Chapter 2

Experimental Methods

2. Sample preparation

2. 1 Materials properties

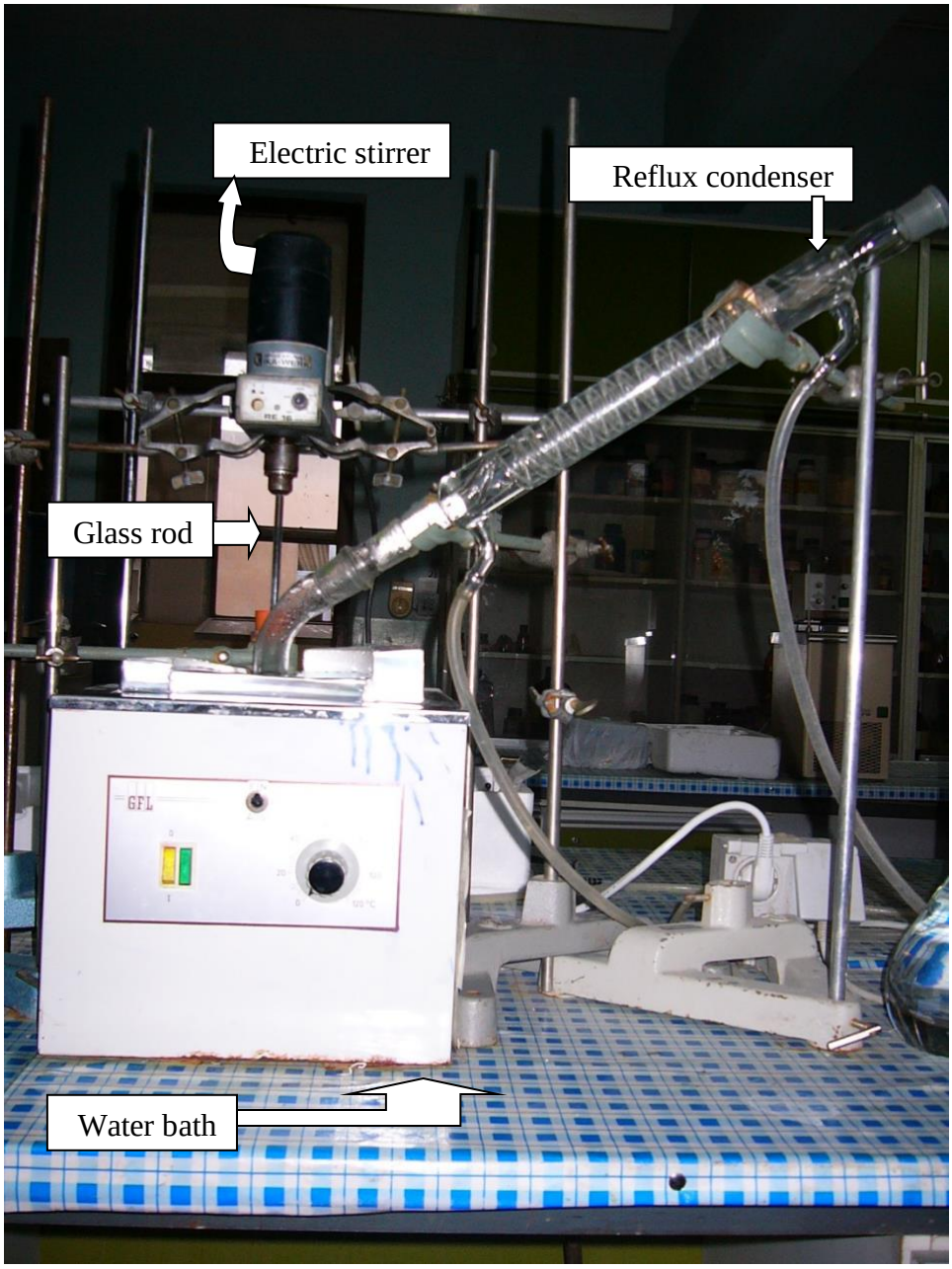
Kaolin, which is an abundant material in Iraq, is used as a starting materials in this study . The chemical composition XRD data for the kaolin used in this work are given in chapter (3). Barium Chloride, which is added as $BaCl_2 \cdot 2H_2O$ was used as stabilizing materials and added through the process of preparation. Kaolin powder was calcined at 800 °C using electric muffle furnace, for 3 hours time. After cooling down to a room temperature, the calcined powder grind in a porcelain ball mill, and then screened to have samples of different particle size range (< 40μm, < 75μm). Sulpharic acid produced by BDH company, having the specification :

(i) density was (1.84 gm/ ml), (ii) molecular weight (98.08 gm/mole) and concentration assay 98% , been used in this process after determination of accurate concentration equal to 95.27 % (35N), prepared according to the analytical method.

2. 2 Experiment System and Design

Figure.(2-1) shows the experimental set up used for extraction of Al_2O_3 from kaolin .The system consists of the following parts, each with its specification and operational manual : (1) Water bath, model 1012 from Germany made , working at a power of (1000 W) of stainless – steel bath of capacity : (10 L). The liquid solution temperature inside the bath can be controlled from room temperature to a maximum of 100°C. The alcohol thermometer was used to measure the solution temperature . (2) The electric stirrer type : RE16 IKA-WERK, Japann made, was used to stirring the mixture . The stirrer speed can be controlled from 100 – 2000 r.p.m. . (3) The balance of W-Germanian type, having a sensivity of a rang (110gm - 0.1 mg) was used to measure the powder weight . The capacity of the used burette was (50 ml), to dropping the leach liquor into the ethanol . A (chibna) (8 and 20 mm) for internal and external diameters, respectively, is used as a barrier to prevent the escape of the solute vapor through the existence of disconnection between the throught of the reaction flask and the glass rod used for stile the solution , as that shown in Figure (2-2) . (4) The rod have a diameter of (7.8 mm), it is passed through the flask

throat for a purpose of solution homogeneity by means of mechanical stirrer. As shown in the same figure , (5) An elastic stopper size No.(8) cylindrically holed in a deep of ¾ of it's length and the two (chibnas) fitted sitting inside each of the other . Reflux condenser, consist as a form of coiled glass tube inside the other ordinary glass tube Figure.(2-1) . The two ends for both tubes are opened to reflect the evaporated solution . During the desired time of reaction the cold water continuously passing through the ordinary glass tube around the coiled glass tube. (6) The Box type Electric Resistance Furnace Figure. (2-12) Model SX2-512 No.60 was used for calcination . This furnace can be heated up to a temperature of 1200°C and a general stability to about ±(5) °C .



Figure(2-1) The Experimental set up .

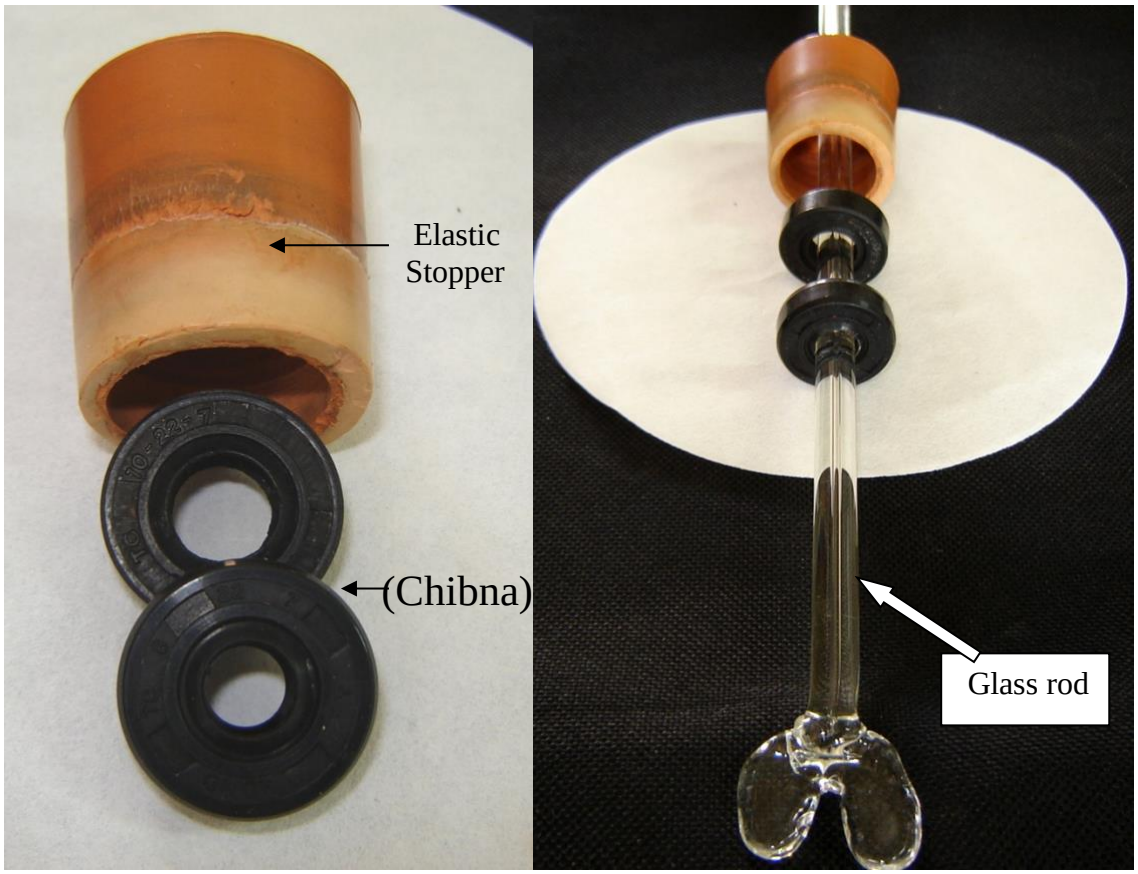


Figure (2-2) The glass rod, chibnas, and elastic stopper.

2. 3 Sample preparation

For preparating the alumina, Figure (2-3). Two different methods been used. The first is carried out with respect to the precipitation while the second followed the evaporation processes.

In the first method the diagram (2-1) shows the γ- Al_2O_2 powder preparation procedure while Figures (2. 4 - 12) showing the process as photographically figures. The synthesized γ- Al_2O_2 powder was prepared by calcining aluminum sulphate powder, Figure. (2- 11and 12) .

Figure (2-3) Some typical prepared powder samples and there residue precipitate.

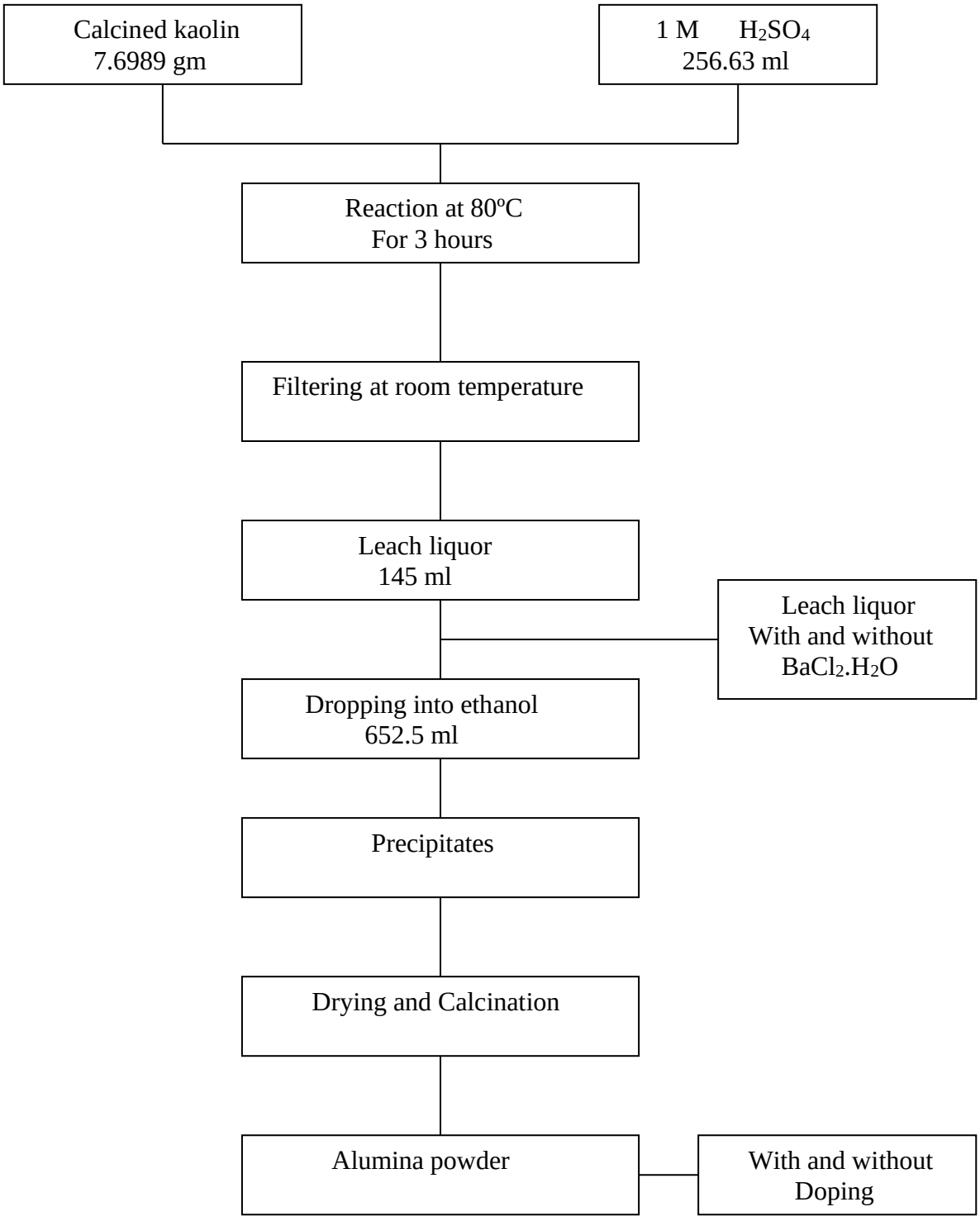
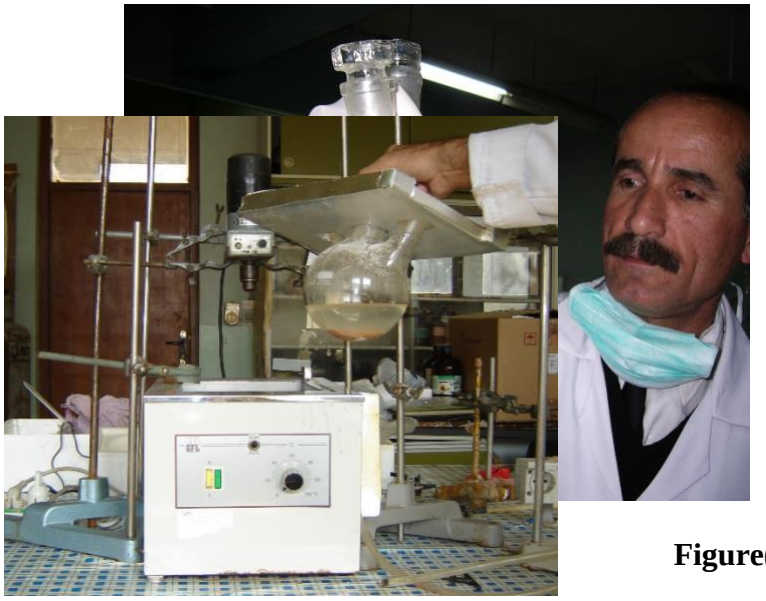


Diagram 2-1: Flow chart of γ -Al_2O_3 and BaO doped alumina powders.



Figure(2-5) The product solution after 3hr duration reaction in flask of (volume 1000 ml).

rature .

Figure(2-7) (a) Aluminum sulphate solution and (b) Precipitate separated.

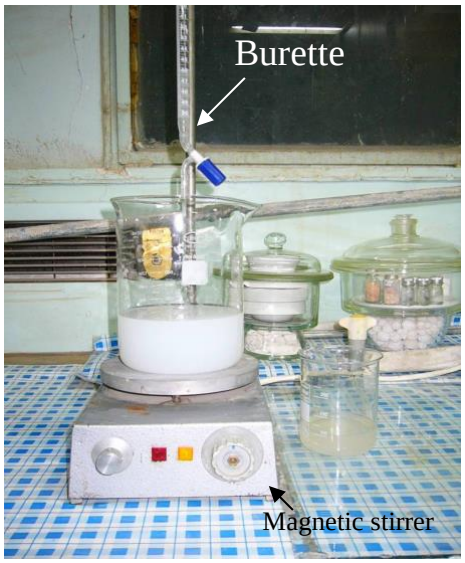


Figure (2-8) Droping into ethanol.

Figure (2-9) Precipitate separation by filtration.

Figure (2-10) Drying at 80˚C.

Figure (2-11) Precipitate as synthesized.

Figure (2-12) Calcinations furnace.

The aluminum sulphate solution Figure.(2-7a) prepared in Pyrex glass flask reactor, immersed in water bath fitted with a thermometer as well as a reflux condenser as in Figure.(2-4). So as, amount of 256.63 ml of 1.0M H_2SO_4 solution with 7.6989 gm of calcined kaolin powder were mixed using a reaction flask, Figure.(2-5), then heated up to 80°C been continuously mixing by electric stirrer of (500 r.p.m.) as shown in Figure (2-1). This reaction process was carried out for two duration times (3 and 6) hours, respectively . After the solution been cooled down to the room temperature the leach residue was separated by filtering the solution, as in Figure (2-6) and the aluminum sulphate solution (leach liquor) obtained Figure.(2-7a) . A (145 ml) leach liquor been added to a (652.5 ml) of ethanol with rate of (5 ml m^{-1}) within stirring rate of (500 r.p.m.) using magnetic stirrer as shown in Figure.(2-8). However, the precipitation was occurred immediately when adding the leach liquor to the ethanol and separated by filtration as in Figure.(2-9) and product (precipitate) , washing again with ethanol then dried in an oven at a temperature of 80°C for a period of 24 hours, Figure.(2-10). The dried precipitated material powder, Figure.(2-11), was calcined at various temperatures Figure. (2-12) .

The BaO doped alumina powder, were synthesized through the precipitation mixing process diagram (2-1) by adding barium chloride powder ($BaCL_2.2H_2O$) to the prepared aluminum sulphate solution (leach liquor) in which the BaO content (wt) % with respect to the calculated Al_2O_3 content in the aluminum sulphate solution was adjusted in the rate given in table (2 - 1) .

Table (2 – 1) : Nominal composition of sample prepared from Aluminum sulphate solution and barium chloride.

Samples	**Mole ratio**	**BaO , wt % based On Al_2O_2 = 100**
T_3 , T_9	**BaO.75Al2O3**	**2.0**
T_7 , T_8	**BaO.30Al2O3**	**5.0**
T_4 , T_{10}	**BaO.2 5Al2O3**	**6.0**

Diagram (2-2) and Figures (2-13) to (2-20) showing the second method process for preparation of (γ-Al_2O_2) powder. The same experimental system been used again for preparation of the product reacted solution except the change of flask from that of (1000 ml) to another of (250 ml). A (25gm) of calcined kaolin reacted with (150.8 ml) of 5.9N H_2SO_4 that gives a solid to liquid ratio of 166 gm/litre .

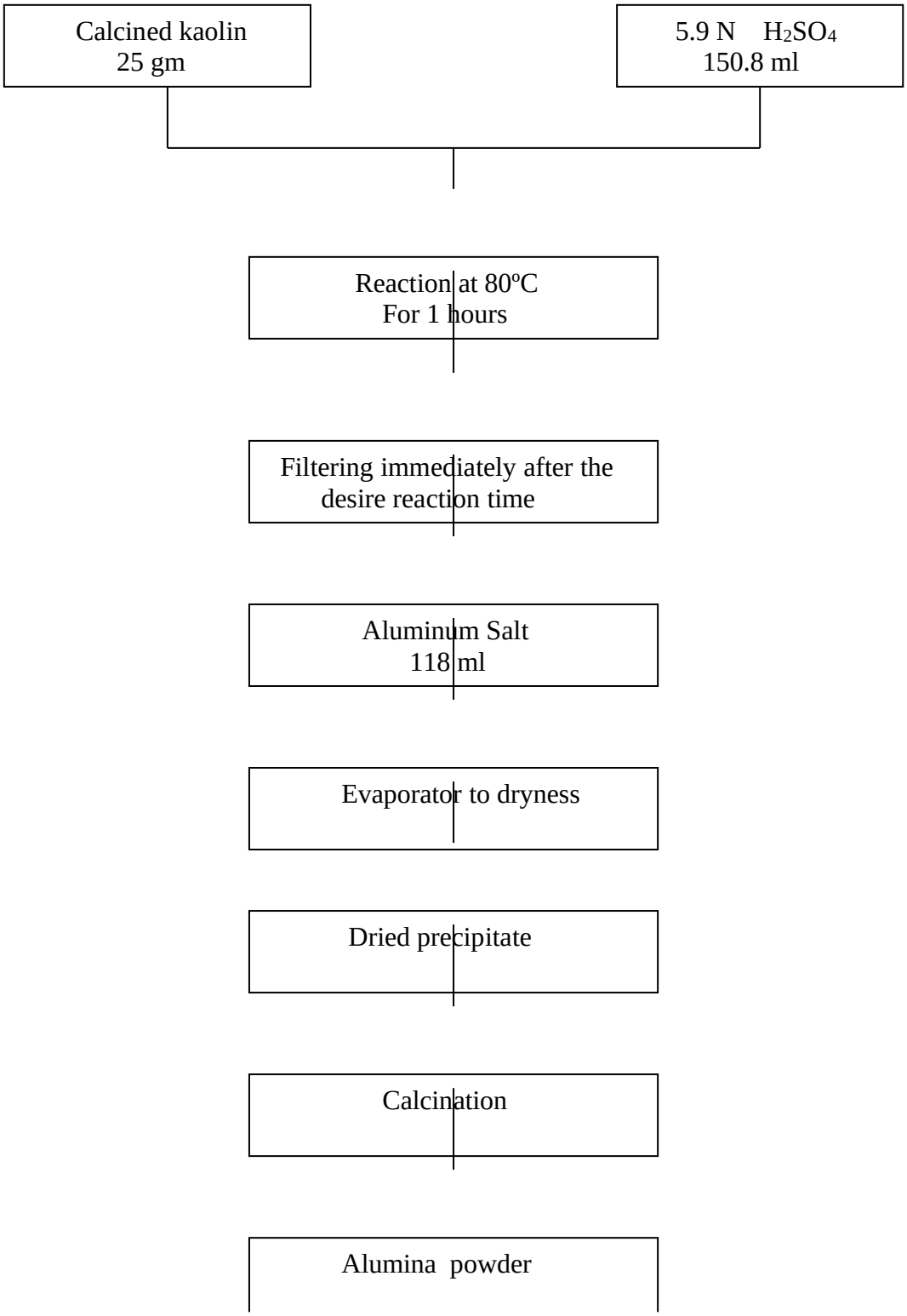


Diagram 2-2 : Flow chart for synthesis of alumina due to evaporation method.

Figure(2-14) Filtration after the desire reaction time , immediately filtrated .

ion
num salt .

Figure(2-13) The solution after 1h reaction .

Figure(2-16) Evaporation at the1st hour.

Figure(2-17) Evaporation at the12th hour .

Figure (2-18) Washing with ethanol.

Figure (2-19) Filtration (clean precipitate).

a

Figure(2-20) (a) Precipitate separated from leach Liquor . (b) Precipitate as-synthesized .

The experimental procedure has been conducted for a duration reaction of 60 min with a reaction temperature of 80°C Figure (2-1). During the experiment, the suspension was constantly stirred with a stirrer speed of 1100 r.p.m. . After the desire reaction time Figure (2-13) , the suspension was immediately filtered Figure.(2-14). The filtrate materials Figure (2-15) is thermally treated, (Figures (2-16) to (2-17)), to a dry purpose . The residue was washed with ethanol , Figure.(2-18) hence after filtration a clean precipitate was obtained , Figure (2-19) . The precipitate was ovened for 20 hours duration for dryness. Figure (2-20b) , shows the precipitate as synthesized. In order to form aluminium oxides from the aluminum sulphate salt the residue was calcined by calcinations furnace at various temperatures.