CHAPTER III

EXPERIMENTAL METHOD

3.1 Chemical and materials

Titanium isopropoxide was purchased from SIGMA-ALDRICH

Polyvinylpyrrolidone (average Mw - 1,300,000) was purchased from SIGMA-ALDRICH

N,N'-Dimethylformamide was obtain from Fluka

Ethanol was purchased from MERCK

2-butanone was purchased from CARLO ERBA

Acetone was purchased from RCL Labscan

Methane gas was purchased from Thai Industrial Gas Co., Ltd.

Methanol was purchased from MERCK

Silver paint was purchased from SPI SUPPLIES

3.2 Experimental procedure

3.2.1 Substrate preparation

Print circuit board (PCB) was used as substrate for gas sensor. PCB was acquired from Innotech PCB Enterprise Co., Ltd. Substrate size was 1.27 × 2.54 cm^2. Gap between Cu electrodes was 0.35 mm. PCB was burnished by rubber to eliminate oxide on the Cu surface. After that, acetone was used to polish the electrode surface.

3.2.2 Preparation of TiO_2 nanofibers

3.2.2.1 Preparation of TiO_2 solution for electrospinning

The method for TiO_2 electrospinning solution preparation was modified from Park *et al.* report [30] which composed of titanium isopropoxide, 2-butanone, polyvinylpyrrolidone, *N,N'*-dimethylformamide, and ethanol at various ratio. Mixture of 6 g of titanium isopropoxide and 2-butanone was mixed with 0.4 g of polyvinylpyrrolidone. Then, 0.1 mL of *N,N'*-dimethylformamide and 0.2 mL of ethanol were added to the mixture. All of the mixtures were stirred at 60 °C for 3 hours after which a homogeneous solution for electrospinning process was obtained.

3.2.2.2 Fabrication of TiO_2 nanofibers

The electrospinning solution was loaded into a 10 mL plastic syringe. The needle tip and a collector were connected to positive and grounded electrode of high voltage power supply, as shown in **Figure 3.1**. Parameters of high voltages, distances between needle tip and collector, and feeding rate were studied to optimize the condition for gas sensor application, as presented in **Table 3.1**.

Table 3.1 Studied parameters of electrospinning process

Parameters	Range
Voltages (kV)	12 – 18
Distances between needle tip and collector (cm)	20 – 30
Feeding rate (mL/h)	0.6 – 1.2

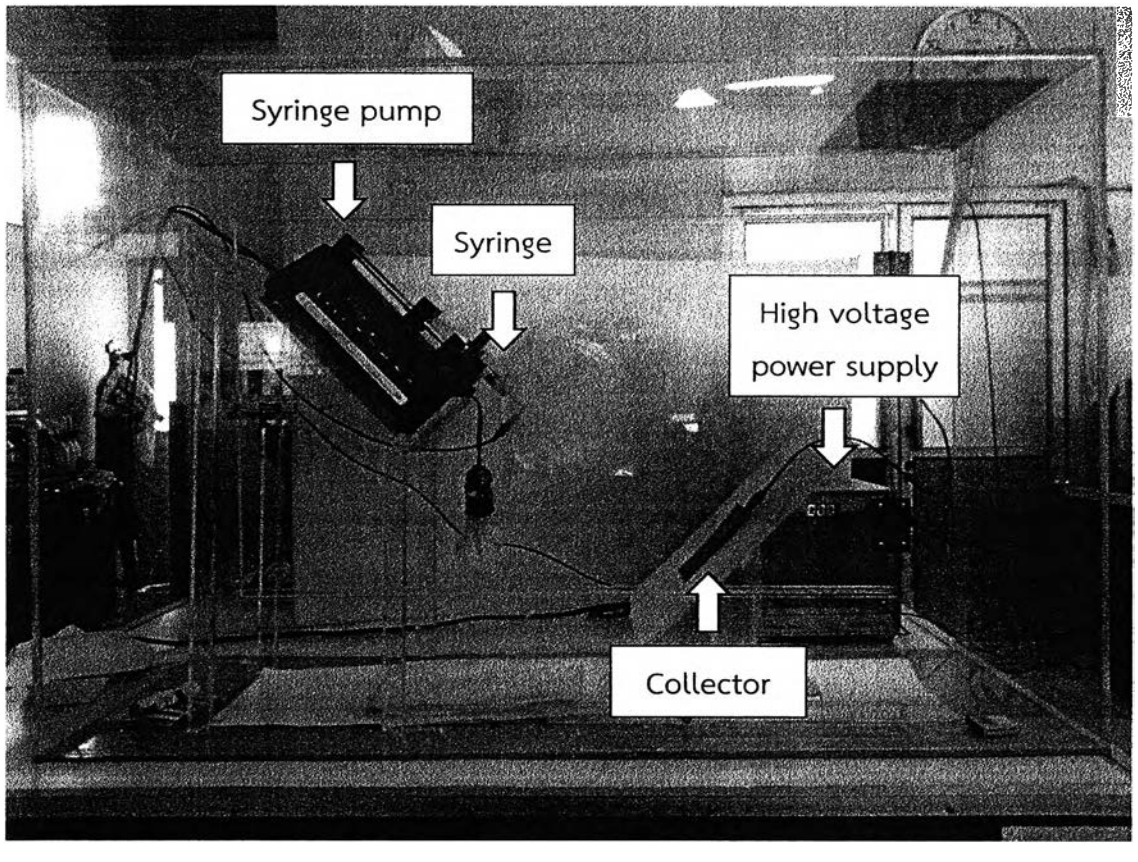

Figure 3.1 Assembly of electrospinning system

Afterwards electrospun TiO_2 nanofibers were obtained, they have been hot-pressed at 180 °C at pressure of 2 kg/cm^2 for 10 minutes. Hot-pressed electrospun TiO_2 nanofibers were then calcined at 600 °C for 1 hour. Finally, TiO_2 nanofibers as sensing materials were obtained.

3.2.2.3 Fabrication of TiO_2 nanofibers for gas sensor

Sensing materials was placed on PCB substrate. Few droplets of de-ionized water were dropped on the sensing materials. Thereafter, silver paint was used to adhere the sensing materials and substrate. Finally, specimen was heated at 60 °C to remove organic solvent.

3.3 Characterization of TiO_2 nanofibers

3.3.1 Scanning electron microscopy (SEM)

Morphology and diameters of TiO_2 nanofibers were observed by scanning electron microscope, JEOL model JSM-6480LV at Faculty of Science, Chulalongkorn University. JMicroVision 1.2.7 program was operated to investigate diameters of nanofibers.

3.3.2 X-ray diffraction analysis (XRD)

Crystal structure of TiO_2 nanofibers were investigated through Rigaku model DMAX 2200/Ultima+ at Faculty of Science, Chulalongkorn University. Moreover, matching search of nanofibers phase structure was also determined to attest the position of two-theta peak.

3.3.3 Brunauer-Emmett-Teller surface area analysis (BET)

Surface area of both unpressed and hot-pressed electrospun TiO_2 nanofibers was measured via Thermo finnigan model Sorptomatic 1990 at Petroleum and Petrochemical College, Chulalongkorn University.

3.3.4 Contact angle measurement

Contact angle of both unpressed and hot-pressed electrospun TiO_2 nanofibers was measured through contact angle machine and contact angle measurement software.

3.3.5 Sensing performance of gas sensor

In this research, gas detection system consisted of Agilent 4284A LCR meter, computer, and in-house chamber, as shown in **Figure 3.2**. Agilent 4284A LCR meter was operated at G-B function, 25 Hz of frequency, and 2 V of level. The correction of open and short circuit has been examined before measurements. Program in LabVIEW

virtual instrument environment was written to display data from LCR meter. Acrylic box was used to limit external air. Humidity at 99 % was generated via built-in fan. 4 W of UV light was installed inside of 2 L of metal box. Gas sensor was placed below the UV light about 2 mm. Light intensity was 233 $\mu W/cm^2$ which measured by UV light meter model UV340B.

Acetone, methane, and methanol at various contents were detected through gas detection system. Conductance of the gas sensor which was excited via UV light before and after the flow of the target gases were calculated to indicate sensitivity of gas sensor.

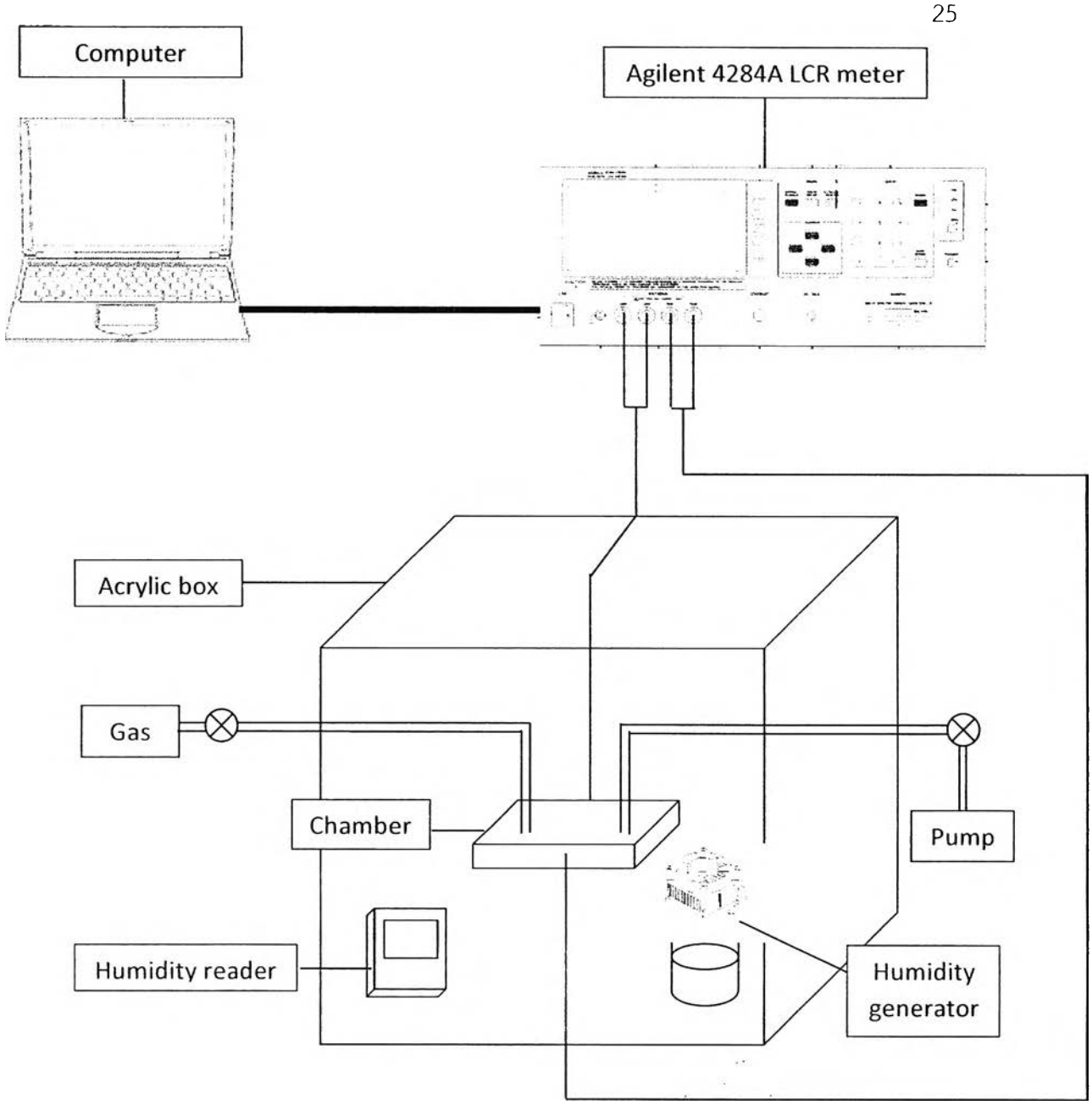

Figure 3.2 Gas detection system of this research