CHAPTER IV

EXPERIMENTAL EQUIPMENT AND PROCEDURE

4.1 Raw material

4.1.1 Polystyrene

The characteristics of the polystyrene used are shown in Table 4.1.

Table 4.1 Characteristics of the polystyrene used

Property	Value
Melt flow rate (g/10 min)	2.4
Specific gravity	1.04
Softening point (°C)	107

4.1.2 Pigment

In the present study, iron oxide and carbon black were used.

1) Iron oxide

The characteristics of the iron oxide pigments used are shown inTable 4.2.

Table 4.2 Characteristics of the iron oxide pigments used

(Bayferrox Rot ; Red 222 : BAYER THAI Co.,Ltd.)

Property	Value
Median particle size (μm)	0.2
Particle shape	spherical
Loss on heating at 1000 °C,1/2 Hr (max. %)	1
Sieve residue on 0.045 mm.-mesh (max. %)	0.1
Tapped apparent density (g/cm^3)	1.4-1.8
Density (CA./approx. g/cm^3)	5.24

2) Carbon black

The characteristics of the carbon black pigments used are shown inTable 4.3.

Table 4.3 Characteristics of the carbon black pigments used

(Lamp Black 101 : Degussa (Thailand) Ltd.)

Property	Value
Form	Powder
Jetness black value	209
Tinting strength	29
BDP adsorption (ml/100g)	117
% volatiles	1.0
pH value	7.0
Sieve residue (max %)	0.05
Ash content (max %)	0.05
Tapped density (g/cm^3)	0.26
Median particle size (nm)	95

4.2 Experimental equipment

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

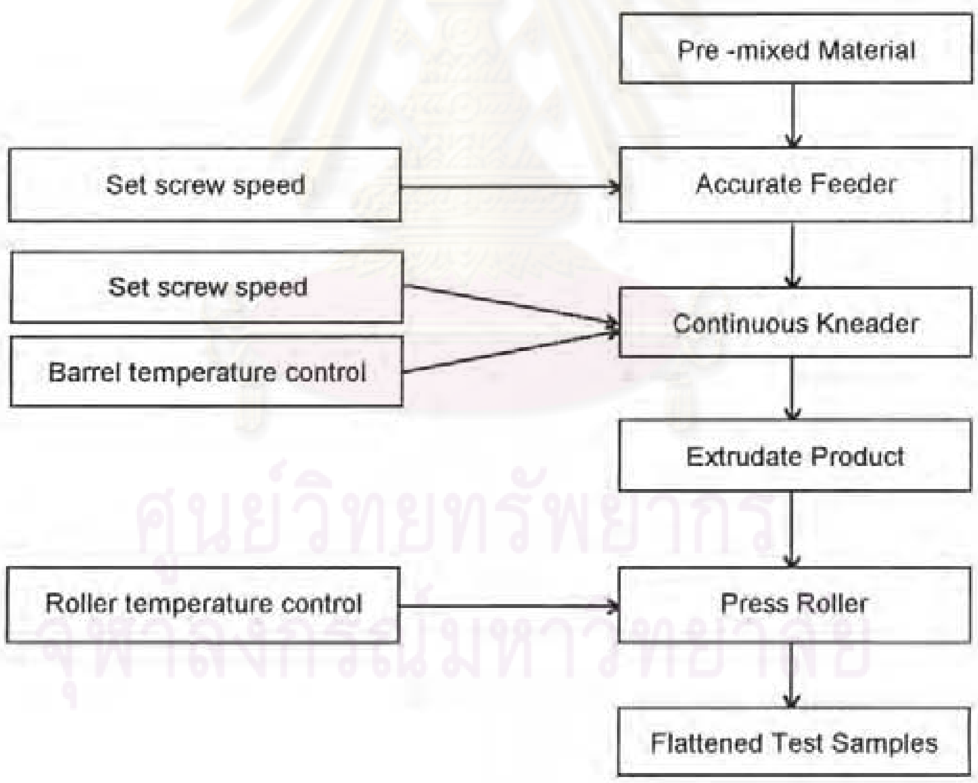

Figure. 4.1 Schematic diagram of the continuous kneading system

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 throughout 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 ± 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).

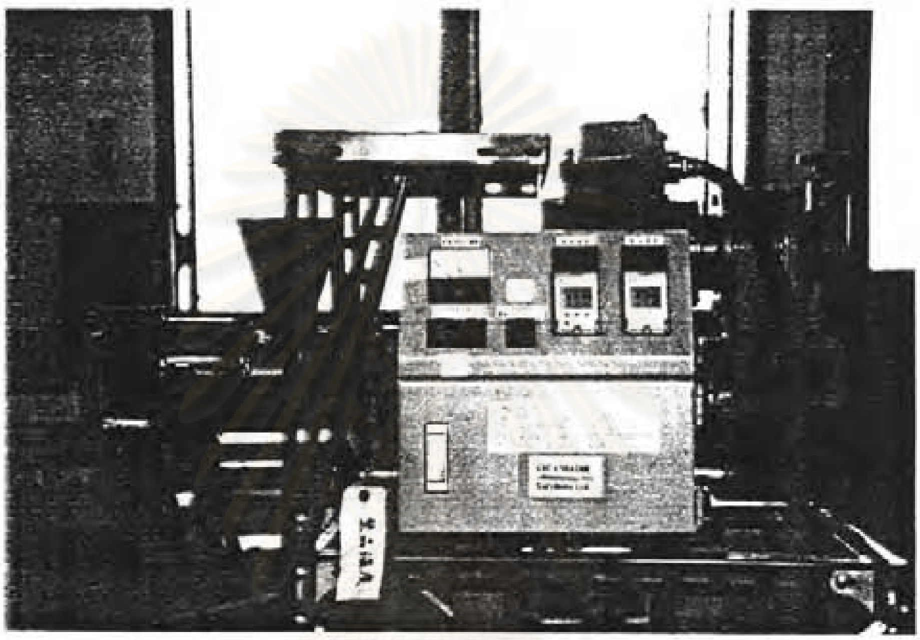

Figure 4.3 Continuous kneader

The standard type of ballel 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 area 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 maerials forward and backward, assuring a continual mixing and re-mixing within any given section. The raw material fed from one end of barrel is sent by the screw into the kneading zone. And then the material is continuously discharged from the lower side, side part or front side of the other end of barrel.

The paddles and screws are mounted on two set of horizontal parallel shafts rotating at the same speed and in the same direction (co-rotating). They consiste of five sections as shown in Figure 4.4, Feed Scew (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.

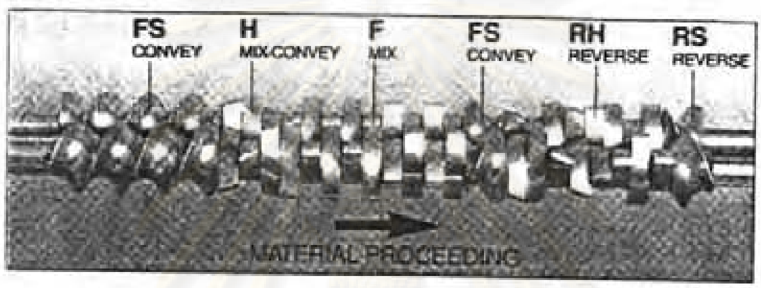

Figure 4.4 Various types of paddle and their arrangement

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 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).

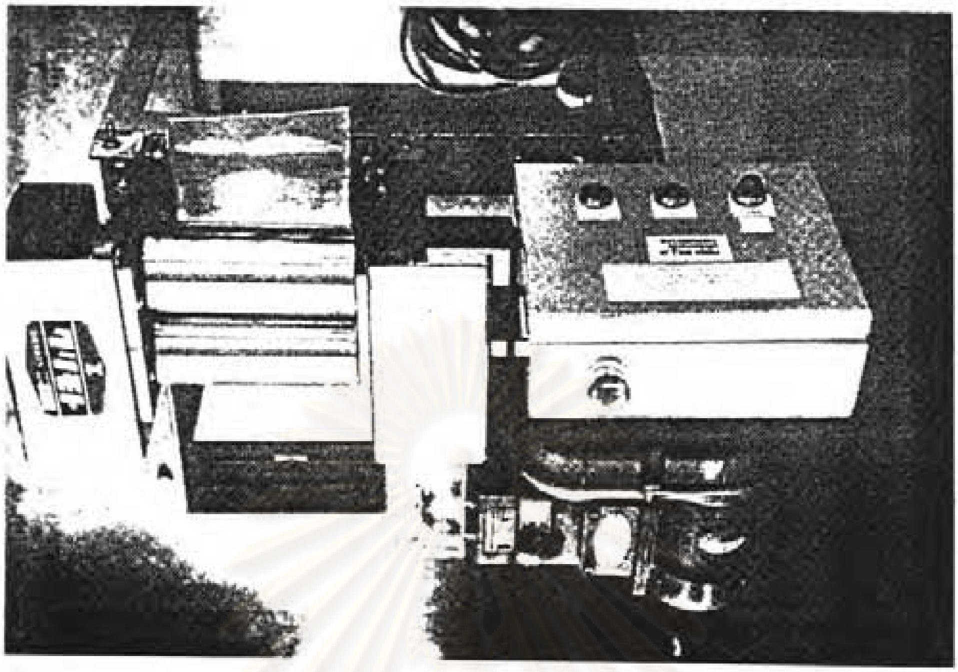

Figure 4.5 Press roller

4.2.4 Roller Temperature Controller

Figure 4.6 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 control for either of the rollers, or the medium temperature, responding immediately to any temperature deviation from the set-point.

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

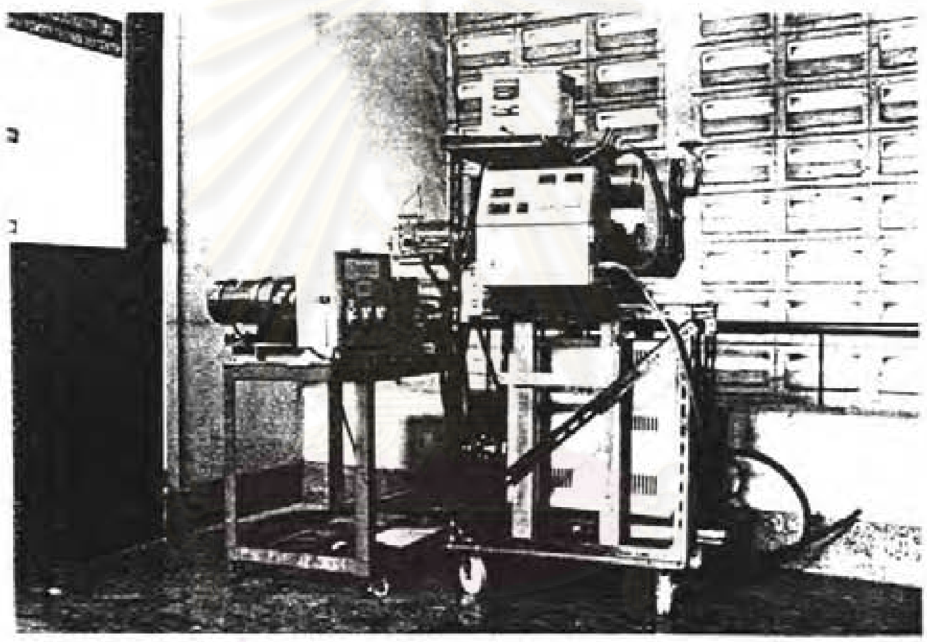

Figure 4.7 The continuous kneading system in the present study

4.3 Experimental conditions

In this work, three major parameters that could effect the dispersibility of pigments in polystyrene were investigated. They were the kneading temperature and rotational speed of the kneader and the feed rate of the accurate feeder. The parameters were varied as follows.

1) Kneading temperature (°C) :	170, 190, 210
2) Rotational speed of kneader (potentiomeer setting) :	81, 162, 324
3) Feed rate (potentiometer seting) :	100, 500, 900

The conditions that remain fixed at constant values are as follows:

1) Ratio of polystyrene and pigment (wt./wt.)	25:1
2) Pre-mixing time (min.)	10
3) Rotational speed of press roller (rpm)	2
4) Press roller temperature (°C)	70

4.4 Experimental procedure

The experimental procedure is as follows :

1. Premix polystyrene and pigment at a constant ratio (25:1 by weight) by using a V blender (V-5 type, Tokuju Kosakusho Co.,Ltd.) for 10 minutes.

2. Set the rotational speed and temperature of the press roller at a constant value of 2 rpm and 70 °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 feed hopper of the accurate feeder, which conveys the material to the kneading vessel inlet at the present 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 study)

6. After steady-state has been attained, direct the extrudate from the kneader onto the press roller to obtain a long thin plate.

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

8. To evaluate the resulting dispersibility, take microphotographs with the aid of a scanning electron microscope (SEM) at magnifying power 5000X, 20 KeV in the case of iron oxides and 15000x, 20 KeV in the other case of carbon black.

9. Randomly select 4-5 microphotographs for each set experimental conditions and analyze the SEM photographs using the fractal analysis method. Here 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 parts , 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).