

Lecture 6. Purification and Functionalization of Multiwalled Carbon Nanotubes (MWCNTs)

The purpose of the lecture: to provide information on the purification and functionalization of multiwalled carbon nanotubes (MWCNTs).

Expected results: to master the methodology for purification and functionalization of multiwalled carbon nanotubes.

As-produced MWCNTs have impurities in the form of catalyst and amorphous carbon, and purification of these MWCNTs is very important. Generally 10–12 wt% of a catalyst is mostly found as impurity along with the MWCNT bundle. Additionally, these CNTs do not have efficient surface functionality to make chemical bonds with the polymer matrix for the development of composite materials. To realize the outstanding mechanical properties in a fruitful way, it is essential to use surface-modified MWCNTs as reinforcing materials in a polymer matrix.

A number of studies on CNT–polymer composites have focused on improving the dispersion and load transfer efficiency by improving the compatibility between CNTs and the polymer matrix through covalent chemical functionalization of the CNT surface. The surface modification involves attaching different functional groups on the surface and the end caps of CNTs. A major challenge in the emerging area of nanotubes chemistry was the development of an oxidation process for CNTs. It can either be carried out by refluxing it in nitric acid or in the combination of nitric and sulfuric acid. These drastic conditions led to the opening of the tube caps as well as the defects in the sidewalls due to oxidative etching and associated release of carbon dioxide.

The final products provide the nanotubes of short lengths whose ends and sidewalls are decorated with a high density of various oxygen-containing groups (mainly carboxyl groups). Under less vigorous conditions, such as refluxing in nitric acid, the shortening of the tubes can be minimized. The chemical functionalization is then limited mostly to the opening of the tube caps and the formation of functional groups at defect sites along the sidewalls.

These carboxylic functional groups can be further replaced by more reactive groups like $-\text{COCl}$ or $-\text{CONH}_2$, as shown in Figure 1. The addition of these functional groups on CNTs cause intermolecular repulsion between functional groups on the surface, which overcomes weak van der Waals attraction between CNTs. It is also vital to stabilize the dispersion to prevent reagglomeration of CNTs. A wide range of surface functionality can thus be created on CNTs.

It has been observed by the researchers that use of amine functionalized CNTs resulted in a significant improvement in the mechanical properties of the CNT epoxy composites due to the formation of covalent bond by the opening of epoxide ring with amine groups available on the surface of CNTs, as shown in Figure 2.

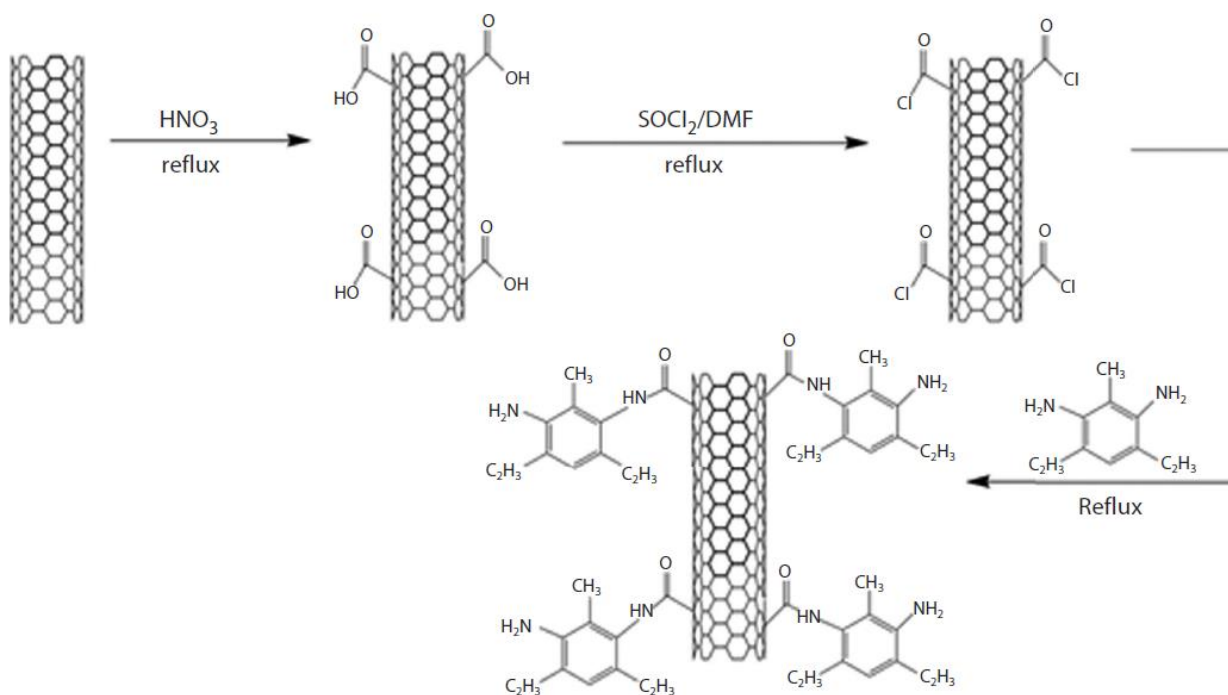


FIGURE 1. Chemical modifications of nanotubes through chemical oxidation followed by subsequent chlorination and amidation.

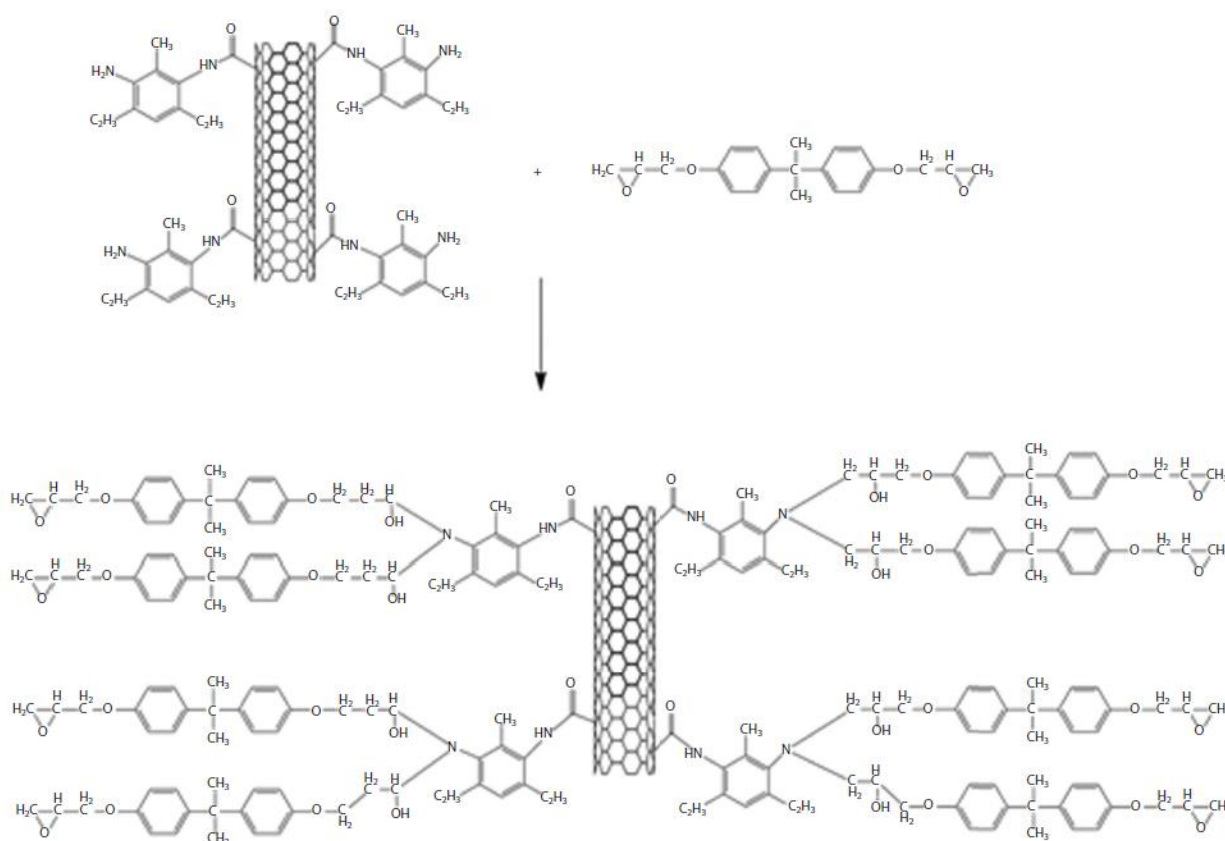


FIGURE 2. Pictorial view for the formation of amine-modified MWCNTs and their interaction with the epoxy matrix.