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CHAPTER 3

APPARATUS AND EXPERIMENT METHODS

It is apparent that rheological behavior depends strongly on the rank and composition of the coal, its particle size, temperature, and the type of oil used. It is also shown that an important property of COM are the formation of networks by the aggregation of coal particles and that in a desirable COM, the aggregate binding is strong enough to yield readily to flow while simultaneously retainly homogeneity of dispersion. The addition of chemical additive improves COM stabiling. The data of rheological properties and stability for COM behavior are explained.

3.1 Materials

3.1.1 As experimental materials bituminous coal, subbituminous coal, heavy fuel oil and light fuel oil used are shown in the Table 3.1 and 3.2.

Table 3.1 Types of coal

Location	Name of Deposit	Rank
LAMPANG	Mae Moh	Subbituminous C
LAMPHUN	Ban Pu	Subbituminous B
PETCHABURI	Nong Ya Plong	High volatile bituminous C

Table 3.2 Types of fuel oil

ESSO Fuel oil	Viscosity @ 50 °C, cSt.
Light fuel oil (NO.1)	80
Heavy fuel oil (NO. 6)	280

3.1.2 Fourteen additives composed of anionic, cationic and nonionic additives were used for stability test. Their formulae are shown in Table 3.3.

3.2 Apparatus

3.2.1 Coal preparation apparatus

- A hammer mill was used to crush lump coal into coarse particles.
- A cross beater mill was used to crush coarse coal particles into fine coal particles.
- Sieve screens : sieve no. 60, 100, 150, 170 and 200 mesh were used to screen five different particle size distributions; -75 microns, 75-90 microns, 90-106 microns, 106-150 microns and -250 microns.

3.2.2 Coal, fuel oil and COM analysis apparatus

- An oxygen bomb calorimeter was used to measure heating value of sample.
- Pour point apparatus was used to measure the temperature at which the oil or COM cease to flow.

3.2.3 Rheological property apparatus

- A mechanical stirrer was used to mix desirable COM samples of coal and fuel oil to form an homogeneous mixture to be

Table 3.3 Types of additives

Additive	Composition or formula
	Anionics
Span 60	Sorbitan monostearate
Span 40	Sorbitan monopalmitate
Arlacel 83	Sorbitan sesquioleate
Arlacel 20	Sorbitan monolaurate
	Cationics
Ethomeen C-20	$CH_3(CH_2)_{19}N(C_2H_{40})_mH(C_2H_{40})_nH$
Ethomeen C-15	$CH_3(CH_2)_{14}N(C_2H_{40})_mH(C_2H_{40})_nH$
Triton X-400	Benzylalkonium chloride
	Nonionics
Brij 78	Polyoxyethylene (20) oleyl ether
Brij 76	Polyoxyethylene (10) stearyl ether
Brij 56	Polyoxyethylene (10) cetyl ether
Surfonic N-95	$CH_3(CH_2)_8C_6H_{40}(C_2H_{40})_{9.8}H$
Igepal CO-610	Nonylphenoxypoly(ethyleneoxy)ethanol
Tween 40	Polyoxyethylene (20) sorbitan monopalmitate
Tween 20	Polysorbate (20) polyoxyethylene (20) sorbitan monolaurate

used in the experiments.

- A water bath was used to control and maintain the system at the temperature needed to obtain rheological data.

- A Brookfield viscometer (LV Model) was used to measure rheological data, e.g. viscosity etc. as shown in Figure 3.1.

3.2.4 Stability apparatus

- An oven was used to control and maintain the apparatus at the desired temperature for sedimentation test.

- A sedimentation column was filled with sample used in sedimentation test as shown in Figure 3.2.

- A 10 ml pycnometer was used to measure the density of the sample.

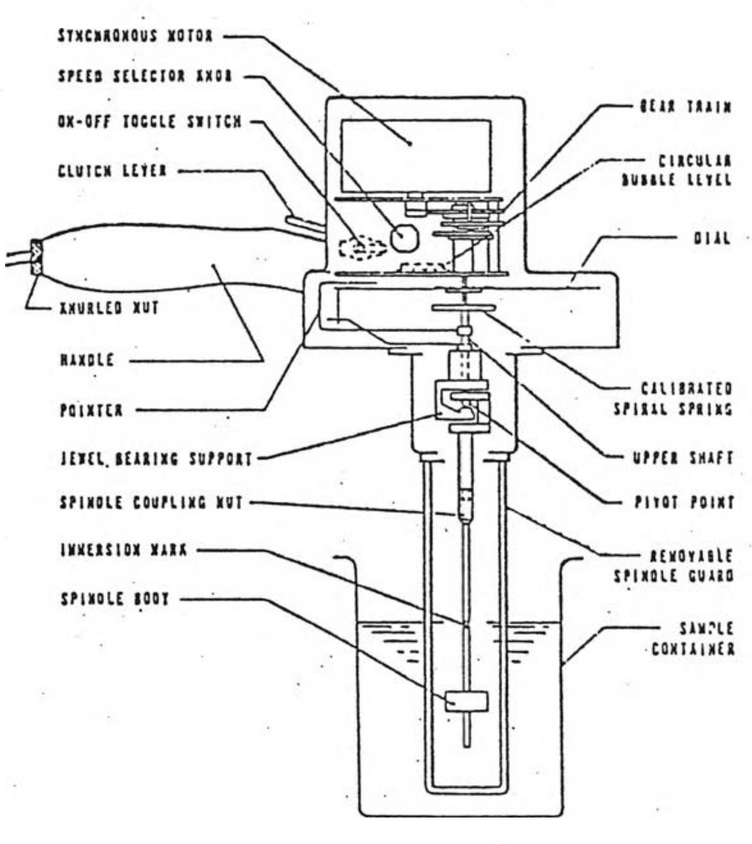
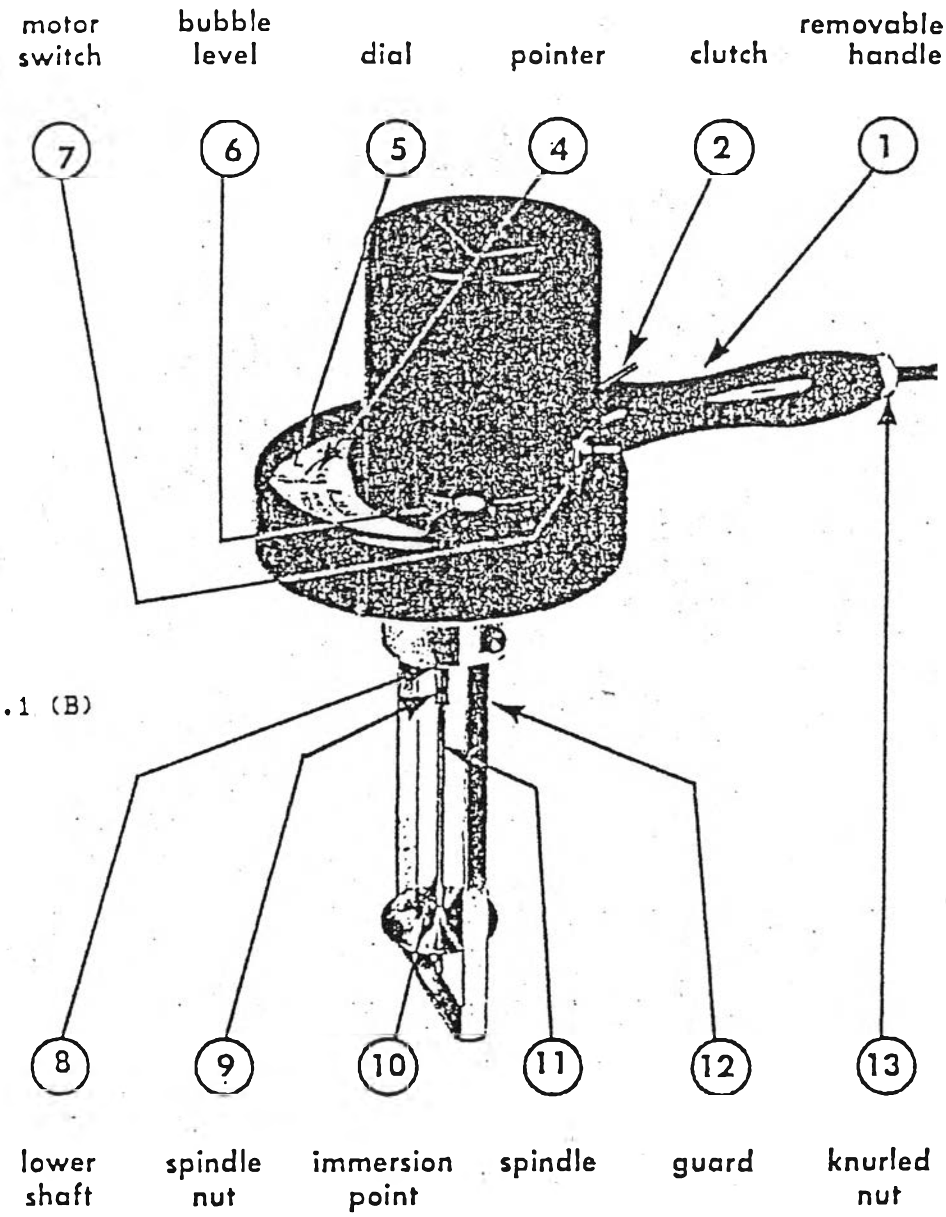
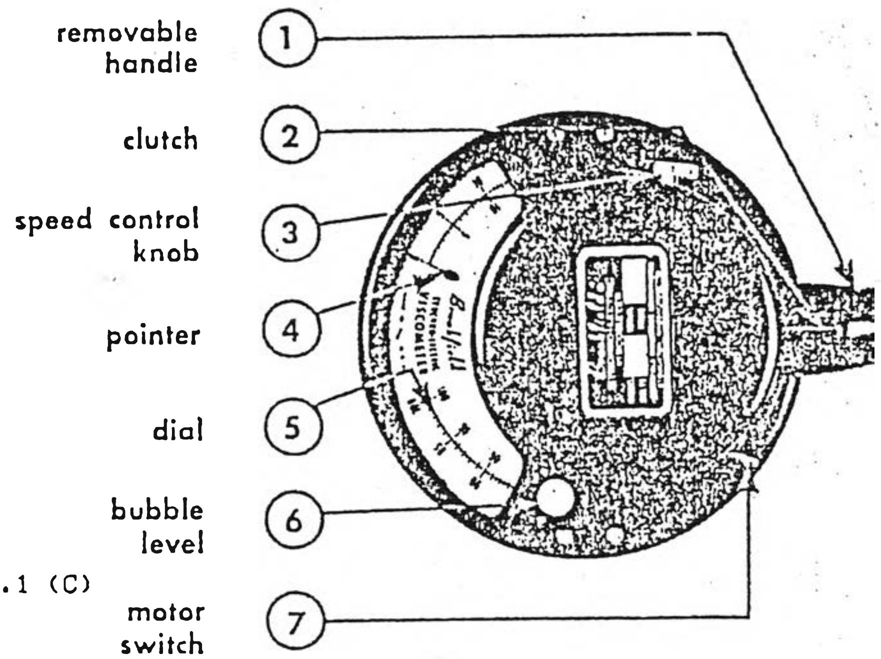


Figure 3.1 (A) SCHEMATIC DRAWING OF THE BROOKFIELD SYNCHRO-LECTRIC VISCOMETER



3.1 (B)



3.1 (C)

Figure 3.1 (B), (C) Brookfield Synchro-Lectric Viscometer

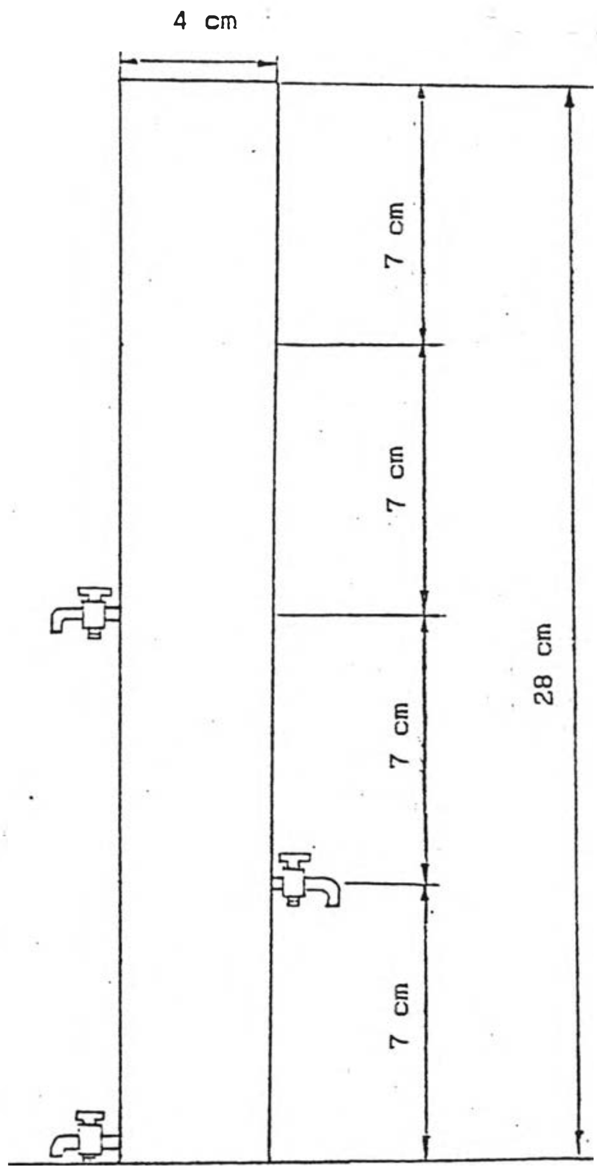


Figure 3.2 Sedimentation column (scale 1:2)

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3.3 Experimental variables

The variables in the study of the properties of coal, fuel oil and coal-oil mixture were :

- Coal types and fuel oil types; three types of coal in Thailand and two types of fuel oil were used as shown in Table 3.1 and 3.2.
- Coal concentrations; the weight percent of coal in COM between 10-50 % were used.

The variables in the study of the rheological property were:

- Coal concentrations; the weight percent of coal in COM between 10-50 % were used.
- Coal types and fuel oil types as shown in Table 3.1 and 3.2.
- Particle size distributions; three different particle size distributions (PSD) were used, i.e. -75 microns, 75-90 microns and 90-106 microns.
- Temperature; five temperatueres were used, i.e. 40, 50, 60, 70 and 80 °C

The variable in the study of stability were :

- Effect of time ; a time range between 3-24 hours was used to find the equilibrium sedimentation time.
- Effect of weight percent coal in COM ; a weight percent of coal range between 10-40 % was used to find the most suitable concentration to study COM stability.
- Effect of weight percent additive in COM; a weight percent of additive range between 0.25-3 % was used to find the optimum concentration to study COM stability.
- Effect of additive types; fourteen additives were used, i.e. anionic, cationic and nonionic additives and the most

effective additives selected. The formulae are shown in Table 3.3.

3.4 Experimental procedures

3.4.1 Coal preparation and coal analysis

The pulverized coal was prepared by crushing a lump of coal with hammer mill to obtain coarse particles, then pulverize the coal with a cross beater mill to obtain fine particles, then screening to four different particle size distributions:

Number	Size	
1	106-150 microns	(100-150 mesh)
2	90-106 microns	(150-170 mesh)
3	75-90 microns	(170-200 mesh)
4	-75 microns	(-200 mesh)

For coal analyse, the coal particles are prepared by screening through a 60 mesh sieve (-250 microns) and analyzed as follows:

- Proximate analysis: moisture; volatile; ash and fixed carbon : ASTM D 3172-75
- Heating value : ASTM D 2015
- Sulfur content in coal : ASTM D 3177
- Sulfur content in fuel oil, COM : ASTM D 129

Detail of the analysis methods were shown in Appendix D.

3.4.2 Coal-Oil Mixture preparation

For preparation of COM, a known quantity of suspending medium, fuel oil, was added to a known amount of coal and the mixture was blended using a mechanical stirrer.

3.4.3 Fuel oil and COM analysis

The properties of fuel oil and COM at various coal concentration were measured as following:

- Pour point	ASTM D 97
- Ash	ASTM D 3174
- sulfur content	ASTM D 129
- Heating value	ASTM D 2015

3.4.4 Rheological properties test

- COM was heated to 40 °C and stirred with a mechanical stirrer to form an homogeneous mixture in a 600 ml beaker which was placed in a water bath.

- The COM rheological data : shear stress and shear rate were obtained by using a Brookfield LV Model viscometer at various coal concentrations.

- To obtain an apparent viscosity of COM at various temperature, COM was heated and stirred to the desirable temperature and the viscosity was measured with a Brookfield viscometer.

3.4.5 Stability test

COM was heated to 50 °C and stirred with a stirrer to form an homogeneous mixture.

- Densities at various coal concentrations using a pycnometer were measured to construct a calibration plot of density or specific volume versus weight percent coal.

- To find the equilibrium sedimentation time, COM composed of -75 microns Ban Pu coal and LFO was filled in a sedimentation column. The weight percent coal from bottom sampling at various times was measured by converting density to a weight percent of coal by using a calibration curve.

- To find suitable weight percent coal for COM stability test, COM composed of -75 microns Ban Pu coal and LFO was filled in a sedimentation column. The sedimentation ratio (SR) from a bottom sampling at a given settling time(24 h) for various weight percent coal in COM was obtained.

- To find the optimum weight percent of additive for the COM stability test, COM composed of 25 wt% -75 microns Ban Pu coal in LFO and 30 wt% coal in HFO were filled in the sedimentation column. The SR from the bottom sampling at a given settling time (24 h), and at various weight percent additive (0.25-3 %) in COM were obtained.

- To find the most effective additive, the SR of COM composed of 25 wt% -75 microns Ban Pu coal in LFO and 2 wt% additive was obtained at a given settling time (24 h) for the various additive types of Table 3.3.

- To find the most effective additives for various coal types, the SR of COM composed of 30 wt% -75 microns coal in HFO and 1 wt% of the most effective additives was obtained at a given settling time (24 h). The SR of COM composed of 30 wt% 106-150 microns Ban Pu coal in HFO and 1 wt% of the most effective additives was obtained.

3.4.6 Combustion test

To study the combustion phenomena , a thermal analysis; thermogravimetric analysis (TGA), were used with the conditions as follows:

Temperature	room temperature-650 °C
Heating rate	15 °C/min
Air flowrate	90 ml/min

Sample	Initial weight (mg)
Ban Pu coal	8.38
HFO	8.15
HFO + 10 % coal	8.95

Sample	Initial weight (mg)
HFO + 20 % coal	8.45
HFO + 30 % coal	8.60
HFO + 40 % coal	8.62
HFO + 50 % coal	8.50
LFO	8.60
LFO + 10 % coal	8.60
LFO + 20 % coal	8.35
LFO + 30 % coal	8.22
LFO + 40 % coal	8.65
LFO + 50 % coal	8.38

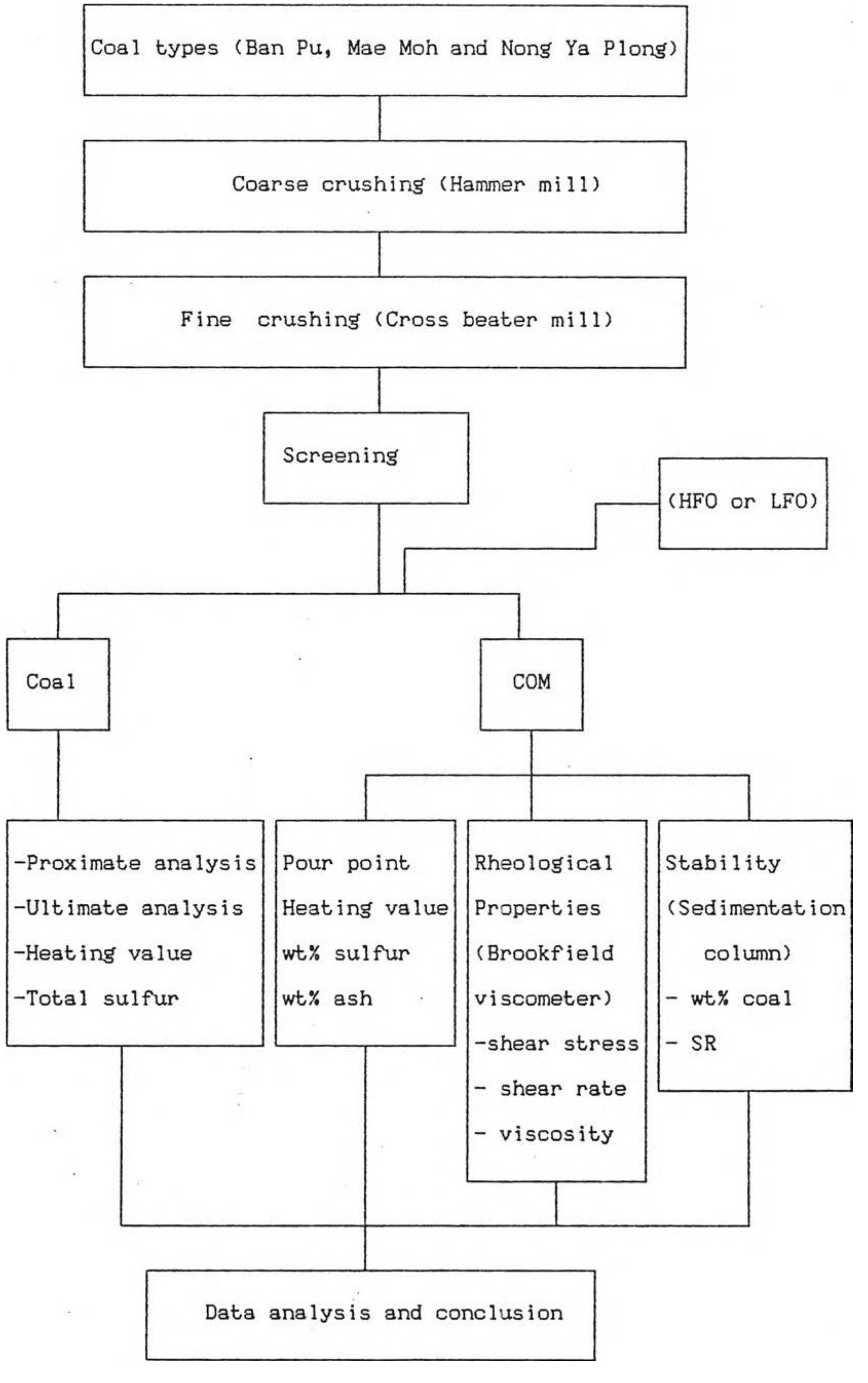
3.5 Data analysis and conclusion

All results are analyzed and explaned by using theory. The rheological datas conclude in the form of; a flow curve, a shear stress versus shear rate ; an apparent viscosity for various variables. The data of stability were given in the form of the ratio SR :

$$SR = \frac{\text{Weight percent coal from bottom sampling (additive)}}{\text{Weight percent coal from bottom sampling (no additive)}}$$

The most effective additive which improve COM stability was found.

All procedures can be abbreviated as follows:


Coal types (Ban Pu, Mae Moh and Nong Ya Plong)
Coarse crushing (Hammer mill)
Fine crushing (Cross beater mill)
Screening
(HFO or LFO)
Coal
COM
-Proximate analysis
-Ultimate analysis
-Heating value
-Total sulfur
Pour point
Heating value
wt% sulfur
wt% ash
Rheological
Properties
(Brookfield
viscometer)
-shear stress
- shear rate
- viscosity
Stability
(Sedimentation
column)
- wt% coal
- SR
Data analysis and conclusion

All variables can be abbreviated as shown in Table 3.4.

Table 3.4 Variables of experimentation

Variables	Coal, fuel oil, COM analysis	Rheological properties		Stability SR
		shear stress shear rate	viscosity	
Coal types	*	*		*
Fuel oil types	*	*	*	*
Coal concentration	*	*	*	*
Temperature			*	
PSD			*	*
Time				*
Wt% additive				*
Additive types				*