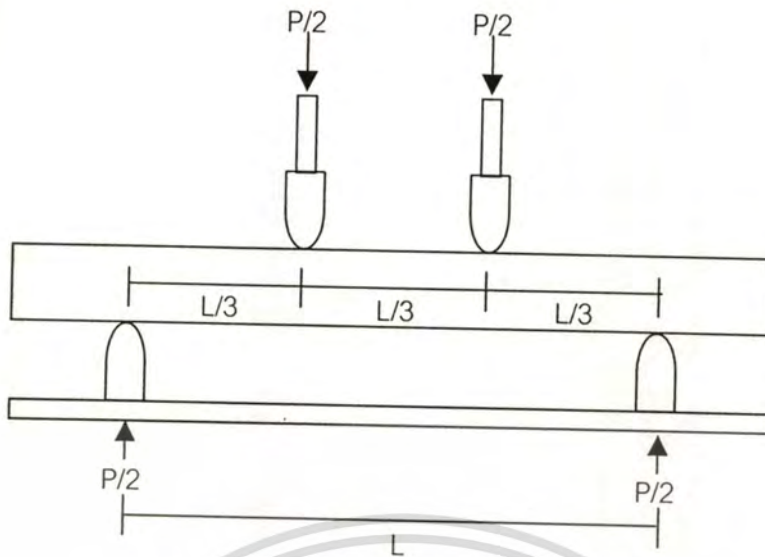
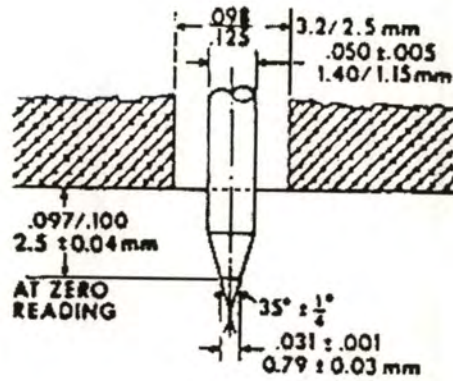


Figure 2.12 Schematic of specimen arrangement for flexural testing (Method 2) [22]

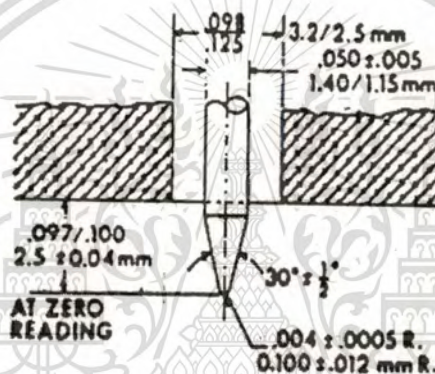
2.11.1.4 Hardness test (ASTM D 2240)

Hardness is defined as the resistance of a material to deformation, particularly permanent deformation, indentation, or scratching. Hardness is purely a relative term and should not be confused with wear and abrasion resistance of plastic materials. Two of the most commonly used tests for plastics are the Rockwell and durometer hardness tests. The Rockwell test (ASTM D 785) is used for relatively hard plastics such as acetals, nylons, acrylics, and polystyrene. For softer materials such as flexible PVC, thermoplastic rubbers, and polyethylene, Durometer hardness (ASTM D 2240) is measured.

Two types of durometers are most commonly used—Type A and Type D. The basic difference between the two types is the shape and dimension of the indenter. A commercially available durometer hardness measuring instrument is shown in Figure 2.13.



(a) Indenter for type A durometer



(b) Indenter for type D durometer

Figure 2.13 Indenter for durometer hardness tester [23]

2.11.2 Thermal analysis

2.11.2.1 Thermogravimetric analysis [20]

Thermogravimetric analysis (TGA) is a test procedure in which changes in the weight of a specimen is monitored as the specimen is progressively heated. The sample weight is continuously monitored as the temperature is increased either at a constant rate or through a series of steps. The component of a polymer or elastomer formulation volatilize or decompose at different temperatures. This leads to a series of weight-loss steps that allow the components to be quantitatively measured. A typical high-performance apparatus consists of an analytical balance supporting a platinum crucible for the specimen, the crucible situated in an electric furnace.

Variations in instrumentation include horizontally mounted furnaces and top-loading balances.

TGA is very useful in characterizing polymers containing different levels of additives by measuring the degree of weight loss. It can also be used to identify the ingredients of blended compounds according to the relative stabilities of individual components. Figure 2.14 shows a typical TGA thermogram. From the thermogram, it clearly presents that the components within the mineral-filled polypropylene decompose at different temperature, and the weight loss can be measured.

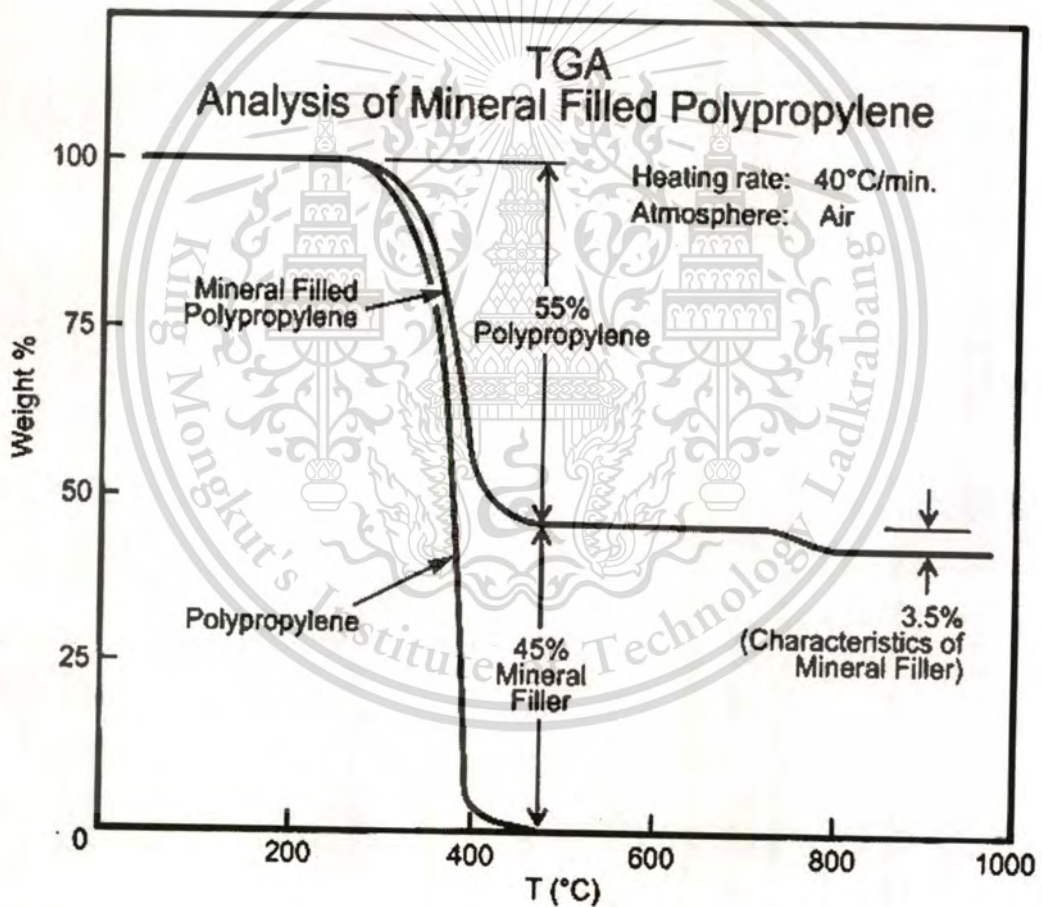


Figure 2.14 A typical TGA thermogram [20]

2.11.2.2 Differential scanning calorimetry

In differential scanning calorimetry (DSC), the most widely used thermal analysis technique, the heat flow rate to the sample (differential power) is

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measured while the temperature of the sample, in a specified atmosphere, is programmed. Because all materials have a finite heat capacity, heating or cooling a sample specimen results in a flow of heat in or out of the sample.

A hypothetical DSC curve showing both endothermic and exothermic changes in a polymer is shown in Figure 2.15 [21].

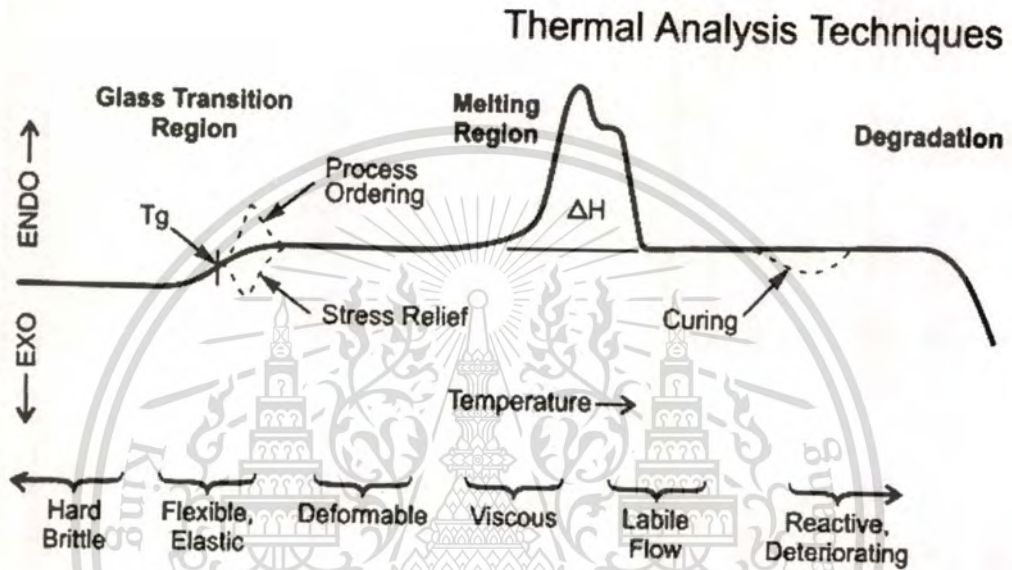


Figure 2.15 A typical DSC thermogram [20]

Initially, constant energy input is required to heat the sample at a constant rate. This establishes a baseline. At a transition point, the sample requires either more or less energy depending on whether the change is endothermic or exothermic. For example, when the glass transition point is reached, the heat capacity increases. The midpoint is taken as the glass transition temperature (T_g). Adding plasticizers to a formulation lowers T_g . If the polymer is semicrystalline, it must be quenched from the melt state rapidly to give a wholly amorphous structure; otherwise the presence of crystals can impede the motion of polymer chains and result in a T_g value that is higher than the true value [25]. When a polymer reaches the melting point, it requires more energy (endothermic) to melt the crystalline structure. The area of the peak in units of energy is the enthalpy of fusion, the heat of melting. The temperature

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dependence of the peak and its shape give information about degree of crystallinity, the molecular-weight distribution, degree of branching, copolymer blend ratio, and or processing history. Often quality procedures involve comparing the melting profile to that of a standard "good" material. When a sample cures, more energy is usually released, and the change is exothermic. The area of the curing peak is proportional to the number of crosslinks that are formed.

A DSC contains two sample holders, each provided with its own heater. The actual sample is placed in one of the sample holders in an aluminum pan, whereas the other sample holder contains an empty pan. The temperatures of both sample holders are increased at a constant rate such as $10^{\circ}\text{C}/\text{min}$ [25]. The temperature of each container is monitored by a heat sensor. If the sample suddenly absorbs heat during a transition, this change will be detected by the sensor, which will initiate a greater current flow through the heater to compensate for the loss. Thus, absorption of heat by the sample results in a greater current flow. Since the change in electric current can be monitored accurately, this provides a sensitive measure of transition temperatures [26].

2.11.3 Thermomechanical properties

2.11.3.1 Heat distortion temperature [8]

The heat distortion temperature (HDT), which is often referred to as the deflection temperature under load, is the temperature at which an arbitrary deformation occurs in a molded part or sheet subject to an arbitrary loading condition. In this measurement, a bar with a rectangular cross section is tested as a beam simply supported at both ends, with the load placed at its center. For adequate temperature control, the bar is submerged in a bath containing a heat-transfer medium, i.e., an oil bath. Figure 2.16 shows a deflection temperature test apparatus.

The procedure consists of submerging a conditioned bar of the appropriate geometry in the bath. A load is then applied to the center of the bar. A dial gauge that measures the deflection of the bar under load is zeroed. Temperature is increased at $2^{\circ}\text{C}/\text{min}$, and the point at which the bar deflects 0.25 mm (0.01 inch) is recorded as the deflection temperature under load, or the heat distortion temperature.

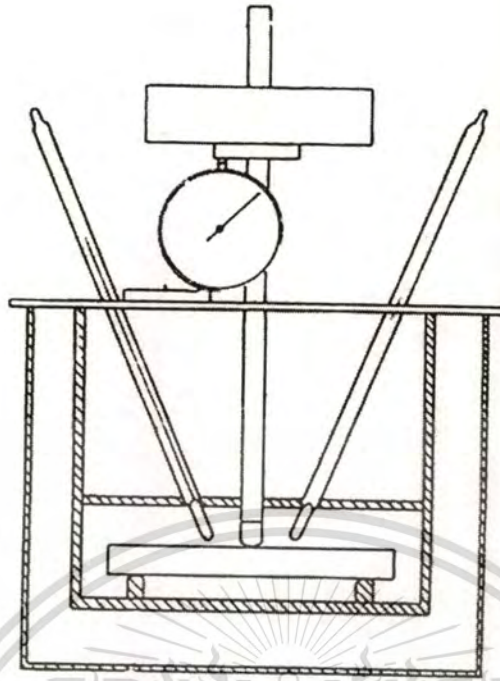


Figure 2.16 Deflection temperature test apparatus [26]

2.11.3.2 Dynamic mechanical thermal analysis

For amorphous samples, a DSC fails to even pick up a glass transition temperature. In such a case the operation turns to dynamic mechanical thermal analysis (DMTA), wherein a polymer sample, whether glassy or rubbery, is deformed in an oscillatory manner in tension or shear, as appropriate such that the maximum strain amplitude is infinitesimal in magnitude [25]. One of the most common uses of DMTA is to study molecular-level thermal transitions in polymeric materials. The most conspicuous of these transitions is the glass transition temperature (T_g). Below this temperature, an amorphous polymer is a glass, and thermal energy is insufficient to cause rotation and translation of molecule segments [8].

2.11.4 Water absorption [20]

Water absorption characteristics of plastic materials are altered by the addition of additives such as fillers, glass fibers, and plasticizers. These additives show a greater affinity to water, especially when they are exposed to the outer surface of the molded article. Washing machine agitators, plastic dinnerware, irrigation valves, and sprinklers are examples of applications requiring low water absorption. Table 2.7 lists

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typical water absorption values of some common plastics.

Table 2.7 Water absorption of common plastics [20]

Plastic material	Percent absorption
ABS	0.20-0.45
Acetal	0.22-0.25
Alkyd	0.50-0.25
Acrylic	0.30-0.40
Cellulose acetate	2.00-7.00
Cellulose acetate butyrate	0.90-2.20
Cellulose propionate	1.20-2.80
CTFE	0.00
Epoxy (unfilled)	0.08-0.15
Nylon	
Type 6	1.30-1.90
Type 66	1.50-2.0
Type 610	0.40
Type 612	1.5
Type 11	1.10
Polycarbonate	0.15-0.35
Polyester (thermoplastic)	0.8-0.38
Polyethylene	0.010
PPO (Noryl)	0.06-0.07
Polypropylene	0.010
Polysulfone	0.22
Polystyrene	0.03-0.6
SAN	0.2-0.3
TFE	0.01
Urea formaldehyde (cast)	0.02-1.50
PVC	0.07-0.75

The test to determine the water absorption of plastics is relatively simple. Details will be described in 4.6.4 in Chapter 4. Only two pieces of equipment are required—an analytical balance and an oven capable of maintaining a uniform temperature. The test specimen may be a molded disk or a piece cut from a sheet, rod, or tube. Dimensions vary according to the type of specimen. A special conditioning procedure must be followed before actual testing.

2.11.5 Specific gravity [27]

A simple method of determining the specific gravity of plastic materials is by the use of a direct reading specific gravity balance, Figure 2.17. This direct reading specific gravity balance eliminates time-consuming labor and calculations. The specific gravity of the plastic samples is read at this dial pointer.



Figure 2.17 Specific gravity balance [27]

2.11.6 Morphology [28]

The large depth of field of the scanning electron microscopy (SEM) makes it ideally suited to the examination of samples with a high degree of surface relief. This feature, together with the ease with which fracture surfaces can be prepared, has led to the widespread use of fractography to study the internal structure of polymer blends. The best results have been obtained when the internal phases are poorly

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bonded and the matrix undergoes brittle failure. These conditions produce surfaces having an assortment of debonded particles and cavities, which are often used to make crude assessments of particle-size distributions and adhesion. While useful for qualitative comparisons, the uncertain nature of the fracture process, the large number of particles that are ejected from the fracture plane, and the crude link between visual appearance and interfacial adhesion limit the extent to which this preparation technique can be used to extract quantitative information regarding blend morphology. Micrographs obtained in this way are more commonly overinterpreted than any other micrographs.

2.12 Extrusion of profiles [29]

There are almost no boundaries for creativity regarding the shape and design of profiles, see Figure 2.18. Applications in the construction industry hold first place, followed by furniture and vehicle manufacturing. However, plastic profiles gain more importance in the area of electronics, and in areas of special packing and communication technology.

2.12.1 Processes for the manufacture of profiles

Critical factors for the quality of the end product and the efficiency of the production process are the correct layout and optimum combination of extruder, shaping and sizing die, calibrating and cooling unit, haul-off and cutting devices, right up to the discharge station. The appropriate processing technique, equipment and the die design are decided upon with the following criterias:

1. Profile shape
2. Profile weight
3. Outer and inner profile dimensions and contours
4. Grade of resins and form of feedstock

The profile shape directly determines the shaping and sizing die design to be used. The grade of the resins determines the extruder type (single- or twin-screw extruder) to be used and its design features.

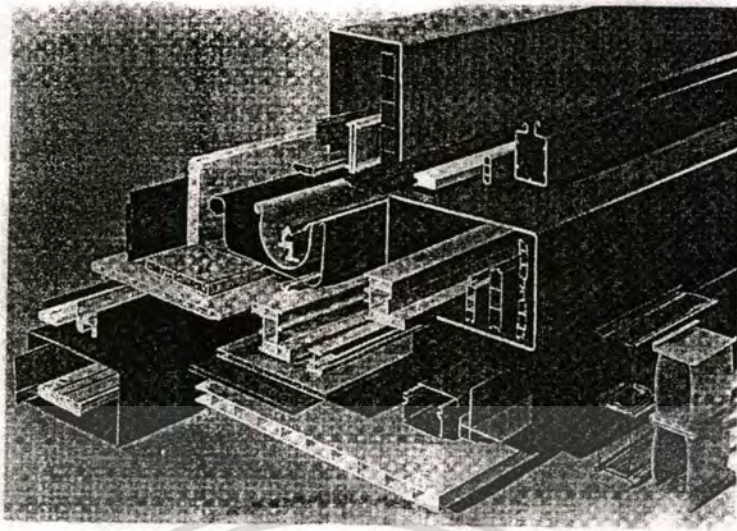


Figure 2.18 Extruded profiles [29]

The following selection criteria for individual component of the profile extrusion line are dependent on profile and resins.

2.12.2 Extruder

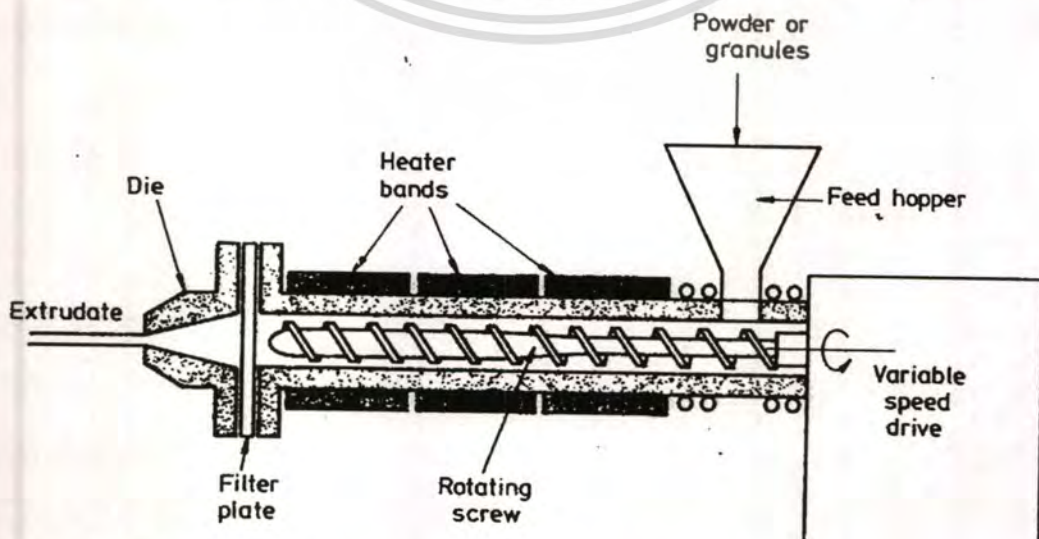
One of the most common methods of processing plastics is extrusion using a screw inside a barrel as illustrated in Figure 2.19 [30]. The plastic, usually in the form of granules or powder, is fed from a hopper on to the screw. It is then conveyed along the barrel where it is heated by conduction from the barrel heaters and shear due to its movement along the screw flights. The depth of the screw channel is reduced along the length of the screw so as to compact the material. At the end of the extruder the melt passes through a die to produce an extrudate of the desired shape.

Screw extruders are divided into single screw and multi screw extruders. The single screw extruder is the most important type of extruder used in the polymer industry. Its key advantages are relatively low cost, straightforward design, ruggedness, reliability, and favorable performance per cost ratio.

The extruder screw of a conventional plasticating extruder has three geometrically different sections, as shown in Figure 2.20 [31]. The first section is referred to as a "single stage." The single stage refers to the fact that the screw has only one compression section, even though the screw has three distinct geometrical sections. The first section which is closest to the feed opening generally has deep

flights. The material in this section will be mostly in the solid state. This section is referred to as the feed section of the screw. The last section which is closest to the die usually has shallow flights. The material in this section will be mostly in the molten state. This screw section is referred to as the metering section or pump section. The third screw section connects the feed section and the metering section. This section is called the transition section or compression section. In most cases, the depth of the screw channel or the height of the screw flight reduces in a linear fashion, going from the feed section towards the metering section, thus this compression, in many cases, is essential to the proper functioning of the extruder. A designation often used is the length of the extruder, generally expressed as length to diameter (L/D) ratio.

The extruder is matched to the required production capacity and the raw material. The design of the extruder used for profile extrusion depends on the technical requirements. In practice, single-screw extruders and counter-rotating twin-screw extruders are typically employed in the field of profile extrusion. Due to its good price per performance ratio and its versatility, the single-screw extruder is used when conveying, plasticizing and homogenizing of thermoplastics in pellet form are required. One of the most frequently used continuously operating plastics processing machines in profile extrusion is the counter-rotating twin-screw extruder, because most of the profile capacities are processed from PVC dry blends. Due to forced conveying, self-cleaning and gentle plastification of the heat sensitive PVC, the counter-rotating twin-screw extruders are far superior to the single-screw extruders in processing PVC dry blends.



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Figure 2.19 Schematic view of a single-screw extruder [30]

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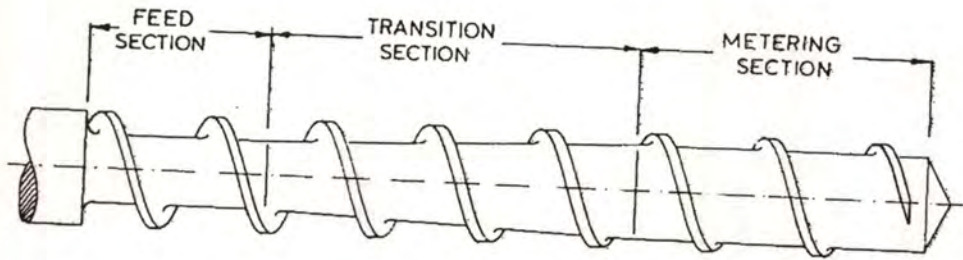


Figure 2.20 Geometry of a conventional extruder screw [31]

2.12.3 Profile extrusion die

The task of the shaping die and die insert is to distribute the melt over the total profile cross-section and transform it into the desired profile. The flow channel must be designed in such a way that the melt flow rate at all points of the cross-section is virtually the same. The residence time of the melt in the die should be short and vary little around the profile. A sufficiently long parallel flow before exit from the die aids steady flow and promotes good surface qualities. The calibration system then takes over the task of stabilizing the shaped melt stream and the final shaping. Calibration is carried out with the help of vacuum and cooling.

In describing the various processes used in profile extrusion, it is useful to have a closer look on how the die is designed. As a general rule, one differentiates between "hot" and "cold" parts of the die. Only the shaping die or die insert, also called the profile extrusion die, is hot. The cold part of the die is formed by the calibration unit and the dimensional and shape-dependent stabilizing elements in the downstream equipment, see Figure 2.21.

The construction of a profile die is determined by the size and shape of the profile and by the raw material.

2.12.4 Calibration unit and cooling unit

A solid base is required for the construction of the calibrating and cooling units. Auxiliary units such as vacuum pumps, circulating pumps and cooling blowers, should be vibration-free. The base must be adjustable both vertically and sideways, and movable lengthwise in order to match the various processing techniques and profile contours. Cooling channels, and spray or water baths, in addition to vacuum tanks with spray or flooding possibilities, are used.

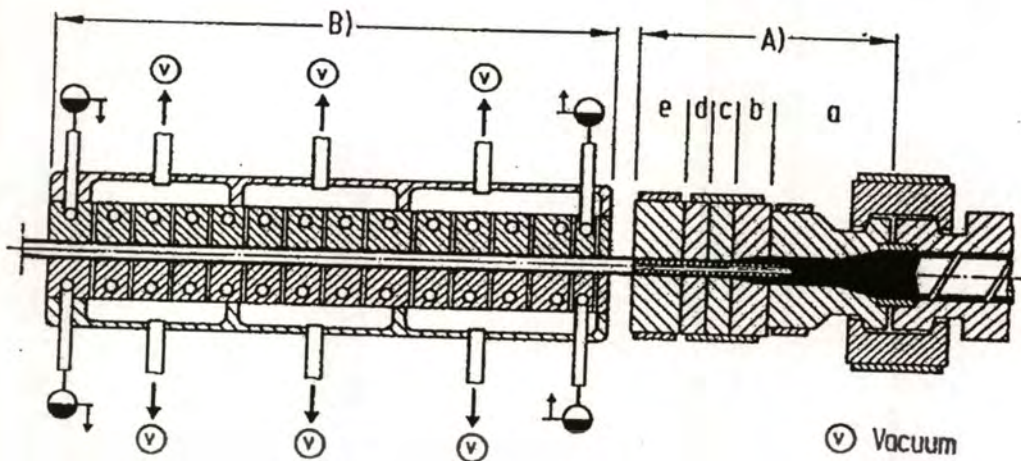


Figure 2.21 Profile die-basic design. A) hot die part (profile extrusion die): (a) connection piece with laminar flow zone, (b) intermediate or transition plate, (c) core or mandrel retainer plate with distributor tip, (d) regulating plate, (e) mouthpiece disk with parallel guide B) cold die part (vacuum tank calibration unit) [29]

2.12.5 Haul-off unit

A variety of haul-off systems are listed below. Any particular choice depends on the shape, calibrating process and surface nature of the profiles to be produced.

1. Roller haul-offs for small profiles, flexible profiles of which the required tractive power and pressure are small.
2. Belt haul-offs for profiles with surfaces sensitive to pressure, scratching and flexible profiles of which tractive forces less than 5000 N are required.
3. Pad-chain haul-offs for profiles which require high tractive forces, unstable profiles which tend to twist.

2.12.6 Cutting unit

Various cutting systems are used. The choice depends on materials to be cut, extrusion speed, cut length, and the required cutting quality.

2.12.7 Discharge unit

Depending on length, take-up capacity, and the product properties.

CHAPTER 3

LITERATURE REVIEWS

In fiber-plastic composites, the most widely used fibers in load-bearing applications is glass fibers which suits special high-performance duties. Natural fibers such as cellulose or lignocellulose are employed. Almost all polymers can be reinforced with fibers. Comparing properties of the fibers with those of polymers, fibers are much stiffer and stronger and they are obtained from renewable natural resources and inexpensive, while polymers have good chemical resistance and other special qualities. Cellulose fibers have been frequently used in thermoset matrices but their use in thermoplastic matrices has been limited by the incompatibility between the fibers and the matrices, and by the difficulty to achieve a good dispersion of the fibers in the polymeric matrices [1,3]. The researches involved are reviewed as follows.

Escamilla et al. [32] studied mechanical properties of plasticized PVC that were improved by grafted or ungrafted cellulose fibers. The reduction of elastic modulus was independent of the amount of grafted PMMA or PBA, but the tensile strength decreased with the PBA content on the PBA-grafted fibers. Either the grafted or ungrafted cellulose fibers improved the elastic modulus of plasticized PVC composites. The best results were obtained for PMMA-grafted cellulose fibers because of the better fiber-matrix adhesion.

Park and Balatinecz [33] investigated the mechanical properties of wood-fiber PP composites and wood-fiber PP composites. Ethylene-propylene diene terpolymer (EPDM) was used to modify impact toughness. The incorporation of wood fiber increased the stiffness and strength whereas the addition of EPDM reduced these two properties but improved the impact toughness.

Karnani et al. [34] indicated that it was possible to enhance properties of biofiber reinforced composites through functionalization of polypropylene matrix with maleic anhydride (MA) and fiber surface modification. Composites based on the modified matrix had, in general, superior mechanical properties than those containing the

unmodified matrix, primarily as a result of improved adhesion and enhanced polar interactions at the fiber/matrix interface.

Gowda et al. [35] studied the renewable source of natural fibers to use as untreated jute fabric-reinforced polyester composites and their mechanical properties. It was concluded that although mechanical properties of jute/polyester composites did not possess strength and modulus as high as those of the conventional composites, they did have better strength than wood composites and some plastics. Therefore, these composites could be considered for future materials use. Since the reinforcing material is eco-friendly, non-toxic, non-health hazardous, low cost and easily available as compared to the conventional fibers like glass, Kevlar, asbestos etc., the composites are a good substitute for wood in indoor applications such as shelves, partitions, wash basins and table tops, and might also be suitable for outdoor uses such as roofing, drainage pipes, automobile components, electrical fittings as well as larger items such as lightweight fishing boats. To ascertain their suitability for outdoor applications, a few more tests were carried out to evolve the hygrothermal and weather resistance properties of these composites.

Bledzki et al. [36] studied thermoplastics reinforced with wood fillers. This research indicated that untreated fillers and matrix composites showed a low tensile strength and high level of moisture sorption. To build composites with high mechanical properties and a long life, surface modification of the wood and/or the polymer matrices was necessary. Modified wood-polymer interaction mechanisms are complex and specific for each definite system and processing conditions. Through a combination of wood fillers, thermoplastics, modifiers and compounding methods, it was possible to create new composite materials with a desirable combination of properties for the use in building, engineering, and automobile industries, as well as for the processing of furniture, storage bins, recreation boats, toys, and games.

Deshpande et al. [37] investigated fiber extraction from bamboo and the use of these bamboo fibers as the reinforcement in polymeric composites. A combination of chemical and mechanical methods was used for the extraction of bamboo fibers. Conventional methods of compression molding technique (CMT) and roller mill technique (RMT) were explored for the mechanical separation. Bamboo fibers obtained

from CMT and RMT were used to make unidirectional composites of polyester. High values of tensile strength were observed in all the composites. The predominant mode of failure for the composites was shown to be the cracking of the fiber-matrix interface.

Clemons et al. [38] studied the mechanism of fracture under impact loading in cellulose-reinforced polypropylene. Dynamic critical energy release rates and dynamic critical stress intensity factors were deduced from Charpy impact test. Fiber orientation increased with cellulose fiber content. At high loading, a material approximating a three-layer structure was formed. The reinforcing fibers had a high degree of curl in the final composites. Greater curl was seen at lower cellulose fiber contents. The linear elastic fracture mechanics analysis appeared to work well for composites up to about 40% cellulose fiber content by weight. Both energy and stress analyses showed increase in dynamic fracture toughness as level of reinforcement increases. The dynamic modulus increased with cellulose fiber content.

Shah and Lakkad [39] presented the mechanical properties of epoxy and polyester resins reinforced unidirectionally by jute and glass fibers singly and in combination as a hybrid. The results showed that the jute fibers, when added into the resin matrix as a reinforcement, considerably improved the mechanical properties, but the improvement was much lower than that obtained by glass fibers and other high performance fibers. Hence, the jute fibers can be used as the reinforcement where modest strength and modulus were required. The main problem of this work was the difficulty in adding a large quantity of jute fibers into the jute-reinforced plastic laminates because the jute fibers, unlike glass fibers, soaked up large amount of resins. This problem was overcome when 'hybridising' with glass fibers was carried out.

Ishak et al. [40] studied the hygrothermal aging and tensile behavior of injection-molded rice husk-filled polypropylene (RH-PP) composites. The incorporation of RH into PP matrix resulted in a significant enhancement in the tensile modulus of the composites. However, this was at the expense of the reduction in the ultimate properties, namely, the tensile strength and energy to break. Qualitative evidence derived from the SEM observations indicated that the poor ultimate performance could be attributed to the poor filler-matrix interfacial bonding, to the size irregularity of the RH, and also to the nature of RH fillers, which had a strong tendency to exist in the form of

bundles. The extent of deterioration incurred by hygrothermal aging was dependent on the immersion temperature. Both the tensile strength and tensile modulus deteriorated as a result of the combined effect of thermal aging and moisture attack.

Li et al. [41] characterized the impact resistance of the sawdust-recycled PP composites through both the standard Izod test and the Charpy experiment. The difference between the two groups of compounds revealed the reinforcing potential of the sawdust filler and the positive effect of the maleic-anhydride-grafted PP (MAPP) compatibilizer. It was observed that the notched Izod strength of the MAPP-containing composites tended to increase with the filler content, and might exceed that of the neat matrix resin. Without the MAPP additive, however, the Izod strength decreased with the filler content and was always lower than the strength of the resins. Impact fracture toughness was studied through the Charpy test. It was found that the fracture energy of the compounds was lower than that of the neat matrix resins. The fracture energy increased significantly with the filler content in MAPP-containing composites, but decreases slightly in composites without the additive. The fracture toughness, however, increased with the filler content for both groups of compounds.

Xu et al. [42] showed the creep resistance of wood-filled polystyrene/high-density polyethylene blends. The advantages of blending a plastic of lower-creep polystyrene (PS) with high-density polyethylene (HDPE) at weight ratios of 100:0, 75:25, 50:50, 25:75, and 0:100 were investigated. These PS-HDPE blends were then melt blended with a short fiber-length wood flour (WF). The creep speed decreased with an increase in PS content, and, to a lesser extent, with an increase in WF content. The WF/PS-HDPE composites with plastic matrices of 50PS-50HDPE and 75PS-25HDPE showed lower creep speeds than the pure PS matrix, which might be attributed to the changes in polymer elongation induced by processing. The WF/75PS-25HDPE blend showed the least creep.

Hassan and Nada [43] used the old newsprint (ONP) fibers as reinforcing filler in polyester composites. Using ONP fibers in polyester composites resulted in a decrease in modulus of rupture (MOR), an increase in modulus of elasticity (MOE) and tensile strength as compared with a neat polyester. Water absorption and thickness swelling were increased as a result of using ONP fibers in the composites. Acetylation,

steaming, and esterification (using maleic anhydride) of ONP fibers were performed to improve the dimensional stability of the composites. Acetylation and steaming of ONP fibers resulted in a decrease in the thickness swelling of the composites; MOR, MOE, and tensile strength were decreased as a result of these treatments. Esterification of ONP fibers using maleic anhydride decreased the thickness swelling of the composites and, at the same time, increased MOR, MOE, and tensile strength.

Chuai et al. [44] presented conifer fibers as reinforcing materials for polypropylene-based composites. The interfacial adhesion or the compatibility of PP with conifer fibers was improved by MAPP grafting or MAPP treating of the fibers, as shown by SEM observation and DSC measurement. The high notched impact susceptibility of PP was significantly reduced by adding conifer fibers. The heat distortion temperature of PP was also enhanced by the addition of conifer fibers. The composites of PP/conifer fibers with either MAPP-grafted fibers or MAPP-treated fibers produced a significant improvement in mechanical properties and processing flowability compared to untreated conifer fiber composites.

Wu et al. [45] indicated the effects of fiber surface pretreatment on the interfacial bond strength and mechanical properties of wood fiber/polypropylene composites. The interfacial bond strength played a critical role in determining the tensile strength and impact strength of the composites. The wood fibers pretreated with the acid-silane aqueous solution gave a high interfacial bond strength with the PP matrix, resulting in the improvement of mechanical properties of the composites.

Sain et al. [46] investigated creep behavior of unmodified and functionally modified thermoplastic-wood fiber composites. For PVC, PE and PP-based composites creep was strongly dependent on the amount of load, time and temperature. A small rise in the temperature above ambient temperature increased creep significantly for PVC-wood fiber composites. Instantaneous creep resistance of wood fiber-filled PP was higher than that of PE-based composites. PP and PE-based wood composites were modified with maleic and maleimide compounds. Maleic and maleimide modification improved transient creep behavior of PP-wood fiber composites but it did not show practically any effect on instantaneous creep.

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Saha et al. [47] improved functional properties of jute-based composites by acrylonitrile pretreatment. Cyanoethylation of jute fibers in the form of nonwoven fabric was successfully achieved using an acrylonitrile monomer which was said to react with the hydroxyl groups of fiber constituents. The moisture regain, water absorption, and thickness swelling of the jute fiber-based composites were significantly reduced when cyanoethylated fiber was used. The dimensional stability of the composites was remarkably improved due to cyanoethylation of the jute fibers. A significant improvement on the tensile and the flexural properties of the jute-polyester composites was observed due to use of the cyanoethylated fibers. The scanning electron micrographs of the fractured surface of the samples revealed stronger bonding at the interface between the cyanoethylated fiber and the polyester resins. Thermal study showed that the initial thermal decomposition temperature was shifted to a higher value while the final decomposition temperature remained unaltered due to cyanoethylated fiber.

Hill and Khalil [48] studied the effect of fiber treatments on mechanical properties of coir or oil palm fiber reinforced polyester composites. Acetylation of coir or oil palm fibers increased the interfacial shear strength between the fibers and matrix. In addition, slight increase in the tensile strength, tensile modulus, and impact strength of composites reinforced with modified fibers were noted. Conversely, the tensile modulus, elongation at break, and flexural modulus were all reduced when compared with the composites reinforced with unmodified coir or oil palm fibers. The treatment of fibers with silane or titanate coupling agents did not show significant changes in the mechanical properties of the composites.

Hill and Khalil [49] investigated the effect of environmental exposure upon the mechanical properties of coir or oil palm fiber reinforced composites. Acetylation of coir or oil palm fibers reinforced with polyester matrix composites gave good mechanical properties during soil or water exposure tests. Water sorption was substantially reduced and structural integrity maintained, in contrast to the composites in which no fiber treatment was used. Silane treatment of fibers was found to protect the composites from the environmental exposure. Titanate treatment was not found to be as effective as silane in this respect. In view of the dramatic decrease in mechanical properties

observed when untreated fiber reinforced composites were exposed to environmental stresses, some forms of fiber treatment would appear to be essential. Since acetylation provided only a marginal benefit over silane treatment by means of protection, it was recommended that the latter method was used for a lower cost option.

Patil et al. [50] studied the effect of maleic anhydride (MA) treatment on steam and water absorption of wood polymer composites prepared from wheat straw, cane bagasse, and teak sawdust using novolac as the matrix. The MA treated wood polymer composites showed higher hardness about 2-3 times than that of the untreated respective wood polymer composites. MA treatment restricted swelling, water absorption and steam absorption in the agrowaste. Teak sawdust showed the best results in all respects among the three wood polymer composites.

Jangchud et. al. [51] studied natural fiber-polymer composites as artificial wood from poly(vinyl chloride) and eucalyptus fibers. By adding the fibers, the mechanical properties of the composites were improved. However, the mechanical properties were declined after the percentage of fiber loading exceeded the optimum point (30 phr). The water absorption (% WA) of the composites were increased when the fiber loading was increased. An optimum DOP loading in the composites was 10 phr. Adding calcium carbonate filler improved high temperature resistance, however, other mechanical properties were declined. The composites made from thermomechanical pulp (TMP) fibers had better mechanical and thermal properties than those made from chemical pulp (CP) fibers. Morphology of the fractured composites was observed by SEM. The composites of TMP fibers had better fiber dispersion than those of CP fibers.

CHAPTER 4

RESEARCH METHODOLOGY

4.1 Materials

4.1.1 Natural rubber fiber

A natural rubber wood was thermomechanically processed into fibers (Figure 4.1) so-called thermomechanical pulp (TMP). The wood was steamed under steam pressure until the temperature was over 140°C , which is the glass transition temperature of the lignin, subsequently, it was ground into fibers under high temperature and pressure by the defibrators or attrition mills.



Figure 4.1 Natural rubber fiber

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4.1.2 Poly(vinyl chloride), PVC

A suspension PVC resins was used to form polymer matrix. Characteristics of PVC resins were shown in Table 4.1.

Table 4.1 The characteristics of PVC resins [52]

Characteristics	Units	Values	Standards
Viscosity index	dm ³ /kg	102	ISO174
K value	-	64	DIN53726
Polymerization degree	-	900	JIS K6721
Apparent bulk density	g/ml	0.50	ISO 60
Volatile matters	%	≤ 0.3	ISO 1269
Particle sizes			ISO 1624
on 60 mesh (0.250 mm)	%	< 2	
on 230 mesh (0.063 mm)	%	> 90	

4.1.3 Stabilizer

A stabilizer is a one-pack stabilizer that is composed of Monohydrous Tribasic Lead Sulphate and Normal Lead Stearate. Specifications of the stabilizer were shown in Table 4.2.

Table 4.2 The specifications of the stabilizer [53]

PbO content (%)	61.2 ± 1.5
Bulk density (g/ml)	0.65 ± 0.15
Appearance	Homogeneous fine powder material without foreign matters
Moisture (%)	Less than 0.5

4.1.4 Plasticizer

Diethyl phthalate (DOP) was used as a plasticizer. Table 4.3 shows the specifications of this substance.

Table 4.3 The specifications of DOP [54]

Items	Units	Results
Purity	%	99.88
Specific gravity @ 20/20°C	-	0.985
Acid value after heat	mgKOH/g	0.0451
Volatile matters	%	0.0288
Water content	%	0.0265
Viscosity	cP	78

4.2 Instruments

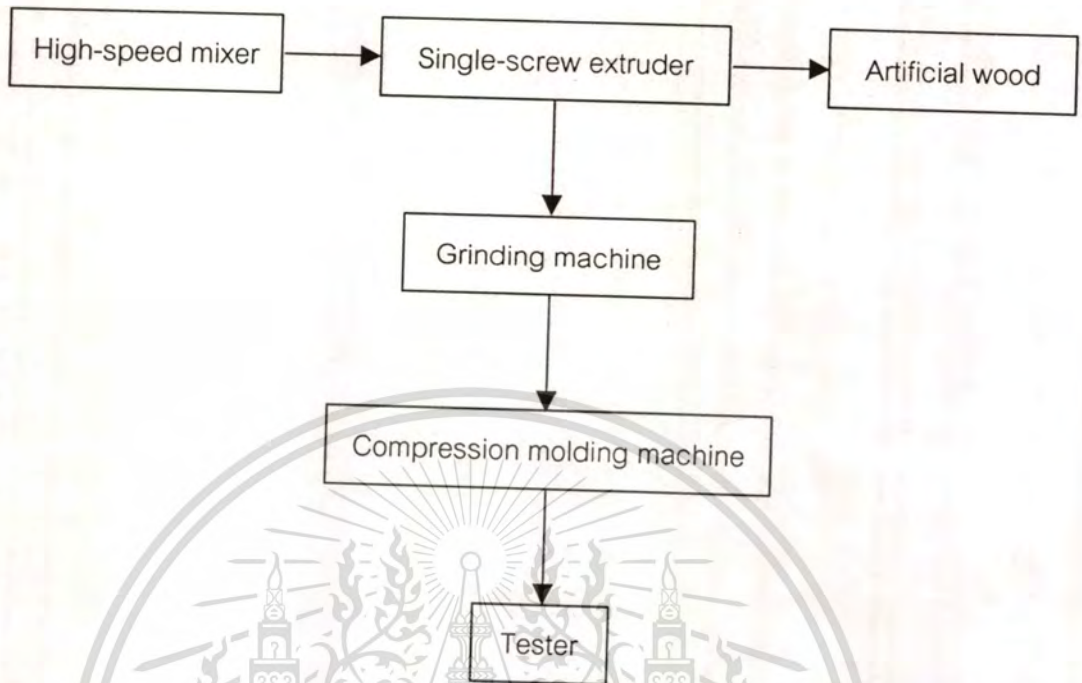
The instruments used in this research were listed below:

1. High-speed mixer (Lab Tech Engineering Co., Ltd., LMX 1000)
2. Single-screw extruder (Axon Ab Plastmaskiner, BX-18)
3. Grinding machine (Bosco Engineering)
4. Compression molding machine (Lab Tech Engineering Co., Ltd., LP 20)
5. Universal tensile tester (LLYOD Instrument Ltd., LR 30 K)
6. Izod impact tester (Yasuda Seiki Seisakusho)
7. Hardness tester, Shore D (Yasuda Seiki Seisakusho)
8. Thermogravimetric analyzer (Shimadzu Co., Ltd., TGA-51)
9. Differential scanning calorimeter (Perkin Elmer, DSC 7)
10. Heat distortion temperature tester (Yasuda Seiki Seisakusho)
11. Dynamic mechanical thermal analyzer (Rheometric Scientific Co., Ltd., DMTA-V)
12. Scanning electron microscope (Jeol, JSM-5410)
13. Specific gravity balance (Wallace)

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4.3 Experimental procedures



An initial formula of a composites (PVC, natural rubber fiber, and additives) was primarily blended in a high-speed mixer.

The mixture was melt-blended and extruded into rods by a single-screw extruder, and subsequently crushed by a grinding machine.

The granulated rods, therefore, were compressed into specimens by a compression molding.

During mixing, compounding and shaping processes, as mentioned above, the formulas and processing conditions were discreetly varied until a smooth and good-blended composites was achieved.

For the endurable purpose, the mechanical and thermal properties of the specimens were tested. Then the best formula and processing conditions giving high mechanical properties were used to fabricate an artificial wood sheet. Again, the high-speed mixer and single-screw extruder were employed for processing. An extrudate was extruded through a die head which was designed into a sheet shape.

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4.3.1 Initial formulas

The initial formulas (Table 4.4) were fixed at the fiber content of 30 phr but the additives (stabilizer and plasticizer) were varied to find an appropriate formula to be studied later.

Table 4.4 Initial formulas of the composites

No.	PVC*	Fiber*	DOP*	Stabilizer*
1	100	30	5	5
2	100	30	10	5
3	100	30	15	5
4	100	30	5	10
5	100	30	10	10
6	100	30	15	10
7	100	30	5	15
8	100	30	10	15
9	100	30	15	15

*phr unit, phr = parts per hundred parts of resins, by weight

4.3.2 Effect of fiber content

To study the effect of fiber content on properties of the composites, the best values of DOP and stabilizer obtained from 4.3.1 were fixed, as shown in Table 4.5.

4.3.3 Effect of DOP

To study the effect of DOP on properties of the composites, the best value of stabilizer obtained from 4.3.1 and fiber content from 4.3.2 were fixed, as shown in Table 4.6.

Table 4.5 The formulas of the composites to study the effect of fiber content

No.	PVC*	Fiber*	DOP*	Stabilizer*
1	100	0	The best value from 4.3.1	The best value from 4.3.1
2	100	10		
3	100	20		
4	100	30		
5	100	40		
6	100	50		

*phr unit, phr = parts per hundred parts of resins, by weight

Table 4.6 The formulas of the composites to study the effect of DOP

No.	PVC*	Fiber*	DOP*	Stabilizer*
1	100	The best value from 4.3.2	5	The best value from 4.3.1
2	100		10	
3	100		15	
4	100		20	

*phr unit, phr = parts per hundred parts of resins, by weight

4.4 Mixing and compounding procedures

4.4.1 High-speed mixer

Raw materials were blended at a constant speed of 4×10^3 rpm in a high-speed mixer (Figure 4.2).

PVC and DOP were blended for about 10 minutes and with the stabilizer for another 10 minutes. Finally, the natural rubber fibers were mixed with the blended mixture for 5 minutes.

4.4.2 Single-screw extruder

The dry-blended mixture was melt-blended subsequently in a single-screw extruder (Figure 4.3) at a screw speed of 40 rpm.

Feed zone, compression zone, and metering zone of the extruder were heated at a temperature of 150, 160, and 170 °C, respectively.



Figure 4.2 High-speed mixer

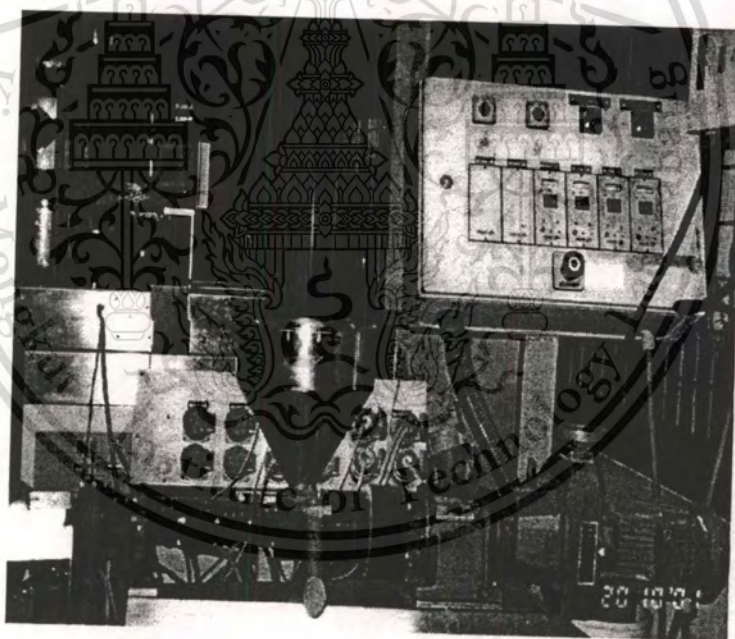


Figure 4.3 Single-screw extruder

4.4.3 Grinding machine

An extrudate was crushed by a grinding machine, as shown in Figure 4.4, into granules.

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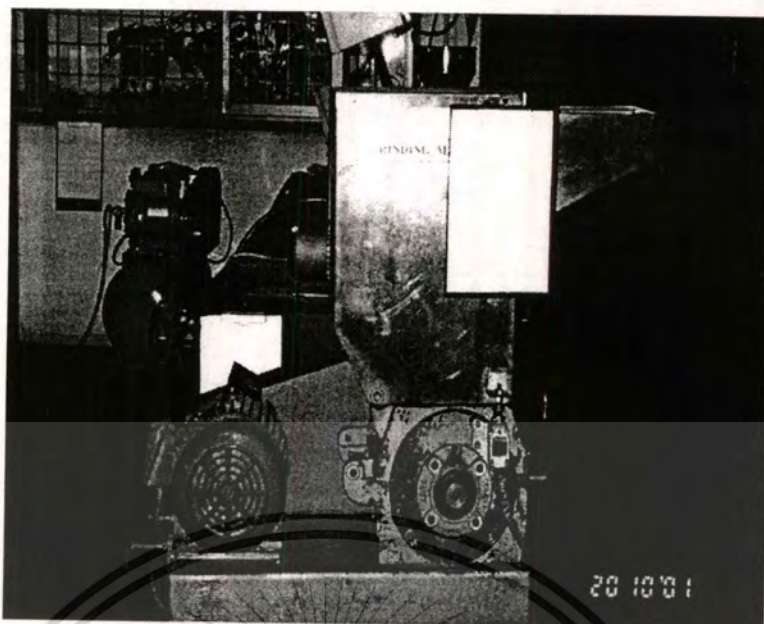


Figure 4.4 Grinding machine

4.5 Shaping procedure

4.5.1 Compression molding

The granules were molded by a compression molding machine (Figure 4.5) at a temperature of 190°C for 3 minutes.

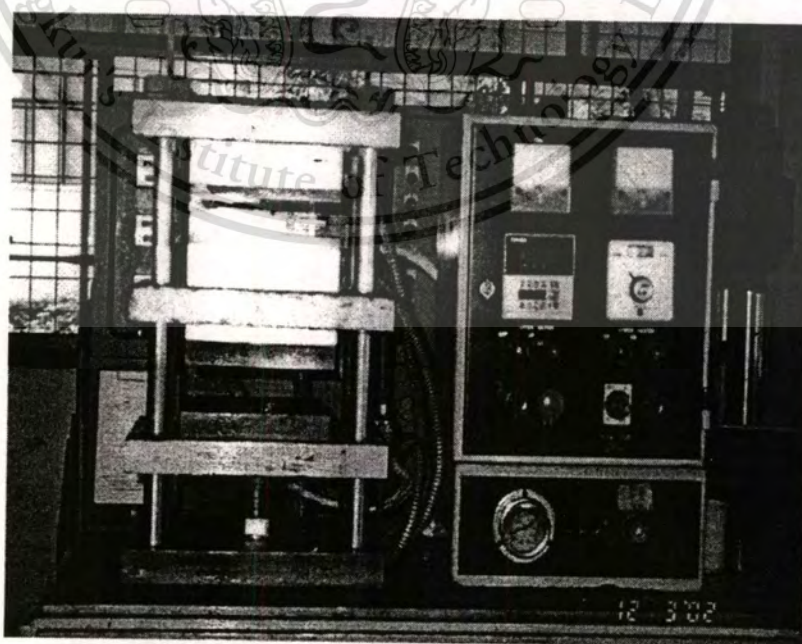


Figure 4.5 Compression molding machine

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4.6 Test procedures

4.6.1 Mechanical test

The results of the mechanical test will be plotted in terms of the mean value with the standard error bar.

4.6.1.1 Tensile test

Tensile properties were performed in accordance with ASTM D 638-91 through a universal tensile tester (Figure 4.6). The specimen was molded in a shape of ASTM type IV. Test was operated at a speed of 5 mm/min with load cell of 1000 N and 45-mm gauge length.

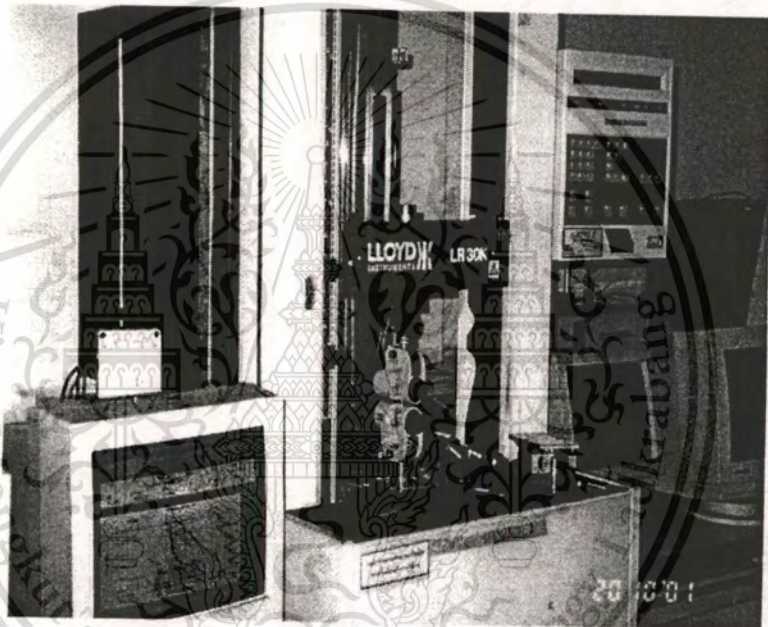


Figure 4.6 Universal tensile tester

The tensile strength, modulus at 3% strain and percent elongation at break were calculated as follows:

Tensile strength (σ)

$$\sigma = \frac{F}{A} \quad (4.1)$$

Where σ = Tensile strength (N/mm² or MPa)

F = Load at break (N)

A = Initial cross-sectional area of gauge length section (mm²)

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Modulus at 3% strain (E)

$$E = \frac{(F/A)}{(3/100)} \quad (4.2)$$

Where E = Modulus at 3% strain (MPa)

F = Load 3% strain (N)

A = Initial cross-sectional area of the specimen (mm^2)

Percent elongation at break (% E)

$$\%E = \left(\frac{l - l_0}{l_0} \right) \times 100 \quad (4.3)$$

Where % E = Elongation at break (%)

l = Distance between gauge length at break (mm)

l_0 = Initial distance between gauge length (mm)

4.6.1.2 Impact test

The standard test method for impact resistance followed ASTM D 256-90b (Method A). The notched specimen was tested by an Izod impact tester (Figure 4.7) with a pendulum capacity of 11 J.

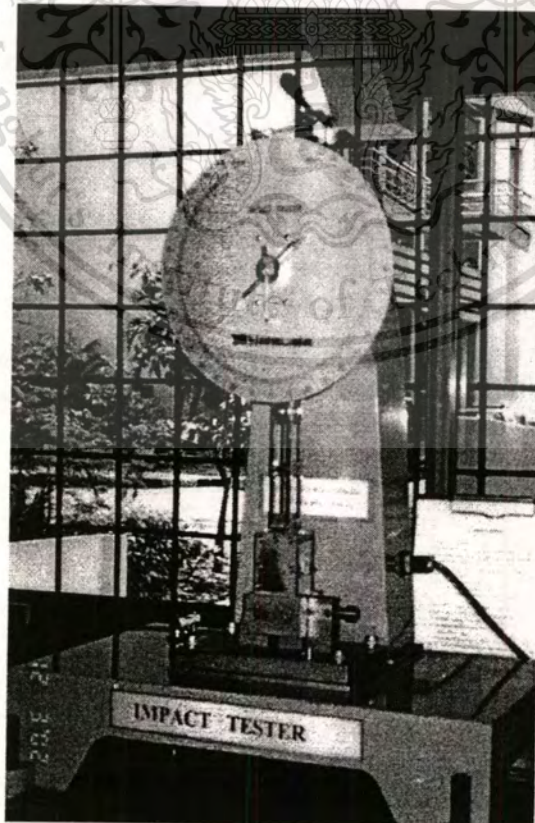


Figure 4.7 Izod impact tester

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The impact strength (W_k) was calculated from:

$$W_k = \frac{W}{A} \quad (4.4)$$

Where W_k = Impact strength (kJ/m^2)

W = Measured impact energy (kJ)

A = Cross-sectional area of the notched specimen (m^2)

4.6.1.3 Flexural test

Flexural properties were accomplished under ASTM D 790-91 with a universal tensile tester. A three-point loading system was performed through Test Method I at a rate of crosshead motion of 1 mm/min with load cell of 1000 N and 40-mm support span length.

The flexural strength and flexural modulus were calculated as follows:

$$\text{Flexural strength } (\sigma_f) \\ \sigma_f = \frac{3}{2} \frac{FL}{bh^2} \quad (4.5)$$

$$\text{Flexural modulus } (E_b) \\ E_b = \frac{L^3}{4bh^3} \times \frac{\Delta F}{\Delta d} \quad (4.6)$$

Where F = Load (N)

L = Support span length (mm)

b = Width of the specimen (mm)

h = Thickness of the specimen (mm)

ΔF = Load difference at linear relationship between stress and strain (N)

Δd = Flexural distance (mm)

4.6.1.4 Hardness test

The durometer hardness type D was measured by a durometer hardness tester (Figure 4.8), as described in ASTM D 2240-91.

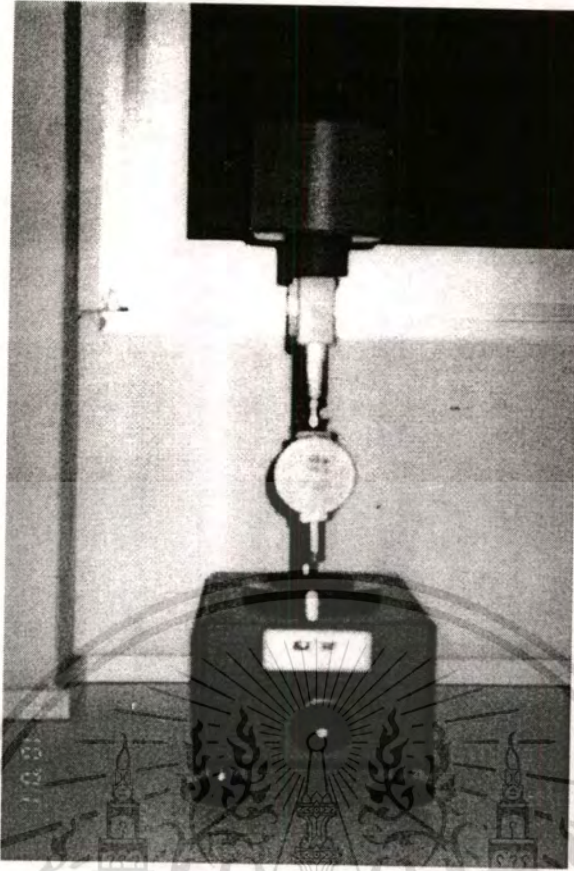


Figure 4.8 Hardness tester

4.6.2 Thermal test

4.6.2.1 Thermogravimetric analysis (TGA)

The TGA thermogram of the composites was measured by a thermogravimetric analyzer (Figure 4.9). The heating rate was 10 °C/min from 80 to 700 °C.

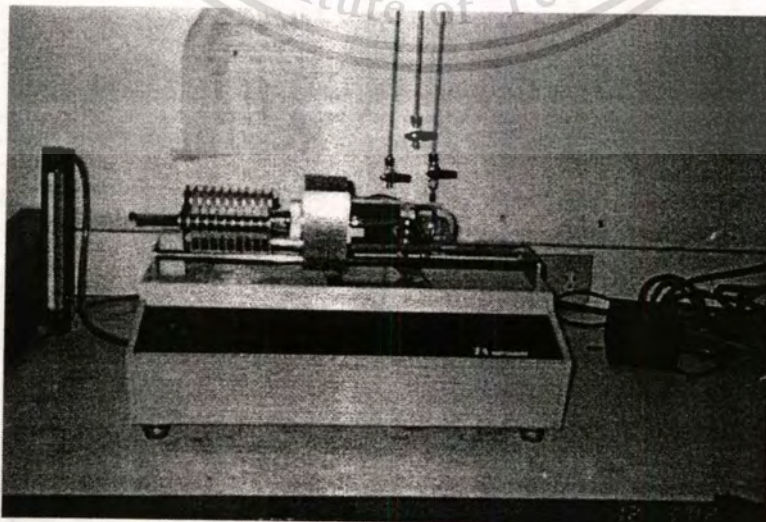


Figure 4.9 Thermogravimetric analyzer

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4.6.2.2 Differential scanning calorimetry (DSC)

The DSC thermogram of the composites was measured by a differential scanning calorimeter (Figure 4.10). The heating rate was 20 °C/min from 35 to 160 °C.



Figure 4.10 Differential scanning calorimeter

4.6.3 Thermomechanical test

4.6.3.1 Heat distortion temperature (HDT)

The heat distortion tester (Figure 4.11) achieved the heat distortion temperature of the composites under a flexural load (ASTM D 648-82). The heating rate was 2°C/min.

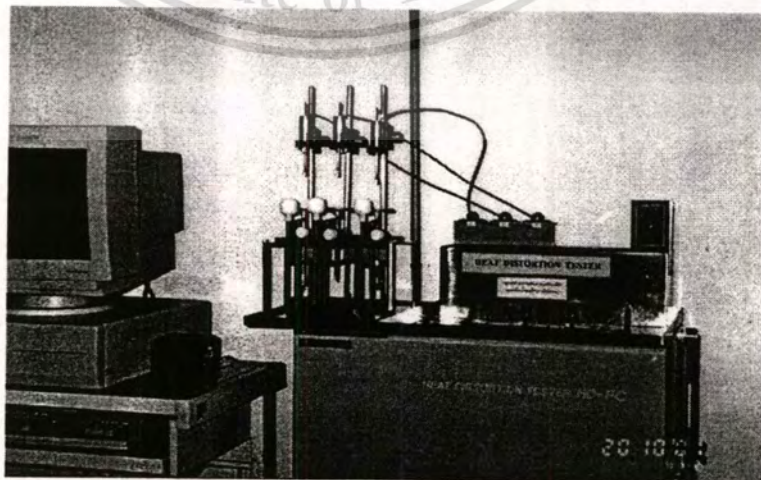


Figure 4.11 Heat distortion tester

4.6.3.2 Dynamic mechanical thermal analysis (DMTA)

The single cantilever bending geometry was used in DMTA by a dynamic mechanical thermal analyzer (Figure 4.12). The heating rate was 5 °C/min from 30-100 °C with a frequency of 1 Hz.



Figure 4.12 Dynamic mechanical thermal analyzer

4.6.4 Water absorption

Specimens of the composites were dried in an oven for 1 hour at 105 ± 2 °C, cooled in a desiccator for 30 to 45 minutes, and weighed to the nearest 0.001 g. The conditioned specimens were placed in distilled water maintained at the temperature of 23 ± 1 °C for totally 60 days. After every 2-day immersion, the specimens were removed from distilled water, wiped off water and weighed immediately to the nearest 0.001g.

The percent increase in weight during immersion was calculated from:

$$\% \text{ Water absorption} = \frac{\text{Wet weight} - \text{Conditioned weight}}{\text{Conditioned weight}} \times 100 \quad (4.7)$$

4.6.5 Specific gravity

The specific gravity was determined by a direct reading specific gravity balance (Figure 4.13).

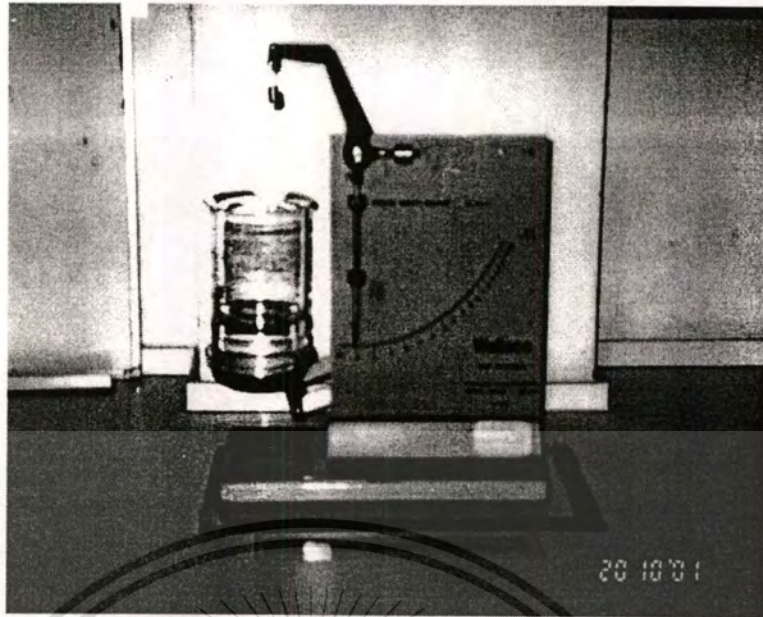


Figure 4.13 Specific gravity balance

4.6.6 Morphology

The composite specimens for scanning electron microscopy (SEM) were cooled in liquid nitrogen for 5 minutes, then broken immediately. Fractured surfaces were sequentially coated with a thin film of gold. The SEM images were obtained using a scanning electron microscope (Figure 4.14).



Figure 4.14 Scanning electron microscope

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CHAPTER 5

RESULTS AND DISCUSSION

From the initial formulas in Table 4.4, the best values of DOP and stabilizer are 10 and 15 phr, respectively.

5.1 Effect of fiber content on mechanical properties

Table 5.1 shows the mechanical properties of the composites, which the natural rubber fibers was varied from 0 to 50 phr.

Table 5.1 Mechanical properties of the composites with different fiber content

Mechanical properties	Fiber content (phr)*					
	0	10	20	30	40	50
Tensile strength (MPa)	26.7	34.2	32.6	30.4	26.0	25.6
Modulus at 3% strain (MPa)	450.0	708.3	820.0	634.3	765.2	777.0
%Elongation at break (%)	7.1	7.3	6.2	6.8	5.3	5.2
Impact strength (kJ/m ²)	2.5	2.7	3.2	3.7	3.9	3.8
Flexural strength (MPa)	43.3	54.8	54.9	51.5	46.6	38.6
Flexural modulus (MPa)	457.2	2128.8	2345.8	2491.4	2098.7	1833.9
Hardness (Shore D)	70.0	69.5	71.0	72.1	73.0	75.0

*phr = parts per hundred parts of resins, by weight

5.1.1 Tensile properties

Figures 5.1 to 5.3 present the tensile properties from Table 5.1. The tensile strength of the composites in Figure 5.1 increased up to the fiber content of 30 phr compared to unreinforced PVC (0 phr fiber content). This is due to the reinforcement of the fibers within the PVC matrix and a moderate interfacial adhesion between the fibers and PVC matrix. At high fiber content, the difficulty in mixing derived from low bulk density of the natural rubber fiber. Poor fiber dispersion in PVC matrix might yield fiber agglomerates with voids insides, therefore, the tensile strength decreased.

The modulus at 3% strain was shown in Figure 5.2. As expected, the modulus, which indicates the material stiffness, increased with the fiber content compared to unreinforced PVC. The result agreed with [40], which suggested that the incorporation of the fibers suppressed the flexibility of the PVC matrix. However, at the fiber content of 30 phr, the modulus at 3% strain was reduced. This may be due to randomly oriented natural fiber reinforced composites. The small decrease in the modulus with the fiber contents of 40 and 50 phr may be related to inadequate shear to the excess fibers in a high-speed mixer and a single-screw extruder.

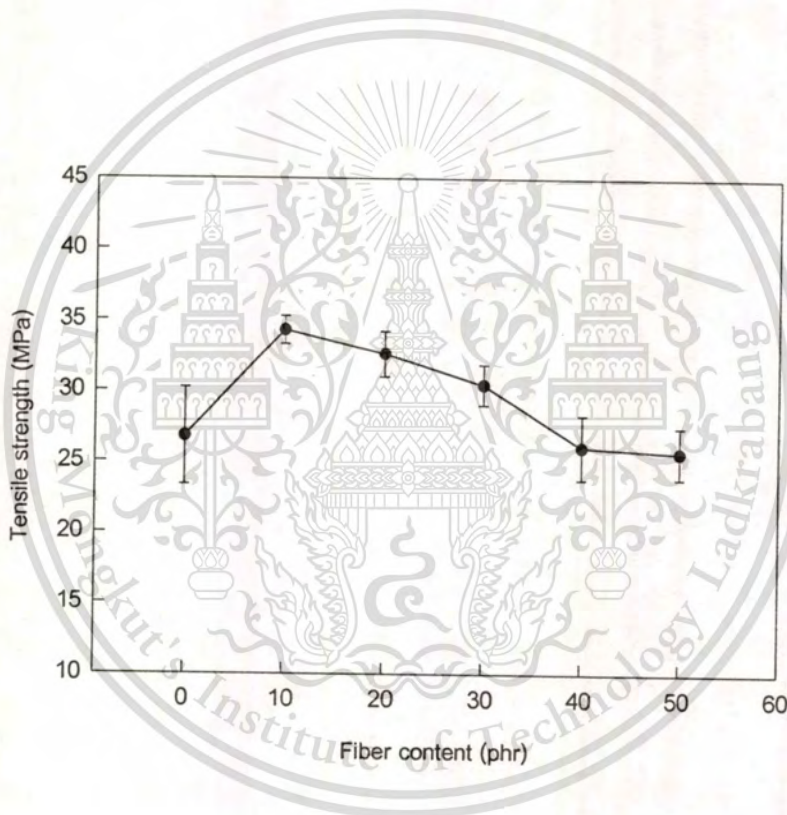


Figure 5.1 Effect of fiber content on tensile strength

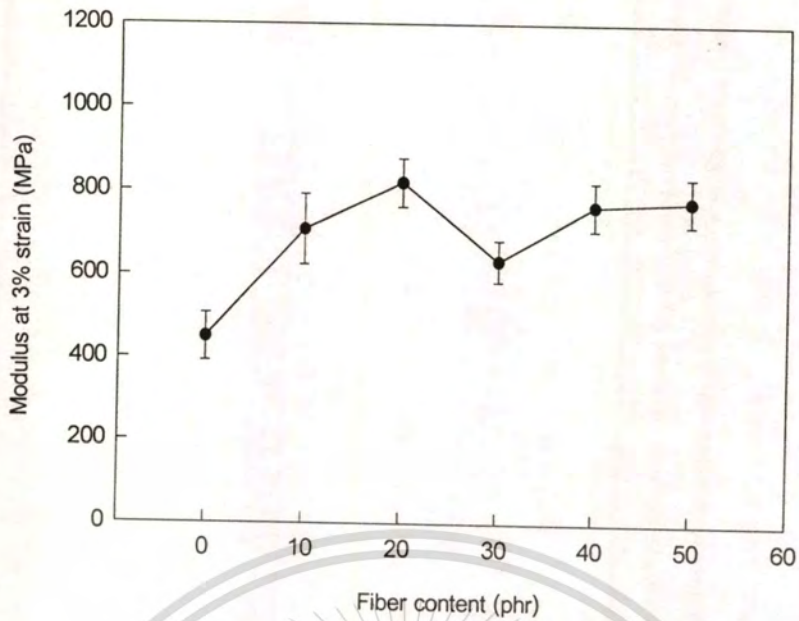


Figure 5.2 Effect of fiber content on modulus at 3% strain

The percent elongation at break (Figure 5.3) inclined to decrease with the fiber content. It appeared that the failure mode of the composites had shifted from ductile to brittle with the increase in fiber content. This can be attributed to the presence

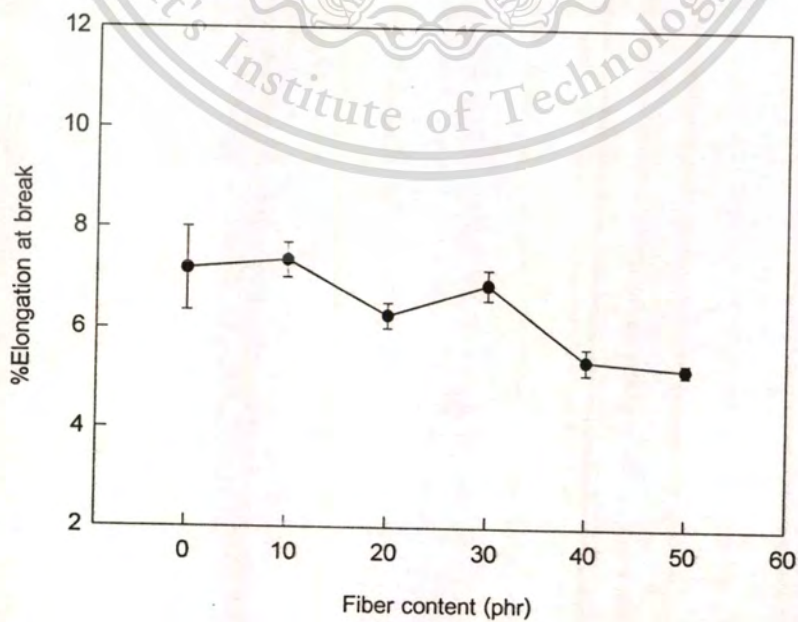


Figure 5.3 Effect of fiber content on %elongation at break

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of the fibers which suppressed the ability of the composites to undergo a plastic-deformation ability same as the results formed by [40].

5.1.2 Impact strength

Figure 5.4 shows the Izod impact strength of the composites. The impact strength of a composite is influenced by many factors including the toughness properties of the reinforcement and the nature of the interfacial region. A strong interfacial bond allows stress to be transferred efficiently, but produces poor toughness in the composites as discussed in [48]. Therefore, the impact strength moderately increasing.

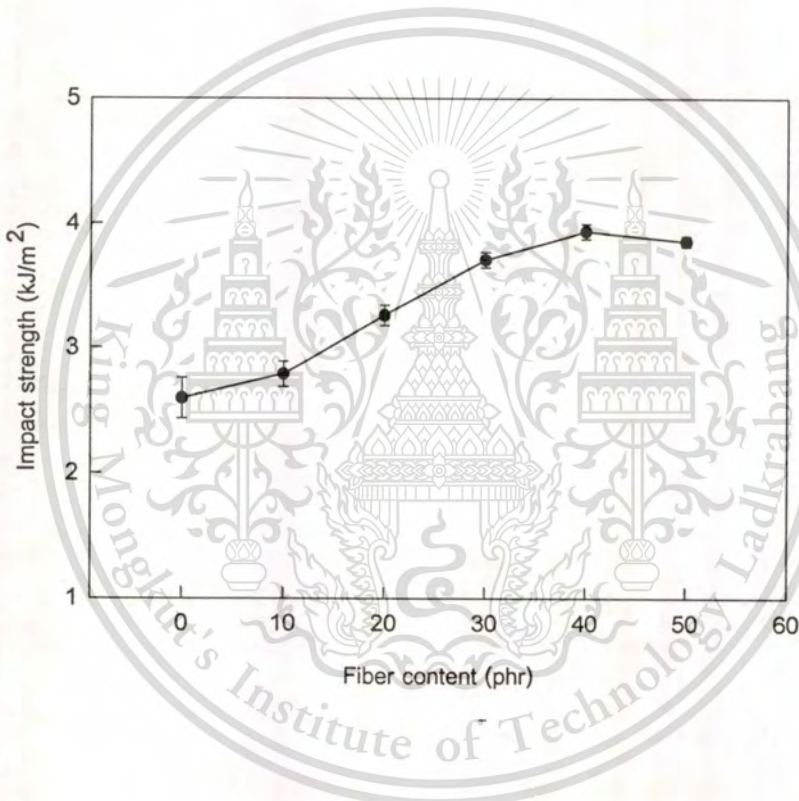


Figure 5.4 Effect of fiber content on impact strength

5.1.3 Flexural properties

Figures 5.5 and 5.6 show the effect of fiber content on flexural properties. The flexural properties were similar to the tensile strength and modulus at 3% strain. The flexural strength of the composites (Figure 5.5) increased up to the fiber content of 40 phr compared to the unreinforced PVC and then slightly decreased.

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The flexural modulus (Figure 5.6) increased with the fiber content to 30 phr. Although the flexural modulus of the specimens at the fiber contents of 40 and 50 phr decreased, their values were still higher than those of the unreinforced PVC.

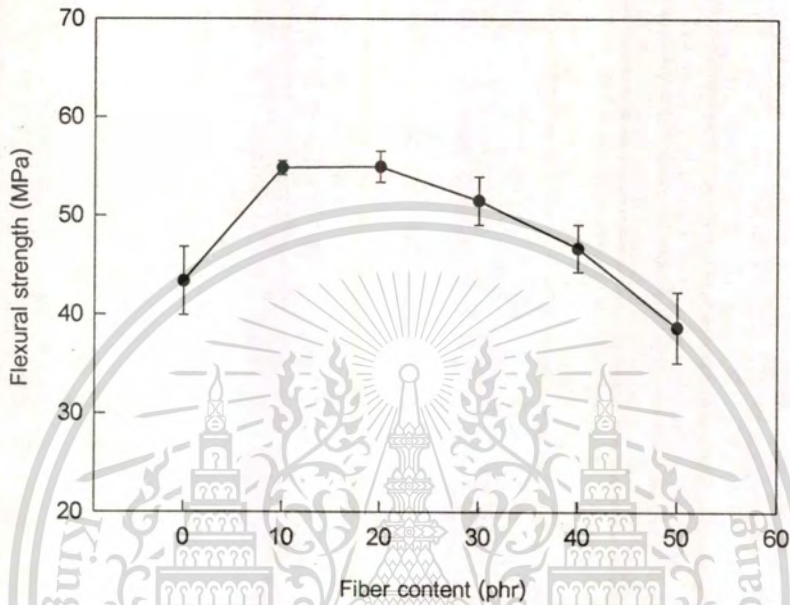


Figure 5.5 Effect of fiber content on flexural strength

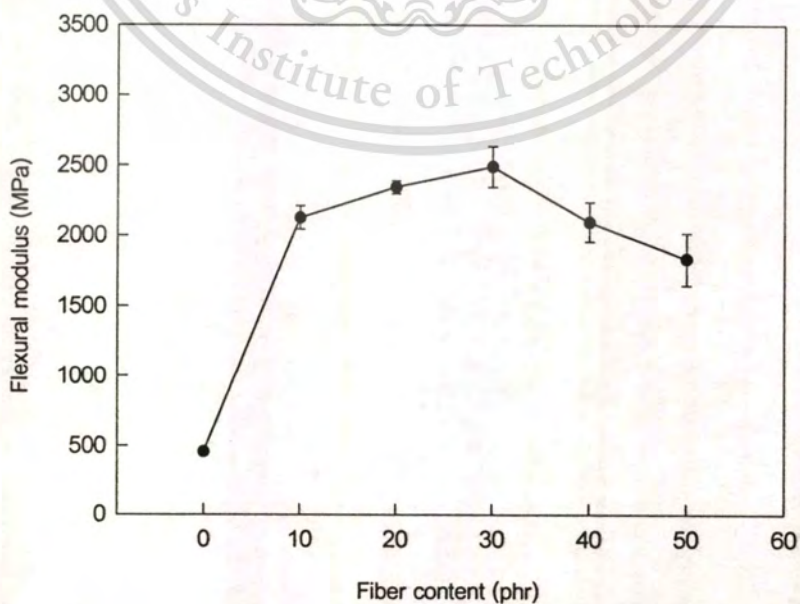


Figure 5.6 Effect of fiber content on flexural modulus

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5.1.4 Hardness

The hardness of the composites was shown in Figure 5.7. The hardness increased with the fiber content. As expected that the presence of the fibers could improve the hardness of the composites since the fibers had higher rigidity compared to that of the PVC matrix.

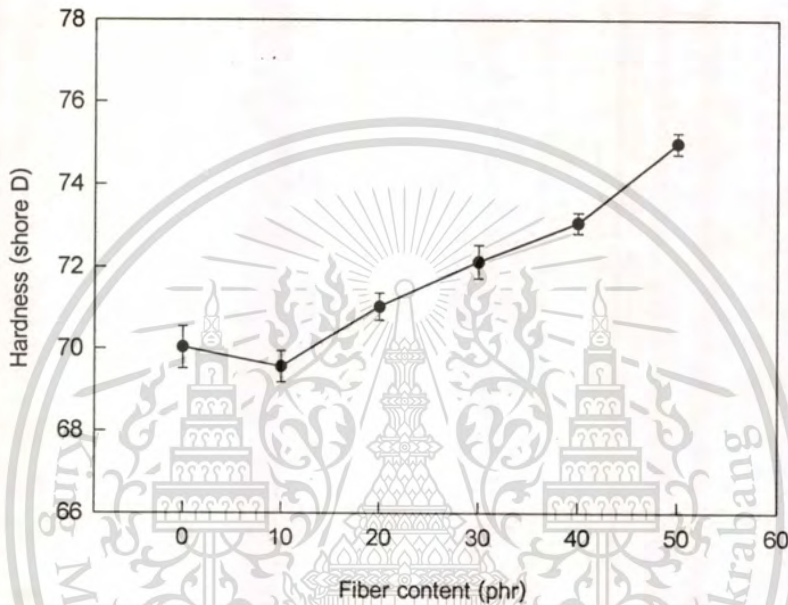


Figure 5.7 Effect of fiber content on hardness

5.2 Effect of fiber content on thermal properties

5.2.1 Thermogravimetric analysis (TGA)

Figure 5.8 shows TGA thermograms of the composites. From the TGA curve of the fibers, the decomposition temperature occurred in the range of 250 to 420 °C. This is due to lignocellulose decomposition as discussed in [44,47]. The main decomposition temperature of PVC was in the range of 300 to 400 °C. The composites between these two components were thus decomposed in the range about 280 to 400 °C, which was the decomposition temperature range of the fibers and PVC. It can be seen that the decomposition temperatures among the composites did not change obviously with the fiber content. It indicated that the fiber content had no effect on the decomposition temperature of the composites.

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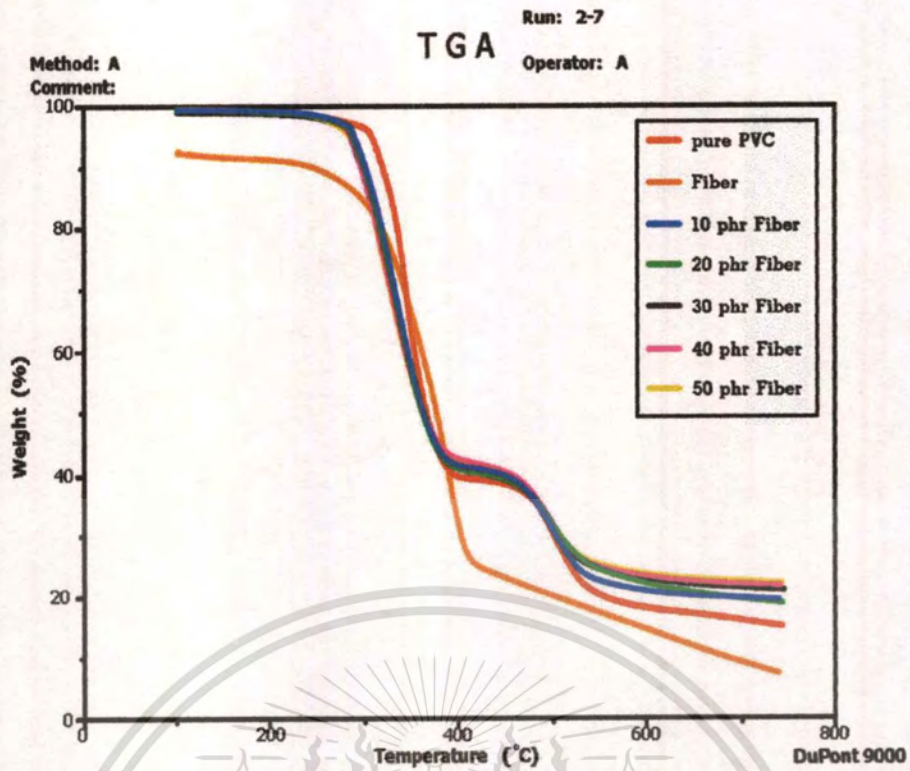


Figure 5.8 Effect of fiber content on TGA

5.2.2 Differential scanning calorimetry (DSC)

DSC thermograms (Figures B-1 – B-6 in App. B) indicated that the melting temperature (T_m) of the composites decreased with the fiber content (Table 5.2). This is attributed to the impediment to the crystal growth of PVC chains by the fibers. From Table 5.2, it could be concluded that the specific enthalpy ($\Delta\bar{H}$) of the composites, determined from the integrated area of the melting peak, corresponding to the degree of crystallization and had no relation with an increase in the fiber content.

Table 5.2 Effect of fiber content on T_m and $\Delta\bar{H}$

Fiber content (phr)	T_m (°C)	$\Delta\bar{H}$ (J/g)
0	105.00	2.80
10	98.33	2.20
20	98.33	3.03
30	97.33	1.77
40	96.00	2.59
50	90.33	2.02

5.3 Effect of fiber content on thermomechanical properties

5.3.1 Heat distortion temperature (HDT)

Figure 5.9 shows the effect of the fiber content on heat distortion temperature of the composites. The heat distortion temperature of the composites increased with the fiber content. This established that the fibers could improve the thermal resistance of the composites.

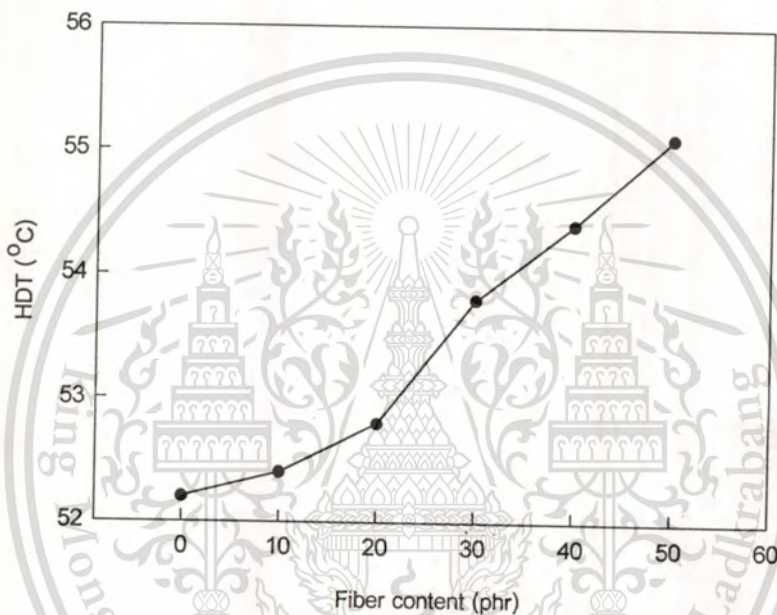


Figure 5.9 Effect of fiber content on HDT

5.3.2 Dynamic mechanical thermal analysis (DMTA)

Table 5.3 shows the viscoelastic properties of the composites. The glass transition temperature (T_g), which indicates the transition from the glassy state to rubbery state, increased with the fiber content up to 30 phr. This is due to the effect of the free volume reduction and the rigidization of the matrix by the fibers. At the fiber contents of 40 and 50 phr, the excess fibers could not disperse thoroughly in the PVC matrix and then agglomerated to form the fiber bundles. This resulted in increasing free volume and voids in the matrix, which reduced the stiffness or increased the chain motions via translation, rotation and vibration, and also T_g of the PVC matrix.

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The storage modulus in the PVC matrix increased with the fiber content leading to the decrease in $\tan \delta$. This can be discussed that the increment in the storage modulus of the composites is due to the reinforcement of the fibers as mentioned in [32]. At the fiber content of 20 phr, $\tan \delta$ did not follow the same trend as others. This may be because of the randomly oriented fibers in the composites.

Table 5.3 Effect of fiber content on T_g and $\tan \delta$

Fiber content (phr)	T_g ($^{\circ}\text{C}$)	$\tan \delta$
0	68.28	0.91
10	72.02	0.62
20	72.38	0.49
30	75.82	0.56
40	69.48	0.51
50	70.22	0.43

5.4 Effect of fiber content on water absorption

Figure 5.10 shows the water absorption curves of the composites. It can be

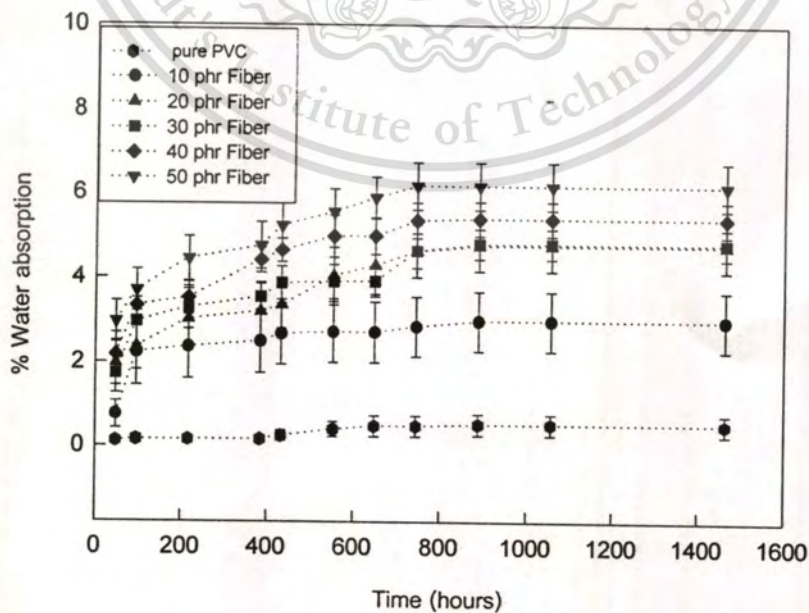


Figure 5.10 Effect of fiber content on water absorption

seen that the water absorption increased with the fiber content. The water absorption increases with the fiber content because the hygroscopic hydroxyl groups (-OH) of cellulose, hemicellulose, and lignin in the cell wall of the fibers increases. These -OH groups have a strong tendency to form hydrogen bond with the water molecule [40].

5.5 Effect of fiber content on specific gravity

Figure 5.11 shows the effect of the fiber content on the specific gravity of the composites. Because the density of the cell wall of the fibers is less than that of the PVC matrix, therefore, the specific gravity decreased. The slight different in the specific gravity of the composites indicates that the fiber content had no effect on the specific gravity.

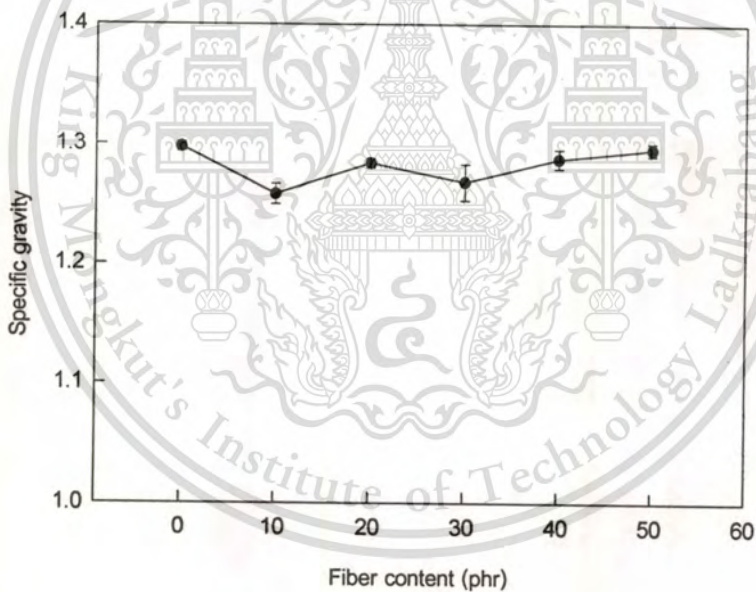


Figure 5.11 Effect of fiber content on specific gravity

5.6 Effect of fiber content on morphology

The microstructure of the composites such as the filler dispersion in the polymer matrices and the filler-matrix interfacial bonding are known to play a significant role in determining the mechanical properties of the composites [40].

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Figures 5.12 (a) to 5.16 (a) show the fiber dispersion in the PVC matrix. It can be seen that the increase in fiber content decreased the dispersion of the fibers, and the fiber bundles could be observed on the crack plane, as shown in Figures 5.15 (a) and 5.16 (a) (indicated by the arrows). It could be expected that these fiber bundles would resist dispersion of the individual fibers when the fiber content increased and would reduce the efficiency of stress transfer from the matrix to the fibers. This would then lead to a detrimental effect on the tensile strength of the composites [40].

Figures 5.12 (b) to 5.16 (b) are high-resolution SEM micrographs of the fractured composites between the fibers and the matrix. It is known that the main problem associated with lignocellulosic thermoplastic composites is the poor compatibility between the constituent phases. The limited polar nature of the fibers and PVC restricted the formation of forming a good interfacial bonding of the composites. The weak interfacial bonding was found when the fiber content reached 40 phr (Figures 5.15 (b) and 5.16 (b), indicated by the arrows). The increased impact strength is thus in agreement to the common sense that weak interface causes higher impact resistance [41].



Figure 5.12 SEM micrographs of fiber-PVC composites with fiber content of 10 phr, (a) at low magnification and (b) at higher magnification

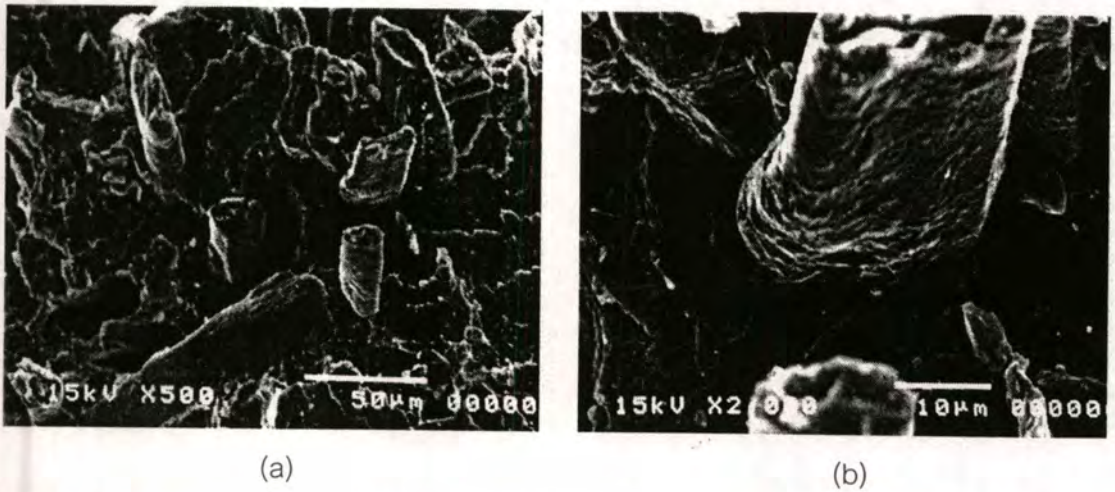


Figure 5.13 SEM micrographs of fiber-PVC composites with fiber content of 20 phr, (a) at low magnification and (b) at higher magnification



Figure 5.14 SEM micrographs of fiber-PVC composites with fiber content of 30 phr, (a) at low magnification and (b) at higher magnification

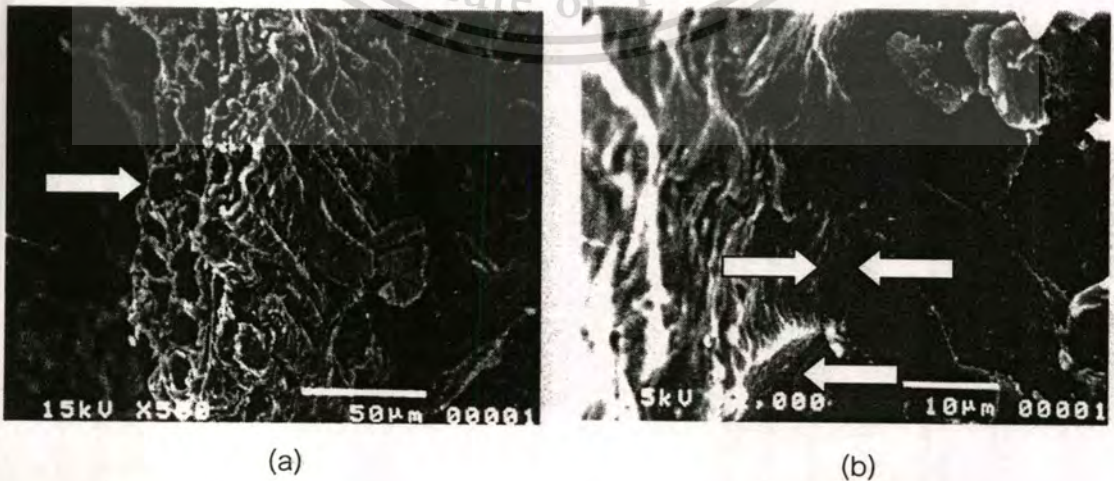
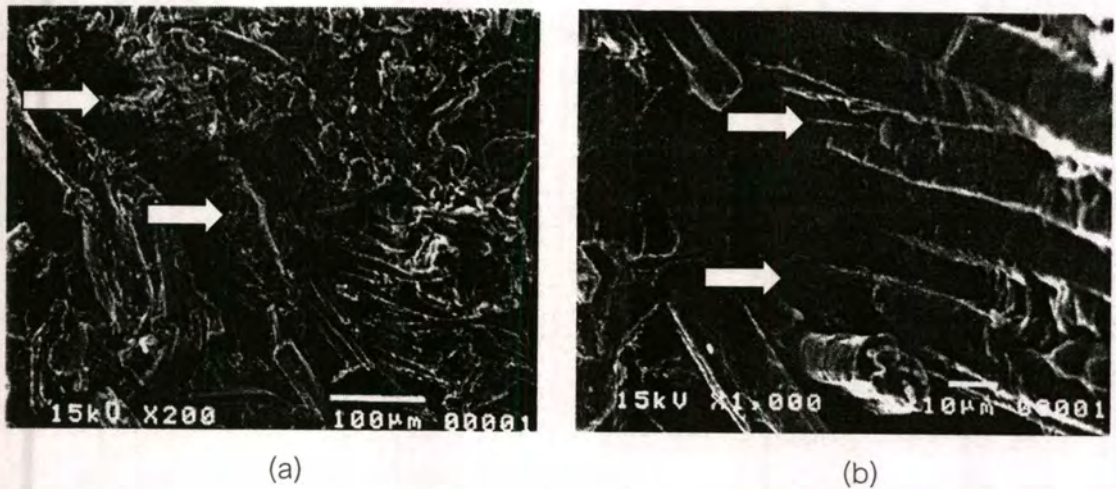


Figure 5.15 SEM micrographs of fiber-PVC composites with fiber content of 40 phr, (a) at low magnification and (b) at higher magnification



(a)

(b)

Figure 5.16 SEM micrographs of fiber-PVC composites with fiber content of 50 phr, (a) at low magnification and (b) at higher magnification

In this work it can be stated that the maximum amount of fibers, which are compatible with PVC matrix is 30 phr. At this composition the specimens show high mechanical properties and low water absorption. To study the effect of DOP the fiber content was fixed at 30 phr.

5.7 Effect of DOP on mechanical properties

Table 5.4 shows the mechanical properties of the composites, which DOP was varied from 5 to 20 phr.

Table 5.4 Mechanical properties of the composites with different DOP

Mechanical properties	DOP (phr)*			
	5	10	15	20
Tensile strength (MPa)	34.4	35.0	27.9	19.7
Modulus at 3% strain (MPa)	1017.5	712.5	793.7	392.5
%Elongation at break (%)	5.1	6.3	4.8	6.1
Impact strength (kJ/m ²)	2.7	2.9	2.9	3.0
Flexural strength (MPa)	51.8	52.2	44.8	34.4
Flexural modulus (MPa)	757.2	689.3	604.7	402.6
Hardness (Shore D)	74.1	74.2	71.4	67.1

*phr unit, phr = parts per hundred parts of resins, by weight

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5.7.1 Tensile properties

Figures 5.17 to 5.19 present the tensile properties from Table 5.4. Adding DOP decreased the tensile strength (Figure 5.17) and the modulus at 3% strain (Figure 5.18) because of the plasticization effect. This effect provides chain mobility and disentanglement within the PVC matrix resulting in a lower tolerance in the strength.

In contrast to the tensile strength and the modulus at 3% strain, the percent elongation at break (Figure 5.19) inclined to increase due to increasing in flexibility of the composites from the plasticization effect of DOP.

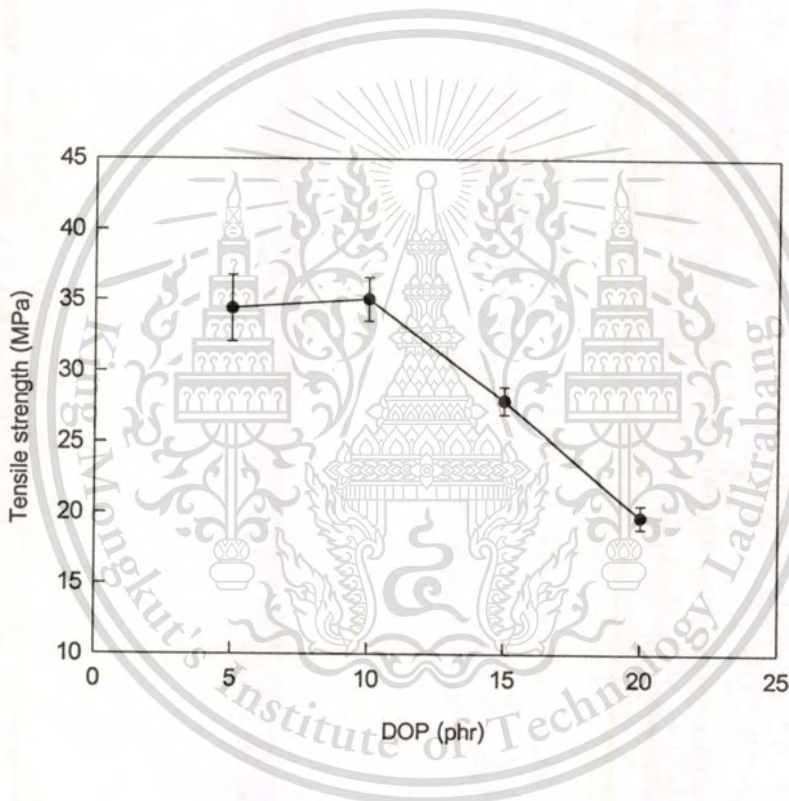


Figure 5.17 Effect of DOP on tensile strength

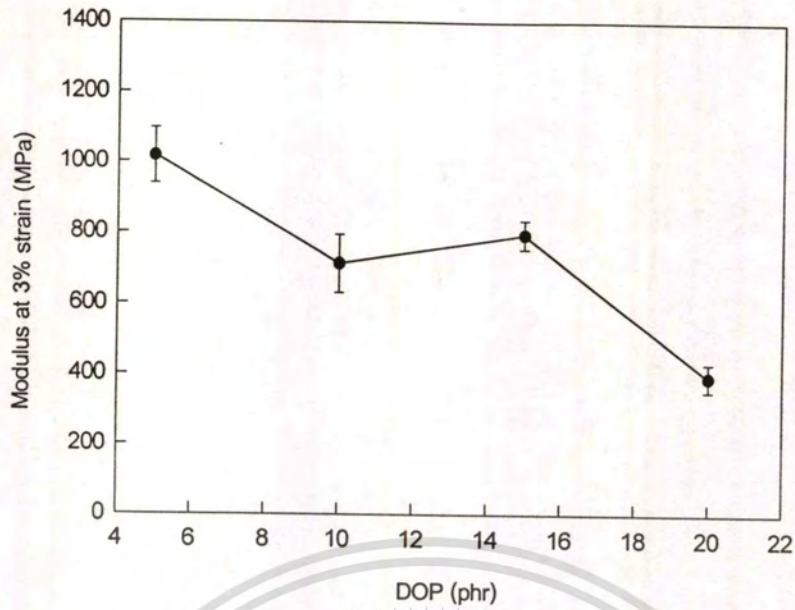


Figure 5.18 Effect of DOP on modulus at 3% strain

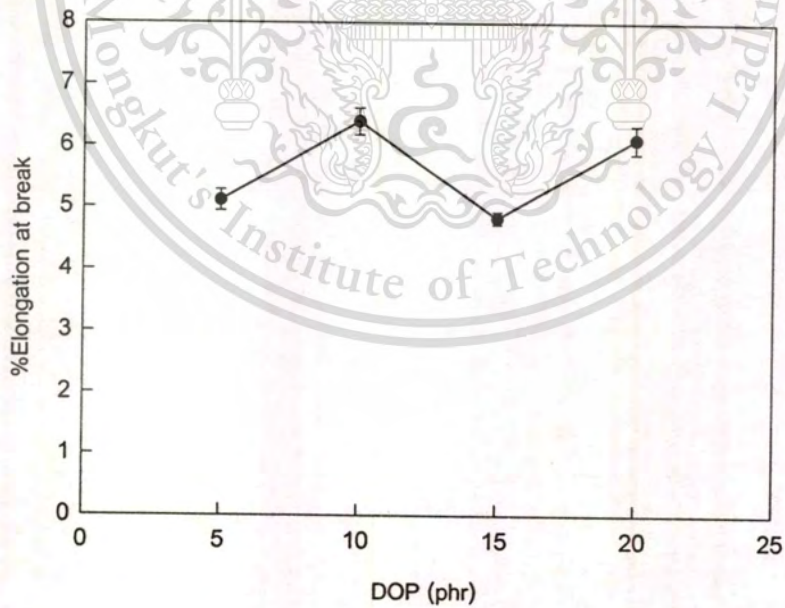


Figure 5.19 Effect of DOP on %elongation at break

5.7.2 Impact strength

Figure 5.20 shows the Izod impact strength of the composites. The impact strength increased with DOP. The increase in DOP generates softer and more flexible composites with higher toughness. Thus, the composites could disperse more strength within their panels so that the impact strength increased.

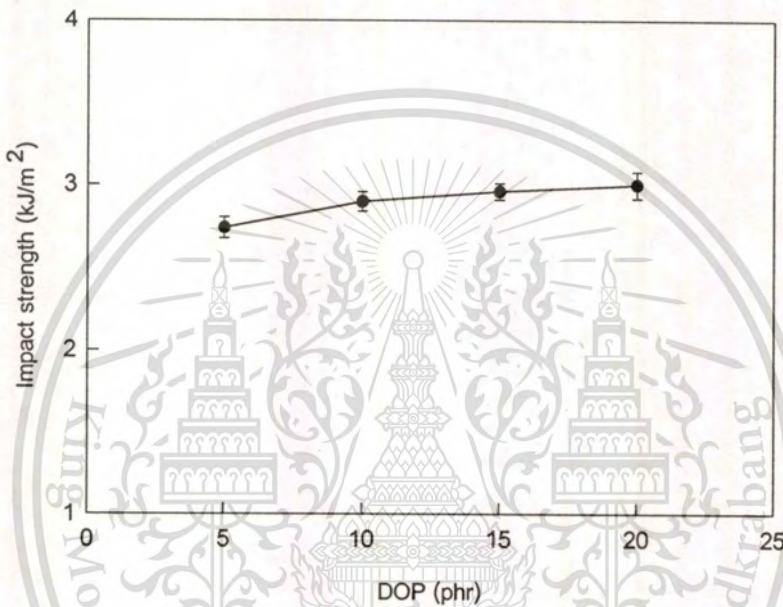


Figure 5.20 Effect of DOP on impact strength

5.7.3 Flexural properties

Similar to the tensile strength and the modulus at 3% strain, the flexural strength (Figure 5.21) and the flexural modulus (Figure 5.22) decreased with DOP. It was found that the interfacial bonding of the composites decreased by the plasticizer action, which generated high chain mobility.

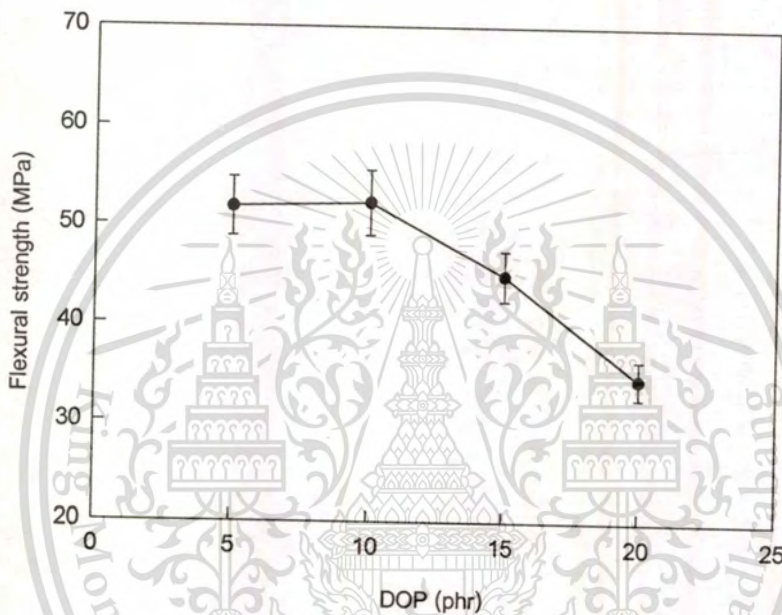


Figure 5.21 Effect of DOP on flexural strength

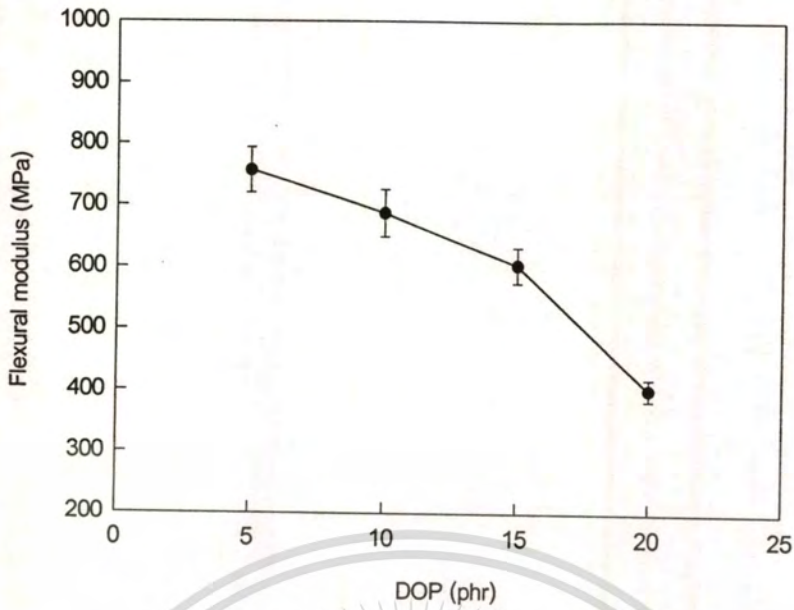


Figure 5.22 Effect of DOP on flexural modulus

5.7.4 Hardness

The hardness of the composites is shown in Figure 5.23. The hardness decreased with DOP. As expected that the plasticizer action of DOP would reduce the hardness of the composites.

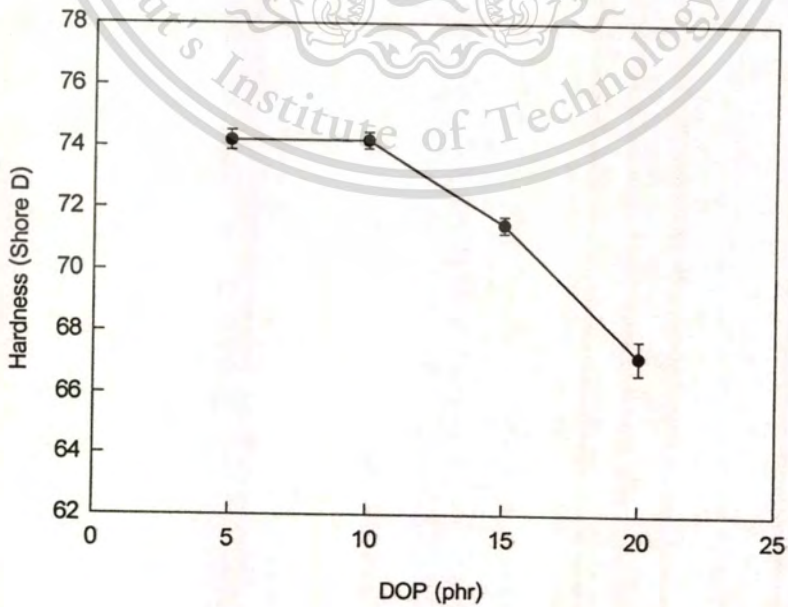


Figure 5.23 Effect of DOP on hardness

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5.8 Effect of DOP on thermal properties

5.8.1 Thermogravimetric analysis (TGA)

Figure 5.24 shows TGA thermograms of the composites. The main decomposition temperature of the composites was in the range of 220 to 370 °C. It was found that this range was lower than that from the effect of the fiber content. This accounts for the plasticizer action of DOP, which allows better heat distribution within the composites.

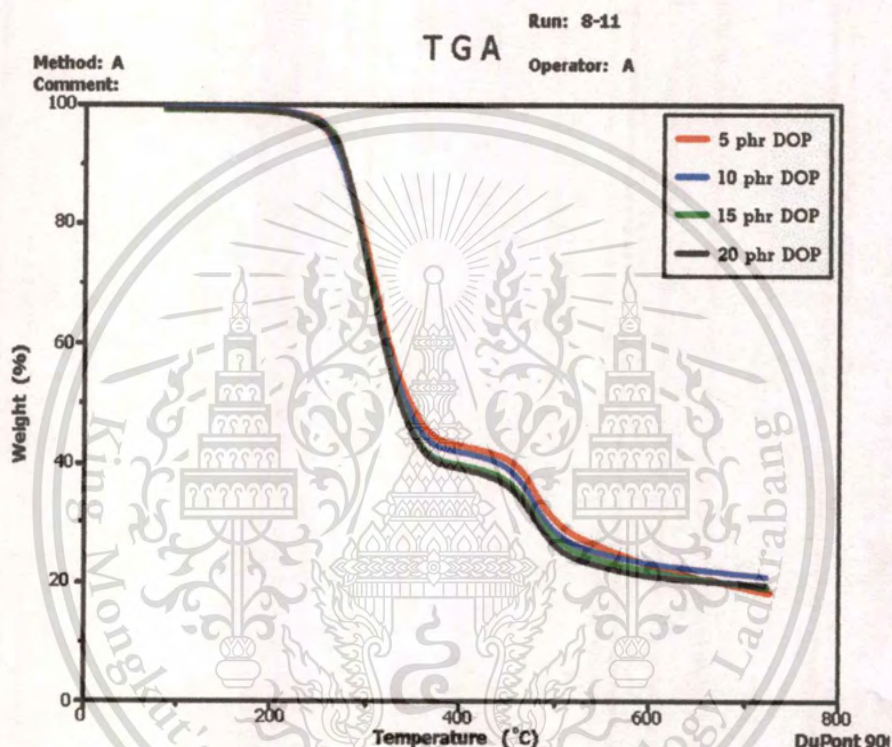


Figure 5.24 Effect of DOP on TGA

5.8.2 Differential scanning calorimetry (DSC)

Table 5.5 shows the effect of DOP on melting temperature (T_m).

Table 5.5 Effect of DOP on T_m and $\Delta\bar{H}$

DOP (phr)	T_m (°C)	$\Delta\bar{H}$ (J/g)
5	97.66	2.22
10	96.66	3.53
15	96.33	3.87
20	97.66	3.35

T_m of the PVC matrix slightly decreased with DOP except for 20 phr. The composites became more rubbery than glassy state because of the plasticizer action of DOP. Thus, the melting of polymer crystals decreased. From Table 5.5, it was found that there was no relation between DOP and ΔH .

5.9 Effect of DOP on thermomechanical properties

5.9.1 Heat distortion temperature (HDT)

Figure 5.25 shows the effect of DOP on heat distortion temperature of the composites. The HDT decreases with DOP this is because the plasticizer action of DOP causes the mobility of polymer chains. This reduces the thermal and distorted resistances of the composites leading to the decrease in the temperature.

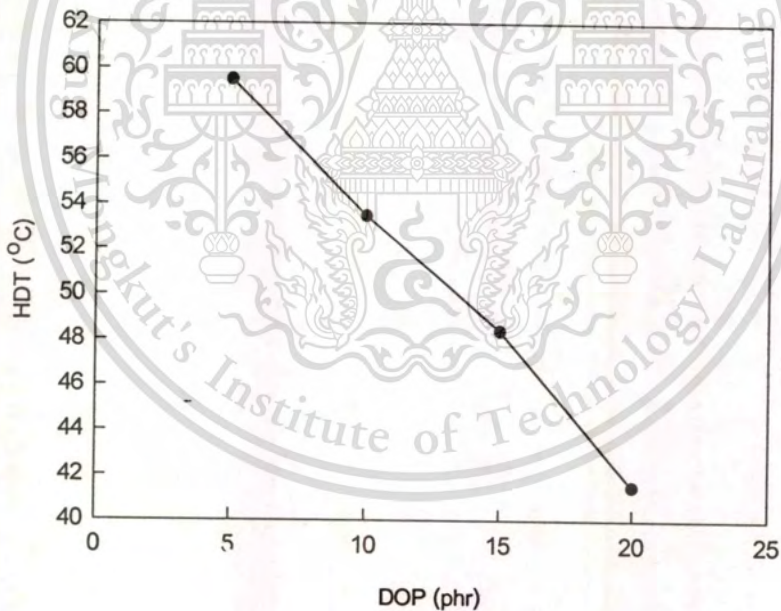


Figure 5.25 Effect of DOP on HDT

5.9.2 Dynamic mechanical thermal analysis (DMTA)

Table 5.6 shows the effect of DOP on the dynamic properties of the composites. The temperature relating the transition of glassy state to rubbery state decreased with DOP because of the ease of chain mobility of the PVC matrix.

Tan δ of the composites decreased with DOP except for 15 phr. The plasticizer action increased the storage modulus of the composites due to high stress transfer in the PVC matrix.

Table 5.6 Effect of DOP on T_g and tan δ

DOP (phr)	T_g ($^{\circ}\text{C}$)	tan δ
5	78.78	0.52
10	71.83	0.50
15	64.70	0.56
20	61.57	0.47

5.10 Effect of DOP on water absorption

Figure 5.26 shows the effect of DOP on the water absorption of the composites.

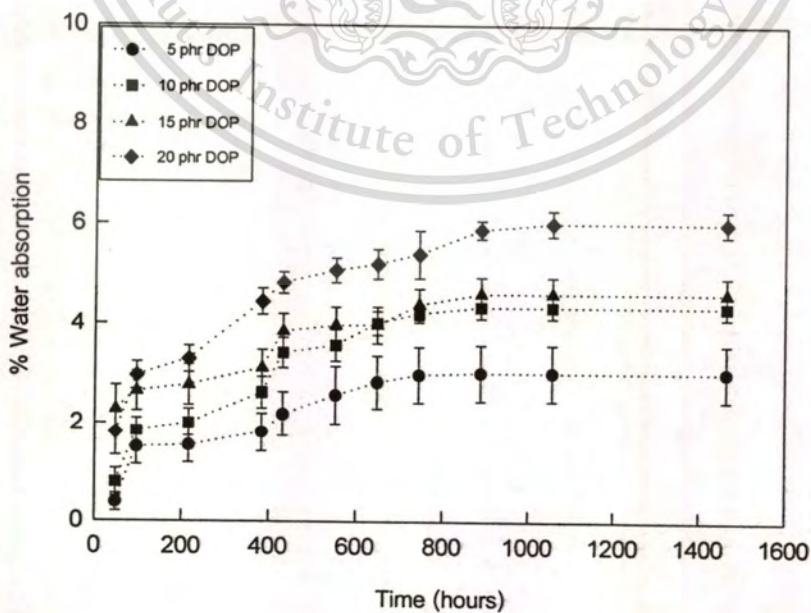


Figure 5.26 Effect of DOP on water absorption

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The water absorption increased with DOP. This suggested that the free volume and voids of the composites, which facilitated water absorption of the, increased with the DOP because of the mobility of PVC chain by the plasticizer action of DOP.

5.11 Effect of DOP on specific gravity

Figure 5.27 shows the effect of DOP on the specific gravity of the composites. As expected, the specific gravity decreased with DOP because of the increase in free volume of the composites due to chain mobility of PVC.

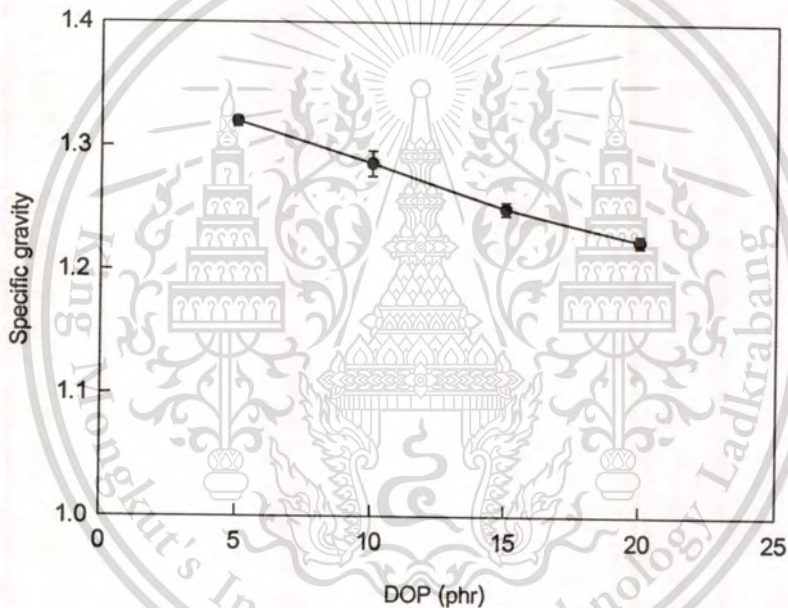


Figure 5.27 Effect of DOP on specific gravity

5.12 Effect of DOP on morphology

Figure 5.28 shows the effect of DOP on morphology of the composites. It can be seen that numbers of voids from fiber pull-out during fracture increased with DOP, Figure 5.28 (b) and (c). This is due to the chain mobility and disentanglement within the PVC matrix, which was affected by the plasticized action of DOP. The dispersion of the fibers increased with DOP, which obviously observed from Figure 5.28 (a) and Figure 5.28 (d).

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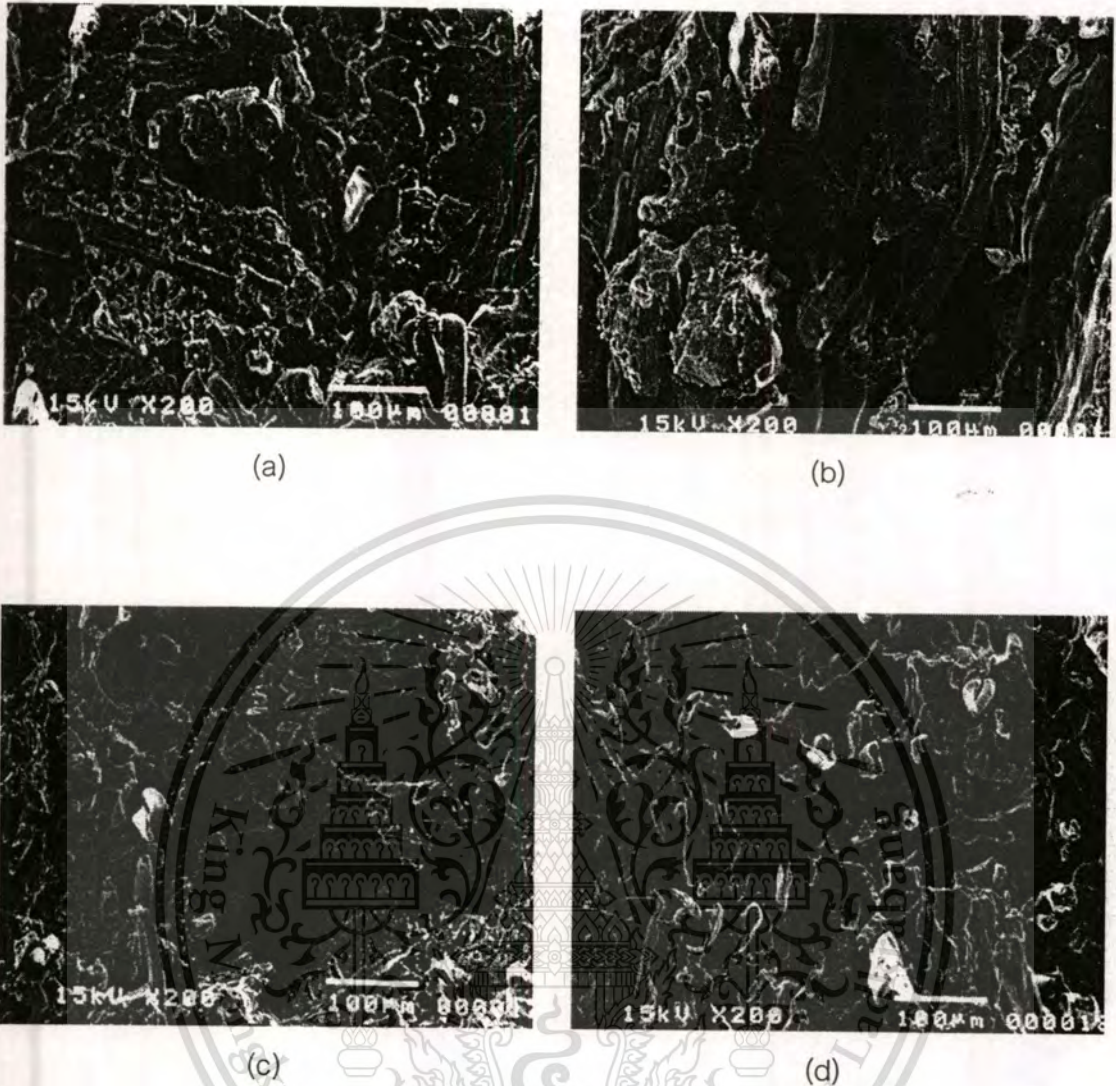


Figure 5.28 SEM micrographs of fiber-PVC composites with DOP of
 (a) 5 phr, (b) 10 phr, (c) 15 phr, (d) 20 phr

From all the tests the amount of DOP confirmed to be 10 phr. DOP of 5 phr caused the difficulty of processing whereas DOP of 15 and 20 phr generated too soft composites, which was not a good property as an artificial wood.

5.13 Fabrication of an artificial wood

An artificial wood was fabricated from the composites containing PVC, natural rubber fiber, stabilizer and DOP of 100, 30, 15 and 10 phr, respectively. The composites were extruded in a shape of a sheet. A shape and dimension of the artificial wood can be determined as the work requirement by a specific die design.

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CHAPTER 6

CONCLUSIONS AND SUGGESTIONS

This research is a study on fabrication of the artificial wood from poly(vinyl chloride) mixed with natural rubber fiber. The natural rubber wood was thermomechanically processed into fibers known as TMP (thermomechanical pulp). The natural rubber fiber-reinforced poly(vinyl chloride) composites were compounded in a single-screw extruder. The test specimens were then shaped by a compression molding machine. The effects of fiber content from 10-50 phr, and DOP from 5-20 phr were studied. The conclusions based on this study were stated as follows.

6.1 Effect of fiber content on mechanical properties

The tensile strength, modulus at 3% strain, flexural strength and flexural modulus increased and then decreased with an excess of the fibers because of the incompatibility between PVC matrix and the excess fibers. The impact strength and hardness increased with the fiber content, however, the percent elongation at break decreased with the fiber content.

6.2 Effect of fiber content on thermal properties

From the TGA thermograms, the fibers decomposed in the range of 250 to 420 °C and PVC decomposed in the range of 300 to 400 °C. The decomposition temperatures of the composites with different fiber content are in the same range of 280 to 400 °C, which is between those of fibers and PVC, so that no effect of the fiber content on the decomposition temperature.

The DSC thermograms showed that the melting temperature (T_m) of the PVC matrix decreased with the fiber content.

6.3 Effect of fiber content on thermomechanical properties

The heat distortion temperature (HDT) of the composites increased with the fiber content.

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The glass transition temperature (T_g) increased with the fiber content up to 30 phr and then decreased due to an excess of the fiber content.

6.4 Effect of fiber content on water absorption

The hygroscopic hydroxyl groups in the cell wall of the fibers leading to an increase in water absorption of the composites.

6.5 Effect of fiber content on specific gravity

The specific gravity of the composites was less than that of PVC. The slight difference in the specific gravity of the composites indicated that the increase in fiber content had no effect on the specific gravity.

6.6 Effect of fiber content on morphology

It could be clearly observed from SEM micrographs that the fiber dispersion and interfacial bonding of the composites decreased with the fiber content which directly affected the properties of the composites.

6.7 Effect of DOP on mechanical properties

The tensile strength, modulus at 3% strain, flexural strength, flexural modulus and hardness decreased with DOP, however, the percent elongation at break and impact strength slightly increased. These results were expected due to the plasticization effect of the DOP.

6.8 Effect of DOP on thermal properties

The TGA thermograms indicated that the decomposition temperature of the composites with different DOP from 5-20 phr was in the same range of 220 to 370 °C.

The melting temperature of the composites decreased slightly with DOP.

6.9 Effect of DOP on thermomechanical properties

The heat distortion temperature (HDT) of the composites decreased with DOP.

The glass transition temperature (T_g) of composites decreased with DOP.

6.10 Effect of DOP on water absorption

The water absorption of the composites increased with DOP. This is due to an increase in free volume and voids by the plasticization effect of DOP.

6.11 Effect of DOP on specific gravity

As expected, the specific gravity of the composites decreased with DOP because of free volume and voids from the plasticization effect of DOP. This is the same reason as the water absorption.

6.12 Effect of DOP on morphology

It could be observed by the SEM micrographs that increasing in DOP caused a number of voids from fiber pull-out during fracture in the composites. The dispersion of the fibers in the composites also increased with DOP.

6.13 Fabrication of an artificial wood sheet

From this work, the composites with good mechanical properties and can be processed into a sheet by a single-screw extruder composes of PVC, natural rubber fiber, stabilizer, and plasticizer of 100, 30, 15 and 10 phr, respectively.

Further studies:

1. The fiber surface should be modified to increase the interfacial compatibility between the fibers and the PVC matrix,
2. The effect of the fiber length on the properties of the composites should be studied,
3. Other reinforcing agents should be experimented.

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- [53] Documents supported by the Siam Stabilizers and Chemicals Co., Ltd.
- [54] Documents supported by the South City Petrochem Co., Ltd.

APPENDICES

APPENDIX A Thermogravimetric analysis (TGA) thermograms

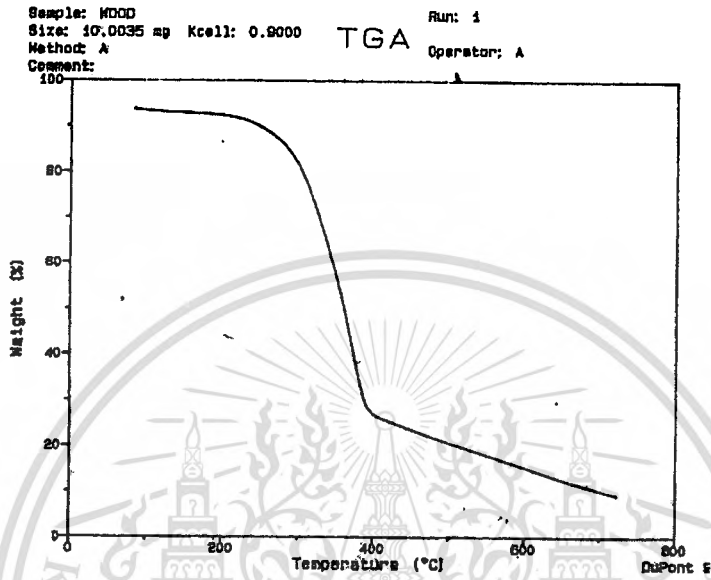


Figure A-1 TGA thermogram of wood

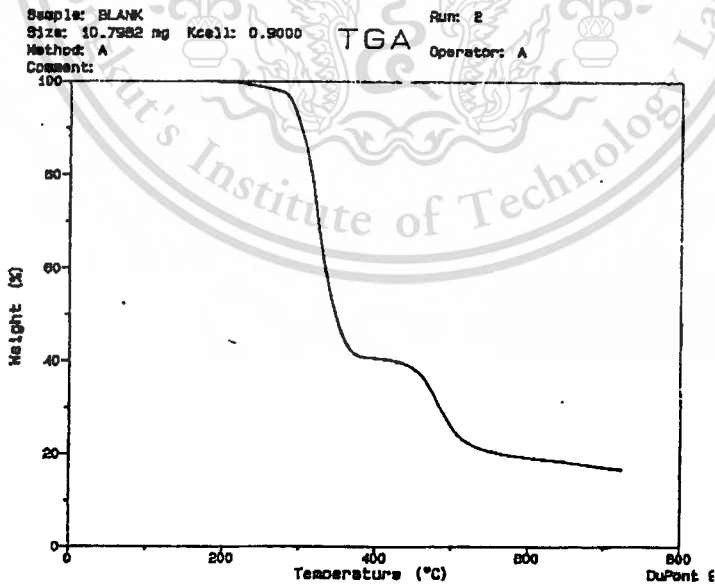


Figure A-2 TGA thermogram of PVC

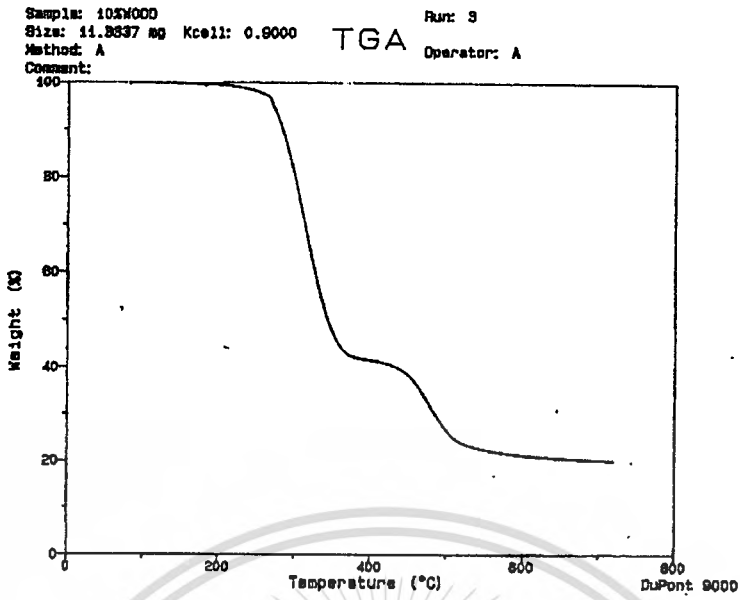


Figure A-3 TGA thermogram of 10 phr fiber

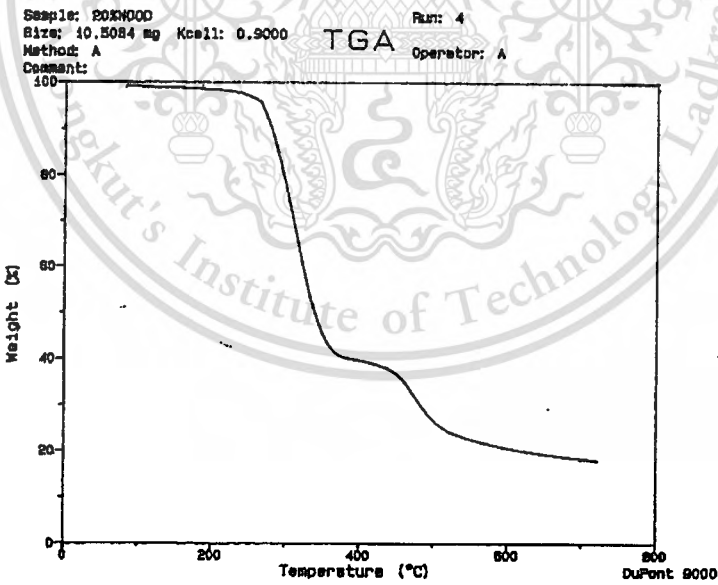


Figure A-4 TGA thermogram of 20 phr fiber

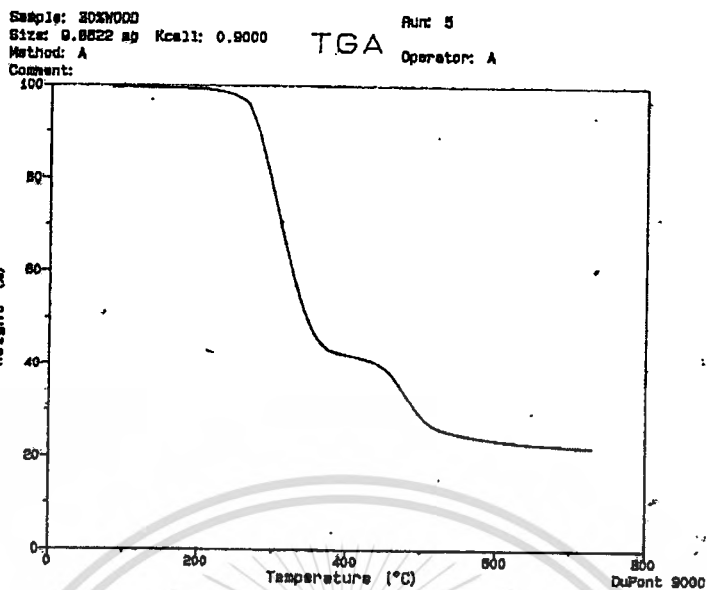


Figure A-5 TGA thermogram of 30 phr fiber

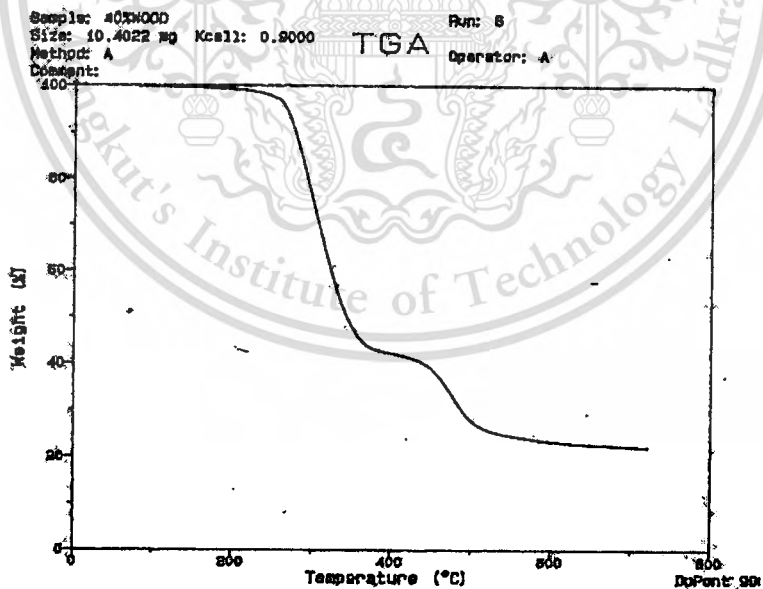


Figure A-6 TGA thermogram of 40 phr fiber

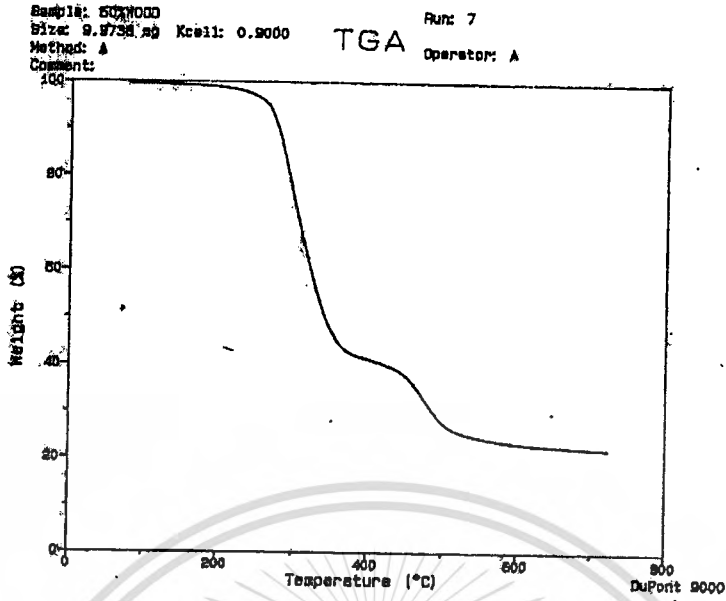


Figure A-7 TGA thermogram of 50 phr fiber

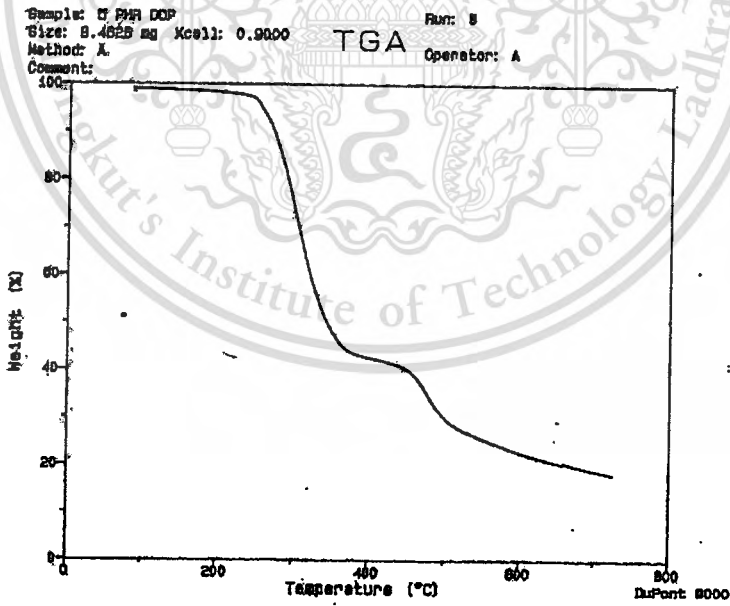


Figure A-8 TGA thermogram of 5 phr DOP

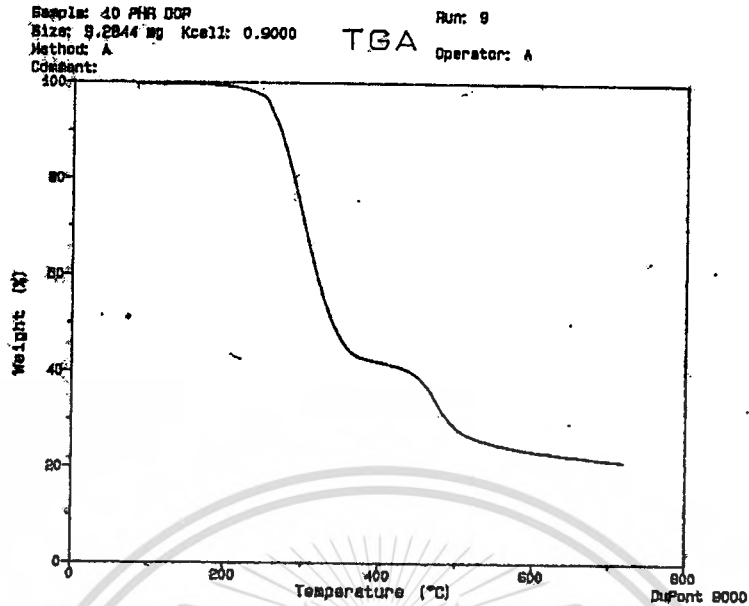


Figure A-9 TGA thermogram of 10 phr DOP

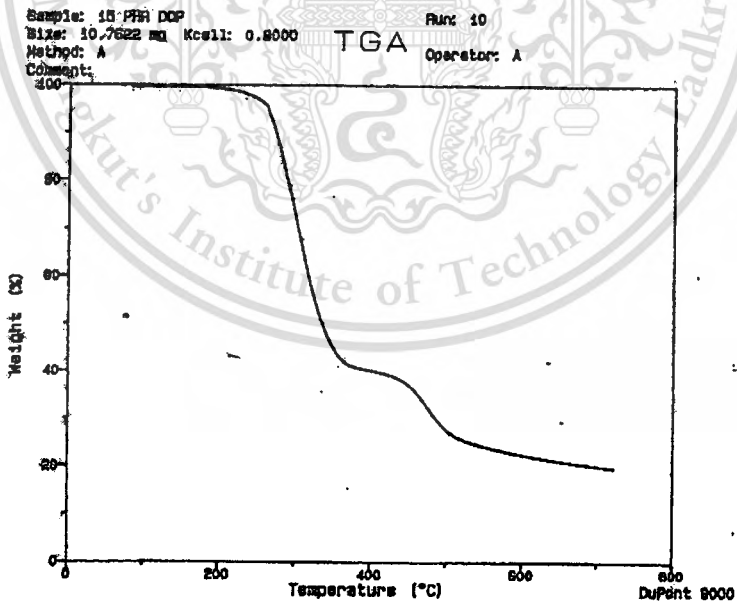


Figure A-10 TGA thermogram of 15 phr DOP

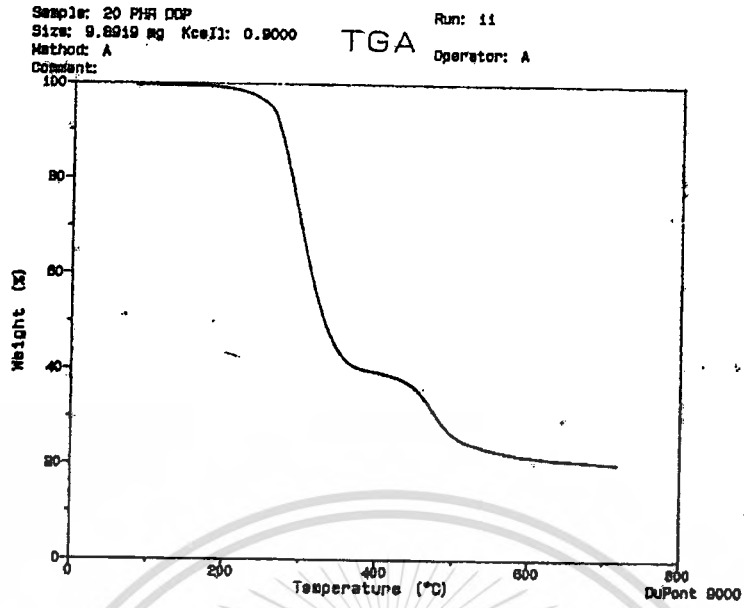
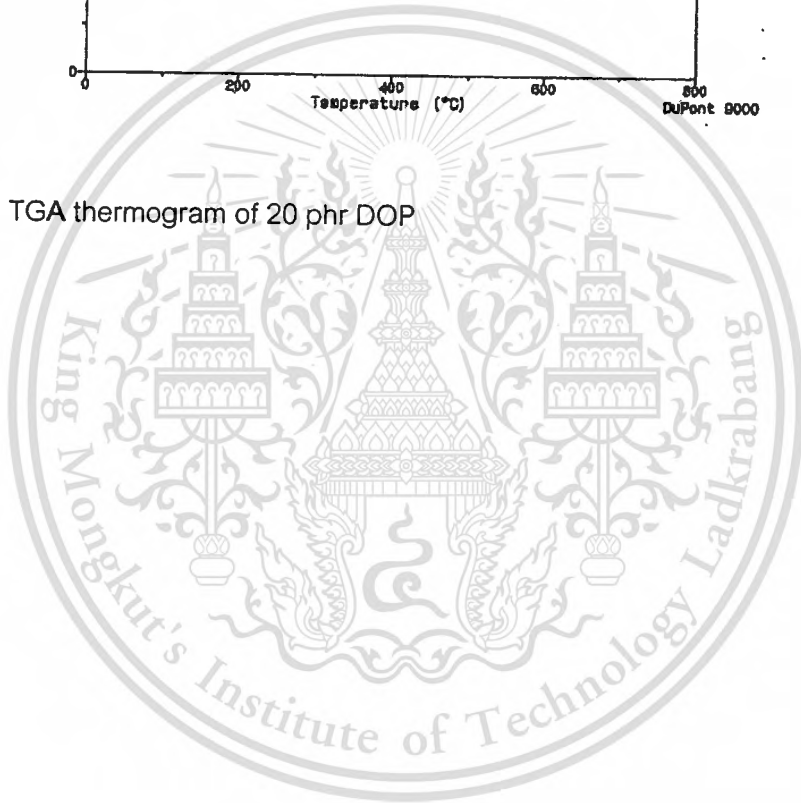


Figure A-11 TGA thermogram of 20 phr DOP



APPENDIX B Differential scanning calorimetry (DSC) thermograms

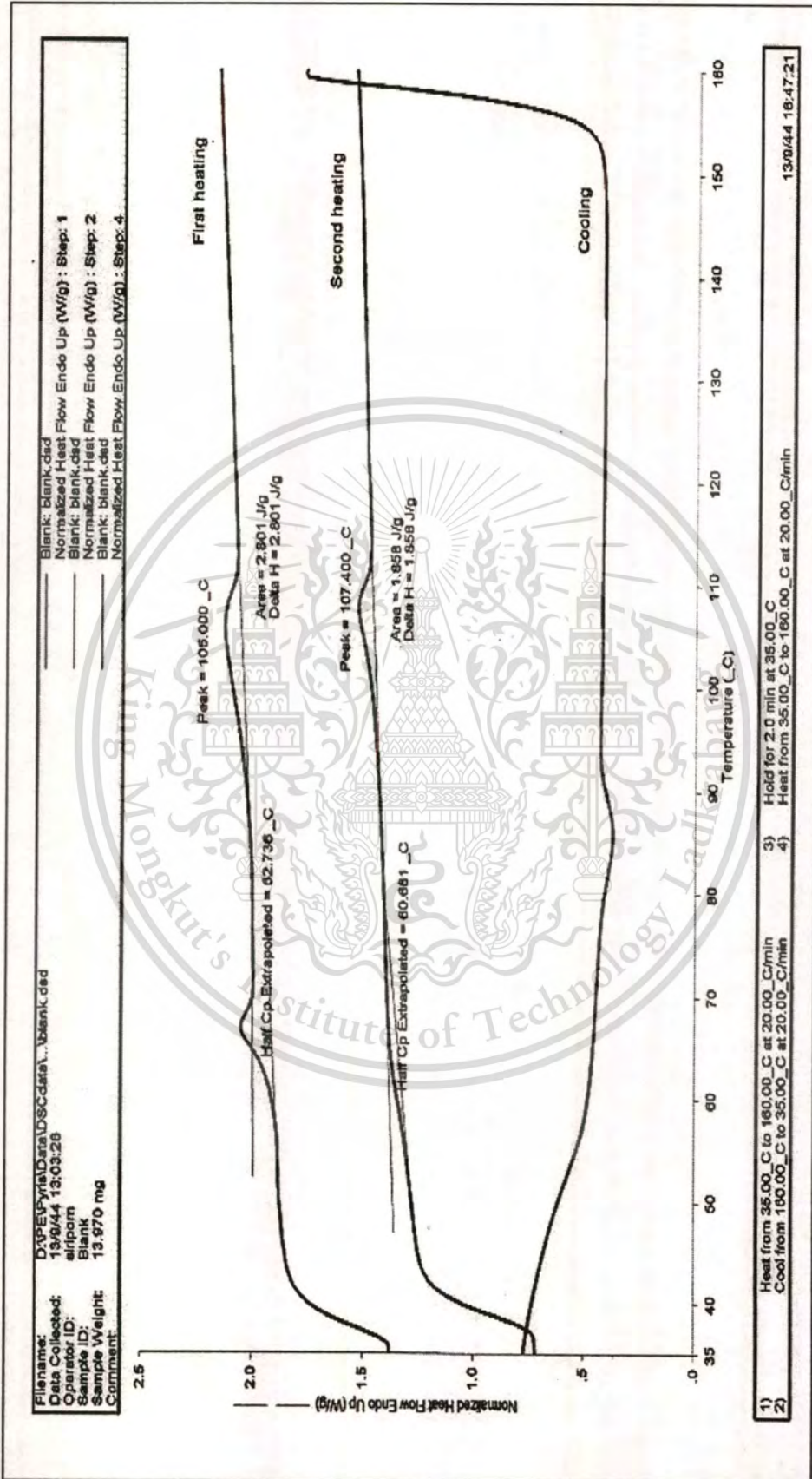


Figure B-1 DSC thermograms of PVC

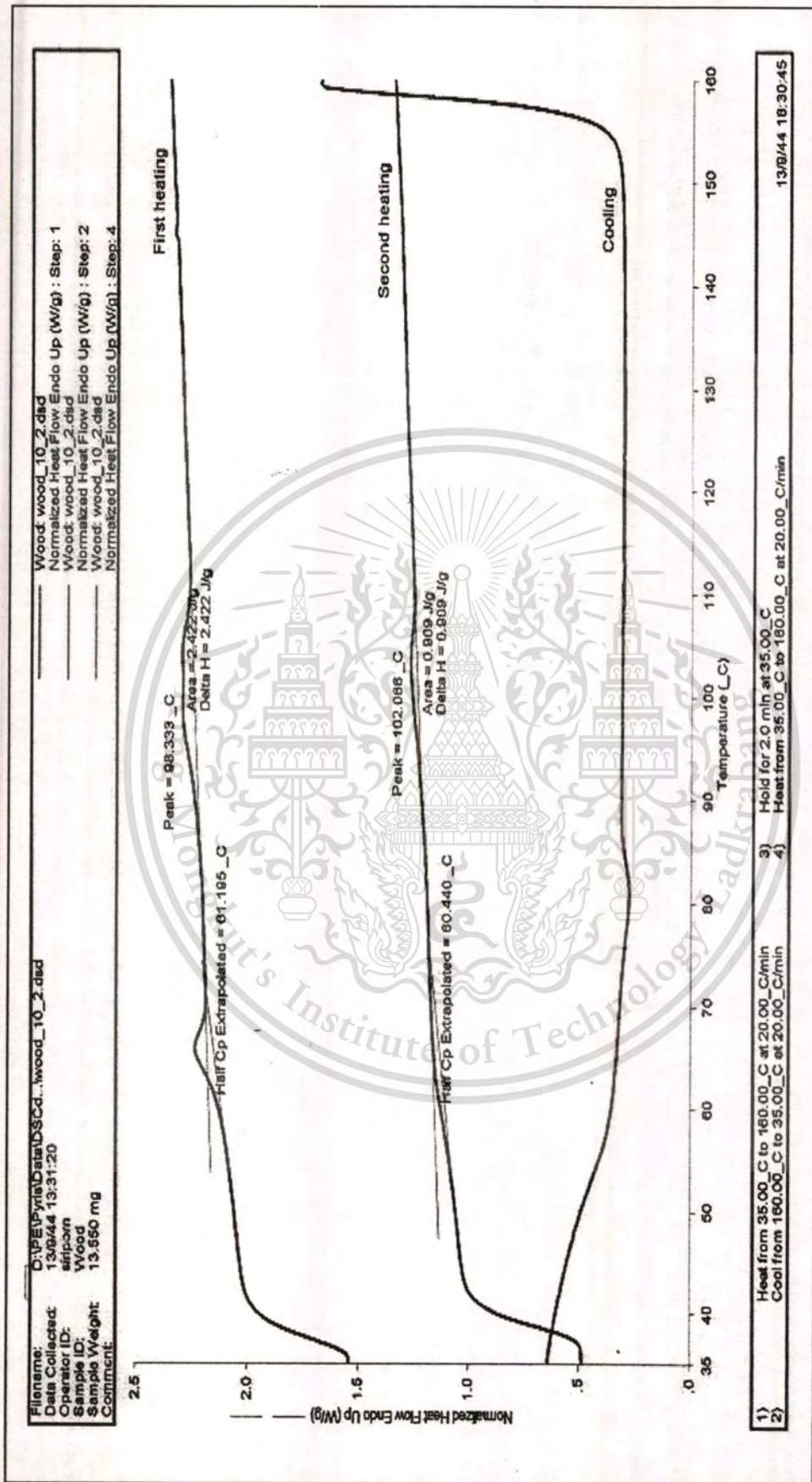


Figure B-2 DSC thermograms of 10 phr fiber

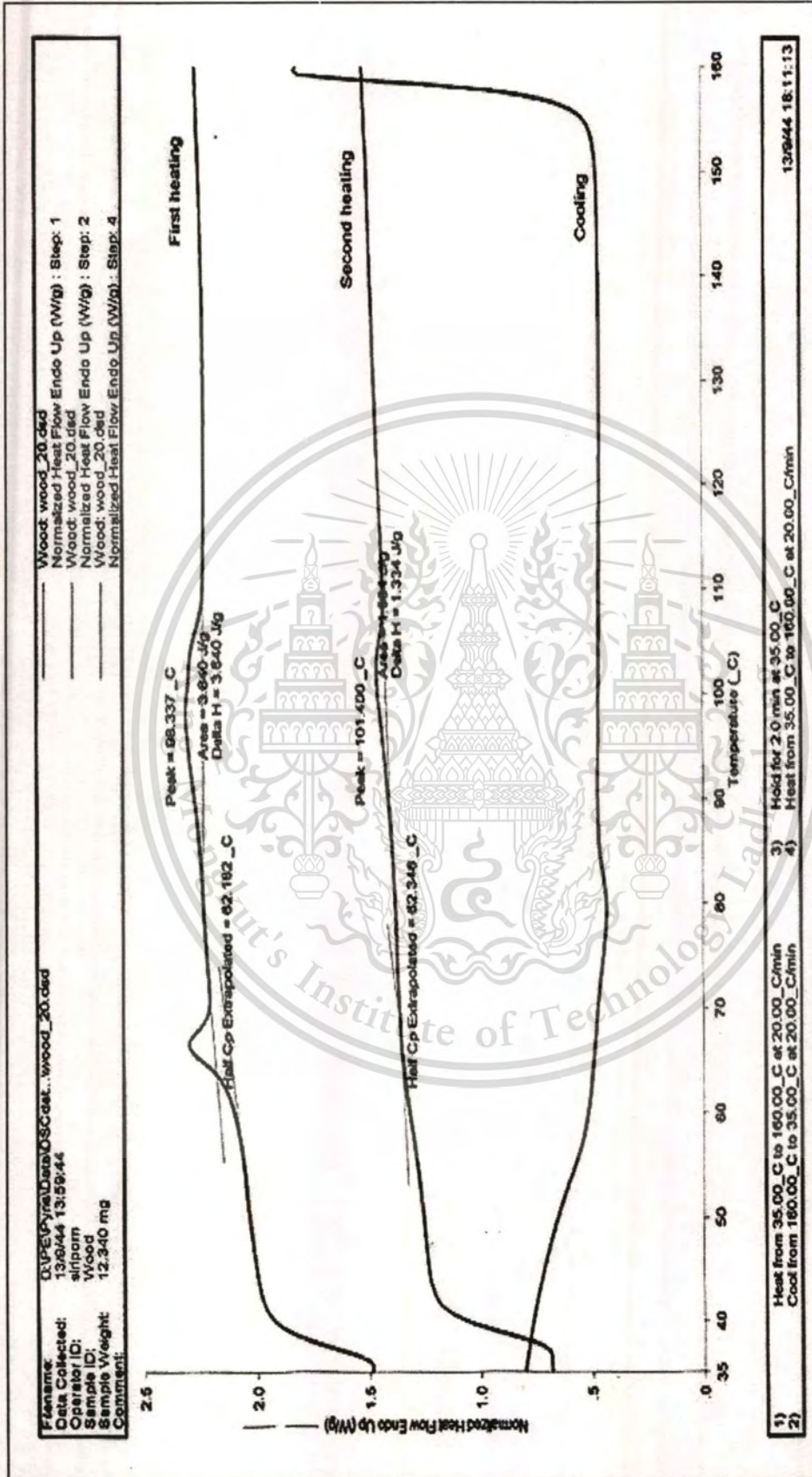


Figure B-3 DSC thermograms of 20 phr fiber

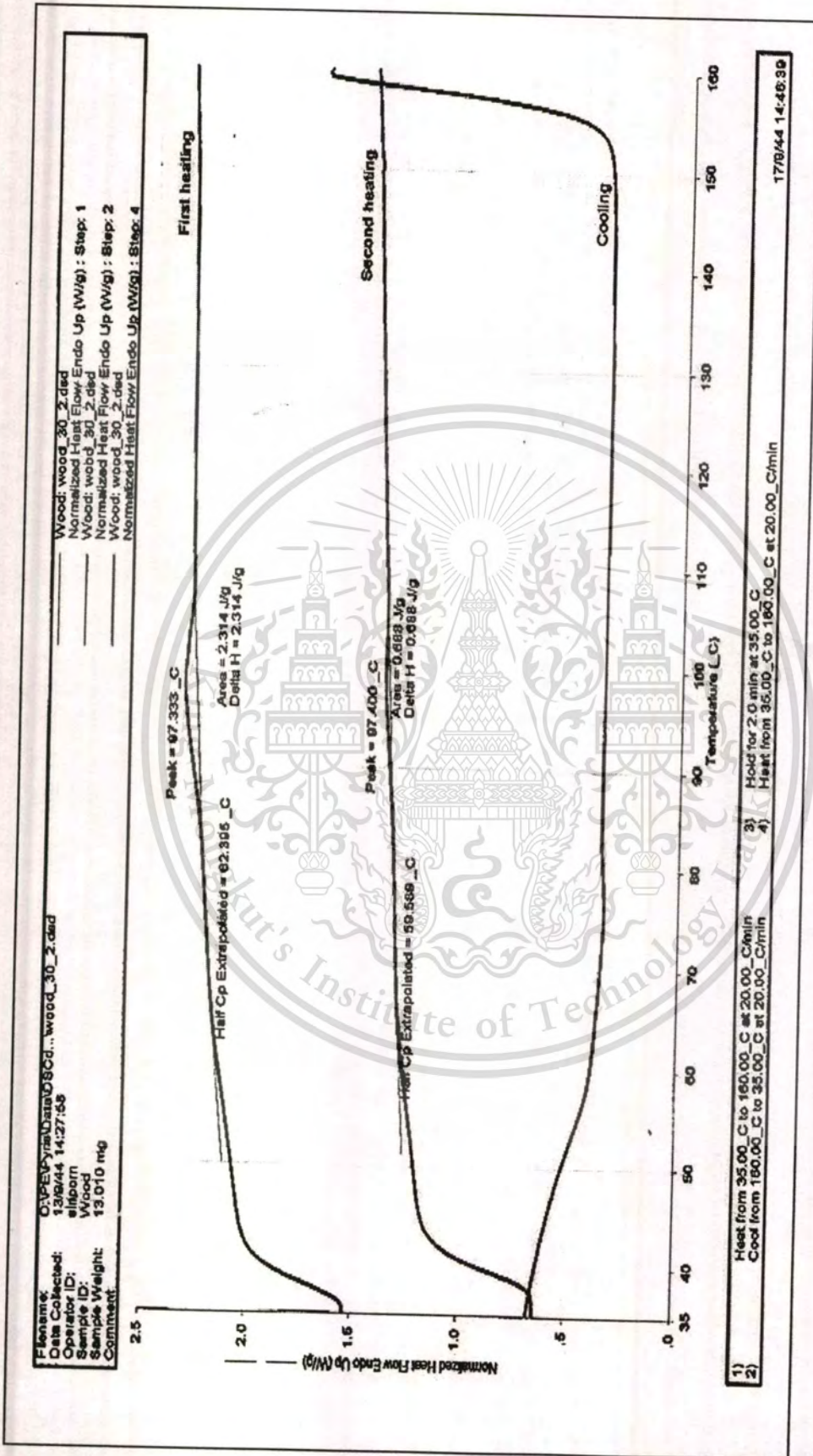


Figure B-4 DSC thermograms of 30 phr fiber

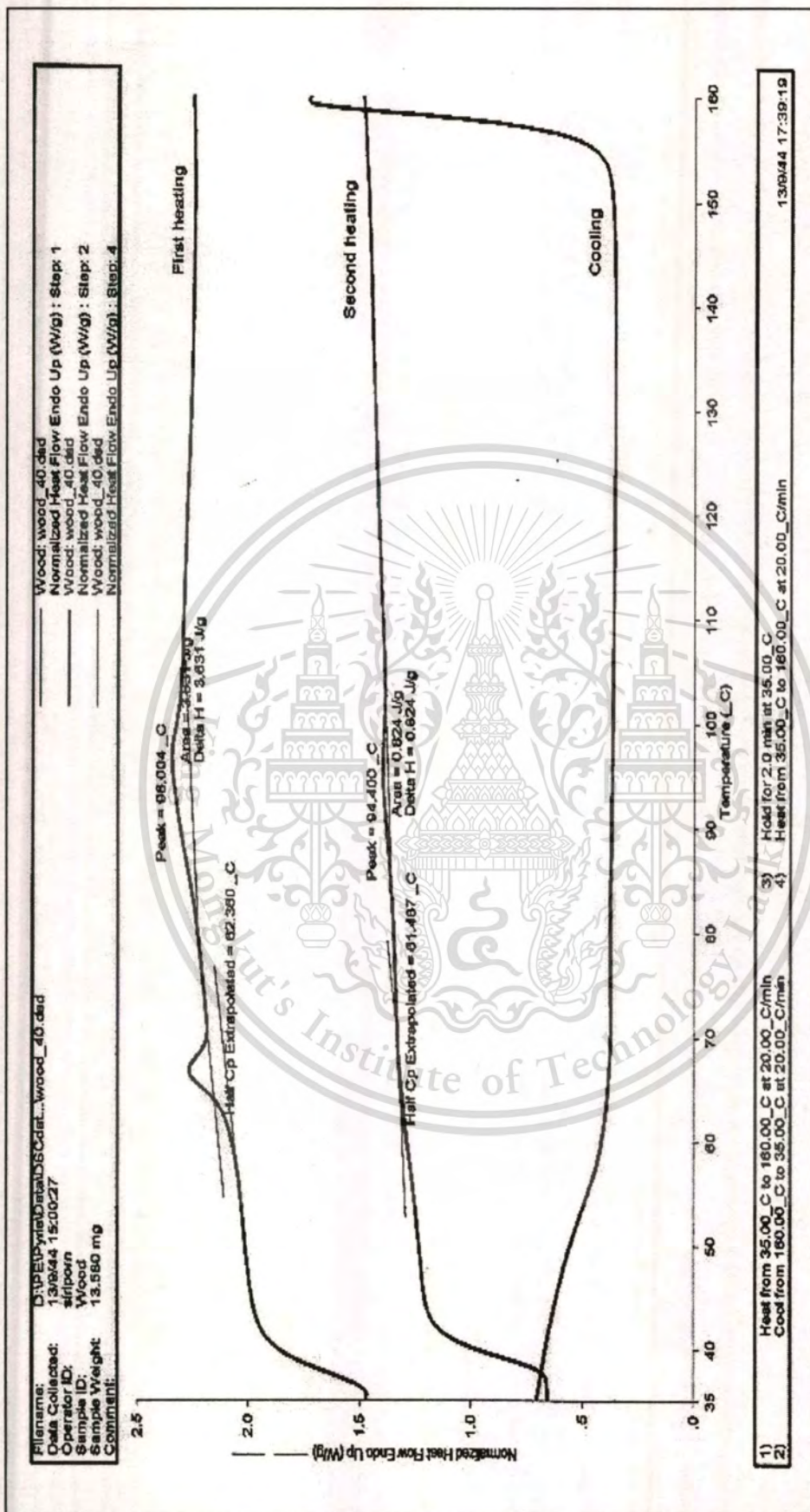


Figure B-5 DSC thermograms of 40 phr fiber

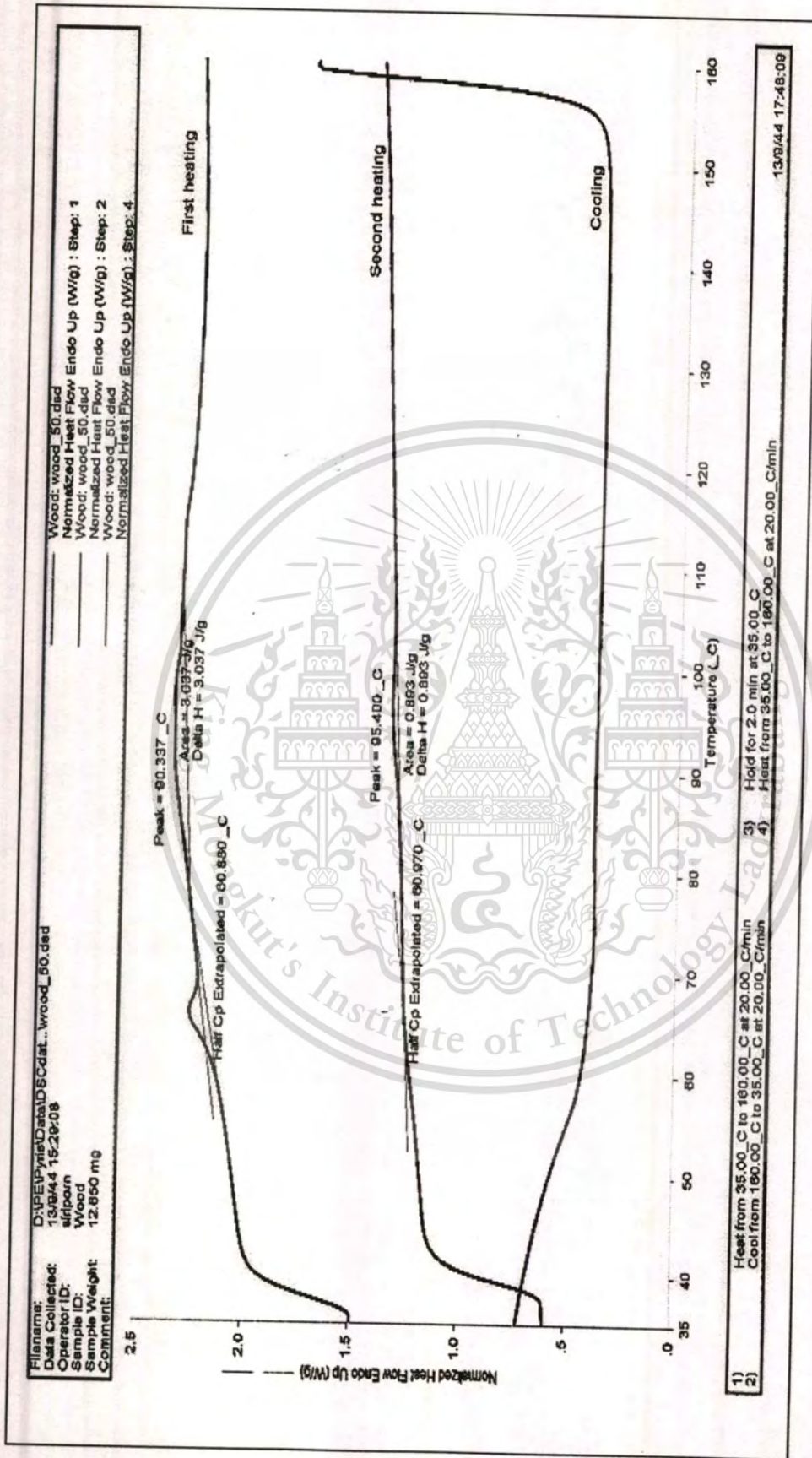


Figure B-6 DSC thermograms of 50 phr fiber

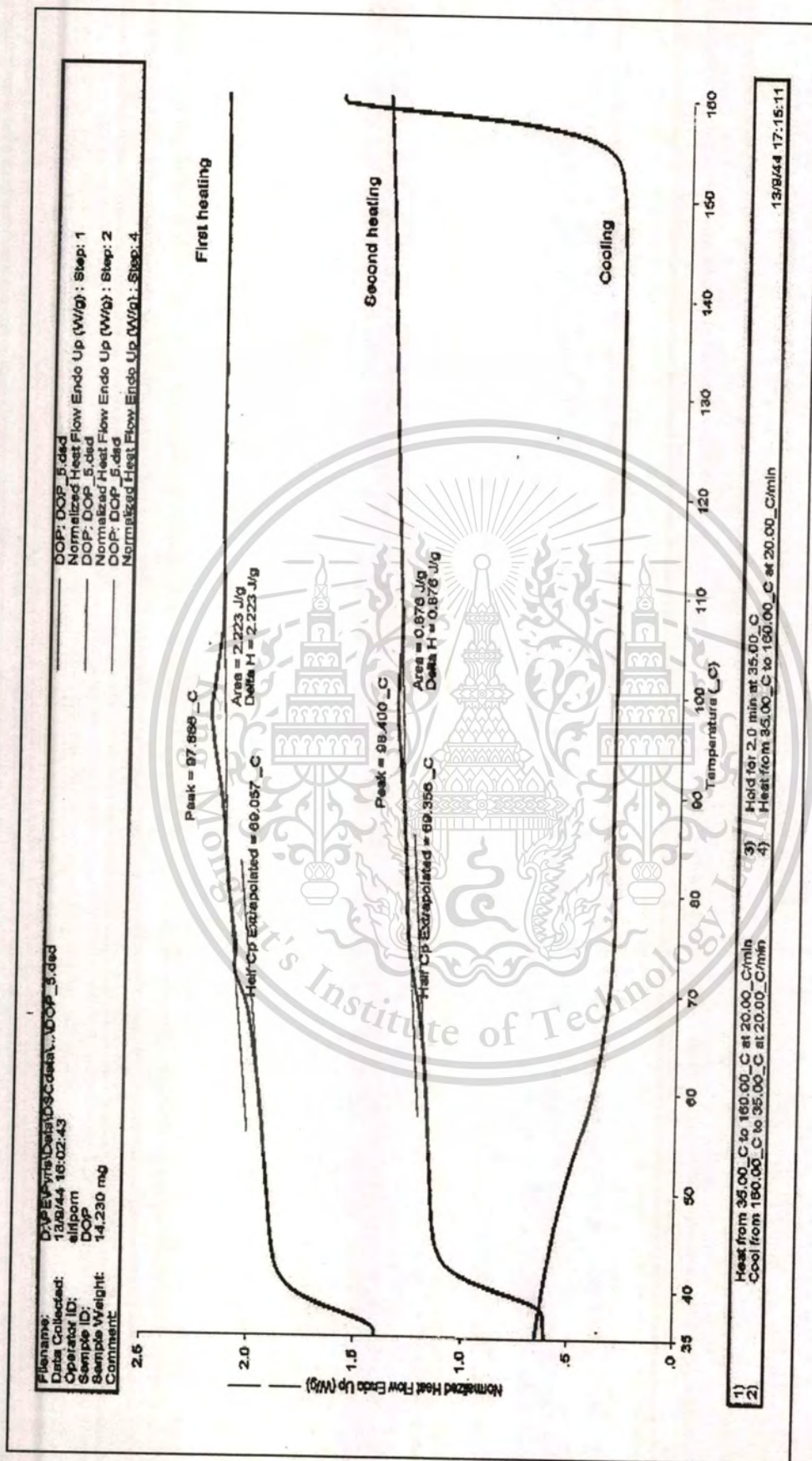


Figure B-7 DSC thermograms of 5 phr DOP

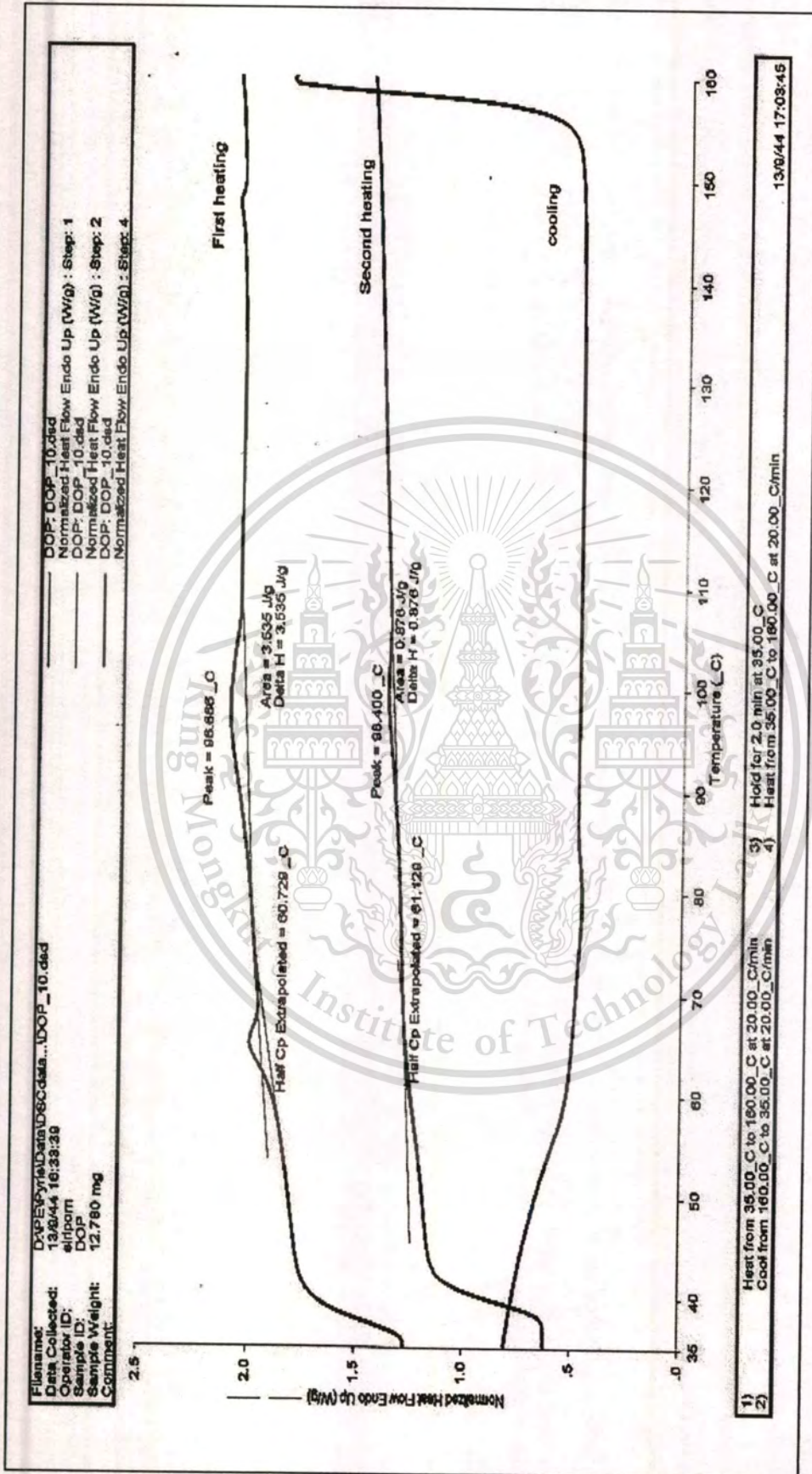


Figure B-8 DSC thermograms of 10 phr DOP

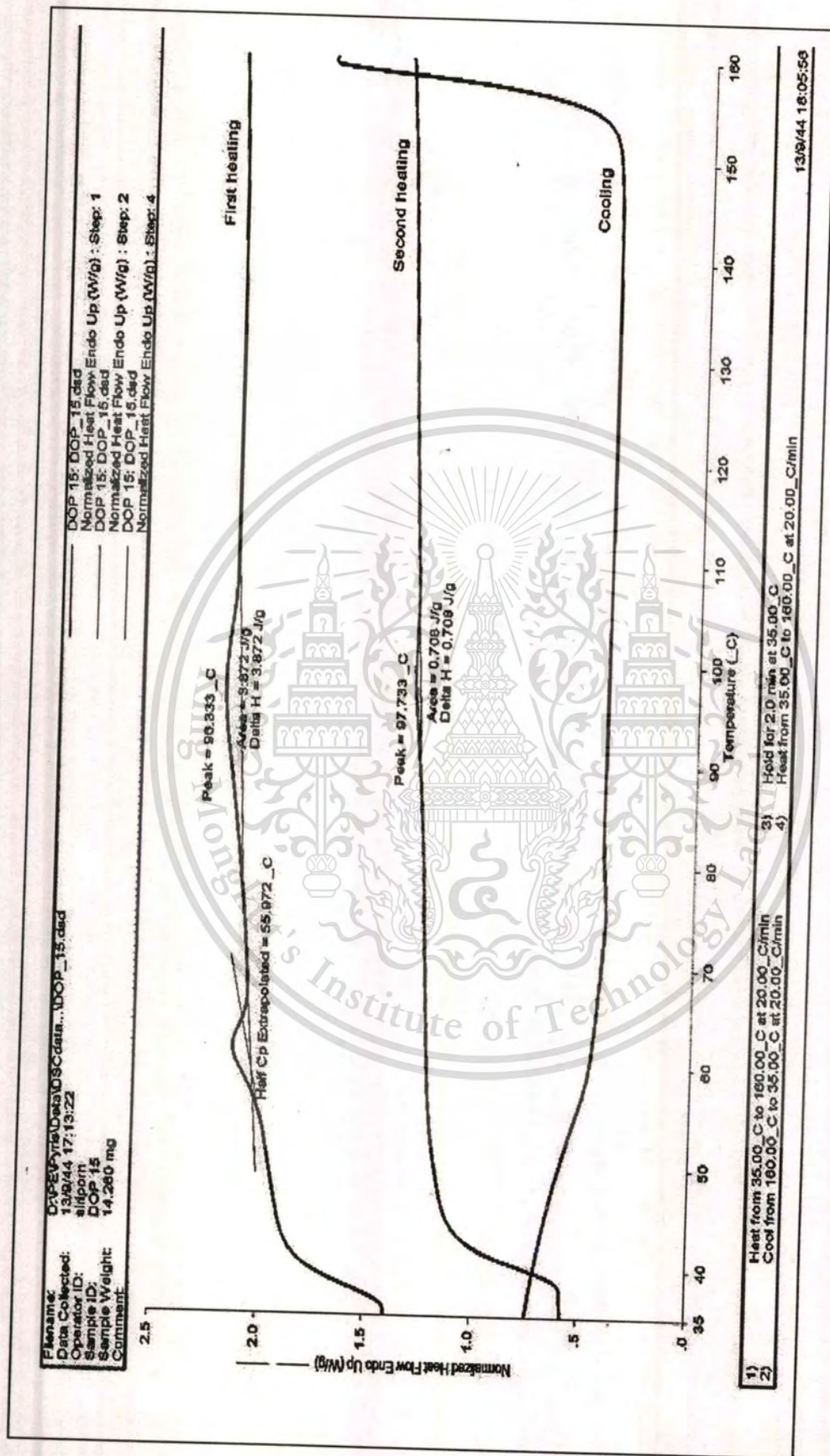


Figure B-9 DSC thermograms of 15 phr DOP

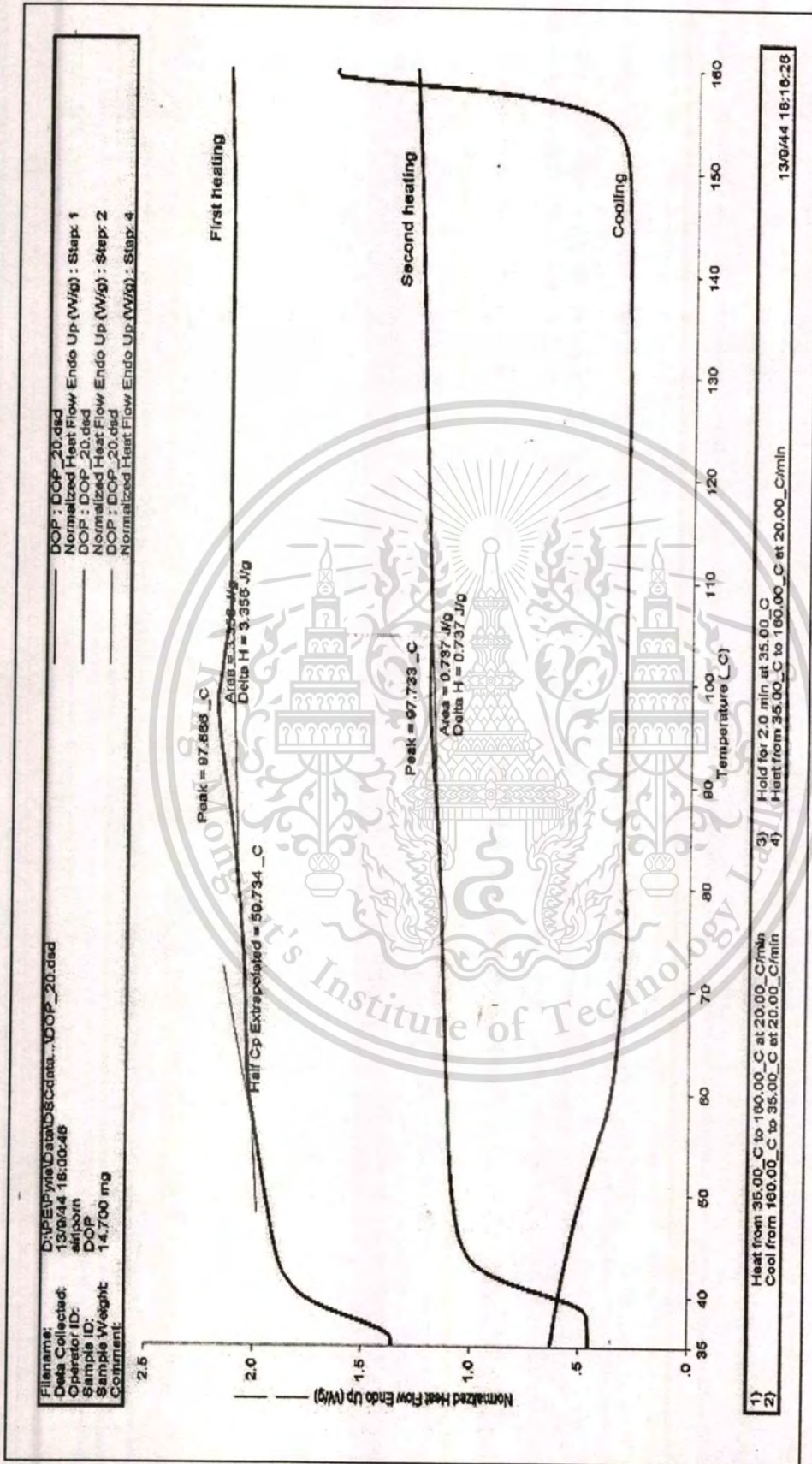


Figure B-10 DSC thermograms of 20 phr DOP

APPENDIX C Dynamic mechanical thermal analysis (DMTA) graphs

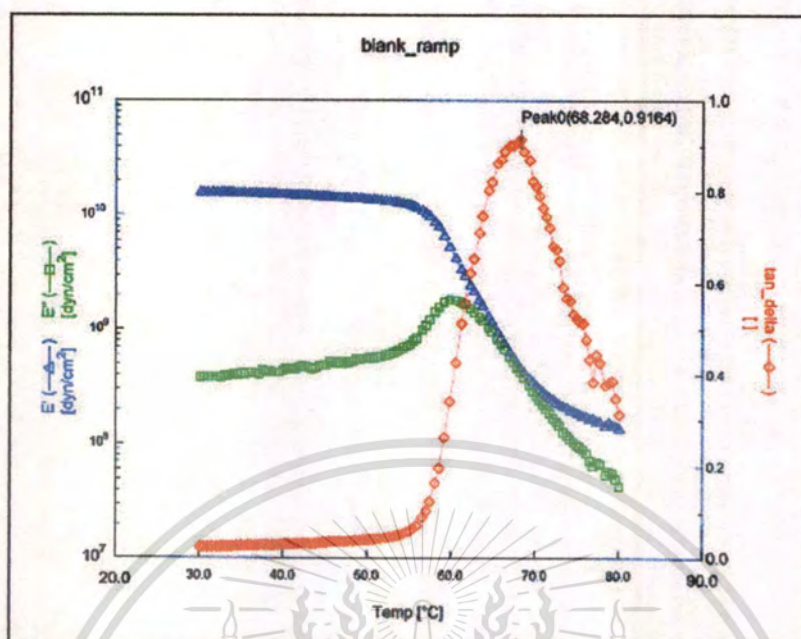


Figure C-1 DMTA graphs of PVC

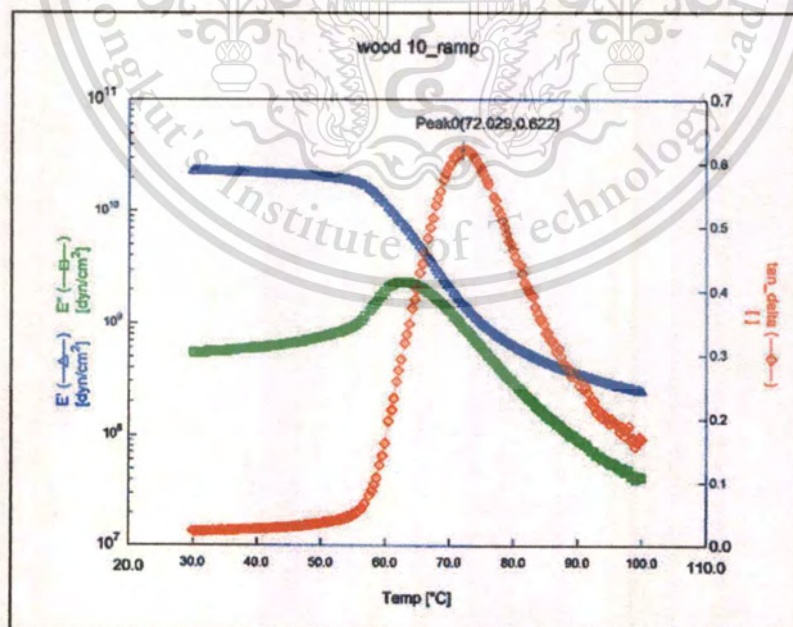


Figure C-2 DMTA graphs of 10 phr fiber

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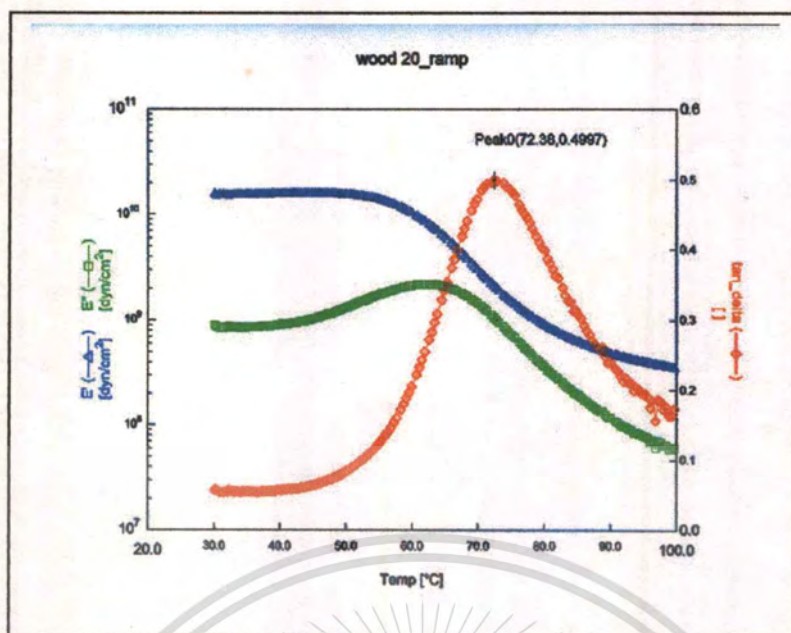


Figure C-3 DMTA graphs of 20 phr fiber

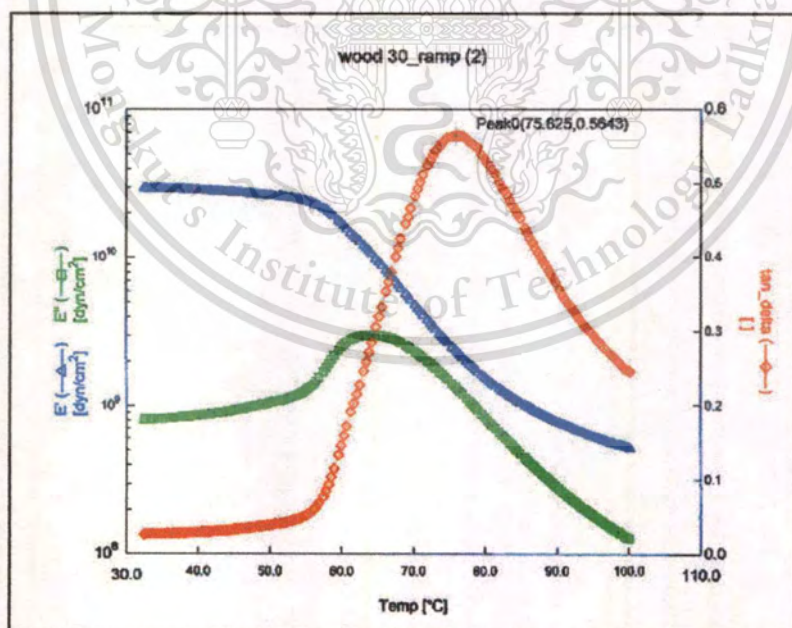


Figure C-4 DMTA graphs of 30 phr fiber

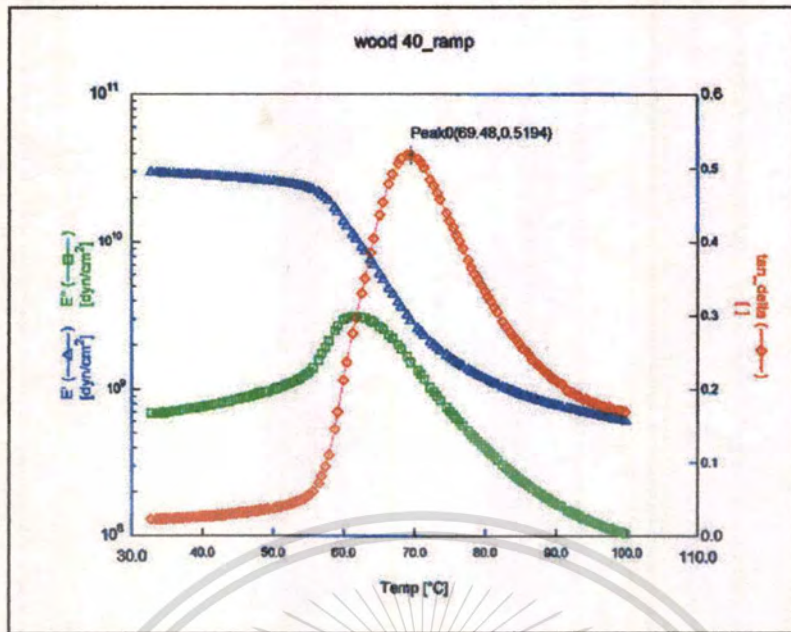


Figure C-5 DMTA graphs of 40 phr fiber

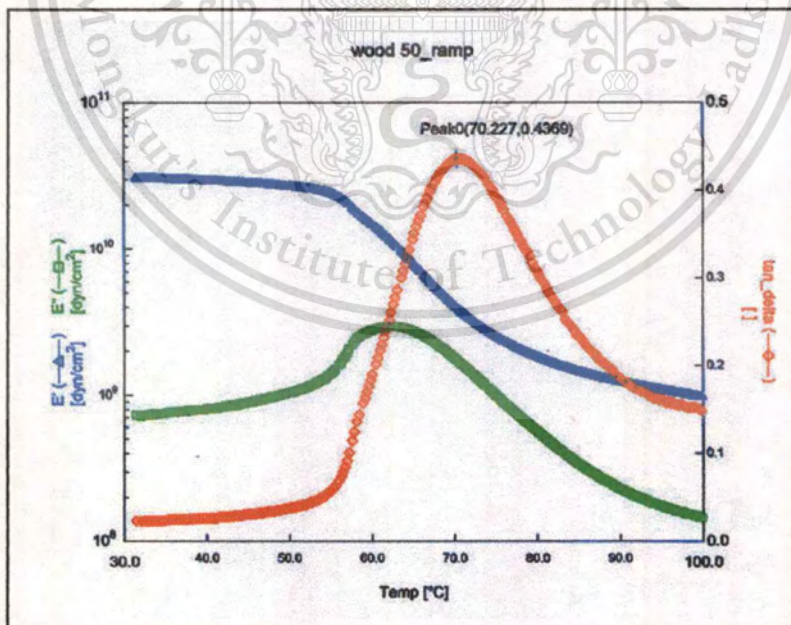


Figure C-6 DMTA graphs of 50 phr fiber

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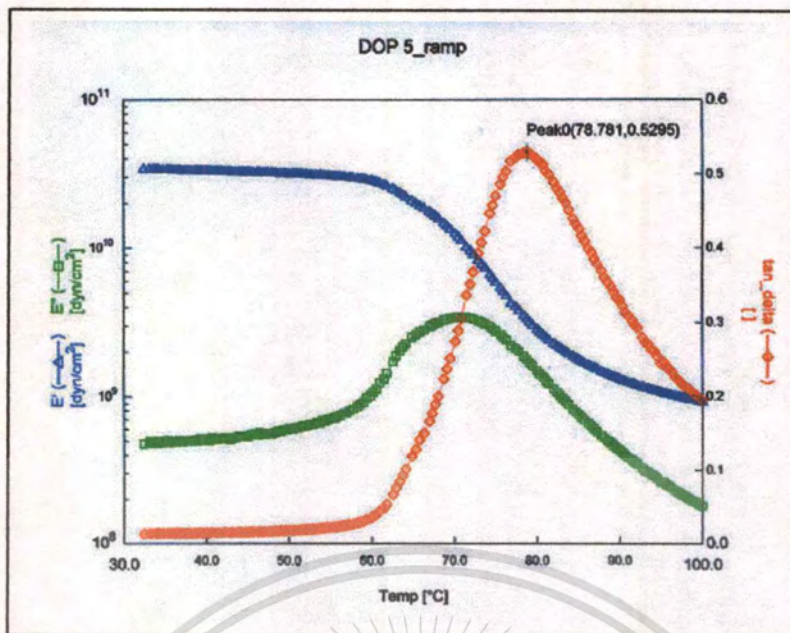


Figure C-7 DMTA graphs of 5 phr DOP

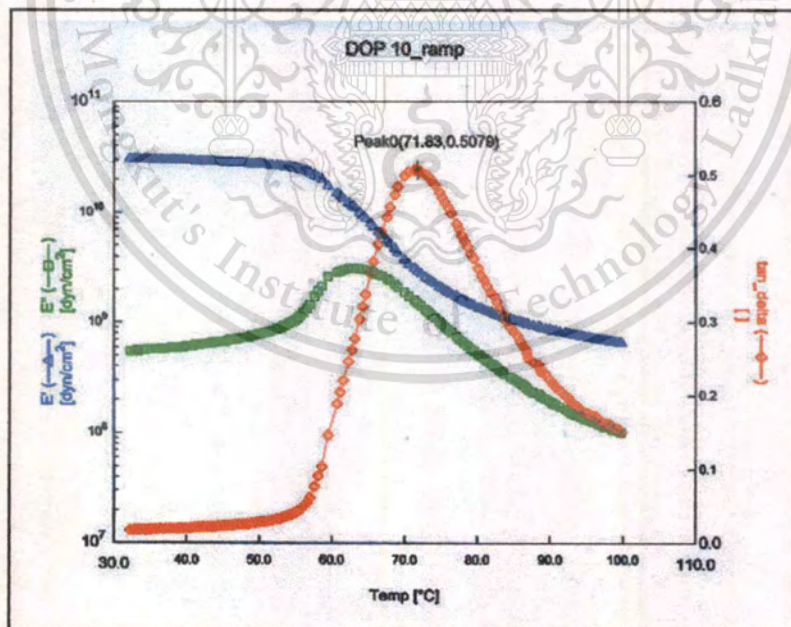


Figure C-8 DMTA graphs of 10 phr DOP

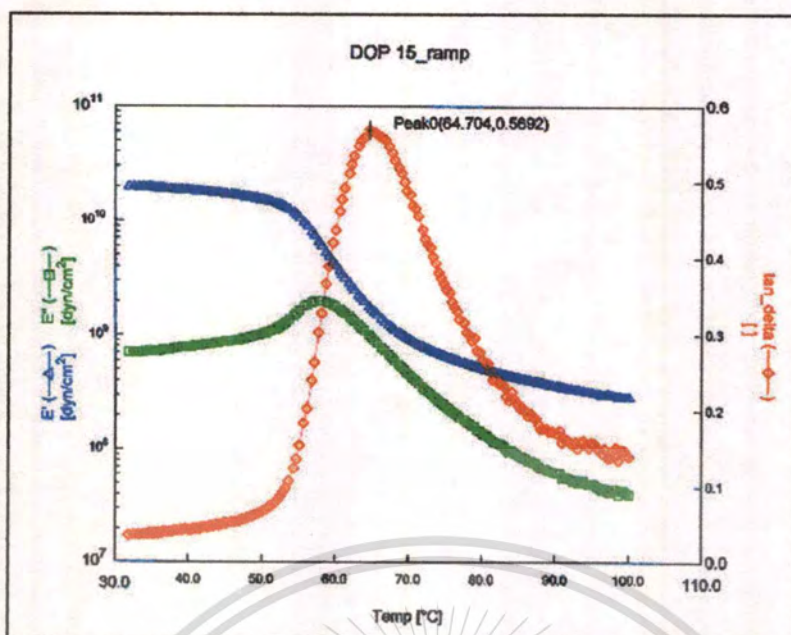


Figure C-9 DMTA graphs of 15 phr DOP

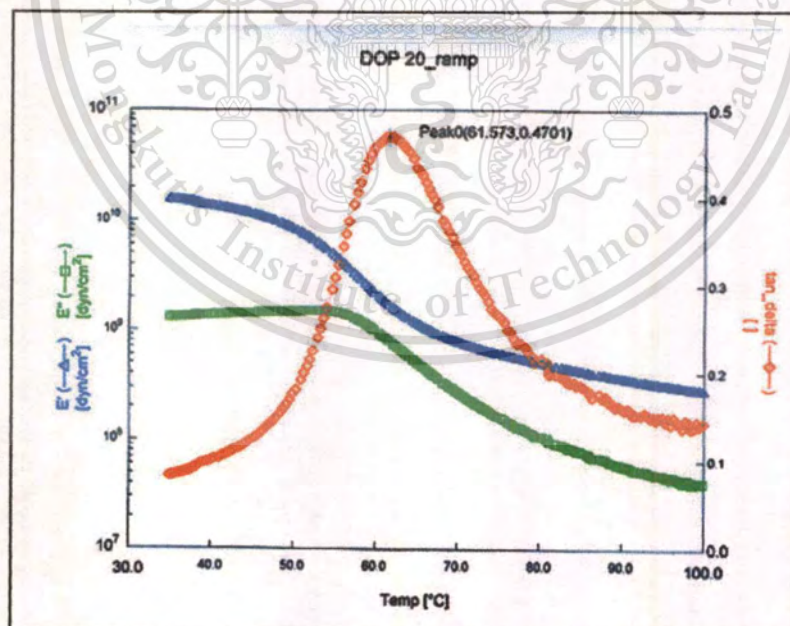


Figure C-10 DMTA graphs of 20 phr DOP

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Publication	Title: Mechanical Properties of Untreated Natural Rubber Fibers-Reinforced Poly(vinyl chloride) Composites: Effects of Fiber Content and Diocthyl Phthalate, Engineering Journal Chiang Mai University, Thailand, No. 1, Vol. 10, March 2002