

## A STUDY OF INTERFACIAL PROPERTIES IN THE LIQUID CO<sub>2</sub>-WATER-COAL SYSTEM

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### INTRODUCTION

During the past few years, a great deal of interest has developed in fine coal cleaning techniques. The concerns about the environmental effect of sulfur dioxide emissions and the efficient use of clean-burning fuel for utility boilers provide the main impetus for the increased research in this area. Currently, several advanced surface-property-based techniques [1,2,3,4] are under development. Among these, the LICADO (Liquid Carbon Dioxide) process [4], which utilizes liquid CO<sub>2</sub> at about 6 MPa and room temperature to separate mineral matter from coal, is currently being investigated as a new technique for cleaning coal.

The LICADO process relies on the relative wettability of "clean" coal and mineral particles with liquid CO<sub>2</sub> and water, respectively. When liquid CO<sub>2</sub> is dispersed into a coal-water slurry, it tends to form agglomerates with the clean coal particles and float them to the liquid CO<sub>2</sub> phase. The mineral particles, on the other hand, remain in the aqueous phase as refuse. Since the surface/interfacial properties of fine coal particles play such an important role in this coal cleaning operation, an understanding of their behavior becomes indispensable. Therefore, basic measurements must be made on the surface/interfacial properties of coal particles under conditions similar to those encountered in actual operation of the LICADO process.

### BACKGROUND

It is generally postulated that the differences in surface/interfacial properties between the coal maceral and coal mineral (hydrophobic vs. hydrophilic) form the basis for the separation of these two components in the CO<sub>2</sub>-water system. One of the most important surface/interfacial properties is the contact angle. In 1805, Young proposed that the equilibrium contact angle ( $\theta$ ) of a liquid drop resting on a flat solid surface be expressed in terms of three interfacial tensions -  $\gamma_{LV}$  at the interface of the liquid and vapor,  $\gamma_{SL}$  at the interface of the solid and liquid, and  $\gamma_{SV}$  at the interface of the solid and vapor. As Baughman and Razouk [5] pointed out, the term  $\gamma_{SV}$  can be related to the surface tension of the solid  $\gamma_S$  by adding the film pressure,  $\pi_{SV}$ , due to adsorbed vapor. Hence the surface tension of the solid  $\gamma_S$ , which is free from any adsorbed surface film, can be described as

$$\gamma_S = \gamma_{SL} + \gamma_{LV} \cos \theta + \pi_{SV} \quad 1)$$

The measurement of  $\pi_{SV}$  is likely to be difficult, but could conceivably be done [6] by measuring the amount of vapor ( $\Gamma$ , mole of vapor adsorbed per unit solid surface area) adsorbed at pressures ranging from near zero up to the saturated vapor pressure of the liquid, and using the integrated form of the Gibbs adsorption equation

$$\pi_{SV} = RT \int_{P_0}^P \Gamma \, d \ln P \quad 2)$$

where  $P_0$  is the saturated vapor pressure of the liquid. At 298 K,  $P_0$  is 3.13 KPa for water and is 6.42 MPa for liquid CO<sub>2</sub>.

Coal is a heterogeneous material composed of many different components which exhibit their own solid surface tension on the coal surface. Thus, the  $\gamma_S$  of the coal surface is actually a statistical average value. The surface area of each component and its surface tension on the coal surface can supply valuable information in determining the coal surface tension. It is due to the heterogeneity of coal that Neumann et al [7,8] have recently reported the duality of coal surface tension (based on the assumptions of negligible  $\pi_{SV}$  and of  $\gamma_{SL}=0$  at the critical wetting condition) in the two different

ranges of 0.03 - 0.04 N/m and 0.06 - 0.07 N/m. According to Neumann [9],  $\gamma_{sl}$  can be determined from the known contact angle and liquid surface tension through the use of Young's equation and the following equation of state:

$$\gamma_{sl} = [(\gamma_{sv})^{1/2} - (\gamma_{lv})^{1/2}]^2 / [1 - 15(\gamma_{sv} \gamma_{lv})^{1/2}] \quad (3)$$

where 15 has the unit of m/N. In the case of the coal-liquid  $\text{CO}_2$ -gaseous  $\text{CO}_2$  system, coal samples were observed to be completely wetted by liquid  $\text{CO}_2$  ( $\cos\theta=1$ ). This was predicted from Zisman plot [6] based on the very low surface tension of liquid  $\text{CO}_2$  ( $\gamma_{lv} \approx 0.0005$  N/m). Consequently,  $\gamma_{sl}$  was estimated to be negligible ( $\approx 0$ ). This fact suggests that the  $\text{CO}_2$  adsorption film pressure,  $\pi_{sv}$ , can be used to approximate the coal surface tension  $\gamma_s$ . However, the determination of  $\pi_{sv}$  depends on the measurements of the high pressure  $\text{CO}_2$  adsorption isotherm and the specific surface area of the coal. The coal surface area can be determined using the Dubinin-Polanyi (DP) equation with  $\text{CO}_2$  adsorption data at 298 K and vacuum conditions [10,11].

In order to understand the separation mechanism involved in the LICADO process, it is necessary to study the interfacial interactions occurring in the  $\text{CO}_2$ -water-coal system. Young's equation can be applied to the water-liquid  $\text{CO}_2$ -coal system at 298 K and 6.42 MPa with the following designations: o - the liquid  $\text{CO}_2$  phase saturated with dissolved water; w - the water phase saturated with dissolved liquid  $\text{CO}_2$ ; s - the solid surface of the coal samples. The equation which relates the interfacial tensions and contact angles among the phases can be expressed as:

$$\gamma_{so} = \gamma_{sw} + \gamma_{wo} \cos\theta_{wo} \quad (4)$$

where  $\gamma_{ij}$  is the interfacial tensions between phase i and phase j, and  $\theta_{wo}$  is the contact angle of the water on the solid surface surrounded by liquid  $\text{CO}_2$ .

The postulate of relative hydrophobicity of coal and mineral surfaces leads to the following inequality of contact angles for the clean coal (c), the feed coal (f), and the refuse (r):

$$(\theta_{wo})^c > (\theta_{wo})^f > (\theta_{wo})^r \quad (5)$$

and using Equation 4, this inequality can be expressed in terms of interfacial tensions:

$$(\gamma_{so} - \gamma_{sw})^c < (\gamma_{so} - \gamma_{sw})^f < (\gamma_{so} - \gamma_{sw})^r \quad (6)$$

It should be emphasized that these are equilibrium equations and that they do not reveal how the particle initially comes into contact with different fluid media. The body force and the shear force are usually required to initiate this contact. Since the shear and body forces are determined by the physical properties (such as particle size, specific gravity) of the coal particles as well as the hydrodynamic conditions in the separation system, it is therefore believed that a relationship between the process performance and the wetting characteristics of the coal/refuse particles can be understood through the following experimental studies.

#### EXPERIMENTAL Batch Research Unit

The Batch Research Unit was used to obtain process performance data and to collect coal/refuse samples for surface/interfacial property studies. The unit consists of two high pressure cells (2-inch I.D.) connected by a 2-inch ball valve so that the clean coal and refuse can be removed completely without remixing of the products when the system is depressurized. The system is provided with two variable speed mixers (up to 1200 rpm) for agitation of both the aqueous and liquid  $\text{CO}_2$  phases. Hence, the effect of mixing on the separation process can be studied. In the experiment, approximately one liter of coal-water slurry was used for each test run. Liquid  $\text{CO}_2$  was injected from the bottom while both phases were mixed. After a pre-determined time, the liquid  $\text{CO}_2$  injection and the agitation were stopped and phases were allowed to separate. The center valve was then closed and the clean coal product and the refuse fraction were removed.

#### High Pressure Interfacial Property Measuring Device

A high pressure apparatus (Figure 1), equipped with necessary optical and photographic accessories, was constructed for the measurement of interfacial properties in the coal-liquid CO<sub>2</sub>-water system. The measurement cell, equipped with two-inch windows on opposite sides, was mounted on a rotary table capable of ±180 degree rotation. The connections from this cell were high pressure flexible metal tubes. This apparatus was used to perform the following kinds of measurements:

- Vapor-liquid and liquid-liquid interfacial tensions for the vapor CO<sub>2</sub>-liquid CO<sub>2</sub>-water system.
- Equilibrium, advancing or receding contact angles of water on coal samples in an environment that can be either liquid CO<sub>2</sub> or gaseous CO<sub>2</sub> at various pressures.

An overhead mixing cell was used to equilibrate both immiscible phases so that both the liquid CO<sub>2</sub> and water could be fed from the mixing cell to the measurement cell by gravity. Hence, a stable pendant drop could be hung from the tip of a S.S. tube for photography. Two high-pressure pycnometers were used to determine the density of water and CO<sub>2</sub> at room temperature and various pressures.

For the contact angle measurement, a liquid drop (sessile drop) was deposited on a 1" diameter x 1/4" thick coal pellet sample, which was held by a specimen holder. This specially designed specimen holder could be mounted, from the side of a two-inch flanged opening, on the tip of a shaft assembly. The shaft assembly was designed in such a way that both rotational and translational movement can be externally controlled. The horizontal rotation of the specimen holder enabled the measurements of the contact angle from any direction of the sessile drop. The vertical movement permitted the adjustment of distance between coal sample and the tip of the feed tube during the formation of the sessile drop. The contact angle could be measured either in-situ using the angular protractor inside the eyepiece of the microscope or from the photograph of the drop.

Photographic image could be analyzed either by Andreas' manual method [12] or by Rotenberg's numerical method [13]. Using the latter method, the images were digitized using the LEITZ TAS-plus image analyzer connected to a macroviewer. A computer program written in TAS-plus language was developed to digitize the contour of the drop and to transfer the coordinate data to the University VAX/VMS system, where Rotenberg's program was implemented to calculate either the interfacial tension or the contact angle.

#### Dubinin-Polanyi Coal Surface Area and CO<sub>2</sub> Adsorption Isotherm

The surface area of coal was determined from the adsorption of CO<sub>2</sub> using a BET apparatus operated at 298 K. The Dubinin-Polanyi plot could be constructed from the amount of CO<sub>2</sub> adsorbed on the coal surface at various vacuum pressures in order to obtain the CO<sub>2</sub> adsorption surface area.

The equilibrium unit used for studying CO<sub>2</sub> adsorption on coal was a high pressure cell in which a precision quartz spring held the coal sample in a porous thimble. Adsorption experiments were performed by introducing a known weight (~2g) of coal sample in the system and then degassing the sample under vacuum. This was followed by the introduction of liquid CO<sub>2</sub>. Once equilibrium had been attained, the elongation of the quartz spring was measured to determine the amount of CO<sub>2</sub> adsorbed on coal. By incorporating the integration of the adsorption isotherm and the surface area data from the DP plot, a computer program written in FORTRAN 77 language was developed to determine the film pressure ( $\pi_{sv}$ ).

#### RESULTS AND DISCUSSION

Upper Freport coal from Indiana County, PA with 23.5% ash content was selected for the experimental work. Primarily, -200 mesh samples were used in this study while other size fractions such as -28 mesh, -28+100 mesh, and -100 mesh were used only for comparison purposes. The coal samples for interfacial property measurements were obtained from the LICADO batch cleaning experiments under specified operating conditions.

Wetting Characteristics and Mixing of the LICADO process

The clean coal and refuse samples from each stage of the multistage cleaning experiments were analyzed. The ash content of the coal samples together with the measured contact angles of water under various CO<sub>2</sub> pressures are presented in Table 1. Based on the deposition technique of the sessile drop, two cases were studied:

1. The coal surface was saturated with CO<sub>2</sub> gas at a desired pressure followed by placing the saturated water drop on the surface (CO<sub>2</sub>-first-wet).
2. A water sessile drop was first formed on the coal surface at its own vapor pressure. Then, CO<sub>2</sub> gas was introduced into the system at the desired pressure. After equilibrium was established, the contact angle was measured (water-first-wet).

Table 1: CONTACT ANGLE OF WATER AT TWO DIFFERENT DEPOSITION TECHNIQUES

Ash Content (Stage No.)		(P, θ)	(P, θ)	(P, θ)	(P, θ)	(L, θ)
2.7% (4c)	1*	(0, 110)	(2.1, 118)	(4.1, 134)	(6.2, 146)	(L, 154)
	2**	(0, 110)	(2.1, 113)	(4.1, 122)	(6.2, 130)	(L, 128)
3.4% (3c)	1	(0, 111)	(2.1, 115)	(4.3, 133)	(6.2, 143)	(L, 152)
	2	(0, 111)	(2.6, 118)	(5.2, 127)	(6.2, 127)	(L, 117)
5.0% (2c)	1	(0, 105)	(2.1, 113)	(4.8, 138)	(6.2, 142)	(L, 152)
	2	(0, 105)	(2.1, 108)	(4.1, 118)	(6.2, 132)	(L, 123)
6.9% (4r)	1	(0, 97)	(2.2, 104)	(4.7, 114)	(6.2, 128)	(L, 154)
	2	(0, 97)	(2.1, 100)	(4.1, 108)	(6.2, 115)	(L, 105)
8.4% (1c)	1	(0, 103)	(2.2, 113)	(4.3, 128)	(6.3, 128)	(L, 138)
	2	(0, 103)	(2.2, 110)	(4.7, 118)	(6.3, 126)	(L, 120)
11.6% (3r)	1	(0, 85)	(1.4, 81)	(2.8, 99)	(6.3, 128)	(L, 137)
	2	(0, 85)	(2.8, 88)	(5.6, 92)	(6.0, 97)	(L, 92)
18.1% (2r)	1	(0, 71)	(3.2, 80)	(5.2, 102)	(6.0, 112)	(L, 130)
	2	(0, 71)	(2.2, 72)	(4.8, 75)	(6.3, 84)	(L, 80)
23.5% (f)	1	(0, 84)	(2.2, 88)	(5.2, 113)	(6.3, 119)	(L, 145)
	2	(0, 84)	(2.8, 88)	(4.5, 91)	(6.3, 94)	(L, 82)
54.2% (1r)	1	(0, 0)	(1.5, 80)	(4.6, 93)	(6.3, 94)	(L, 89)
	2	DOES NOT EXIST				

\*1: CO<sub>2</sub>-First-Wet; \*\*2: Water-First-Wet; c: Clean Coal; f: Feed; r: Refuse;  
P in MPa; θ in Degree; L: Liquid CO<sub>2</sub> Phase.

It is shown that the water contact angle on the feed coal surface (CO<sub>2</sub>-first-wet) increases from 84° at atmospheric CO<sub>2</sub> pressure to 120° at a gaseous CO<sub>2</sub> pressure of 6.2 MPa. When the water drop is in the liquid CO<sub>2</sub> environment, the contact angle increases further to 145°. This similar trend can also be observed for the first stage clean coal (1c). On the other hand, the water contact angle on the refuse sample (1r) decreases from 94° to 89° as the environment changes from equilibrium gaseous CO<sub>2</sub> to liquid CO<sub>2</sub>. Hence, the largest contact angle difference between the clean coal (8.4% ash) and refuse (54.2% ash) samples is in the liquid CO<sub>2</sub> phase, which accordingly, indicates that clean coal particles show a hydrophobic behavior (145°) that results in their agglomeration and transfer to the liquid CO<sub>2</sub> phase while refuse particles, more hydrophilic, remain in the water phase. Measurements made from other stage samples display a similar phenomenon. Generally, it is observed that when the ash content of the coal sample increases, the water contact angle decreases. A strong support for the use of liquid CO<sub>2</sub> as the processing medium for fine coal cleaning is therefore confirmed.

It was observed that the mixing in the LICADO process helped to improve the effectiveness of separation between the coal and mineral matter. In an attempt to understand the relationship between

the shear force and interfacial force, the effect of mixing speed in both the liquid CO<sub>2</sub> and water phases was studied. As depicted in Figure 2, it appears that an intermediate agitation speed between 300 to 900 rpm will be best for the separation. It is possible to explain the effect of agitation on the LICADO process from the data obtained from the contact angle measurement on the feed coal. Since the coal sample was prepared in a water slurry form before feeding to the LICADO system, the wetting characteristic of the feed coal/water slurry is similar to that of the water-first-wet case as the CO<sub>2</sub> pressure is raised to the LICADO operating pressure. Consequently, a smaller contact angle (94°) can explain the poor separation result (13% ash content, 11% yield) at zero mixing speed, where no shear force is supplied to expose the water-first-wet coal surface to the liquid CO<sub>2</sub> medium. On the other hand, an optimum mixing speed in both phases will provide enough shear force to expose the clean coal particles to the liquid CO<sub>2</sub> droplets. It follows that a good separation (8.6% ash content, 67% yield) at 800 rpm can be interpreted by the larger contact angle (145°) when coal particles are in contact with liquid CO<sub>2</sub> (CO<sub>2</sub>-first-wet case). As the mixing speed increases beyond 1200 rpm, the very strong agitation action, which overwhelms the interfacial force, may cause the entrainment of refuse particles into large clean coal agglomerates. This results in a higher ash content (12.3% ash content) in the product coal.

Table 2: SURFACE AND PHYSICAL PROPERTIES OF COAL SAMPLES

Ash %	Particle Size $\mu\text{m}$	Specific Gravity	Surface Area $\text{m}^2/\text{g-coal}$	g-CO <sub>2</sub> adsorbed <sup>a</sup> per g-coal	Film Pressure N/m $\times 10^3$
2.7 (4c)	15.2	1.27	163	0.097	61.3
3.4 (3c)	16.0	1.28	156	0.088	65.0
5.0 (2c)	16.2	1.30	156	0.097	65.0
6.8 (4r)	17.1	1.32	143	0.095	79.5
8.4 (1c)	18.8	1.31	138	0.085	81.2
11.6 (3r)	18.8	1.32	136	0.090	80.9
18.1 (2r)	17.4	1.40	82	0.055	88.0
23.5 (f)	18.4	1.45	109	0.082	81.8
54.2 (1r)	25.6	1.88	52	0.046	93.7

<sup>a</sup>At CO<sub>2</sub> pressure of 6.2 MPa.

#### Adsorption and Agglomeration of the LICADO Process

The measurements of CO<sub>2</sub> adsorption on samples from each cleaning stage were performed at pressures ranging from vacuum pressure to the vapor pressure of CO<sub>2</sub> at room temperature. The results are presented in Table 2. It can be seen that the use of carbon dioxide in fine coal cleaning produces clean coal with a relatively larger specific surface area (136 ~ 163 m<sup>2</sup>/g-coal) than that of the corresponding refuse (52 ~ 143 m<sup>2</sup>/g-coal). The mean particle size and the specific gravity increase as the ash content of the coal sample increases. In addition, the CO<sub>2</sub> adsorption data, obtained at various elevated pressures to construct a complete adsorption isotherm, shows that the amount of CO<sub>2</sub> adsorbed on the clean coal samples (0.085 ~ 0.097 g-CO<sub>2</sub>/g-coal) is greater than the amount adsorbed on the corresponding refuse (0.046 ~ 0.095 g-CO<sub>2</sub>/g-coal). Consequently, the film pressure of each coal sample increases from 0.0613 to 0.0937 N/m as the ash content of the coal sample increases from 2.7% to 54.2%. Due to the low surface tension of liquid CO<sub>2</sub>, it has been deduced that the CO<sub>2</sub> adsorption film pressure is suitable to represent the solid surface tension of the coal. Thus, the surface tension ( $\gamma_s$ ) of these coal/refuse samples increases from 0.0613 to 0.0937 N/m as the ash content increases from 2.7% to 54.2%. It is believed that this large amount of adsorbed CO<sub>2</sub> will help to agglomerate the clean coal particles, which further confirms the validity of using liquid CO<sub>2</sub> for fine

coal cleaning.

**Effect of Particle Size on Coal Surface Properties**

Since the clean coal samples have relatively larger surface areas and smaller particle sizes than those of the refuse samples, three factors were envisioned to contribute to these adsorption results: (1) surface property (ash content); (2) mean particle size; (3) specific surface area.

In order to understand the role that the mean particle size plays on the coal/refuse surface properties, portions of Upper Freeport -28 mesh and -28+100 mesh coal were ground to -200 mesh and -100 mesh coal respectively. Hence within each of these two tested groups (-28 mesh and -200 mesh; -28+100 mesh and -100 mesh), the coal samples differ only in their particle size while the other physical properties such as the ash content, sulfur content and specific gravity remain the same. The contact angles, surface areas, and adsorption isotherms for these samples were experimentally determined.

The results (Table 3) show that the contact angles in the large particle size fractions (-28+100 mesh and -28 mesh) are slightly smaller than those in the small particle size fractions (-100 mesh and -200 mesh). This may be attributed to the apparent differences in surface condition of the compressed coal pellet samples formed with large and small particle sizes. Hence the apparent contact angle measured for the large particle size coal tends to underestimate the true contact angle. This phenomenon was also observed by Murata [14]. By considering the small contact angle differences between these two size fractions of each group, it can be said that the particle size has only a minor effect on the contact angle.

Table 3: EFFECT OF PARTICLE SIZE ON COAL SURFACE/INTERFACIAL PROPERTIES

	(Ash=28.6%, S.P.=1.52)		(Ash=23.5%, S.P.=1.45)	
	-28+100 mesh	-100 mesh	-28 mesh	-200 mesh
$\theta$ in Liquid $CO_2$	140	149	138	145
Surface Area ( $m^2/g$ -coal)	87	98	95	108
$\pi_{sv}$ (N/m) $\times 10^3$	90.4	88.9	85.2	81.6
$g-CO_2/g$ -coal at $P=P_0$	0.057	0.065	0.069	0.082
$g-CO_2/m^2$ -coal at $P=P_0$	0.0655	0.0663	0.0726	0.0752

As expected, the surface area is smaller for the large size fraction of coal samples, but the difference of  $12 \sim 14 m^2/g$ -coal indicates that a certain portion of the internal micropore exists in these coal samples. By incorporating the data of the adsorption isotherm and the specific surface area, the film pressure of each size fraction of coal sample was measured and compared. Since the  $CO_2$  adsorption film pressure is only a function of temperature, pressure and composition, little difference between the two size fractions of coal samples is perceived for both of the tested groups. It is thus concluded that the particle size has little effect on the  $CO_2$  adsorption film pressure of the coal.

It is also found that coal with the larger size fraction adsorbed less  $CO_2$  on a unit weight basis. This can be explained by the fact that when the coal particles are ground into smaller sizes, the increase in the total surface area is mostly due to the increase in the external surface area (assuming very few dead pores in the original coal particles), while the internal micropore surface area is the same for both the larger and smaller sizes of coal particles. The increase in the external surface area for the finer coal particles is known to be the reason for the increased amount of  $CO_2$  adsorption on a unit weight basis. However when expressed on a unit surface area basis, the same amount of adsorption is observed. Again, it reveals that the mean particle size has a minor effect on the  $CO_2$  adsorption.

## CONCLUSIONS

From the above discussion, it is clear that the LICADO process is a surface-property-driven process that belongs to the physical cleaning category. The work performed so far indicates that the mechanism of the LICADO process is governed by the interactions among the interfacial, shear and body forces present in the coal particle-liquid CO<sub>2</sub>-water system. The shear and body forces are determined by the physical properties of the coal particles as well as the hydrodynamic conditions in the separation process, which subsequently control the manner that the surfaces of coal particles are exposed to processing media. Therefore, it is possible to optimize the performance of the LICADO process by improving the quality of mixing.

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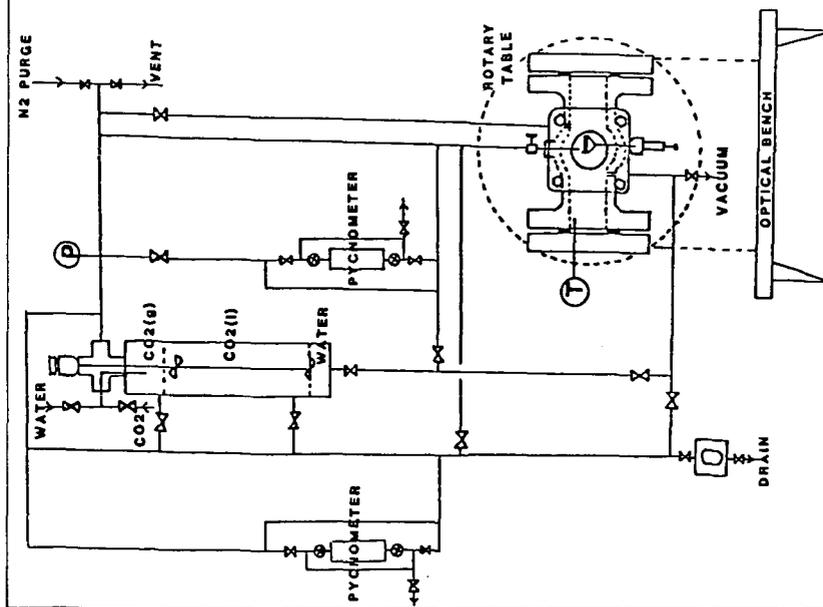


Figure 1: High Pressure Contact Angle Apparatus

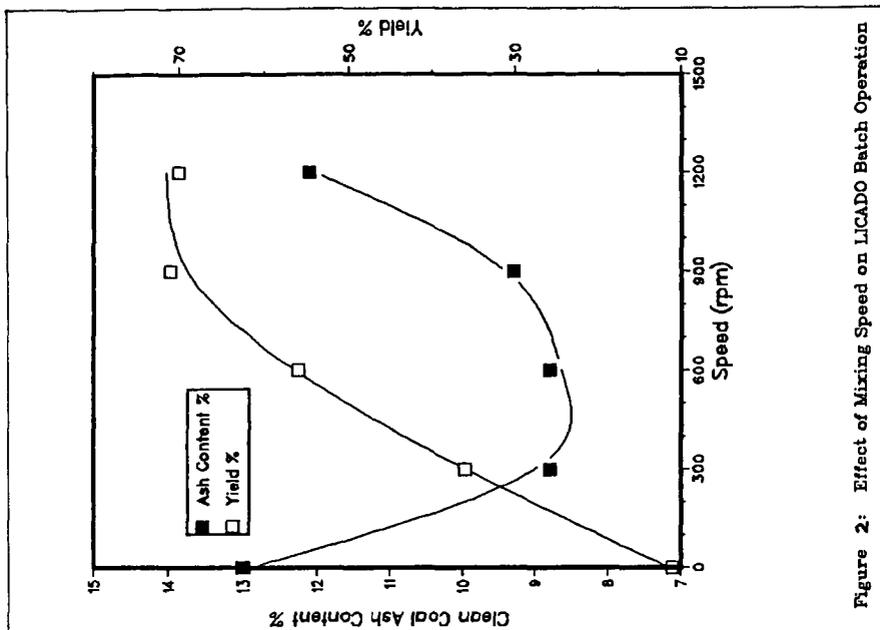


Figure 2: Effect of Mixing Speed on LICADO Batch Operation