CHAPTER III

METHODOLOGY

This experiment consisted of 2 parts. Part one involved sink-float separation, the latter dealt with selective flotation technique. The experiment was done at least three times to ensure reproductivity of the data. Then average values of these data obtained from the replicated experiments were presented in this work.

3.1 Plastics samples

The samples of six different kinds of plastics wastes were

- Poly(ethylene terephthalate) – PET
- High-density polyethylene – HDPE
- Polyvinyl chloride – PVC
- Polypropylene – PP
- Polystyrene – PS
- Acrylonitrile butadiene styrene – ABS

The samples were post-consumer plastics and obtained from drinking bottles, milk bottles, pipes, yoghurt cups, drinking cups and cardboards respectively. The samples used for test consisted of a mixture of 3 g of each plastics type. Moreover they were different colour, which made it easier to analyse the concentrate samples through manual sorting at the end of each experiment. Each of the plastics waste samples were shredded by using scissors and screened. The sieve size fractions used in this experiment are 0.3 – 0.5 cm, the general size that recyclers used as shown in **Figure 3.1**.

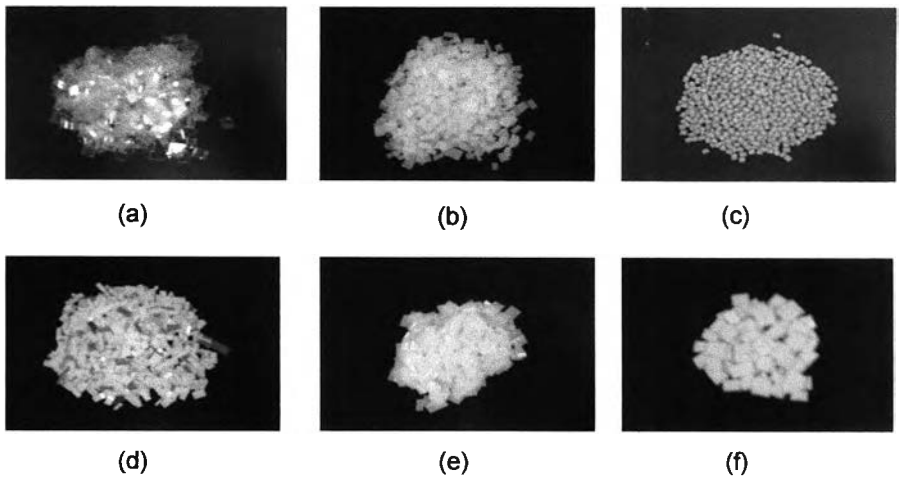

Figure 3.1 Schematic a configuration of each plastics
(a) PET, (b) HDPE, (c) PVC, (e) PP, (d) PS and (f) ABS

3.2 Chemical reagents

3.2.1 Sink-float separation

i) Calcium chloride ($CaCl_2$) with purity 98% was purchased from Ajax, Australia. It was used to prepare dense medium aqueous solution at various concentrations. Moreover, it was an electrolyte for selective flotation test.

ii) Ethyl alcohol (C_2H_5OH) with purity 95%was purchased from LAB SYSTEMS, Thailand. It was used as light medium aqueous solution at different concentrations.

3.2.2 Selective flotation test

i) Calcium lignosulfonate (CaLS [a]) was purchased from Aldrich, USA. CaLS is a by-product of paper industry and has low toxicity and environmental friendly (*sees* MSDS of CaLS at **Appendix A**), thus it was chosen as wetting agent in this research at various concentrations.

ii) Methyl isobutyl carbinol, MIBC [a], and Terpineol [a] (with α terpineol = 65%) were purchased from Fluka, Switzerland. They were used as frother at different concentrations.

Sodium hydroxide (NaOH, with purity 99%) was purchased from Merck, Germany and hydrochloric acid (HCl, with purity 36.5 – 38%) was purchased from J.T. baker, USA, which were pH adjustment reagents.

All chemical reagents were analytical grade except ethyl alcohol. The alcohol was commercial grade.

3.3 Experimental methods

3.3.1 Sink-float separation

The samples were separated by using medium solutions with various concentrations. Light medium solution is a mixture of ethyl alcohol and water, while dense medium solution is calcium chloride aqueous solution. The steps of separating post-consumer plastics are below:

i) The sample was first separated by tap water.

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[a] The chemical structures of CaLS, MIBC and Terpineol were shown in **Figure 2.11** and **Table 2.6**, respectively.

ii) The sample that floated in tap water was separated by light medium solution with various concentrations from 10, 20, 30, 40, 50, 60, 70, 80, 90 to 95% by volume, respectively.

iii) The sample that sank in tap water will be separated by dense medium solution with various concentrations from 10, 20, 30, 40, 50 to 60% by weight, respectively.

iv) The samples that were not properly separated in either step ii) or step iii) was separated by selective flotation method that presented in section 3.3.2

The samples were mixed thoroughly in medium solution by stirring at 150 rpm (IKA; RW 20.n) for 2 minutes. After that they were left for 4 minutes for ensure complete separating. The density of medium solution was determined before and after each experiment by pycnometre. After the separation, the collected products were washed, dried and weighed then the grade and the recovery and purity were calculated based on mass balance. The different in colour allowed easy hand sorting and visual analysis of product streams.

3.3.2 Selective flotation test

The mixed samples that were not properly separated by sink-float separation were separated by selective flotation. CaLS was used, as wetting agent, while MIBC and terpineol were used as frother.

i) The flotation experiments were carried out in two columns of 38 and 60 cm height (h) and 10 cm diameter (d) fitted with a filter disc of size 1 for air bubbles. According to preliminary test, the flow rate of air bubble was 280 ml/min. The apparatus of selective flotation column was shown in **Figure 3.2**.

ii) To get an appropriate concentration and pH of wetting agent. The plastics samples were treated with CaLS at various concentrations (20, 50, 100, 200, 300, 400 and 500 mg/l.) for 4 minutes. At each concentration, it was prepared at different pH (5, 7, 9, 11 and 12). Firstly, the wastes were treated for 4 minutes in 20 mg/l of CaLS solution at different pH. After treating, air was introduced at the bottom of the column to perform flotation. Next, the steps were then repeated but used CaLS solution at different concentrations. Finally, an optimal concentration of CaLS solution and suitable pH for separating the plastics waste was found out.

iii) Conditioning time of treating plastics with wetting agent was varied at 2.5, 3 and 6 minutes for figure out an appropriate time reaction.

iv) Electrolyte, $CaCl_2$, was used to create bridge between the plastics and CaLS at various concentrations 0.1, 0.3, 0.5, 0.7 and 0.9 %w/v.

v) MIBC and terpineol were used to create a stabilisation of floating plastics. Concentration of frother was about 0.2, 0.6 and 1.0 ml/reactor, respectively. In this stage, a proper frother with its concentration was found out. pH of frother solution at each concentration was hold at an optimal pH that obtained from step ii) in section 3.3.2.

vi) NaOH and HCl were used to adjust pH.

vii) Plastics that floated to the top of flotation column will be collected every 2, 4, 6, 8, 10 and 12 minute after flotation preformed.

The density of medium solution was determined before and after each experiment by pycnometre. After the separation, the collected products were washed, dried and weighed then the grade, the recovery and recovered plastics purity were calculated based on mass balance. The different in colour allowed easy hand sorting and visual analysis of product streams as described in previous section and the summery of experiment methodology procedure is shown in **Figure 3.3**.

The recovery of individual sample at the end of the experiment was determined by counting. Each data was collected at least three times to ensure reproductivity of the data. Then the data presented in this work were always the average value obtained from the replicated experiments.

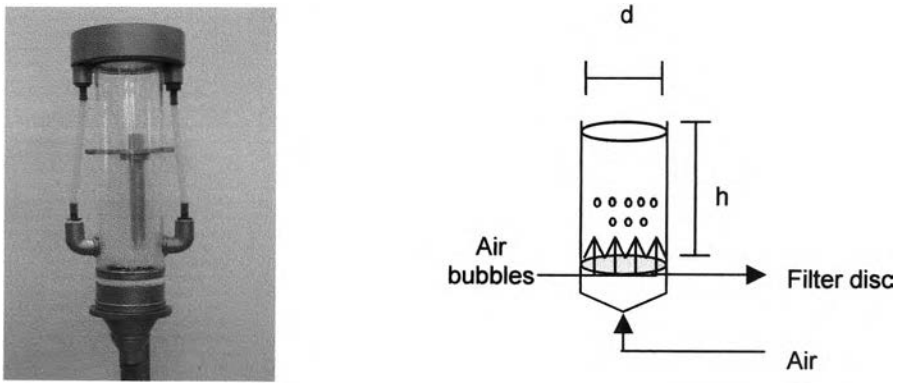

Figure 3.2 Flotation column

3.4 Contact angle measurement

Tensiometry was used for the contact angle measurement with a Contact angle metre: CAM–PLUS Tantec from department of material science, faculty of science, Chulalongkorn University. The plastics waste was cut in approximately 3 x 3 cm, in order to measure a contact angle. The measurement was done at least 5 points on one piece of each plastics plate to ensure the reproductivity. Thus the data that reported in this research was an average value. The liquid, which were used in contact angle measurement were the same as one used in selective flotation. In addition, liquid surface tension was also measured by using ring method.

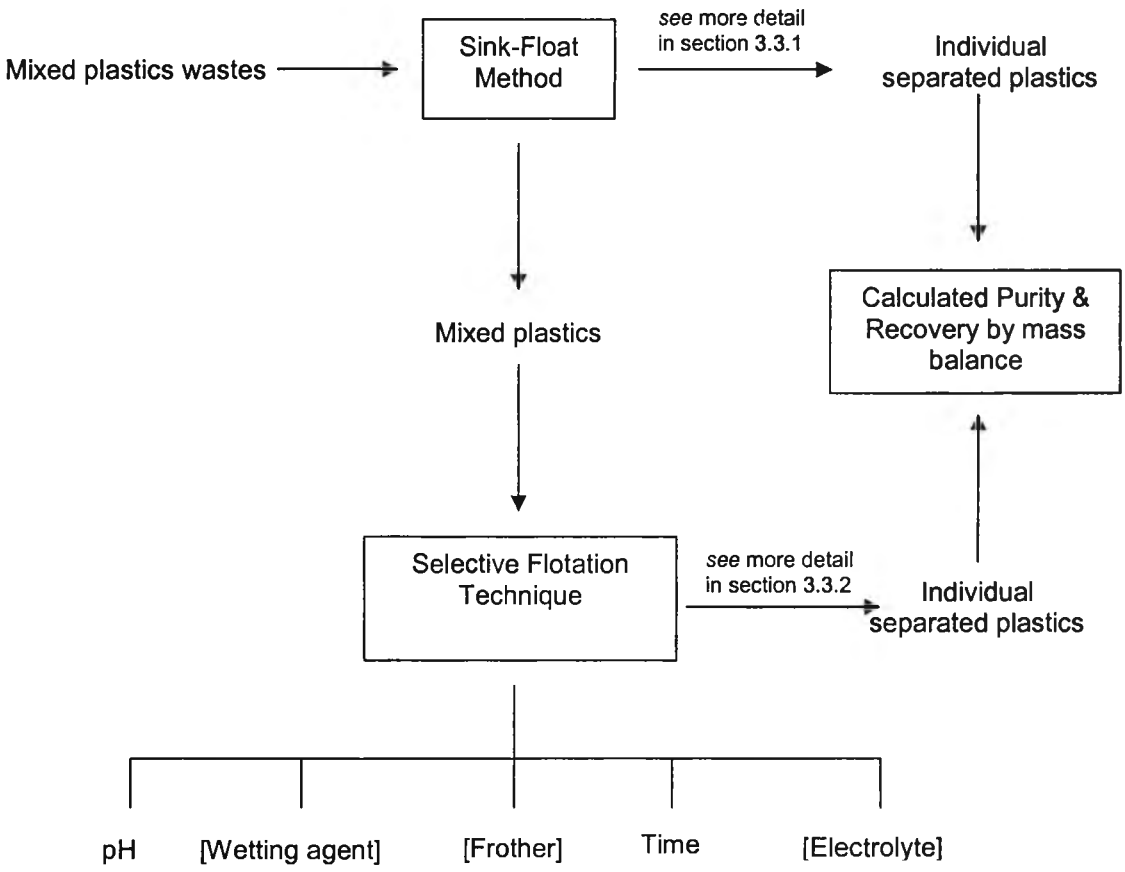

Figure 3.3 Diagram of experiment procedure