CHAPTER 4

EXPERIMENT

4.1 Reagent and Raw Materials

4.1.1 Polyol

The type of polyol used in this study is polyether polyol and its tradename is RAYPOL 3002. RAYPOL 3002 was donated by Thai Polyurethane Industry Co.,Ltd.. The specification of RAYPOL 3002 are presented in Table 4.1

Table 4.1 Specifications of polyether polyol (RAYPOL 3002)

Specifications	Polyether polyol : RAYPOL 3002
Appearance	Clear liquid
Acid number (mg KOH/g)	0.03
Hydroxyl number (mg KOH/g)	54-58
Viscosity at 25 °C (cps.)	500-600
Water content (%)	0.10

4.1.2 Isocyanate

The type of isocyanate used in this study is polymeric MDI and its tradename of polymeric MDI is C-MDI. The specification of C-MDI are precented in Table 4.2. C-MDI was supplied by Thai Polyurethane Industry Co.,Ltd..

Table 4.2 Specifications of polymeric MDI (C-MDI)

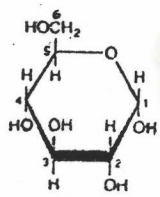
Specifications	Polymeric MDI : C-MDI
Physical state at room temperture	Liquid
Colour	Fawn to dark brown
Odour	None to aromatic at room temperature
Viscosity at 25 °C (cps.)	200
% free NCO	31

4.1.3 Molasses

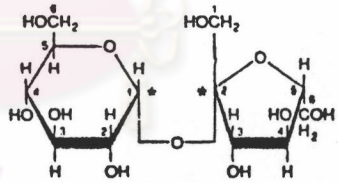
Molasses used in this study was supplied by Thai Sugar Kanjanaburi Factory in Jan.-Mar. 1996. The properties of molasses is presented in Table 4.3

Table 4.3 Characteristic of molasses

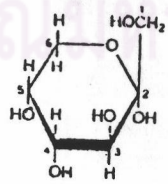
Properties	Molasses
°Brix	86.4
Sucrose (Wt.%)	38
Reducing sugar (Wt.%) (mainly glucose and fructose)	25



Glucose



Sucrose



Fructose

Figure 4.1 Structural of sucrose, glucose and fructose

4.1.4 Other chemical

Other chemical used in this work are as follows:

Dibuthyltin diluarate	serve as	catalyst
Sawdust	serve as	filler
Husk	serve as	filler
Silicone	serve as	mold releasing agent

4.2 Apparatus

4.2.1 Manufacturing Apparatus

Apparatus for producing polyurethane consist of the following units: machanical stirrer, beaker (500 ml.) and aluminum mold.

4.2.3 Testing Apparatus

Universal Testing Machine : HOUNDSFIELD H10KM

4.3 Experiment Procedure

4.3.1 Establishing Production Procedure for unfilled-polyurethane

Figure 4.2 shows the flow diagram of this procedure

(a) Mixing.

Polyther polyol is hydroscopic, and dry nytrogen or low dew point air is recommended for tank padding. The dried polyether polyol and molasses were metered in correct weight in the beaker and mixed until the mixture was uniform. If there was no molasses added (0% molasses), the catalyst (dibutyltin dilaurate, 0.02 % weight) must be added.

(b) Polymerization

The MDI was added to the polyol mixture in the beaker using speed of agitator at 400 rpm. and mixed for 15 minutes. Then the liquid polyurethane was poured onto a aluminum mold which was coated with silicone. Then aluminum mold was closed with flat wood covered with aluminum foil. The resulting product was in block form.

4.3.2 Establishing Production Procedure for polyurethane filled with sawdust

The production procedure for filled polyurethane is essentially the same as that of unfilled polyurethane. The procedure is shown in Figure 4.3

(a) Raw Material Preparation

Sawdust must be sized and dride before added to the polyether polyol. The polyether polyol, molasses and sawdust were metered in correct weight in beaker and mixed until the mixture was uniform.

(b) Polymerization

The MDI was added to the polyol mixture in the beaker using speed of agitator at 500 rpm. and mixed for 15 minutes. Then the polyurethane was poured onto aluminum mold which was coated with silicone. Then aluminum mold was closed with flat wood covered with aluminum foil. The resulting product was in block form.

4.3.3 Establishing Production Procedure for polyurethane filled with husk

Figure 4.4 shows the flow diagram of this procedure

(a) Mixing

Procedure was identical to 4.3.1 (a).

(b) Polymerization

The MDI was added to the polyol in the beaker using speed of agitator at 500 rpm. and mixed for about 2 minutes. Then add husk into a beaker slowly while the polymerization was occouring, and keep on agitating for 15 minutes. The polyurethane was poured onto aluminium mold which was coated with silicone. Then aluminum mold was closed with flat wood covered with aluminum foil. The resulting product was in block form.

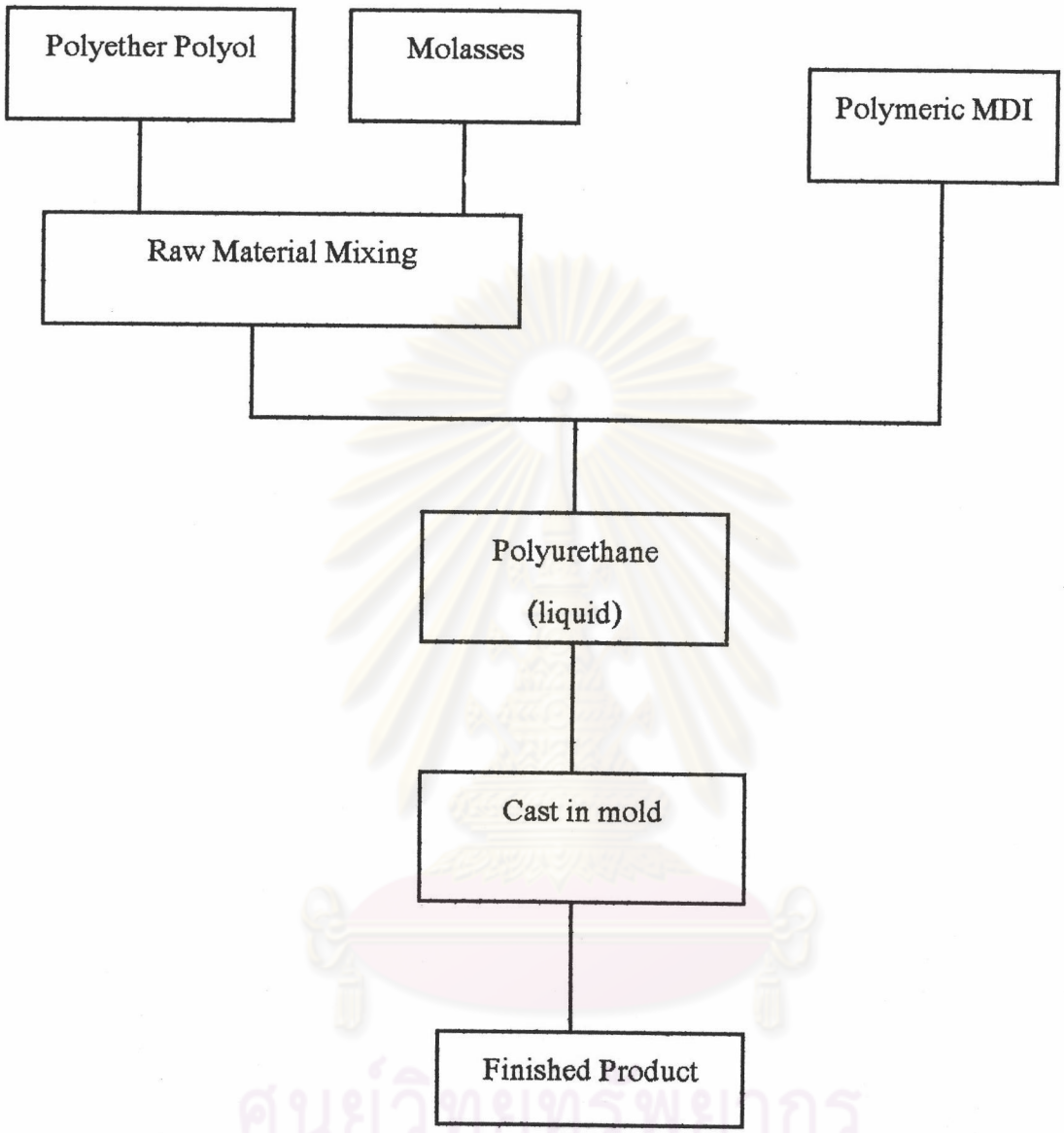


Figure 4.2 Manufacturing one shot production for unfilled polyuretane

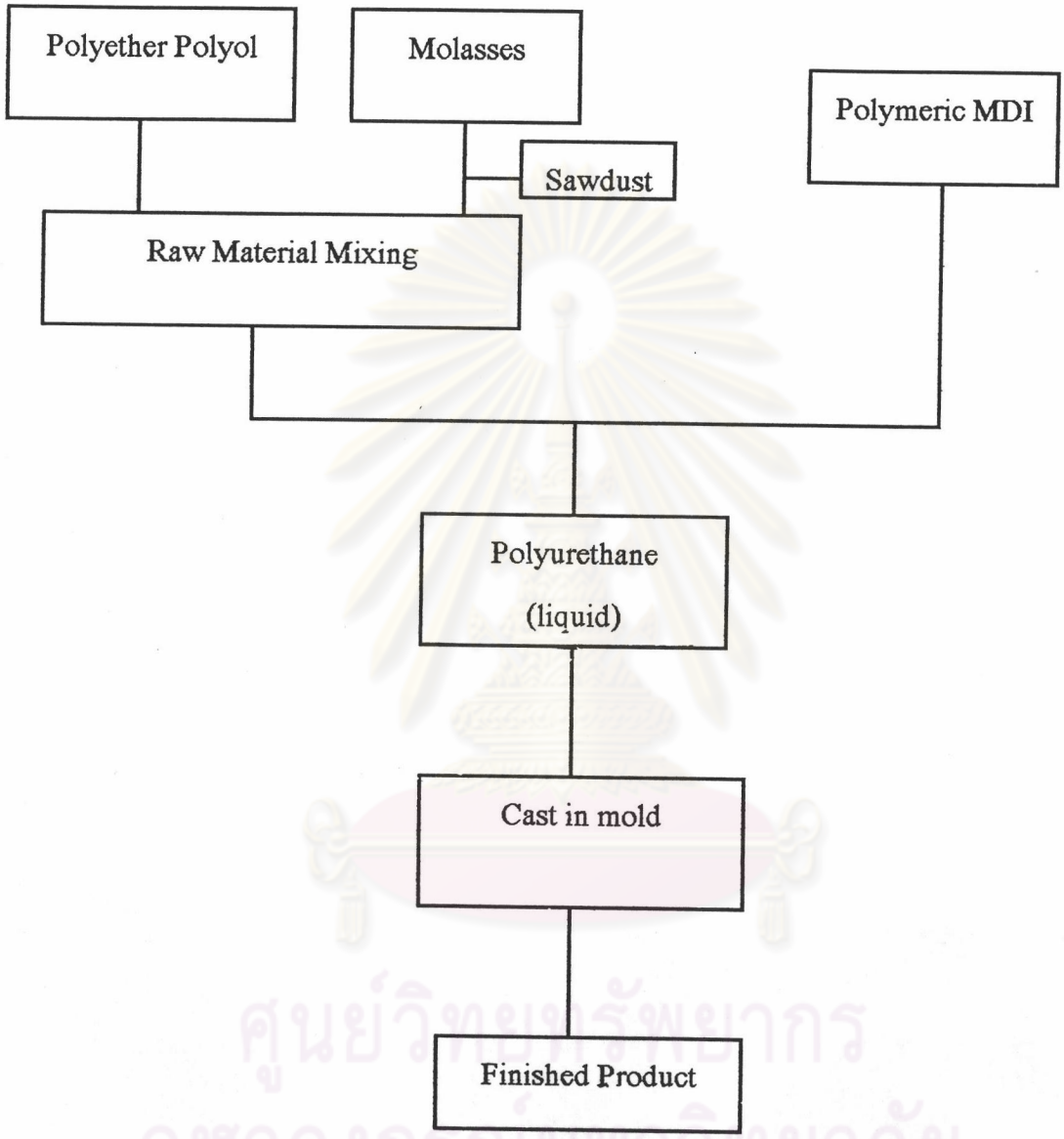


Figure 4.3 Manufacturing one shot production for polyuretane filled with sawdust

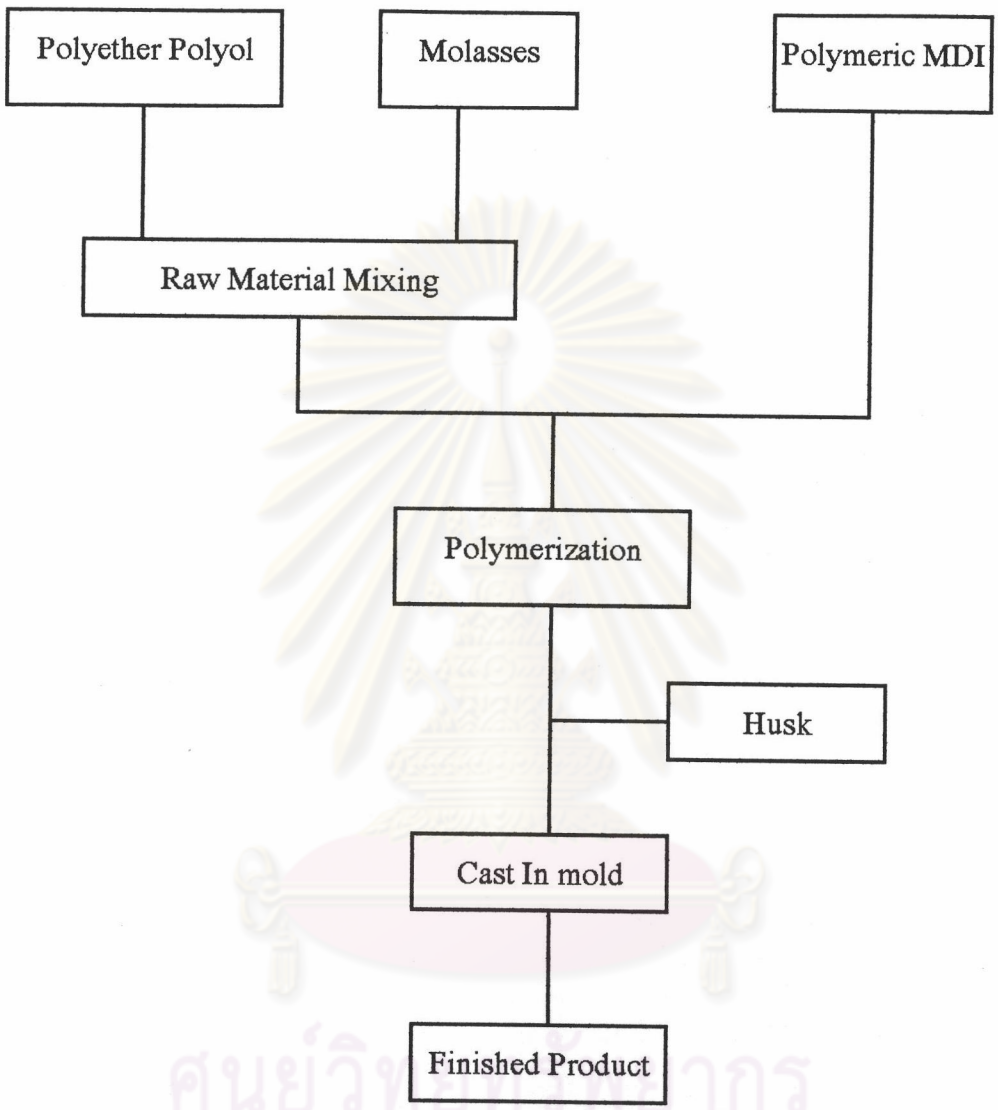


Figure 4.4 Manufacturing one shot production for polyuretane filled with husk

4.3.3 Determination of % Molasses

In this part of study, the NCO/OH ratio was fixed at 1.00. Sample of polyurethane were produced for various % molasses. % Molasses was varied from 0-60 %. Percent of molasses is calculated from the equation below :

$$\% \text{ Molasses} = \frac{\text{weight of molasses (g.)} * 100}{\text{weight of polyol (g.)} + \text{weight of molasses (g.)}}$$

The detail of this part is presented in the Table 4.4

4.3.4 Determination of Quantity of Filler

In this part of study, the NCO/OH ratio was fixed at 1.00 and a fixesd % molasses of 20% and 35% while the weight percentage of fillers, e.g. sawdust and husk, was varied. Percent of filler is calculated from the equation below :

$$\% \text{ Filler} = \frac{\text{weight of filler (g.)} * 100}{[\text{weight of polyol (g.)} + \text{weight of molasses (g.)} + \text{weight of MDI (g.)} + \text{weight of filler (g.)}]}$$

The details of this part are presented in the Table 4.5, 4.6 and 4.7 respectively.

Table 4.4 % Molasses of the investigates polyurethane at NCO/OH ratio 1

% Molasses	Quantity (g.) of Polyol : Molasses : MDI
0	50 : 0 : 14.29
20	50 : 12.5 : 33.57
30	50 : 21.43 : 52.73
35	50 : 26.92 : 64.51
40	50 : 33.33 : 78.46
50	25 : 25 : 57.01
60	37.5 :56.25 : 125.73

Table 4.5 Various weight percentage of filler for filled polyurethane at 20% molasses

% Filler	Quantity (g.) of polyol : Molasses : MDI : Filler
0	50 : 12.5 : 33.57 : 0
9.09	50 : 12.5 : 33.57 : 9.61
16.66	50 : 12.5 : 33.57 : 19.21
20.00	50 : 12.5 : 33.57 : 24.02
23.08	50 : 12.5 : 33.57 : 28.82
28.57	50 : 12.5 : 33.57 : 38.43

Table 4.6 Various weight percentage of filler for filled polyurethane at 35% molasses

% Filler	Quantity (g.) of polyol : Molasses : MDI : Filler
0	50 : 26.92 : 64.51 : 0
3.51	50 : 26.92 : 64.51 : 5.15
9.09	50 : 26.92 : 64.51 : 14.14
16.67	50 : 26.92 : 64.51 : 28.29
20.00	50 : 26.92 : 64.51 : 35.35
23.08	50 : 26.92 : 64.51 : 42.43

Table 4.7 Various weight percentage of filler for filled polyurethane at 50% molasses

% Filler	Quantity (g.) of polyol : Molasses : MDI : Filler
0	25 : 25 : 57.01 : 0
9.09	25 : 25 : 57.01 : 10.70
16.67	25 : 25 : 57.01 : 21.40

4.4 Mechanical Properties Analysis

All samples produced in sections 4.3.1-4.3.3 were subjected to compression testing and drift and set testing to determine the polyurethane product of the most suitable constituents.

4.4.1 Apparent Overall Density (ASTM D1622)

Test specimens were cut from the sample in square prism that volume can be readily calculated. Number of specimens tested is not less than five. The apparent overall density was calculated as follows:

$$D = Ws / V$$

where :

D = density of specimen

Ws = weight of specimen

V = volume of specimen

4.4.2 Compressive Testing (ASTM D1621)

Specimen of polyurethanes for compressive testing are produced by molding in sheet form and cut in square in cross section with 4 $in.^2$ in area, and 1 $in.^2$ in height.

The cross head speed of compressive testing at 2.5 mm./min. was used. Continue until a yield point is reached or until the specimen has been compressed approximately 13% of its original thickness, whichever occurs first. The median of five specimens was taken as the characteristic of the material tested. Compressive strength was calculated by following the step below :

(a) Using a straightedge, extend to the zero load line the steepest straight portion of the load-defletion curve (Point *O* in Figure 4.5a and 4.5b). Measure all distances for deformation calculations fromthis point.

(b) Measure from Point *O* along the zero-load line a distance representing 10% deformation. At that point (Point *M* in Figure 4.5a and 4.5b), draw a vertical line intersecting the load-deflection at Point *P*. If there is no yield point before Point P (as in Figure 4.5b),read the load at Point *P*. If there is a yield point before Point *P*, read the load at the yield point.

(c) Calculate the compressive strength by deviding the load by the initial horizontal cross-sectional area of the specimen.

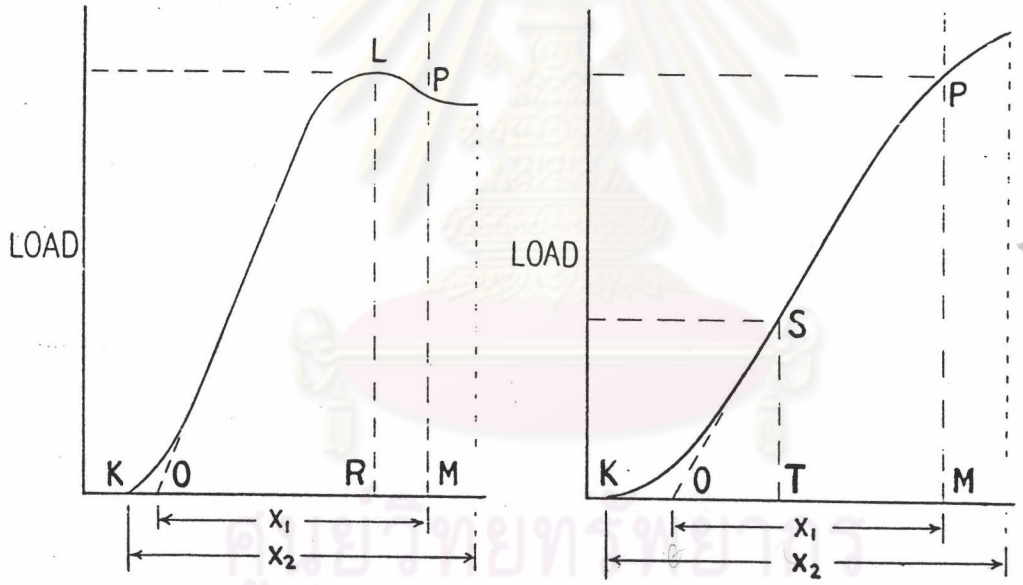


Figure 4.5a Compressive strength

Figure 4.5b Compressive strength

x_1 = 10 % Deformation

x_2 = Deflection (Approximately 13 %)

4.4.3 Drift and Set Testing (ASTM D1372)

% Drift and % Set are the value that show the loss of thickness with time under the continue stress (24 hr.).

Specimen of polyurethanes are cut in a square prism with 2 by 2 in. by 1 in. thick. Each specimen was recorded the " original thickness ", then apply a sufficient load to cause a deflection of specimen thickness of 20 ± 2 % and measured " original loaded thickness ". After 24 hours, the specimen was remeasured thickness while under load as " thickness after drift ". After remove the load for 3 min. the specimen was remeasured " final thickness ". % Drift and % Set were calculated as follows:

Drift, % = 100 * [(original loaded thickness - thickness after drift) / (original loaded thickness)]

Set (24 hr.), % = 100 * [(original thicknesss - final thickness) / (original thickness)]