Chapter IV

Experimentation

4.1 Raw Material

4.1.1 Polyethylene

In this study, the polymer resin used was high density polyethylene (HDPE). The characteristics of the HDPE used are shown in **table 4.1**.

Table 4.1 Characteristics of the HDPE used

Property	Unit	Typical values
Melt flow rate	g/10 min.	5.5
Density	g/cm^3	0.968
Melting point	°C	134
Softening point	°C	124

4.1.2 Pigment

Carbon black and quinacridone violet, both organic pigments, were selected for use in the present study. Their characteristics are shown as follows:

4.1.2.1 Carbon black

The characteristics of the carbon black pigment are shown in **table 4.2**

Table 4.2 Characteristics of the carbon black pigment used

(Printex P : Degussa (Thailand) Ltd.)

Property	Value
Form	Powder
Jetness black value	240
Tinting strength	110
DBP adsorption (ml/100g)	102
Volatiles (%)	0.9
pH value	10
Sieve residue (max%)	0.05
Ash content (%)	0.2
Tapped density (g/cm^3)	0.4
Median particle size (micron)	0.02

4.1.2.2 Quinacridone violet pigment

The characteristics of the quinacridone violet pigment used are shown in **table 4.3**

Table 4.3 Characteristics of the quinacridone violet pigment used

(Quindo Violet RV-6926 : BAYER THAI Co., Ltd.)

Property	Value
Density (g/cm^3)	1.5
Tapped density (g/cm^3)	0.41
Specific surface BET (m^2/g)	78
pH value	6.9
Electric Conductivity (μS/cm.)	<200
Oil adsorption (g/100 g pigment)	43
Median particle size (micron)	0.12

4.2 Equipment

The set of experimental equipment is composed of a temperature-controlled continuous kneader, an accurate feeder, a press roller, and a roller-temperature controller (see their specifications in the Appendices). A schematic diagram of the continuous kneading system used in the present study is shown in **figure 4.1**

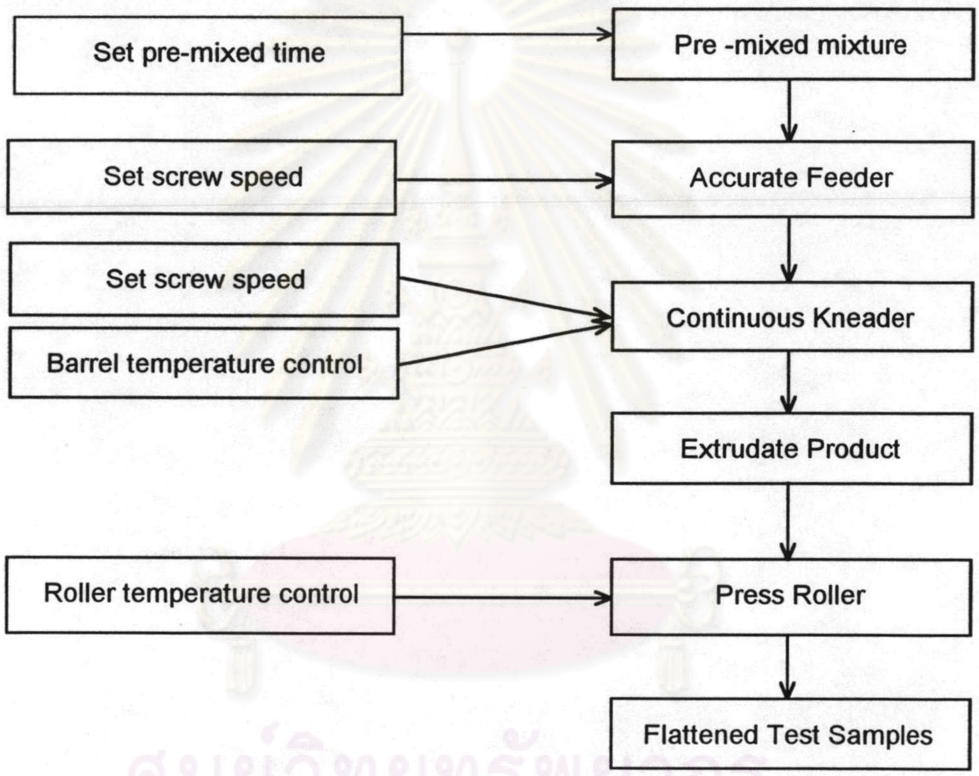

Figure. 4.1 Schematic diagram of the continuous kneading system

4.2.1 Accurate feeder

It can handle a wide variety of dry materials including fine powder, granules, chips, pellets, caustics, plastics, food, and pharmaceutical powders. It achieves high rates of accuracy by using a flexible hopper that contorts continuously during operation, preventing any bridging of materials inside the hopper, which has been designed to keep all powders flowing downward uniformly with no dead areas, with rounded hopper corners to promote material flow, and with no cracks or seams that would interrupt material flow. The amplitude and frequency of the hopper flexing mechanism are adjustable to gain optimum performance for each material. Metering accuracies generally range between $\pm$ 0.5 to 2 percent for most materials (see **figure 4.2**).

Figure 4.2 Accurate feeder

4.2.2 Continuous kneader

It consists of the barrel, screws, paddles, shafts and driving unit (see **figure 4.3**).

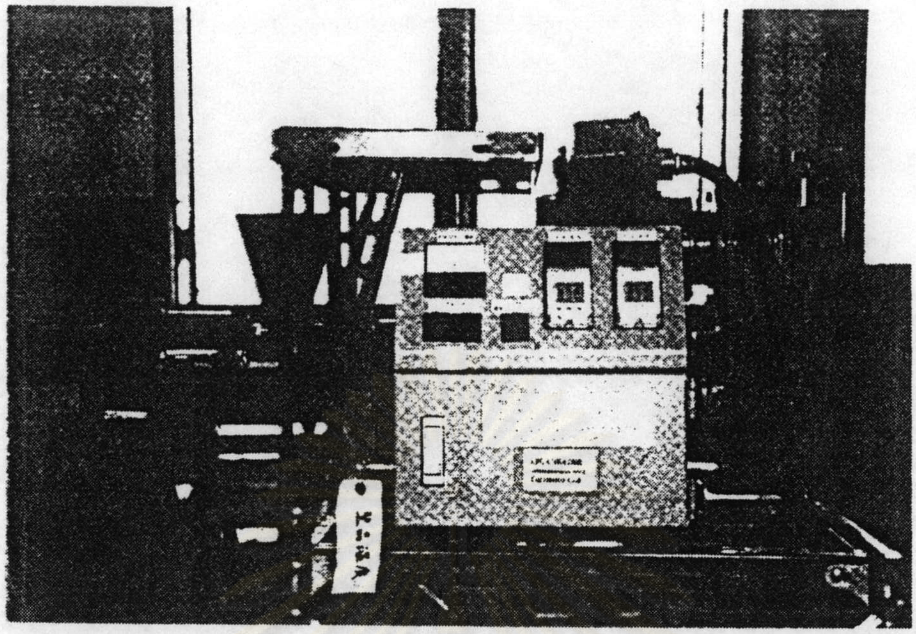

Figure 4.3 Continuous kneader

The standard type of barrel is horizontal closed type. The paddles of one shaft assembly maintain close clearance with the second assembly as well as with the walls of the barrel. This not only assures a more efficient mixing, but also provides a self-cleaning action for the paddles. The rotation of the two shaft assemblies provides a continual variation in volume between the paddles and the barrel in any given region of the entire unit. At the same time, the action of the shafts also creates an alternating compression and suction of materials. This, in effect, moves materials forward and backward, assuring a continual mixing and re-mixing within any given section. The raw material fed from one end of the barrel is sent by the screw into the kneading zone. And then the material is continuously discharged from the lower side, side port or front side of the other end of the barrel.

The paddles and screws are mounted on two sets of horizontal parallel shafts rotating at the same speed and in the same direction (co-rotating). They consist of five sections as shown in **figure 4.4**, Feed Screw (FS) for feed, Helical Paddle (H) for kneading and feed, Flat Screw (F) for kneading, Reverse Helical Paddle (RH) for kneading and reversing, and Reverse Screw (RS) for reversing and feed.

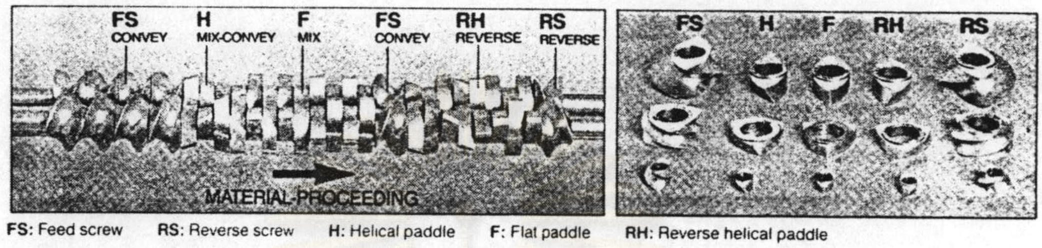

Figure 4.4 Various types of paddles and their arrangement

4.2.3 Press roller

It consists of two parallel rollers rotating in opposite directions and placed close together with the roll axes lying on a horizontal plane, so that a relatively small space or nip exists between the cylindrical surfaces. Material reaching the nip is deformed by friction forces between the material and the rollers and is forced to flow through the nip in the direction of the roll motion. Usually, by adjusting the individual roller temperature, the material could be made to adhere to either of the rolls as a relatively thin sheet. The rollers are either heated or cooled by a heating or cooling medium introduced into their respective hollow cores. They are usually rotated at the same speed to facilitate the formation of a sheet (see **figure 4.5**).

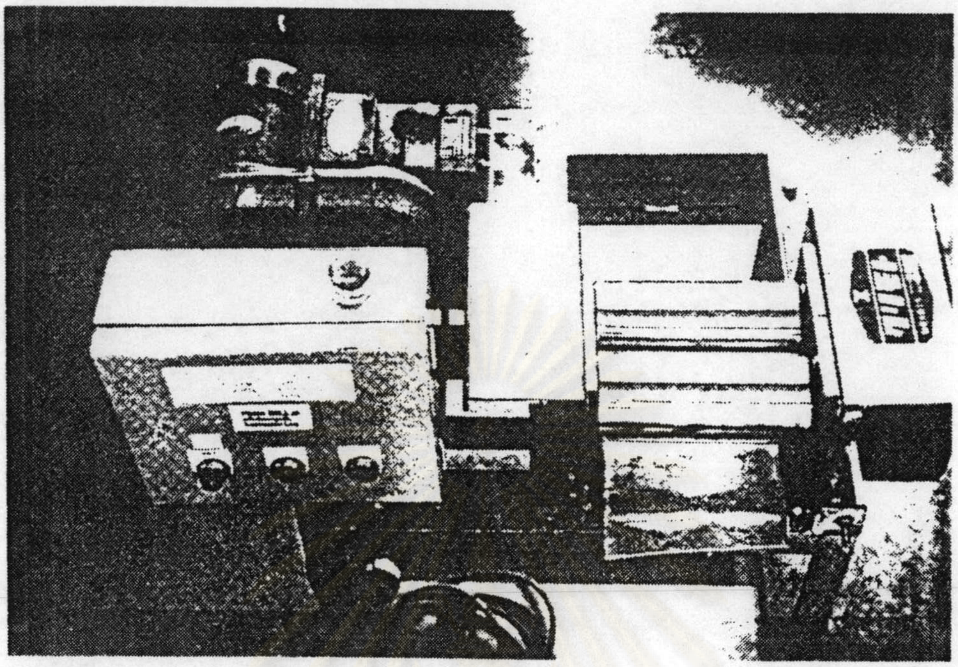

Figure 4.5 Press roller

4.2.4 Roller temperature controller

This controller is provided with an advanced temperature control function based on the computer-integrated manufacturing (CIM) system (see **figure 4.6**). The CIM system ensures very precise temperature control for either of the rollers, or the heat transfer medium, responding immediately to any temperature deviation from the set-point.

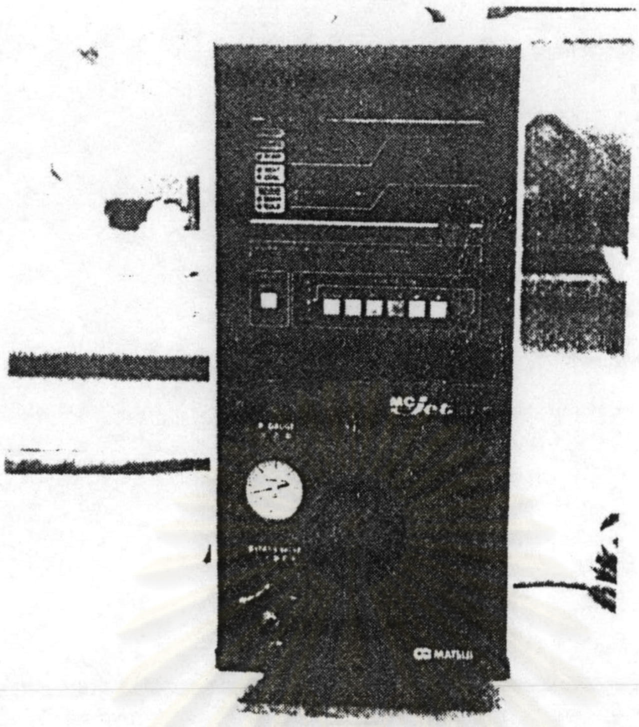

Figure 4.6 Roller temperature controller

A complete setup of the continuous kneading system in the present study is shown in **figure 4.7**.

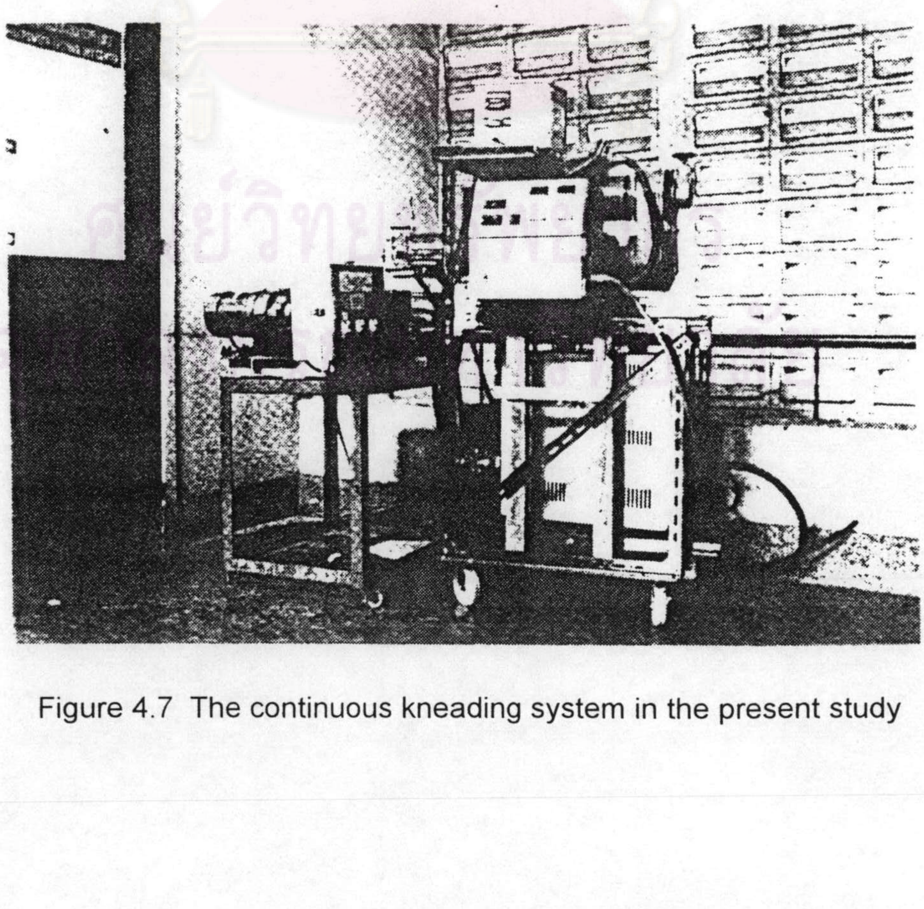

Figure 4.7 The continuous kneading system in the present study

4.3 Experimental conditions

In the present work, four parameters that could affect the dispersibility of individual pigments in polyethylene were investigated. They were the pre-mixed time, the kneading temperature, the rotational speed of the kneader and the feed rate of the accurate feeder. The experimental conditions were as follows:

1) Pre-mixed time (min.) : 10,20,30

2) Kneading temperature (^{o}C) : 200,220,240

3) Rotational speed of kneader (potentiometer setting) : 162,243,324

(to convert to rpm, see Appendix)

4) Feed rate (potentiometer setting) : 500,700,900

(convert to g/min., see Appendix)

The conditions that remained at constant values were as follows:

1) Ratio of HDPE and pigment (wt./wt.)	25:1
2) Rotational speed of press rollers (rpm)	2
3) Press roller temperatures (^{o}C)	70

4.4 Experimental procedure

Part 1) Sample preparation and dispersibility analysis

1. Pre-mix HDPE and pigment at a constant ratio (25:1 by weight) using a V blender (V-5 type, Tokuju Kosakusho Co.,Ltd.) for the set time duration.

2. Set the rotational speeds and temperatures of the press rollers at a constant value of 2 rpm and 70 ^{o}C, respectively.

3. Set the flow rate of the accurate feeder and the rotational speed of the kneader at the desired values.

4. Set the kneading temperature at the desired condition and wait until it reaches steady-state condition.

5. Start to feed the pre-mixed material to the material to the kneading vessel inlet at the preset rate. (Since the capacity of the accurate feeder is much smaller than that of the kneader, the actual extrusion rate is controlled by the accurate feeder in this work).

6. Discharge the extrudate from the kneader onto the press rollers to obtain a long thin plate.

7. After each experimental run has been finished, feed only the HDPE resin to the kneading vessel to purge out any remaining material before starting a new experimental run.

8. To evaluate the resulting dispersibility, take microphotographs using a scanning electron microscope (SEM) at magnifying power 7500x, 20 KeV in the case of quinacridone violet pigment and 15000x, 20 KeV in the other case of carbon black.

9. Analyze the SEM photographs with the aid of the fractal analysis method. The following number of division to similarity (n) has been used: 2, 4, 5, 8, 10, 20, 40, and 80. In other words, the similarity ratios (r) are 1/2, 1/4, 1/5, 1/8, 1/10, 1/20, 1/40 and 1/80, respectively.

10. Count the number of subsections, N(r), that contain at least some small part of a pigment particle or agglomerate.

11. Plot N(r) versus r on the log-log scale to determine the fractal dimension (D).

Part 2) Tensile Testing

Since the thickness of a sample piece is greater than 1.0 mm. (0.04 in), tensile properties determination was based on ASTM Standards D882-91, using Test Method A. The long flat specimens were cut into drum-bell shape according to the dimensions specified in ASTM D638.