3 MATERIALS AND METHODS

3.1 Chemicals

The chemical reagents used in this experiment were thio-urea, cadmium nitrate tetrahydrate, cadmium sulfide (CdS) powder, bismuth III nitrate pentahydrate, sodium nitrite, potassium hydroxide and sodium sulphite. All chemicals were ~99% purity obtained from Sigma, USA.

3.2 Preparation of photocatalysts

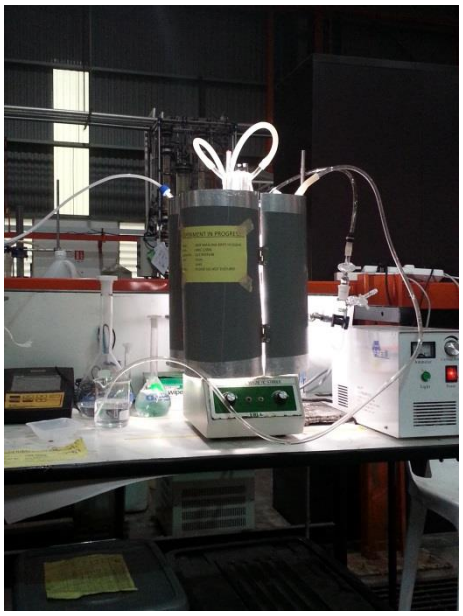
The Bi_2S_3/CdS catalyst was prepared by following direct hydrothermal method. $Cd(NO_3)_2.4H_2O$, $Bi(NO_3)_3.5H_2O$ and thio-urea of different compositions such as 6.02, 3.05 and 2.26 g, respectively were considered for the preparation followed by hydrolysis with 600 mL of deionized water in an autoclave at 90-100℃ for 6 h. Then cooled down to room temperature and successively the precipitate was filtered off, washed with distilled water and dried in an oven at 60℃ overnight. The catalyst was grinded with mortar before calcined at 250℃ for 3 h. Similarly, the weight proportions of Bi_2S_3 to CdS were 15% Bi_2S_3/CdS, 30% Bi_2S_3/CdS and 45% Bi_2S_3/CdS were prepared with the same method.

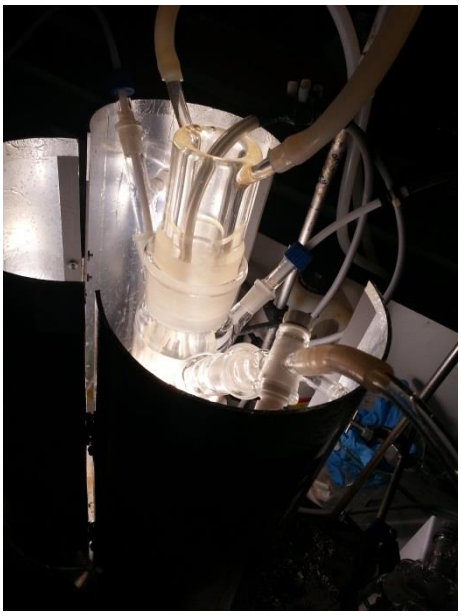
3.3 Characterization

The X-ray diffraction (XRD) patterns were obtained at room temperature using MSAL-XD2 diffractometer with Cu K_α radiation (operated at 36 kV and 30 mA, λ = 0.15406 nm). The Fourier transform infrared spectroscopy (FTIR) patterns were also obtained for both sample and products.

3.4 Photocatalytic experiment

The photocatalytic experiment was performed in a photochemical reactor equipped with a magnetic stirrer, a quartz cool trap and a condensation tube. A 500 W Xe lamp was located in the quartz cool trap as illuminant. The UV light was removed by 1.0 M sodium nitrite solution. Sodium nitrite, potassium hydroxide and sodium sulphite of corresponding quantities 20.701, 1.225 and 3.789 g were dissolved in 300 mL of ultrafiltered water. The solution was then put into the photochemical reactor. Ultrapure CO_2 was bubbled through the solution in the reactor before irradiation for 30 min to ensure that all dissolved oxygen was eliminated. 0.2 g of catalyst powder was added into the solution and the irradiation lamp was turned on to start the photoreaction. The temperature with the range of 30-35℃ was observed for every 1 h to avoid the loss of methanol into the air. Ultrapure CO_2 was continuously bubbled through the solution during the whole irradiation for 6 h. A needle-type probe was inserted into the solution of the reactor with the aid of vacuum pump to withdraw a small amount of liquid samples for 1 h interval up to 6 h. The concentrations of methanol in the samples were analysed using a gas chromatograph flame ionization detector (GC-FID).





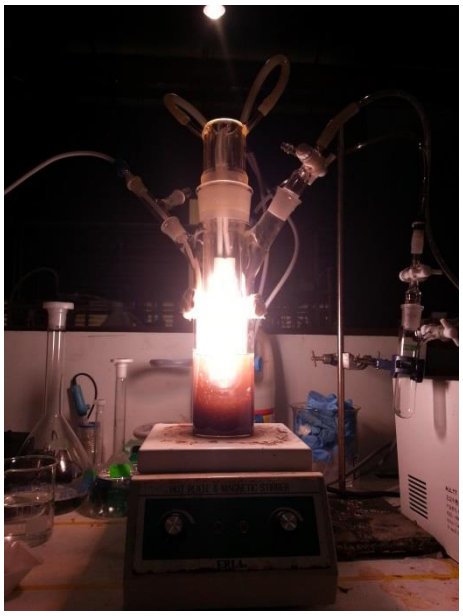


Figure 3-1 Illustration on how the experiment were done