3 MATERIALS AND METHODS

3.1 Chemicals

The reagents that were used in the hypercrosslinked reaction are anhydrous 1,2-dichloroethane (DCE) (99.8 % grade), methanol (99.7 % grade) and iron (III) chloride (97 % grade). They were supplied by Sigma-Aldrich and were used as received.

3.2 Equipments

The hypercrosslinked polymers are performed in a three-necked, round-bottomed flask fitter with a condenser, overhead stirrer and two-blade PTEE-type stirrer. The reaction vessel was heated up using heating block at 80 °C.

3.3 Methodology

The non-aqueous dispersion (NAD) precursor (1.5 g) was added into a round-bottomed flask which contains anhydrous 1,2-dichloroethane (DCE) (40 mL). Then, left it to swell fully under the nitrogen for 1 hour. After that, iron (III) chloride (FeCl₃) (in a 1:1 molar ratio of CH₂Cl: FeCl₃) is suspended in DCE (40 mL) were added. The mixture was heated up rapidly to 80 °C. The reaction was continued for 18 hours with continuous stirring over the entire reaction. The hypercrosslinked particles were filtered using vacuum filtration on a 0.22 µm nylon membrane filter. They were washed with MeOH and several times with aqueous HNO₃ (pH 2). Then, they were extracted overnight with acetone in a Soxhlex Extractor and dried vacuo (60 mbar) at 40 °C (Šálek & Horák, 2011).
3.4 Apparatus set up

Figure 3-1 shows the apparatus is set up to run an experiment. All reactions are performed under a nitrogen atmosphere. The nitrogen circuit included a flask with silica, to absorb any moisture present in the system. A multi-necked flange is placed on top of a round-bottomed reaction vessel and a metal clasp is used to secure the system. An overhead, four-bladed Teflon stirrer is used to agitate the system. The position of the stirrer is highlighted in order to ensure the system is agitated gently and efficiently. The position of the stirrer should not touch the bottomed of the reaction vessel to avoid the abrasion of the particle. If the stirrer is set too high, the agitation will result inefficient.
Figure 3-2: 0.22 μm nylon membrane is used to filter the hypercrosslinked particles.

Figure 3-2 shows 0.22 μm nylon membrane is used to filter the hypercrosslinked particles.

3.5 Characterization of hypercrosslinked particles

3.5.1 Fourier Transform Infrared Spectroscopy (FTIR)

FTIR analysis was carried out in order to identify the functional groups in the hypercrosslinked particles. FTIR relies on the fact that the most molecules absorb light in the infra-red region of the electromagnetic spectrum. This absorption corresponds specifically to the bonds present in the molecule. The frequency ranges are measured as wave numbers typically over the range 4000 – 600 cm\(^{-1}\).

The background emission spectrum of the IR source is first recorded, followed by the emission spectrum of the IR source with the sample in place. The ratio of the sample spectrum to the background spectrum is directly related to the sample's absorption spectrum. The resultant absorption spectrum from the bond natural vibration frequencies indicates the presence of various chemical bonds and functional groups present in the sample. FTIR is particularly useful for identification of organic molecular groups and compounds due to the range of functional groups, side chains and cross-