Chapter III

Experiment

3.1 Materials

3.1.1 Standard Material : Aerosil silica powder OX-50 produced by Degussa AG.

3.1.2 Sample Material : Rice husk ash produced during research work.

3.2 Equipment

3.2.1 The Equipment for Preparation of Rice Husk Ash

The equipment for preparation of rice husk ash (U.Leela-adisorn, 1992) consisted of a washing machine, a mantle heater, reflux condensor, commercial muffle furnace, grinding mill, and particle classifier. The details of each piece of equipments are described below :

A commercial washing machine (figure3.1) was used for cleaning the rice husk. The adhering soil and organic dust were eliminated from rice husk. The washing machine has two compartments. It has a bottom stirrer and a free-flow bottom drain. The other compartment is a spin dryer.

A mantle heater & reflux condensor was used for chemical treatment. The glass bulb flask of 12 l volume was heated escaping by means of a reflux condensor. The set-up is illustrated in figure 3.2. A check with pH paper showed that no HCl gas escaped from the condensor.

A commercial muffle furnace with accurate temperature control was used for incineration of the treated husk.

Grinding was preformed in two steps. In the first step, rice husk ash was grinding with a ball mill. In the second step, rice husk ash was grinded with an agate mortar and pestle.

The particle classifier was an equipment specificly designed (Conradt, 1993) for this research work. The idea of device came from air jet mill which unfortunately was not available. The particle classifier is composed of two long tubes, a filter bag, a vibrator, a flexible plastic pipe, a transformer, and an air flower. The set-up is illustrated in figure 3.3. The flexible plastic pipe was connected to the vibrator. The vibrator mechanically fluidized the ash in the flexible plastic pipe so that the air flow brought about an effective carry-over into the tube system. By the competition of gravity forces and friction forces of the ash particles in the air flow, a classification was obtained. Only a certain fraction (depending on the air flow) was carried beyond the upper U - turn and collected in the filter bag. The particle size depended on the speed of air flow and height of the classifier tube.

3.2.2 The Equipment for Sol-Gel Preparation

The equipment for sol-gel preparation consisted of a mixer, an ultrasonic bath, and plastic ware. The sol was prepared in the plastic ware by the mixer. The particles were dispersed by the action of the ultrasonic bath.

3.2.3 The Equipment for Gel Drying

The equipment for gel drying had two components. The first component generated an atmosphere with a well-defined H_2O particle preserve by means of an accurate temperature control. A small air pump passed on air flow through the water in the bulb, thereby saturating it with vapor at the aspective temperature T_1. The saturated air was transferred to the drying chamber through a transfer tube which was kept at $T_3 > T_1$ in order to avoid condensation. The second component was the drying chamber. It was kept at a well-defined temperature $T_2 > T_1$ so as to accurately control the relative humidity in the chamber. By this, both the absolute and relative humidity in the drying chamber could be varied independently.

The device was made for the use in this research work, as shown in figure 3.4. The device was composed of water bath, hot plate-thermometer control, air pump, drying chamber, and transformer. The gel samples in drying chamber were dried by hot vapor which had a high heating too.

3.2.4 The Equipment for Sintering

The equipment for sintering was a heating furnace which had a high heating rate.

3.2.5 The Equipments for Characterization

The equipments for characterization of the sample were SEM, XRD, a 5 decimal balance.

SEM (Scaning Electron Microscope) was equipment to study the microstructure and size distribution of the powders.

XRD (X-ray diffraction) was used to study the crystallinity of the samples.

The balance was used to determine the bulk density, water absorption, and porosity of samples. The balance was also used to measure the weight loss upon drying.

Shrinking was measured by a vernier.

3.3 Chemical Substances

3.3.1 The Chemical Substance for Treatment

The chemical substance for treatment of rice husk was 2.4 molar hydrochloric acid.

3.3.2 The Chemical Substance for Wet Grinding

The chemical substance for wet grinding in the ball mill was 50 % Isopropanol.

3.3.3 The Chemical Substance for Gel Preparation

The chemical substance for gel preparation were 1 % ammonium fluoride, 5 % boric acid, hydrochloric acid(conc.), and 28 % ammonium hydroxide solution.

The 1 % ammonium fluoride was used to make a sol of OX-50. The 5 % boric acid was used to make a sol of rice husk ash. Pyrogenic silica A200, hydrochloric acid and ammonium hydroxide were used to adjust the pH and to control the gel formation.

3.4 Supports and Molds

3.4.1 Molds

Molds were used for gel preparation such as styropor foam, and plastic ware.

3.4.2 Support

For sintering, a platinum wire was used as sample support. Ceramic supports easily induced heterogeneous crystallization and had to be avoided.

3.5 Method

3.5.1 Method of Preparing Silica from Rice Husk

The concentrations were selected after consulting Uraiwan's thesis.

3.5.1.1 Washing step : The rice husk was cleaned with tap water in the commercial washing machine. The amount of rice husk was about 500 grams. Then, the rice husk was dried by dry-spinning.

3.5.1.2 Chemical treatment : The clean rice husk was put into a 12 l. flask and boiled under reflux in 2.4 molar HCl, 3 h, at a ratio of 100 g husk/l. After chemical treatment, the rice was washed several times with tap water, and dried in a chamber at 110°C, 48 h.

3.5.1.3 Incineration : An incineration temperature of 600°C, 6h, in static air was chosen. The incineration took place in loosely packed beds of appoximately 3 cm height of stainless tray in a commercial muffle furnace.

3.5.2 Method of Classifying Particles of Rice Husk Ash

The ash has strong agglomeration which is not appropriate to make a gel.

3.5.2.1 Grinding : The rice husk ash was wet grinded with 50% isopropanol solution, 6h, in a ball mill, and dried in a chamber at 110°C.

3.5.2.2 Classifying : The ash, as received, was dry grinded with an agate mortar and pestle, taken to the particle classifier and exposed to an air flow. The ash was classified in this device for approx. 15 h. The rest ash (coarse fraction) in the plastic pipe was ground again.

3.5.3 Method of Sol-Gel Preparation

This method was studied with samples produced from OX-50, to find the ratio of mixing yielding a homogeneous gel. The appropriate ratio was used for rice husk ash, too.

3.5.3.1 The mass ratioes of solid (OX-50) and solution (1% ammonium fluoride solution) were varied as 40:60, 50:50, 60:40.

3.5.3.2 While mixing the solid and solution, the suspension had to be dispersed by the action of an ultrasonic bath.

3.5.3.3 To get a shaped body the suspension was pored into a mold where it coagulated within 15 h. The mold used for casting of gel, was made of foam, plastic, or any other mold which did not absorb water.

3.5.3.4 To prevent cracking caused by interaction between the green body and the mold, the stiffened suspension has to be removed from the mold before drying. Rice husk ash was to be removed from the mold before drying.

3.5.3.5 Rice husk ash was found to have a poor to only fair dispersion in water. So, it required a binder, which does not add inpurities to the system. Pyrogenic silicas; such as OX-50, Aerosil-200, Aerosil - 300, in amounts of 10-50% were used as binder. In addition to this, HCl and NH_4OH were used to adjust the pH of the sol.

3.5.3.6 The binders were mixed in 5% H_3BO_3 before rice husk ash was added following 3.5.3.2, 3.5.3.3, 3.5.3.4.

3.5.4 Method of Drying

This method was studied with gel sample produced from OX-50 first. Drying is an important part of a number of manufacturing processes. If the air is too hot of too dry when the object is placed in the chamber, undesirable cracks are generated in it, and it must be discarded. The role of the support was studied too.

3.5.4.1 Humidity control : The gel samples were placed into the humidity control device at 0, 30, 50, 70, 90% relative humidity, to study weight loss, shrinkage, and cracking after drying. The appropriated method was humidity control which used for drying of all gel samples made from rice husk ash.

3.5.4.2 No humidity control : Put the gel samples at 70, 90, 110, 150, 200, 300 °C in the chamber.

3.5.5 Method of Sintering

3.5.5.1 Dried sample of OX-50 were sintered at temperature 1100, 1150, 1200, 1250, 1300, 1350, and 1400°C in an electrical furnace.

3.5.5.2 Dried sample of rice husk ash at temperature 1250, 1300, 1350, 1400, 1450, and 1500 °C in heating electrical furnace.

3.5.6 Method of Characterization

3.5.6.1 Powder

I SEM : microstructure, dispersion

II XRD : amorphous / crystalline

III ASTM : density

3.5.6.2 Gel

I Measurement : shrinkage

II Weighing : mass loss

III XRD : amorphous / crystalline

IV SEM : homogeneous, microstructure

3.5.6.3 Sintered gel

I SEM : microstructure

II XRD : amorphous / crystalline

III ASTM : density

IV Dilatometer : thermal expansion

Flow chart of process in thesis work

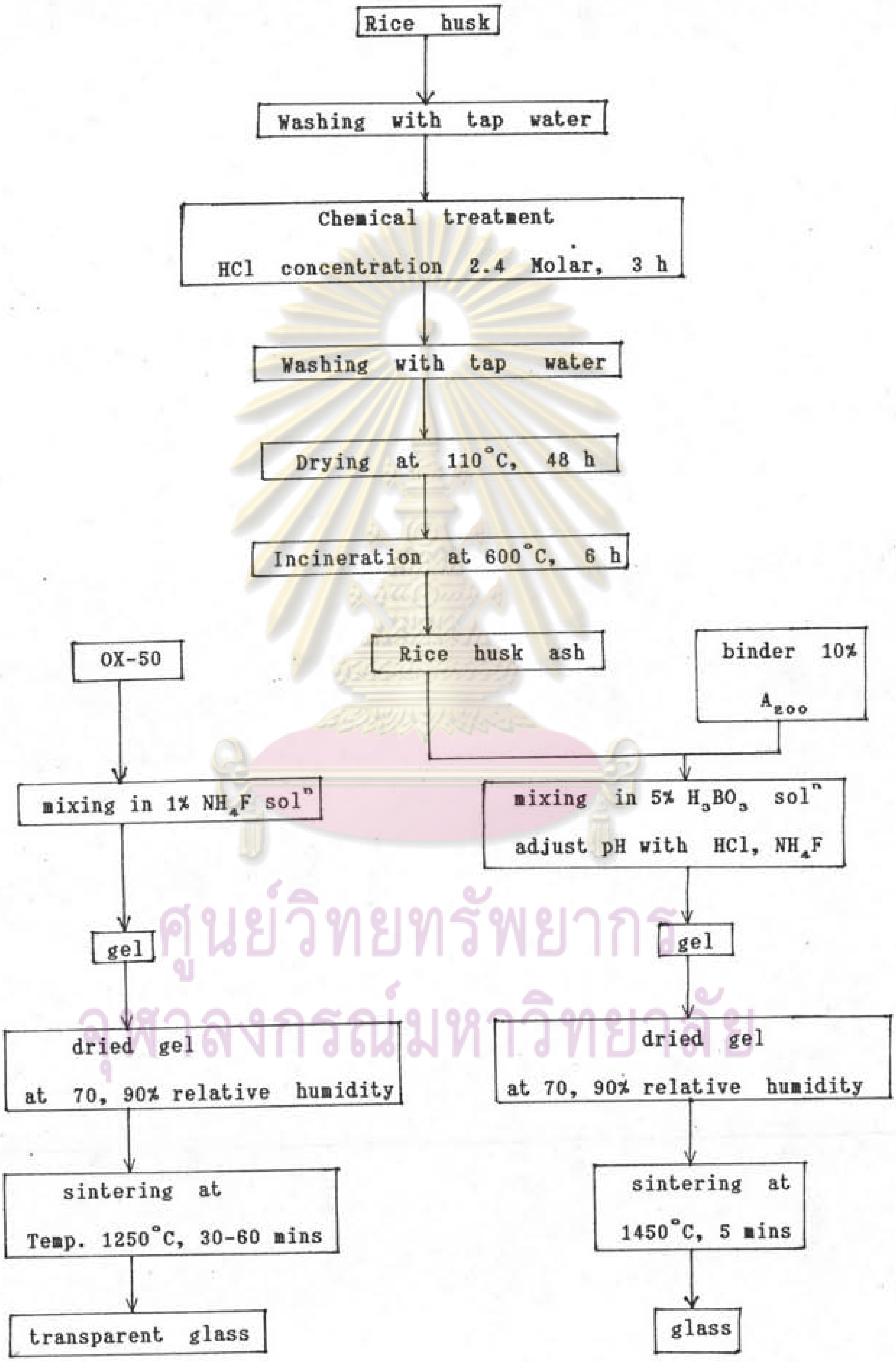

Table 3.1: Comparison of materials between standard (OX-50) and sample (rice husk ash)

material	produced	cost	$\%SiO_2$ content	density g/cm^3	shape
OX-50	flame hydrolysis	high	>99.8	2.20	sphere/colloid
rice husk ash	chemical treatment	low	>99.8	~ 2.20	sphere/colloid

avg. size nm	XRD peak (crystal)	sp.surface BET (m^2/g)	agglomeration	dispersion in in water
40	none	50 ± 15	weak	excellent
3-30	none	220 ± 30	strong	fair

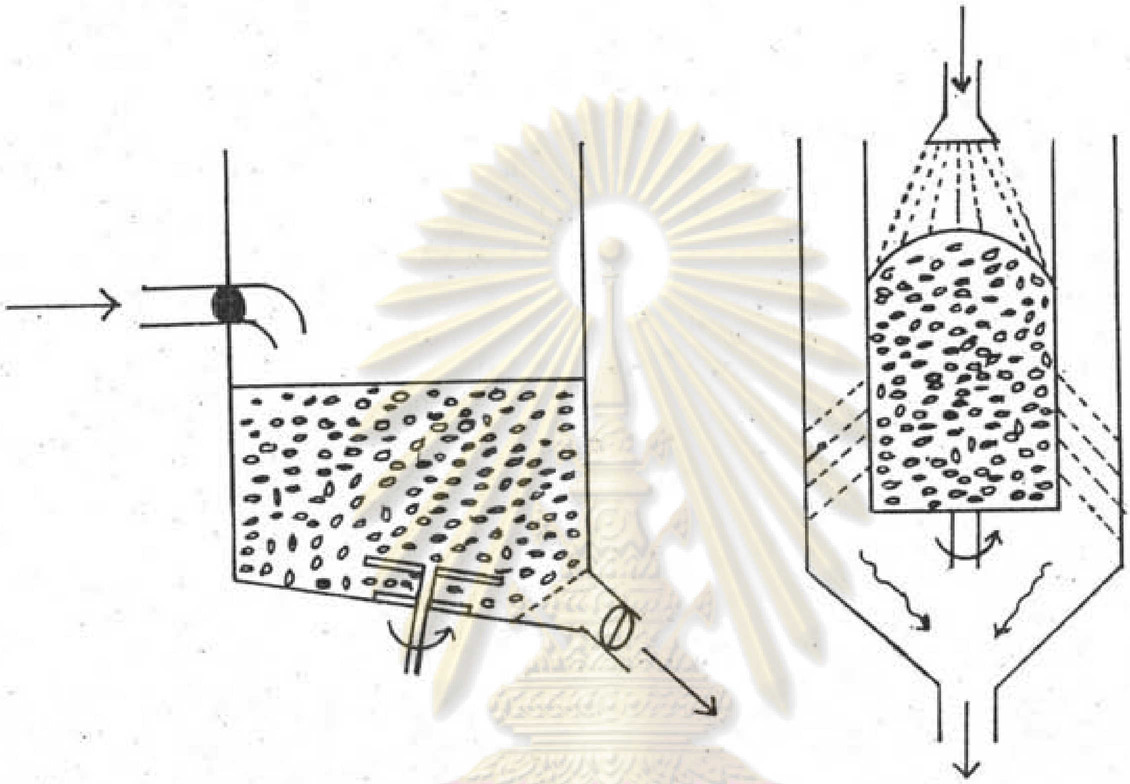

Fig. 3.1 : Washing machine

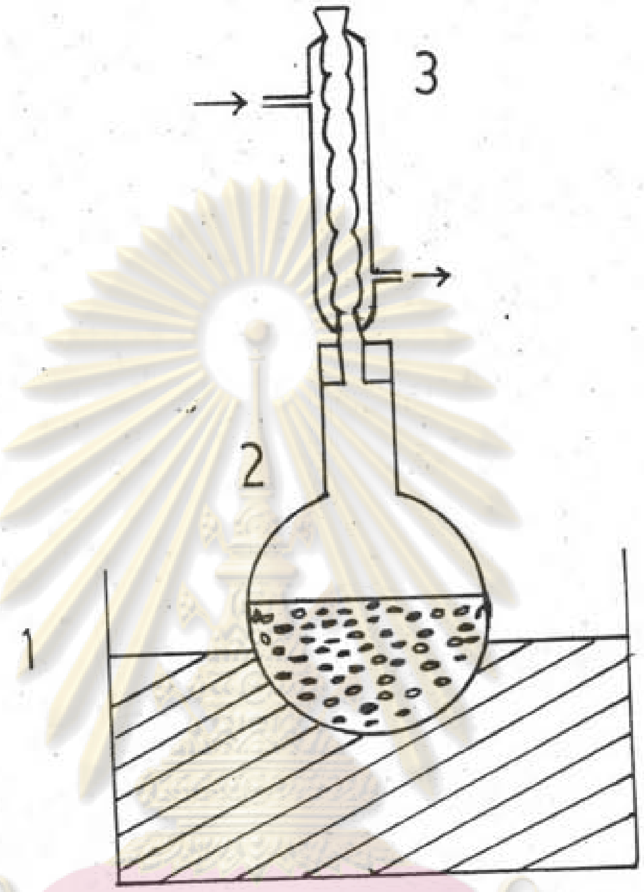

Fig. 3.2 : Sketch of equipment for chemical treatment

1. mantle heater;
2. bulk flask;
3. reflux condencor unit

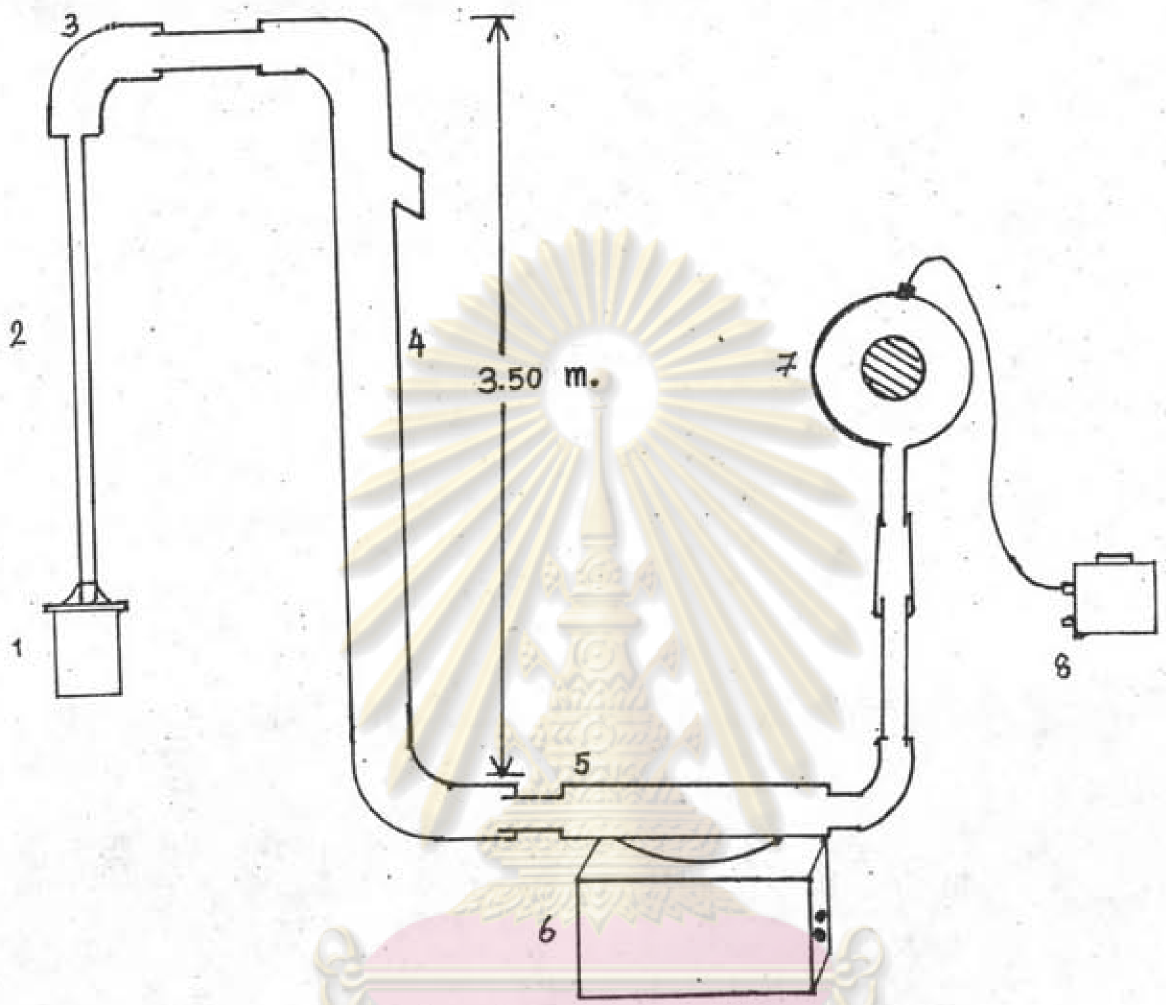

Fig. 3.3 : Sketch of particle classifier

1. filter bag;
2. collection tube (diameter 1.0 inch);
3. upper U-turn;
4. classifier tube (diameter 2.0 inch);
5. flexible plastic pipe;
6. vibrator;
7. air flow machine;
8. transformer

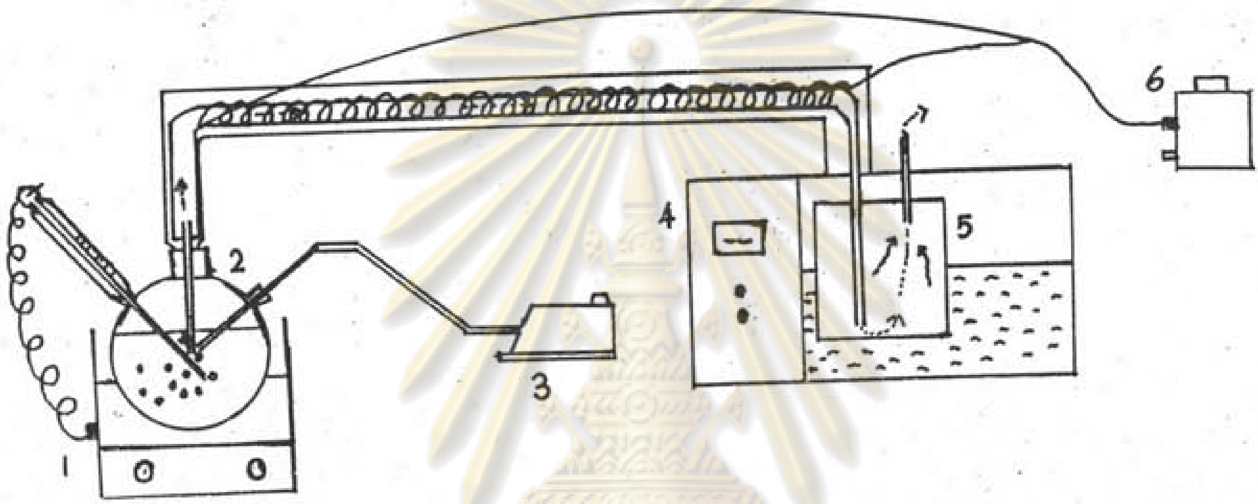

Fig. 3.4 : Sketch of equipment for gel drying

1. hot plate - thermometer control;
2. bulk flask;
3. air pump;
4. water bath;
5. drying chamber;
6. transformer