CHAPTER III

EXPERIMENTAL METHODS AND MATERIALS

Dehydration of lime juice by spray-drying was investigated. Firstly, dehydration was studied without addition of any drying aid, both for fresh and concentrated lime juice. Later, drying aids such as glucose, sucrose and dextrin were added to lime juice. Experiments were carried out by varying percentage of drying aids, concentration of lime juice and temperature of dehydration.

3.1 Materials

1. Fresh lime fruits Citrus aurantifolia were obtained from the market

2. Glucose (analytical grade)

3. Sucrose (analytical grade)

4. Dextrin with a specification of :

D.E. 9.2	
moisture content	4.74%
ash	0.35%
pH	4.24

5. NaOH 0.1 N.

6. Phenophthalein

7. 2, 6 dichlorophenol indophenol

8. metaphosphoric acid

9. Ascorbic acid

3.2 Instruments

1. Spray-dryer at Chemical Technology Department with a diameter of 0.9 metres, 1.41 metres in height. Rotary type atomizer with co-current feed. Inlet air temperature can be controlled by thermostat (Fig. 2)

2. Vacuum Evaporator (Centri-therm Ct-1B of Alfalaval) at Thailand Institute of Scientific and Technological Research.

3. Freezer (contact type) at Chemical Technology Department.

4. Centrifuge, portable type, diameter 16 cm, 6000 rpm. at Chemical Technology Department.

5. Hand Refractometer (Atago N2, Japan) 0-32 and 32-58 ^{o}Brix

6. pH Meter (Prolabo, Paris)

7. Prolabo Spectrophotometer(no 5347, Prolabo, Paris)

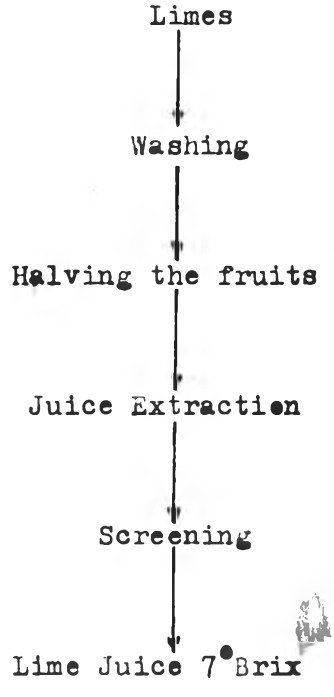
3.3 Methods

3.3.1 Preparation of fresh lime juice

The lime juice used in this study was prepared from lime fruits Citrus aurantifolia as follows:

Fig. 1 Spray dryer

The lime fruits were washed thoroughly under tap water to remove all dirts and contaminants. Defective limes were picked out and rejected. The limes were halved by a sharp stainless steel knife and the juice was extracted by hand -pressed extractor. The juice was passed through two-fold muslin cloth to remove rags and seeds. The lime juice was about 7°Brix. Fresh lime juice prepared from the same lot of lime friut was used in subsequent experiment. The procedure described above is presented in the following scheme.



3.3.2 Preparation of concentrated lime juice

3.3.2.1 By evaporation.

The extracted lime juice (7°Brix) described above was concentrated in a vacuum evaporator (Centri-therm CT-1B alfa-Laval)

at 0.9 kg/sq-cm. steam pressure and the heating fluid temperature of 60-65°C. The evaporating temperature used was 45°C. The concentrated lime juice after evaporation was 30°Brix, 20°Brix and 15°Brix.

3.3.2.2 By freeze-concentration

The lime juice was first freeze-concentrated in an ice-cream freezer. The frozen juice was then centrifuged in a portable-type centrifuge to remove the accompanying ice crystals. This procedure could be repeated to obtain the required lime juice concentration. The lime juice was concentrated to 10°, 20°, 25° and 30°Brix.

3.3.3 Operation of spray-dryer

Turn on the blower. Set required inlet temperature and wait until the required outlet air temperature was constant. Start drying operation by feeding the sample juice at a constant rate such that specified outlet air temperature was maintained.

3.3.3.1 Preparation of lime powder without drying aid

3.3.3.1.1 From fresh lime juice

Fresh lime juice (7°Brix) was spray dried using an inlet air temperature of 140°C air pressure of atomizer at 40 psig and outlet air temperature at about 80-85°C

3.3.3.1.2 From concentrated lime juice

Fresh lime juice was concentrated to 30°Brix by vacuum evaporater and spray-dried with an inlet air temperature 180°C, air pressure of atomizer at 40 psig and outlet air temperature 100°C .

3.3.3.2 Preparation of lime powder with drying aid

3.3.3.2.1 Effect of kinds of drying aid

Glucose, sucrose and dextrin in an amount of 10% were added to fresh lime juice (8°Brix) and the lime juice was spray-dried under the same condition as described in 3.3.3.1.2

3.3.3.2.2 Effect of concentration of lime juice before drying

Fresh lime juice was concentrated to 15°, 20° and 30°Brix by vacuum evaporator and added with 30% dextrin and experiment was run at 140°C inlet air temperature, air pressure of atomizer at 40 psig and outlet air temperature 70-80°C

3.3.3.2.3 Effect of dextrin content

At first concentrated lime juice (30°Brix, by freeze concentration) was mixed with 10%, 20% and 30% dextrin and dried in the same condition as in 3.3.3.2.2.Later, the juice concentration used was lowered to 10°Brix before mixing with dextrin at 20, 25 and 30%. The resulting juice was spray dried at higher temperature (200°C) in order to get drier powder. Air pressure of atomizer was about 50 to 60 psig and outlet temperature was at 70°C.

Physical and chemical properties of products were studied. Observation and analysis were conducted on color, precentage of acidity, ascorbic acid content, pH, total soluble solid (^{o}Brix) and moisture content. Acceptability test was also carried out by taste panel.

3.3.3.2.4 Effect of inlet air temperature

Freeze concentrated lime juice (10^{o}Brix) was mixed with 20% dextrin and spray-dried at constant inlet air temperatures of 200, 250 and 300^{o}C at a constant air pressure of atomizer 50 to 60 psig. The products were observed and analysed as in 3.3.3.2.3

3.3.3.2.5 Experiments of 2^3 factorial design

Concentrated lime juice prepared by freeze concentration was used in this experiment. Lime juice concentration of 8^{o}Brix and 16^{o}Brix were mixed with dextrin at 8 and 16% and spray-dried with inlet air temperature at 150^{o}C and 250^{o}C. Air pressure of atomizer was kept constant at 50 psig and outlet air temperature was maintained at approximately 75^{o}C. Observation and analysis were conducted as in 3.3.3.2.3. Effects of variables were analysed and estimated according to Appendix I

3.3.4 Storage test

Fresh lime juice (8^{o}Brix) mixed with 20% dextrin were spray-dried using inlet air temperature 250^{o}C, air pressure of atomizer at 50 psig and outlet air temperature 80^{o}C. The powder

obtained was packed under 3 conditions. They were packed in aluminium foil with and without vacuum and packed under atmospheric condition in polyethylene bag. Packaged samples were stored at room temperature and quality changes of lime powder was followed periodically in term of ascorbic acid, acidity, optical density of reconstituted lime juice in distilled water at 7°Brix and moisture content.

3.4 Analytical Method

3.4.1 Determination of ascorbic acid content

The volumetric determination using 2,6 dichlorophenol indophenol, as described by Cox and Pearson (58) was used and the ascorbic content in the sample was calculated as mg ascorbic acid per 100 ml of lime juice.

3.4.2 pH measurement.

The pH of the lime juice was measured by using pH meter (3.2.6).

3.4.3 Total soluble solid (°Brix)

The total soluble solid was measured by using Hand Refractometer (3.2.5).

3.4.4 Determination of the optical density

The optical density of the juice was determined by using Prolabo Spectophotometer (3.2.7). The lime powder sample was

prepared by diluting with water in order to make 7°Brix lime juice, filtered, and the optical density of the supernatant was measured against water at 420 nm. The increase in the optical density indicates the increase in degree of browning of the juice.

3.4.5 Titratable acidity

Two ml. of lime juice was titrated against 0.1 N. NaOH using phenolphthalein as an indicator and calculated as percentage of anhydrous citric acid, the predominant acid in the lime juice. (Appendix II)

3.4.6 Moisture Content

The product was dried at 100°C until the weight was constant. Calculation of moisture content was carried out and compared with the weight of finished product.

3.4.7 Sensory evaluation test

Lime powder was dissolved in distilled water to get reconstituted lime juice (7°Brix). 10 ml of reconstituted lime juice was served to 7 untrained panel to evaluate the acceptability of lime powder. Scores were allotted to different liking on the Hedonic scale from 1 at'dislike extremely' to 9 at' like extremely'. The score of each characteristic were analysed. The testing panel form used is shown in Appendix III .