CHAPTER III

APPARATUS AND EXPERIMENTAL PROCEDURES

In the study of particleboard from rubber wood flake with polymeric MDI binder, the rubber wooden flake was obtained from the Section of Forest Products Research and Development, Forest Research Office, Royal Forest Department. Polymeric MDI and phenolic resins are used as binders. Three-layer particleboard was fabricated, cut into testing specimens, and tested under normal and vigorous conditions. The interface adhesion between wood flakes is the important factor affecting the properties of particleboard, because it reflected the properties and utilization of particleboard. The effects of binder type and binder content in surface/core on the properties were investigated. Microstructure of fracture surface of bonded particleboard was observed by scanning electron microscopy.

3.1 Raw Materials and Chemicals

3.1.1 Rubber Wood Flakes

The wood flake in this study was all rubber wood flaked, obtained from Section of Forest Products Research and Development, Forest Research Office, Royal Forest Department. It was divided into two groups, the course and fine flakes. The coarse flake was used for core, and the fine one was used for the surfaces. Its color is light brown.

3.1.2 Binders

3.1.2.1 Polymeric MDI and Polyol

Polymeric MDI and Polyol were supplied by Thai Petrochemical Industry Co., Ltd. Polymeric MDI used in this study was Raycore B 9001, more than 99.9% MDI content, a dark brown liquid mixture of diphenylmethane diisocyanate (MDI) with isomers and homologues of high functionalities. It was used

in conjunction with polyols for various polyurethane applications; rigid foams for thermal insulation, buoyancy etc.; flexible molded foams; adhesives, coatings, and elastomers. Properties of Raycore B 9001 are shown in Table 3.1.

Table 3.1 Typical data of Raycore B 9001.

Appearance		
Colour		dark brown liquid
Odour		non to aromatic at room temperature
Viscosity (25 ^{0}C)	cps	150-250
NCO content	%	30.0-32.0
Acidity (as HCL)	%	0.02-0.10
Density (25 ^{0}C)	g/cm3	1.23

Polyol was Raypol 4150. It was a glycerine based polyether polyol of low viscosity and was normally used as a blending polyol in rigid foam formulations. Some properties of Raypol 4150 are shown in Table 3.2.

Table 3.2 Typical data of Raypol 4150.

Appearance		
Colour		clear liquid
Odour		none
Viscosity (25 ^{0}C)	cps	350-450
Hydroxyl value	mg KOH/g	360-390
Water content	Max., %	0.10
Density (25 ^{0}C)	g/cm3	1.03

3.1.2.2 Phenolic resin

Phenolic resin was Dynosol S-576, used in hot bonding. Some properties of Dynosol S-576 are shown in Table 3.3.

Table 3.3 Typical data of Dynosol S-576.

Appearance		
Colour		brown powder
Odour		none
Solubility in water		infinite
Viscosity in 1:1 solution (25 ^{0}C)	cps	400-800
pH in 1:1 solution (25 ^{0}C)		12.9-13.2
Density in 1:1 solution (25 ^{0}C)	g/cm3	1.22-1.23

3.1.3 Releasing Agents

Polyisocyanates have a tendency to adhere to metals under heat and pressure. This can be overcome by spraying the surface of the metal or the preformed mat with a liquid releasing agent. In this study, silicone released mold, aluminium foil sheet, paraffin oil, teflon coated tape, and polyol were used as the releasing agents.

3.1.4 Chemicals

1) Ethyl acetate, Commercial grade
2) Sodium hydroxide, Commercial grade
3) Isopropanal, Commercial grade

3.2 Apparatus and Equipment

1) Binder and particle blender: FSP 80, Draiswerke GMBH.
2) Hot press: LA 40/40, BURKLE.
3) Internal bond tester: UHP6, LUSENHAUSENWERK.
4) Universal testing system: 60CS-60,000LB CAP, SATEC SYSTEM Inc.
5) Shaker and Test sieve: VS 1000, Retch, Endecotts LTD. (5, 12, 20, 40, 60, 80, 100 mesh)
6) Specimen boiler: E.E. ENTERPRISES LTD., Part.
7) Drying oven: Precision Scientific Co., Ltd. and UT-5100E, HERAEUS.
8) Air compressor: OW-35, Fuji Compressor Mfg. Co., Ltd.
9) Scanning electron microscope: JSM-6400, JEOL Co., Ltd.
10) pH meter: Orion SA230, Orion reserch incorporated.
11) Optical microscope: Olympus SZH10.

3.3 Experimental Procedures

3.3.1 Wood Flake Analysis

1) The particle size of rubber wood flake was determined by using Retch VS 1000 instrument. In doing such analysis, a set of standard screens was arranged serially in a stack, with the smallest mesh at the bottom and larger meshes on the top. Weighed rubber-wood flake was placed on the top screen and stack was shaken mechanically for 10 min. The particles retained on each screen were removed and weighed, and the wood flake masses of each individual screen were converted to percentage of the total sample.

2) Slenderness ratio of the coarse and fine rubber wood flakes was determined. Each screening mesh, 100 particles were used.

$$\text{Slenderness Ratio} = \frac{\text{Length of Flake}}{\text{Thickness of Flake}} \qquad (3.1)$$

3) The pH of the coarse and fine rubber wood flakes was determined. In doing such analysis, 5 g of the flake was soaked in 75 ml of distilled water. After that 1 hour, pH was measured.

3.3.2 Wood Flake Moisture Content

To test moisture content, two samples were randomly taken from each bag. Each sample was weighed accurate to 0.1 g, and put them in an air drier at 103±2 ^{0}C for over night to obtain dry weight. Then, the sample was taken from drier and cooled in desiccator. Each sample was weighed again. The moisture content in each sample was calculated as follows:

$$\text{Moisture Content (\%)} = \frac{\text{Weight of Water}}{\text{Weight of Dry Wood}} \times 100 \qquad (3.2)$$

or

$$= \frac{m_1 - m_0}{m_0} \times 100 \qquad (3.3)$$

where, m_0 : weight after drying

m_1 : weight before drying

3.3.3 Treatment of Binder on Rubber Wood Flakes

For each treatment, the appropriate quantity of flakes was weighed and placed in a rotary drum blender. The prepared resin, (as percent based on the oven-dried weight of flakes; 100% solid) was sprayed onto the flakes from a sprayhead mounted at the center of the blender drum. Compressed air was used to atomize the resin.

3.3.4 Preparation of Particleboard and Finish Product

In this experiment, different particleboards were prepared as follows:

1) The particleboards were prepared from flakes sprayed with pMDI and polyol.
2) The particleboards were prepared from flakes sprayed with pMDI resin.

3) The particleboards were prepared from flakes sprayed with phenolic resin.

3.3.4.1 Mat Formation

After blending, the flakes were matted by hand on cauls that were previously treated with releasing agents. In all case, the mat was formed into 3 layers particleboard. It was formed within forming case size 35X35 cm which placed on the treated lower caul by 250 g of fine flakes for each surface, and 500 g of coarse flakes for the core. Then, the completely formed mat was prepressed to consolidate the mat for subsequent handling, covered with treated upper caul and transferred to the hot press.

3.3.4.2 Hot Pressed

The 10 mm thick stoppers were used to control the thickness of the mats. The resin was cured at the selected temperature. The mat was pressed for the definite time. The pressed pressure was 160 kg/cm^2 for maximum pressure and 140 kg/cm^2 for minimum pressure.

3.3.4.3 Cutting and Conditioning

After pressed, the particleboards were cooled at room temperature for overnight. After that, the finish product was cut to the size 32X32 cm and conditioned at the atmosphere for seven days before cutting to test specimens.

3.3.5 Factors Affecting on Rubber Wooden Particleboard Properties

3.3.5.1 Investigation of Suitable Releasing Agents

In this study, silicone released mold, paraffin oil, aluminium foil sheet, teflon coated tape, and polyol were studies. In these experiments, the mats were prepared from flakes which sprayed with 6% pMDI both core and surface. The cauls were treated with those releasing agents in mat forming. The particleboard was prepared at pressed pressure of 140-160 kg/cm^2, cure temperature of 160 ^0C, and cure time of 5 min.

3.3.5.2 Effect of Releasing Agents on the Properties

The particleboards were prepared as follows: 6% pMDI (both surfaces and core layers), 7% wood flake moisture content based on the oven-dry weight, cure temperature of 160 ^{0}C, cure time of 5 min., pressed pressure of 140-160 kg/cm^2. Different releasing agents (teflon releasing tape and polyol releasing agent) were used.

3.3.5.3 Effect of Isocyanate Index on the Properties

The partioleboards were prepared as follows: 6% binder content (both surfaces and core layers), 7% wood flake moisture content based on the oven-dry weight, cure temperature of 160 ^{0}C, cure time of 5 min., pressed pressure of 140-160 kg/cm^2, polyol releasing agent. Different additive compositions (isocyanate index 1, 2, 3, and ∞ (infinite or pure pMDI)) were used as shown in Table 3.4. As the chemical reaction occurs on the equivalent weight ratio is called the isocyanate index. [22, 51]

$$\text{Isocyanate Index} = \frac{\text{Isocyanate Equivalents}}{\text{Polyol Equivalents}} \qquad (3.4)$$

Table 3.4 Weight of pMDI and polyol in the binders.

Isocyanate index	Weight of binder (g)			
	fine flakes		coarse flakes	
	pMDI	polyol	pMDI	polyol
1	91.57	101.21	88.52	97.83
2	124.18	68.60	120.05	66.30
3	140.92	51.86	136.22	50.13
∞	192.78	---	186.35	---

3.3.5.4 Effect of pMDI content in Surface/Core on the Properties

The particleboards were prepared as follows: 7% wood flake moisture content based on the oven-dry weight, cure temperature of 160 ^{0}C, cure time of 5 min., pressed pressure of 140-160 kg/cm^2. Different pMDI contents on surfaces and core layers (6/6, 7/5, 7/4, and 7/3) were studied. For example, pMDI content on surface and core layer is 7/5, 7% pMDI in surface layers and 5% in core layer.

3.3.5.5 Effect of Cure temperature on the Properties

The particleboards were prepared as follows: 7% pMDI in the surface and 5% pMDI in the core, 7% wood flake moisture content based on the oven-dry weight, cure time of 5 min., pressed pressure of 140-160 kg/cm^2. Different cure temperatures (120, 140, and 160 ^{0}C) were studied.

3.3.5.6 Effect of Cure Time on the Properties

The particleboards were prepared as follows: 7% pMDI in the surface and 5% pMDI in the core, 7% wood flake moisture content based on the oven-dry weight, cure temperature of 160 ^{0}C, pressed pressure of 140-160 kg/cm^2. Different cure times (3, 4, and 5 min.) were investigated.

3.3.5.7 Effect of Wood Flake Moisture Content on the Properties

The particleboards were prepared as follows: 7% pMDI in the surface and 5% pMDI in the core, cure temperature of 160 ^{0}C, cure time of 5 min., pressed pressure of 140-160 kg/cm^2. Different wood flake moisture contents based on the oven-dry weight (2, 7, 12, 17, and 22%) were studied.

3.3.5.8 Effect of Phenolic Resin (PF) on the properties

The particleboards were prepared as follows: 7% binder in the surface and 5% binder in the core, 7% wood flake moisture content based on the oven-dry weight, cure temperature of 160 ^{0}C, cure time of 5 min., press pressure of 140-160 kg/cm^2. Different binders types in surface/core layers (pMDI/pMDI, PR/pMDI, and PR/PR) were studied. For example, binder in the surface and core layers is PR/pMDI, PR in surface layers and pMDI in the core layer. In the case of using PR in the surface layers, paraffin oil was used as releasing agent.

Table 3.5 Processing parameters of the particleboards.

Particleboard	Binder	Binder content (%)	Pressed temperature (^{O}C)	Cure time (min.)	Pressed pressure (kg/cm^2)	Flake moisture content (%)	Releasing agent
A	pMDI	6/6	160	5	160-140	7	teflon tape
B	pMDI	6/6	160	5	160-140	7	polyol
C	pMDI+polyol (iso. index 1)	6/6	160	5	160-140	7	polyol
D	pMDI+polyol (iso. index 2)	6/6	160	5	160-140	7	polyol
E	pMDI+polyol (iso. index 3)	6/6	160	5	160-140	7	polyol
F	pMDI	7/5	160	5	160-140	7	polyol
G	pMDI	7/4	160	5	160-140	7	polyol
H	pMDI	7/3	160	5	160-140	7	polyol
I	pMDI	7/5	140	5	160-140	7	polyol
J	pMDI	7/5	120	5	160-140	7	polyol
K	pMDI	7/5	160	4	160-140	7	polyol
L	pMDI	7/5	160	3	160-140	7	polyol

Table 3.5 (continued)

Particleboard	Binder	Binder content (%)	Pressed temperature (^{O}C)	Cure time (min.)	Pressed pressure (kg/cm^2)	Flake moisture content (%)	Releasing agent
M	pMDI	7/5	160	5	160-140	2	polyol
N	pMDI	7/5	160	5	160-140	12	polyol
O	pMDI	7/5	160	5	160-140	17	polyol
P	pMDI	7/5	160	5	160-140	22	polyol
Q	PF/pMDI	7/5	160	5	160-140	7	paraffin oil
R	PF/PF	7/5	160	5	160-140	7	paraffin oil

3.3.6 Testing

The properties of the rubber wooden particleboards were measured by following the test methods:

The water absorption (except size of specimen), thickness swelling, internal bond (dry), modulus of rupture (dry), and modulus of Elasticity (dry) were determined according to TIS 867-2532. The internal bond (wet) was determined according to DIN 68763. The modulus of rupture (wet) was determined according to CAN 3-0188.0-M78.

Table 3.6 Dimension of testing specimens.

Standard	**Testing Method**	**Dimension of Specimens** (mm)
TIS 876-2532	Water Absorption	100X100
	Thickness Swelling	100X100
	Internal Bond	50X50
	Modulus of Rupture & Modulus of Elasticity	100X16 times of thickness
DIN 68763	Internal Bond (wet)	50X50 (DIN 52365)
CAN3-0188.0-M78	Modulus of Rupture (wet)	75X24 times of thickness

3.3.6.1 Water Absorption (WA)

The testing specimens were weighed to 0.1 gram accuracy, refer to the weight before water soaking. Then, the specimens were submerged vertically in cleaning water at room temperature, about 25 mm under water level. After a 2-hour submersion, the specimens were taken from the water, dabbed dry, and immediately weighed, refer to the weight after 2 hours soaking. The specimens were submerged for an additional period of 22 hours, dabbed dry, weighed, refer to the weight after 24 hours soaking. The water absorption at 2 hours and 24 hours of the specimens were calculated as follows:

$$\text{Water Absorption (\%)} = \frac{W_0 - W_1}{W_1} \times 100 \qquad (3.5)$$

where, W_0 : Weight after water soaking (gram)

W_1 : Weight before water soaking (gram)

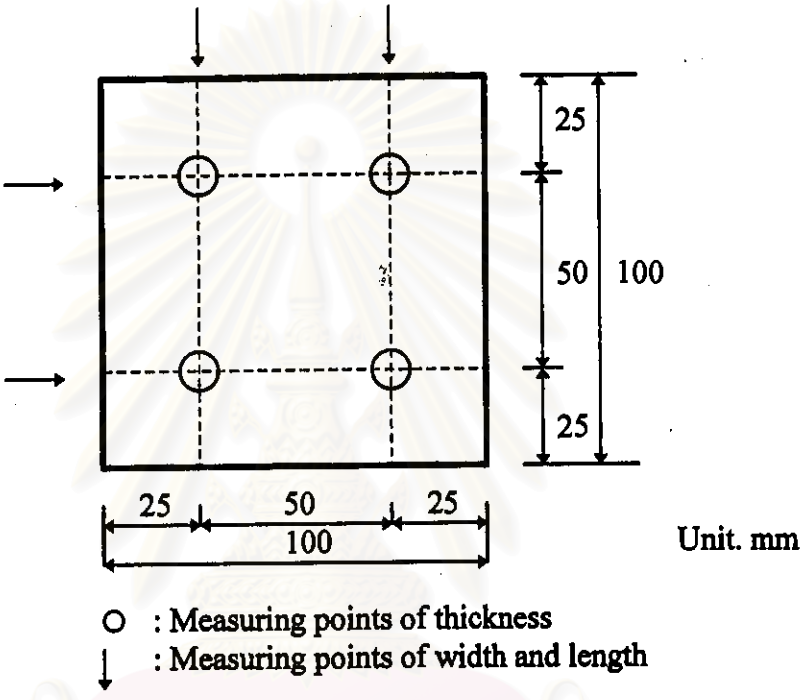

Figure 3.1 Points to be measured of length, width and thickness.

3.3.6.2 Thickness Swelling (TS)

The testing specimens were marked the thickness testing positions according to Figure 3.1, averaged as before water soaking thickness. The specimens were submerged vertically in cleaning water at room temperature, about 25 mm under water level. After 1 hour submersion, they were taken from the water, quickly dabbed dry, and left them in room temperature by one edge placed on unabsorb water material, e.g. plastic or mirror. The specimens were placed for an additional 1 hour. The thickness of specimens was measured at the same position, averaged as after water soaking thickness. The thickness swelling of specimens was calculated as follows:

$$\text{Thickness Swelling (\%)} = \frac{T_0 - T_1}{T_1} \times 100 \qquad (3.6)$$

where, T_0 : Thickness after water soaking (mm)

T_1 : Thickness before water soaking (mm)

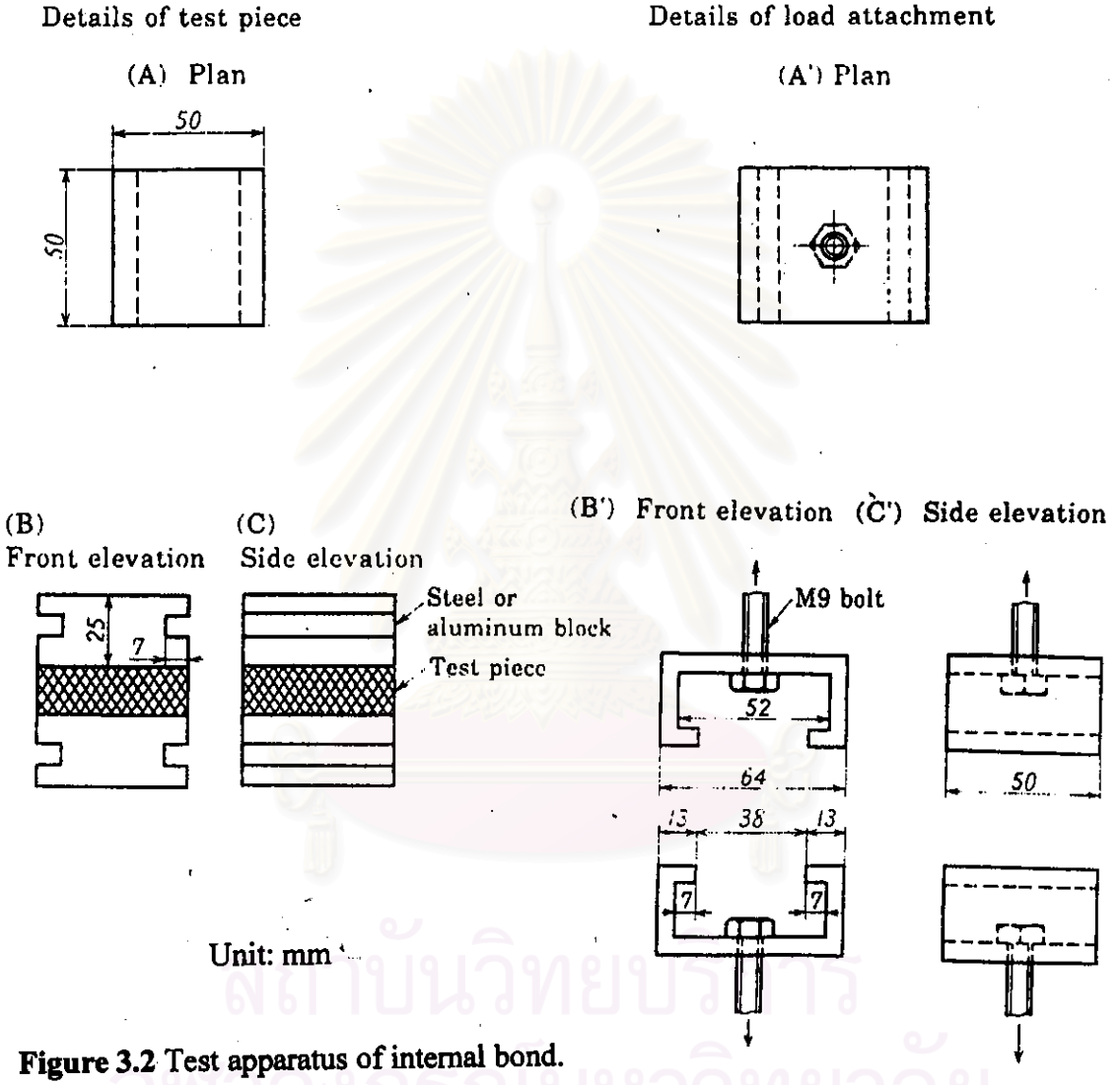

Figure 3.2 Test apparatus of internal bond.

3.3.6.3 Internal Bond (IB)

Loading blocks were effectively bonded both surfaces of testing specimens with a suitable adhesive. The loading fixtures attached to the head of the testing machine, with the block attached to the specimen were engaged, as shown in Figure 3.2. The specimen was stressed by separation of the heads of testing machine until failure occurred, usually in core layer. The increasing of the stretching force must

be constant. The time for specimen failure was not lower than 30 sec and not greater than 120 sec. The internal bond of specimens was calculated as follows:

$$\text{Internal Bond (MPa)} = \frac{P}{bXl} \quad (3.7)$$

where, P : Maximum load at the time of failing force(N)

b : Width of test piece (mm)

l : Length of test piece (mm)

3.3.6.4 Modulus of Rupture (MOR) and Modulus of Elasticity (MOE)

The span for testing was 16 times of the norminal thickness of the specimen. The testing specimen was placed on the span, as shown in Figure 3.3, both end tips were left over the span about 25 mm by each side. The specimen was loaded center of span with the loading applied to the finished face at the uniform rate. The time for specimen broke down was not lower than 30 sec and not greater than 120 sec. The relative graph between loading force and the bending is shown in Figure 3.4. The MOR and MOE of the specimen were calculated as follows:

$$\text{Modulus of Rupture (MPa)} = \frac{3\,Wl}{2bd^2} \quad (3.8)$$

$$\text{Modulus of Elasticity (MPa)} = \frac{l^3\,\Delta W}{4bd^3\,\Delta S} \quad (3.9)$$

where, W : Maximum loading force (N)

l : Length of span (mm)

b : Width of specimen (mm)

d : Thickness of specimen (mm)

ΔW : Loading force which increased in rang of linear graph (mm)

ΔS : Bending distance which increased in rang of linear graph (mm)

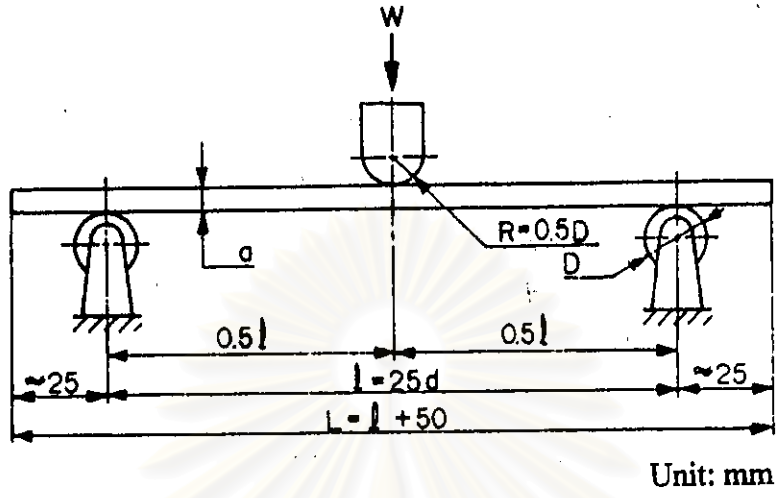

Figure 3.3 Testing of modulus of rupture (MOR) and modulus of elasticity (MOE).

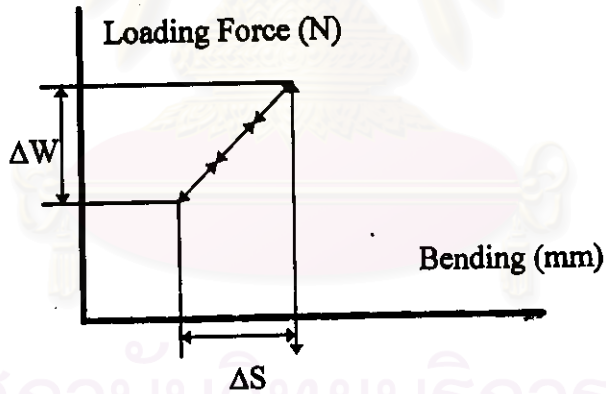

Figure 3.4 Graph shown relation between loading force and bending.

3.3.6.5 Internal Bond (wet)

Type V100 test pieces, with the platens bonded to them, were placed in water at a temperature 20±5 ^{0}C, which was heated slowly to 100 ^{0}C over a period of one or two hours, and left in boiling water for two hours. The test pieces were placed in water bath, at least 15 mm apart on all sides, to ensure complete immersion and to leave room for swelling. After conditioning, the test pieces were

cooled for at least 1 hour in water at a temperature of 20±5 ^{0}C, dabbed dry, and tested while still wet.

3.3.6.6 Modulus of Rupture (wet)

The testing specimens were submerged in boiling water for not less than 2 hours and then immediately submerged in cold water for a period of not less than 1 hour period to testing for modulus of rupture. Testing process was followed by MOR (dry) as previously mentioned but span distance was 24 times of the thickness of specimens.

3.3.7 Microstructure of Fracture Surface of the Particleboard

The fracture surfaces from internal bond testing specimens were observed by a JOEL JSM-6400 scanning electron microscope. The samples were coated with gold before scanning observations.

3.3.8 Staining Method for Bondline Investigation

The performance of the binders was determined by using staining technique, TAPPI standard methods (T 401 om-93). The stained samples were observed by a OLYMPUS SZH10 optical microscope. In doing such study, the samples were stained with Lofton-Merritt stain before optical observations.