

## CHAPTER IV

### EXPERIMENTS

#### 4.1 Chemicals

- 4.1.1 Zinc dialkyl dithiophosphate (ZDDP) from Castrol (Thailand) Co., Ltd.
- 4.1.2 Diammonium hydrogen phosphate (commercial grade)
- 4.1.3 Sodium dodecyl benzene sulphonate (SDBS) from Kao Industrial (Thailand) Co., Ltd.

#### 4.2 Experiments

This experiments consisted of batch and continuous processes. An oil feed was prepared by dissolving a certain amount of of zinc complex ion (i.e. zinc dialkyl dithiophosphate; ZDDP) in base oil (Base oil 100 SN , Castrol (Thailand) Co.,Ltd. Typically, concentration of ZDDP was used approximately at 0.9% wt/wt. The extracting solution contained 8% wt/wt of diammonium hydrogen phosphate in aqueous solution. Sodium dodecyl benzene sulphonate (SDBS) was used as surfactant in this experiments. The experiments describing methods for testing extractants and surfactants could be seen in Appendix B.

#### 4.2.1 Batch process experiments

The experiments were carried out in a baffled vessel equipped with a pitched-blade turbine. A schematic of this system was shown in figure 4.1. The oil feed and the extracting solution were contacted in the vessel in the equal volume (phase ratio of extracting solution:oil was 1:1), then thoroughly mixed at agitator speed of 600 rpm for 60 minutes. After each experiment, the mixture of two liquids was settled, separated into two phases, the upper (oil phase) and the lower one (aqueous phase). Samples were obtained from the oil phase, centrifuged at 4000 rpm for 20 minutes, then analysed for zinc content by Inductively Coupled Plasma Emission Spectral Analysis (ICP). Based on the results obtained, the percentage of extraction and distribution ratio were calculated. Experimental calculations were shown in Appendix C.

##### 4.2.1.1 Effect of agitator speed

The procedure of batch process was followed except that the agitator speed was varied from 400 to 800 rpm.

##### 4.2.1.2 Effect of surfactant concentration

The procedure of batch process was followed except that SDBS was added into extracting solution at concentration in the range of 1 to 8% wt/wt.

#### 4.2.1.3 Effect of phase ratio

The procedure of batch process was followed except that SDBS concentration was fixed at 1% wt/wt. and phase ratio (volume fraction of extracting solution:volume fraction of oil) was varied from 1:1 to 1:5.

#### 4.2.1.4 Effect of initial zinc concentration

The procedure of batch process was followed except that SDBS concentration and phase ratio were fixed at 1% wt/wt and 1:1, respectively. Initial zinc concentration was varied from 100 to 1500 ppm.

#### 4.2.2 Continuous process experiments

The experiments were performed in a 9 cm ID, 150 cm long agitated extraction column equipped with a variable speed electronically controlled motor. At the axis of the column, 37 stainless steel plates of 7.62 diameter were installed. Each plate had 32 holes with 0.3 cm ID.

The oil feed prepared in storage tank (T1) was pumped by pump (P1) up to storage tank (T2). Pump (P2) was used to pump oil from T2 to adjustable rotameter (R1) adjusting flow rate in the range of 0 to 1 lit/min. The oil was fed further to the bottom of the column. While extracting solution was transferred from storage tank (T3) up to storage tank (T4) by pump (P3), then pumped by pump (P4) through adjustable rotameter (R2) into the top of the column.



When operation started, the column was firstly filled with the extracting solution at flow rate of 0.2 lit/min. The oil was fed from the bottom end of the column. The overflow of oil was out of the column at the top end and kept in extract tank. The flow of raffinate was controlled by valve (V1). After the flows of extracting solution and oil were steady, agitation was started. Operating contact time was defined as interval between agitator started and agitator stopped.

#### 4.2.2.1 Effect of agitator speed

The procedure of continuous process was followed except that the agitator speed was varied from 100 to 400 rpm.

#### 4.2.2.2 Effect of surfactant concentration

The procedure of continuous process was followed except that SDBS was added into extracting solution at concentration in the range of 0.1 to 4% wt/wt.

#### 4.2.2.3 Effect of phase ratio

The procedure of continuous process was followed except that SDBS concentration was fixed at 1% wt/wt and phase ratio (flow rate of extracting solution:flow rate of oil) was varied from 1:1 to 1:5.

#### 4.2.2.4 Effect of initial zinc concentration

The procedure of continuous process was followed except that SDBS concentration and phase ratio were fixed at 1% wt/wt and 1:1, respectively. Initial zinc concentration was varied from 100 to 1500 ppm.

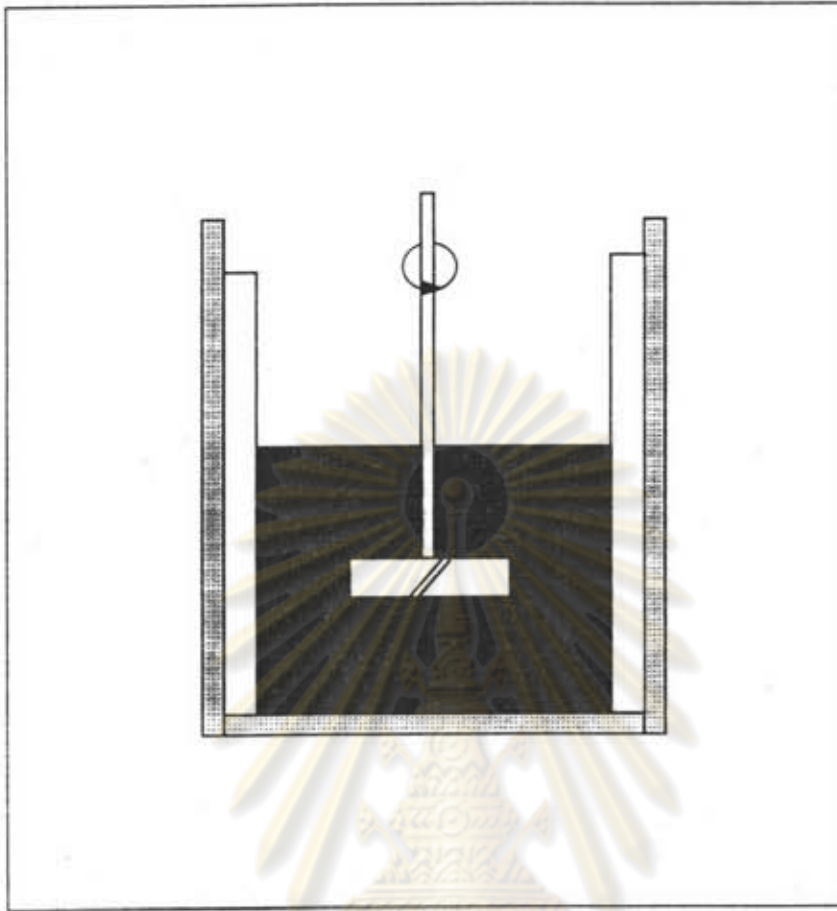


Figure 4.1 Schematic of batch system

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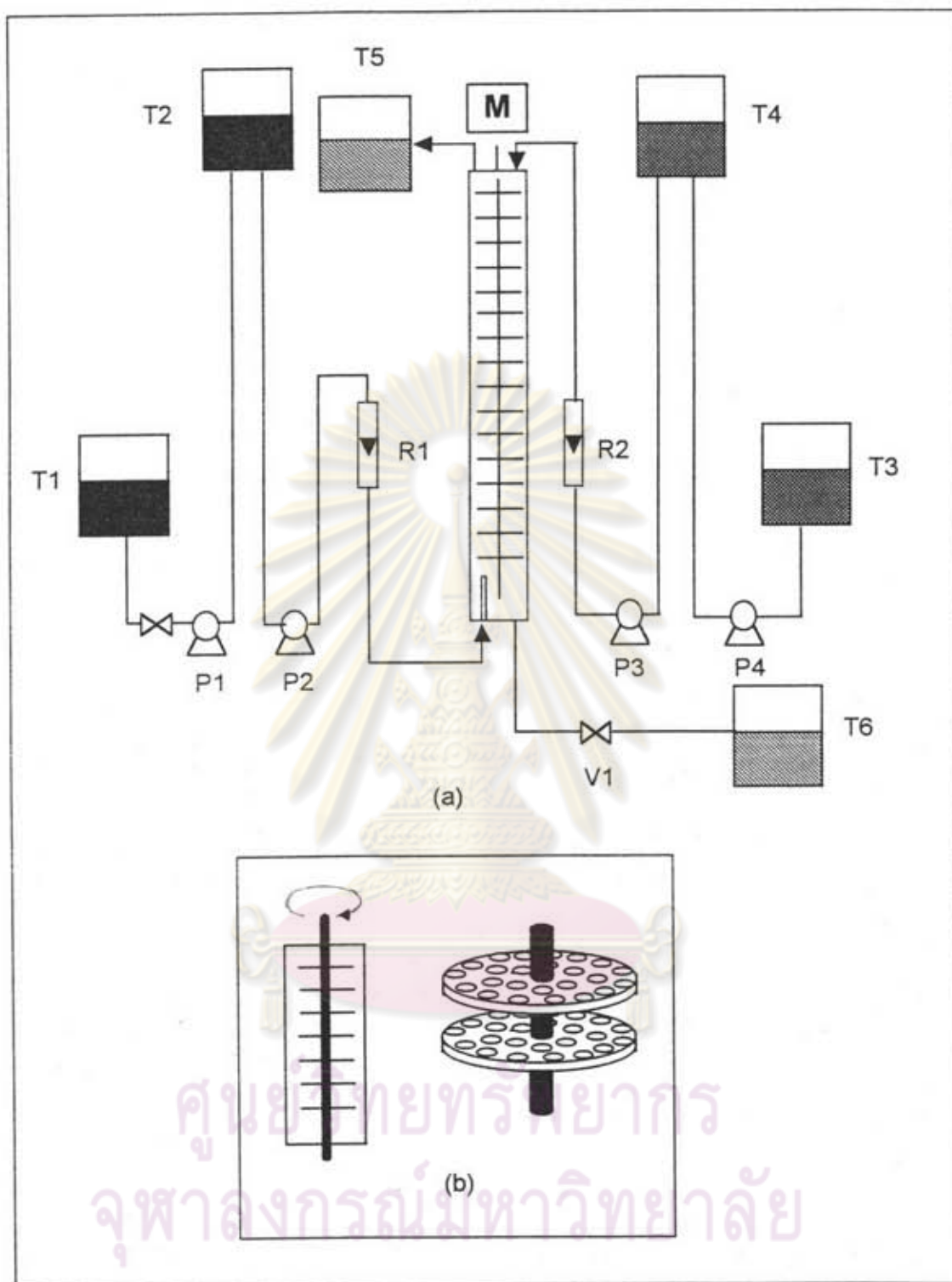


Figure 4.2 Schematic diagram of continuous process. (a) flow diagram ; (b) characteristics of column and agitator.