CHAPTER II
EXPERIMENT

2.1 Materials

Furnace carbon black of high structure and low structure in the trade mark of Printex 30 and Printex 300 respectively. They are products of Degussa. The physical specifications of them are shown in Table 1.

Table 1 Physical specifications of carbon blacks

furnace carbon black	DBPA	particle size (nm)	surface area B.E.T. (m^2/g)	pH value
Printex 30	106	27	80	10
Printex 300	65	27	80	9.5

Polydimethysiloxane (PDMS) ,Fluka silicone oil #85415, has molecular weight 17,000 , viscosity 370-390 mPa. s. and density 0.973 g/cm^3. The viscosity was rechecked for exact value by using viscometer "Brookfield Model DV-III". The giving value is 356 mPa.s. Surface tension (γ_{lv}) is measured by using tensiometer and the obtained value is 19.8 mN/m.

1,2 Polybutadiene (PB),Ricon 156, kindly supported by Ricon Resins INC., has molecular weight 1,400 , viscosity 1600 ± 600 mPa.s. and density 0.89 g/cm^3. It has surface tension(γ_{lv}) of 33.4 mN/m which is measured by using tensiometer.

Maltose resin ,the product of Thailand, has viscosity 600,000 cp. s. and density 1.441 g/cm^3.

2.2 Apparatus

Apparatus used in this research are as following:

- Pycnometer
- Calibrated 100 ml cylinder with no lip
- Cylindrical container for sedimentation experiment
- Viscometer
- Tensiometer
- Rolling machine
- Vibration sieves
- Metal mold (Fig. 3)
- Hydraulic compression machine
- Stop watch
- Optical microscopy
- Centrifuge
- Syringe

2.3 Carbon black blending

Carbon black blends are prepared by mixing carbon black high structure(Printex 30) with carbon black low structure(Printex 300). They were prepared in six blends as following:

1. 100%H CB composes of pure high structure carbon black.
2. 80%H CB is the mixture of 80% high structure with 20% low structure of carbon black.
3. 60%H CB is the mixture of 60% high structure with 40% low structure of carbon black.

4. 40%H CB is the mixture of 40% high structure with 60% low structure of carbon black.
5. 20%H CB is the mixture of 20% high structure with 80% low structure of carbon black.
6. 100%L CB composes of pure low structure carbon black.

The mixing process is performed in a glass bottle. Carbon black blends are put into glass bottles which are put on the rolling machine. The bottles are rolled at least 2 hours. The powder black in the bottles tends to stick at the wall during rolling. Therefore stopping the rolling every 20 minutes is necessary to unstick the black from the wall.

2.4 Measurement of pour density and tap density

Carbon black blend is poured in a calibrated 100 ml cylinder(no lip). The excess black is wiped out by stainless blade. The weight of carbon black in cylinder is recorded. The cylinder that is filled with carbon black is tapped on bench for 300 times with the same average force in each tapping. The volume occupied by the black is recorded.

2.5 Agglomerates preparation

The mold shown in Fig. 3 is used for preparing carbon black compacts which later are broken to agglomerates. The procedure of agglomerates preparation is described below.

1. Carbon black blend of 7 g. is placed in the mold .
2. The mold is compressed until compact reaches the height of 2.5 cm. by using a hydraulic compression machine.
3. The mold is opened and compact is removed .

4. In order to get series of agglomerate densities , the height of compacts is varied from 2.5 to 1.25 cm. in each blend.
5. The compact is broken to fragments by spatula.
6. Fragments are placed in a sieve shaker for a shaking time about 2-3 hours.
7. Nearly spherical agglomerates(diameter 2-2.8mm.) are collected from the sieve (ϕ 2mm) and kept in a desiccator for later measuring of sedimentation velocities and agglomerate densities.

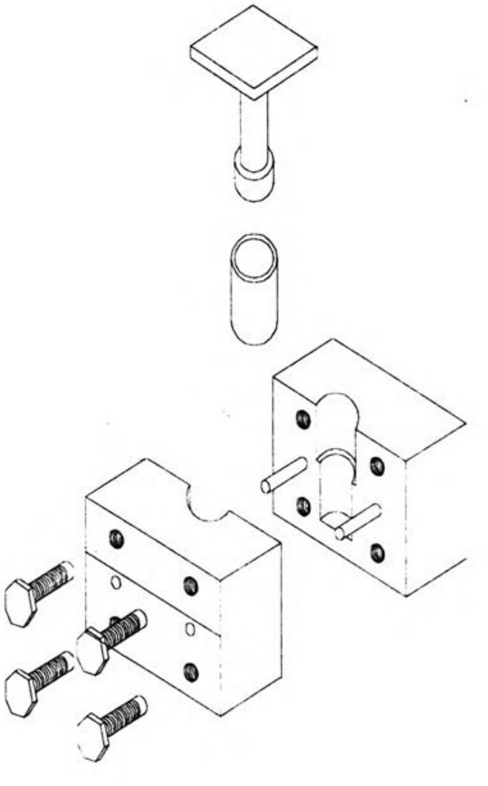

Fig. 3 Schematic diagram of the mold which is used to prepare agglomerates.

2.6 Measurement of agglomerate densities

The volume of the pycnometer is 0.4146 ml. Maltose resin is the viscous fluid that has viscosity of 600,000 cp.s and density of 1.441 g/cm^3 and is used in this measurement. The following shows the steps of measurement.

1. Maltose is poured in plastic syringe and is degassed by centrifuging at 3,000 RPM. for 20 min.
2. Syringe contained fluid is left to stand still at least one day for completely eliminating air bubbles before use.
3. Pycnometer is weighed then some liquid is injected into the bottom.
4. A few agglomerates (~0.01g) are added and the pycnometer is weighed again.
5. The liquid is injected to fill out pycnometer . Generating air bubbles must be avoided. Finally pycnometer is weighed again.

2.7 Sedimentation experiment

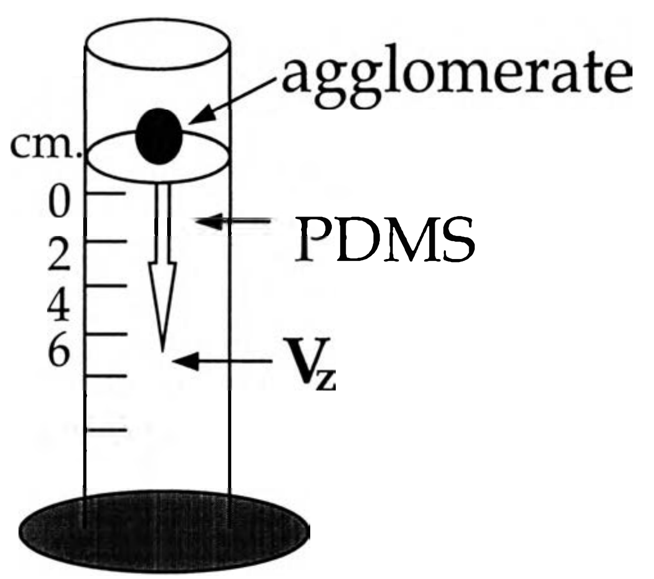

Fig. 4 schematic diagram of the sedimentation experiment.

Sedimentation of agglomerates in PDMS is performed in a cylindrical container ϕ 3 cm. ,height 20 cm. Agglomerate sizes are ranging from 2.0-2.8 mm. in diameter. Fig. 4 shows the schematic diagram of the sedimentation experiment and the procedure are shown below.

1. Cylinder is filled with liquid (silicon oil #85415) and has a mark of length scale on it.
2. Agglomerate diameter is measured by using an optical microscope at the magnification of 1.5x.
3. The agglomerate is placed in PDMS. We wait until agglomerate is totally immersed into matrix and is starting to sediment.
4. The sedimentation distance and the times consumed at several points are recorded.
5. The sediment agglomerate is kept in PDMS for 3 days in order to get the total matrix penetrated agglomerates.
6. The total matrix penetrated agglomerate is taken out for measuring the ultimate velocity by performing the sedimentation experiment again.

2.8 Study of interfacial properties of carbon black -PDMS and carbon black-PB

Carbon black agglomerates of six blends that have nearly the same density (~0.35 g/cm^3) are used in this experiment. The sedimentation of agglomerates in the matrix of PDMS as well as in the matrix of PB are undertaken. The experiment method is the same as in section 2.6.

2.9 Calculation

2.9.1 Pour density (ρ_{pour}) and void fraction

$$\rho_{pour} = \frac{\text{weight of carbon black in cylinder}}{\text{volume of cylinder, 100ml,}}$$

$$\text{void fraction} = 1 - \frac{\rho_{pour}}{\text{density of carbon black solid particle, } 1.86\text{g/cm}^3,.}$$

2.9.2 Tap density (ρ_{tap}) and specific volume

$$\rho_{tap} = \frac{\text{weight of carbon black in cylinder}}{\text{volume of carbon black after tapping 300 times}}$$

$$\text{specific volume} = \frac{1}{\rho_{ap}}$$

2.9.3 Agglomerate density (ρ_{aggl})

$$\rho_{aggl} = \frac{W_{aggl}}{V_{pyc} - \left(\frac{W_{mal}}{\rho_{mal}}\right)}$$

where : w_{aggl} is the weight of agglomerates in pycnometer.

v_{pyc} is the volume of pycnometer equal 0.4146 ml.

ρ_{mal} is density of maltose equal 1.441 g/cm^3.

w_{mal} is the total weight of maltose filled in pycnometer.

2.9.4 Calculation of t_{max} and R_h

t_{max} is taken from the slope of the straight line of the plot between "$2(R/a)^3 - 3(R/a)^2 + 1$" against the observed times. (R/a) is the degree of penetration of fluid in agglomerates which is calculated using the following equations.

$$\left(\frac{R}{a}\right)=\left(\frac{v_z - v_z^{\infty}}{v_z^{0} - v_z^{\infty}}\right)^{1/3}$$

where : v_z is the agglomerate sedimentation velocity at observed time.

v_z^{∞} is the ultimate velocity of the agglomerate.

v_z^0 is the agglomerate initial velocity and is computed using the following equation.

$$v_z^{0} = \frac{2a^2 g}{9\mu_f}\left(\rho_s - \rho_f\right)$$

where : ρ_s is agglomerate density .

ρ_f is density of fluid (for PDMS =0.973 g/cm^3,PB=0.89 g/cm^3)

μ_f is viscosity of fluid.(for PDMS 356 mPa.s, PB =1,600mPa.s).

a is agglomerate diameter.

g is the acceleration of gravity(9.81 m/s^2)

R_h is calculated by using the following equation.

$$R_h = \frac{25a^2\mu}{36\, t_{max}\, \varepsilon\, \gamma_{lv} \cos\theta}$$

where γ_{lv} is surface tension of liquid (for PDMS =19.8 mN/m, PB =33.4 mN/m).

cos θ is contact angle between carbon black and liquid taken as equal 1 in both PDMS and PB.

$\varepsilon = 1-(\rho_{aggl} / 1.86)$.