

CHAPTER 5

CONCLUSION

5.0 Conclusion

The chemical functionalization was successfully anchored acidic functional groups on the functionalized samples to enhance mercury (II) removal from aqueous solution. The optimum condition of treatment was found to be at 200 °C for 2 hrs that creates the highest number of acidic functional groups on the MWCNTs samples. At this temperature, the gas phase (B7) exhibits the highest number of acidic site as compared to liquid phase treatment (B10) samples. The IR studies showed the formation of oxygen and sulphur containing functional groups. The BET method shows the gas phase has the highest specific surface area than liquid phase functionalized samples and all samples exhibit mesoporous surface characteristic. From the BJH method, the pore size distribution of mesopores is high in the gas phase than liquid phase treated sample. The HRTEM images shows MWCNTs were hollow and tubular in shape. The agglomeration and the presence of a thick layer amorphous carbon were observed on pristine MWCNT samples. Purification using nitric acid was loosely packed and open end of MWCNTs, also the amorphous carbon layer was removed. The XRD diffractogram of liquid phase treated samples exhibits the highest intensity of graphite peak than the gas phase treated samples indicating that fewer defects created on the wall layer of liquid phase samples. Liquid phase treatment improved the order of the graphitic character of the carbon wall. Raman spectrum of the gas phase samples ($I_D/I_G = 1.59$) exhibits the highest number of I_D/I_G ratio compared to the liquid phase samples ($I_D/I_G = 1.16$). This indicates that gas phase treatment has created high degree of defect on the sidewall of MWCNTs as compared to liquid phase treatment. The gas phase (B7) exhibits excellent surface characteristic such as highest acidic functional groups, greatest specific surface area and highest mesopores size distribution than liquid

phase (B10) functionalized MWCNTs sample. This two adsorbent sample and raw MWCNTs were tested for the adsorption performance.

In the equilibrium adsorption studies, result on the adsorption of mercury (II) on pristine, gas phase (B7) and liquid phase (B10) functionalized MWCNTs showed that the adsorption process was dependent to the initial mercury (II) concentration. In this study the pristine MWCNTs perfectly fit Freundlich Isotherms while the B7 and B10 were well fitted the Langmuir Isotherm. The B10 adsorbent sample exhibits the highest maximum sorption capacity (q_m) value of 135.04 mg/g than B7 sample. Two postulates are proposed to explain the highest adsorption capacity achieved by B10 adsorbent sample. The first postulate, the oxidation of B10 using HNO_3 and H_2SO_4 by liquid phase treatment tends to increase oxygen functional groups such as hydroxyl group (O – H) on B10 adsorbent surface. As observed from IR result, the presence of strong broad unbounded hydrogen (free hydroxyl, OH:OH) group and hydroxyl group (O-H) on the B10 adsorbent surfaces helps the B10 adsorbent become better hydrophilic which in turn promotes the formation of water cluster on B10 adsorbent surface. In contrast, the presence of sulphur functional (sulfonates ester) group such as $\text{R-SO}_2\text{O}^-$ and other sulphur (C – S and S – O – C) on B7 adsorbent introduced by mixture of $\text{H}_2\text{SO}_4/\text{SO}_3$ (Oleum) using the gas phase reaction does not have a strong affinity interaction as hydroxyl groups (O – H) in aqueous solution. Therefore, the B7 adsorbent could not disperse well in the aqueous mercury solution. As a result, the less interaction between mercury ion and the acidic functional group on the B7 surface was considered. Thus, the B10 adsorbent has better interaction in aqueous mercury solution than B7 adsorbent sample. The second postulate is the sulphur group might be less stable in the aqueous

solution. Thus, the sulphur groups tend to detach or leach out from the B7 surface during the agitation step in the adsorption experiment.

5.1 Recommendation

It is recommended for further study to improve the functionalization method by using organic ligand, to achieve high dispersible functionalized MWCNTs in aqueous solution. Therefore, this can help in adsorption of metal ions onto functionalized MWCNTs. In addition, the thermal treatment of MWCNTs through gas and liquid phase can also be performed in rotating autoclave under controlled atmosphere where the temperature and pressure of the functionalization process can be optimized. This technique provides sufficient amount of energy needed to break apart the carbon atoms network of MWCNTs in order to create defects essential for functionalization. As a result, this creating stronger bond between oxygen and sulphur containing functional groups. Therefore, leaching of functional groups (acidic site) during the adsorption experiment can be reduced or avoided completely.