



## A review: Multiwall carbon nanotubes in desulphurization of fuel

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### Abstract

Increasing worldwide demand for energy and the demand for cleaner fuels with the recent stringent regulations of commercial fuel specifications have driven the research of alternative methods to upgrade the current industrial desulphurization technology. Adsorptive desulphurization, the removal of refractory sulphur compounds using appropriate selective tailor-made adsorbents, has shown up as a promising alternative in the recent years. Carbon nanomaterials, namely, carbon nanotubes, show a significant potential as desulphurization adsorbents. Their surface area and porosity, their ability of easy functionalization, and their suitability to serve as a support of different types of adsorbents have rendered them attractive candidates for this purpose. In this study, after a presentation of the current industrial desulphurization practice and its limitations, the structure and properties of the carbon nanotubes will be described, followed by a detailed account of their applications in adsorptive desulphurization. The major literature findings and conclusions will be presented and discussed as a road map for future research in the field.

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### 1. Introduction

Crude oil is a complex mixture of hydrocarbons in a wide range of molecular weights. It also includes other polydispersed organic compounds such as resins, asphaltenes, and organometallic compounds (1). They are composed of carbon, hydrogen, metals, nitrogen, oxygen, and sulphur in their structures; inorganic oxides; salts; and metals. The crude oil composition varies from one oil field to another and depends on how it is produced (2). In crude oils, the range of sulphur content is 0.05–10 wt.%, but usually it ranges from 1 to 4 wt.% (3). If the sulphur content is less than 1 wt.%, the crude oil is termed as sweet, while it is named sour if the sulphur content is more than 1wt.%. The sulphur compounds can be either inorganic such as COS (carbonyl sulfide), H<sub>2</sub>S (hydrogen sulfide), and dissolved pyrites or organic in which sulphur exists as a heteroatom bounded to a hydrocarbon molecule (2), see Figure 1.

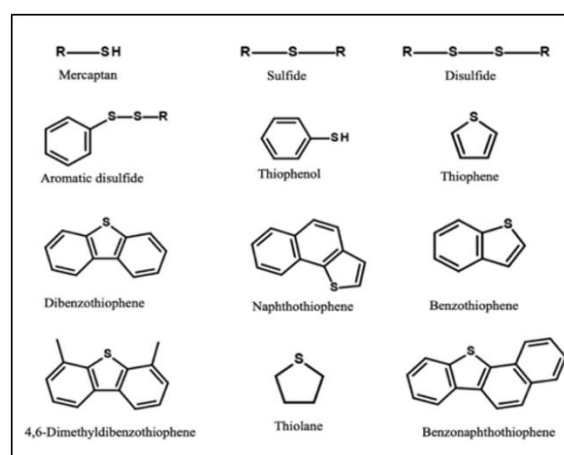


Figure 1: The most common organic sulphur compounds in crude oil (2)

The sulphur content in fuels often indicates SO<sub>x</sub> emissions, which are responsible for the formation of sulfates and acids (for example, acid rain caused by SO<sub>2</sub>) (4). Moreover, several corrosion problems

in pipelines, pumping, and refining equipment may also occur due to sulphur content. Sulphur emissions also cause deleterious effects on human health (such as breathing problems, and irritation to the eyes and the throat due to Thiols) (5). In addition, the presence of sulphur compounds is often a serious poison for the secondary processes (such as catalytic reforming). These compounds could be selectively adsorbed on the surface of metal catalysts (such as Cu, Ni, Co, Pb, and Pt) causing deactivation of the catalyst. In addition to all of the above, the presence of sulphur compounds in gasoline decreases the octane number which means reducing its quality. Thus, additional processes are required to restore and maintain the quality of fuels (6). In order to reduce the sulphur contents in fuels, the environmental regulation agencies (especially in the United States and in the Europe) have introduced an allowable limit of 10 ppm with the ultimate goal of further reducing SO<sub>x</sub> gases emissions (7). As a result of the aforementioned environmental regulations, there have been several reports successfully reducing the organic sulphur contents in diesel fuels to less than 10 ppm. In the recent decades, there has been a growing interest in the development of fuel cells, aiming to the transition towards cleaner fuel technologies (8). Under this concept, research has focused on developing hydrogen-powered fuel cell cars. The problem is that these systems, which use hydrocarbon fuels as feedstock, are very sensitive to the presence of sulphur. Therefore, the regulations regarding the specifications of automobile fuels as well as the universal need to promote the use of cleaner fuels constantly drive the effort to develop more efficient technologies for deep desulphurization (9). The aim of this study was to remove sulphur from fuel through multiwall carbon nanotubes.

## **2. Desulphurization methods**

There are several methods to remove sulphur compounds from petroleum derivatives. These methods are hydrodesulphurization (HDS), oxidative desulphurization, biodesulphurization, and adsorptive desulphurization. Currently, HDS is the common process in the refineries where it removes many organosulphur compounds and their derivatives from transportation fuels. In HDS, the distillate is fed together with hydrogen gas in the catalytic reactor at high temperatures (320 – 440°C) and high pressures (15 - 200 atm) in the presence of catalysts (7). However, HDS has certain limitations and is less effective at removing sterically hindered dibenzothiophene derivatives, such as 4,6-dimethyldibenzothiophene, which are most prominent in diesel fuels (10). The sulphur compounds in petroleum derivatives differ in their chemical structure (see Figure 1) and their physicochemical properties. Thus, these sulphur compounds differ in their reactivity during HDS processes. Thiols, disulfides, and sulfides have

higher electron density of the sulphur atom and weaker C-S bonds thus, HDS is very efficient for these compounds (11). On the other hand, for the thiophenic compounds, S atom is bonded to the aromatic rings which are stronger due to the pi-electrons. Besides, there is steric repulsion due to alkyl-substituents, so it is difficult for S atoms to reach the centre of the catalyst. Hence, HDS of thiophenic compounds (also known refractory sulphur compounds) such as 4,6-dimethyldibenzothiophene present a very low HDS reactivity and inefficient for reaching very low concentration. As it requires extreme operating conditions besides a high H<sub>2</sub> consumption (12).

Some suggestions such as the use of more active catalysts, longer residence times, and higher temperatures and pressures have been reported for improving the effectiveness of HDS for producing low sulphur product. Therefore, to meet the demands of producing ultraclean fuels with lower than 15 ppm of organosulphur compounds via HDS will increase both monetary investment and operational costs. As a consequence, alternative ways for deep sulphur removal have been investigated in the recent years (13). One of the promising ways is selective adsorptive desulphurization where  $\pi$ -complexation is free of steric hindrance for adsorption. This method seems to be very efficient method as it is less energy intensive and could be carried at moderate temperature and pressure as well as no solvents or hydrogen consumption. Recently, many researchers focus on different adsorbents such as carbon nanomaterials (14).

The aim of this report is to focus on carbon nanomaterials (namely carbon nanotubes) that have been tested for the adsorption of sulphur compounds from petroleum distillates. The major findings reported in the literature will be summarized and discussed in the following sections, after a brief introduction to the structure and properties of the carbon nanomaterials of interest.

## **3. Carbon nanomaterials**

Graphene is the name given to a flat monolayer of carbon atoms tightly packed into a two-dimensional (2D) honeycomb lattice (15). The carbon atoms are sp<sup>2</sup>-hybridized; this hybridization creates a structure with strong in-plane  $\sigma$ -bonds between the carbon atoms and  $\pi$ -orbitals that lie perpendicular to the plane. The  $\pi$ -orbitals are conjugated as in the aromatic molecules, and the  $\pi$ -electrons are delocalized on graphene surface. Hence, the  $\sigma$ -bonds collectively form a rigid hexagonal backbone, while the out-of-plane  $\pi$ -orbitals mediate interactions between successive graphene sheets or with other molecules (e.g., aromatic rings) (16). 2D graphene is the building block of graphitic materials of all other dimensions. Hence, it can be wrapped into 0D fullerenes (buckyballs), rolled up into 1D

nanotubes or stacked into the 3D graphite, building different architectures (17), as shown in Figure 2. In the past few decades, carbon nanotubes (CNTs) have received tremendous attention from the science community for fundamental research as well as their applications in various fields of study such as catalyst supports, hydrogen storage medium, field emission displays, composite materials, sensors and biosensors, nanoprobe and, most recently, as a potential drug delivery vehicle for therapeutics agents in nanomedicine (18).

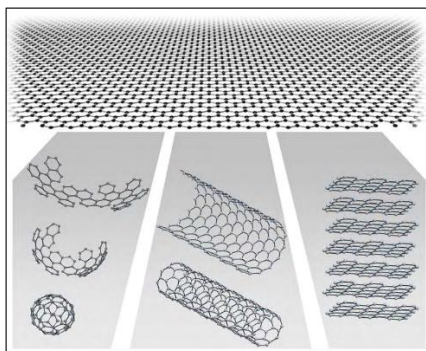


Figure 2: Different architectures of graphite (17)

Carbon Nanotubes were discovered in 1991 by (19), they have excellent conductive, mechanical, and thermal properties as well as their large surface area. Under well-defined synthesis conditions and by rolling up one or more graphene sheets, two forms of carbon nanotubes can be prepared: single wall carbon nanotubes (SWCNTs) and multiwall carbon nanotubes (MWCNTs) (20), see Figure 3. It was mentioned that CNTs are 1D materials which are at atomic-scale that make them chemically inert (21). In addition, CNTs are two orders of magnitude stronger than steel while carrying ultralight weight with their large surface area (22) as well as their high melting where estimation of melting point of nanotubes of about 3700 °C.

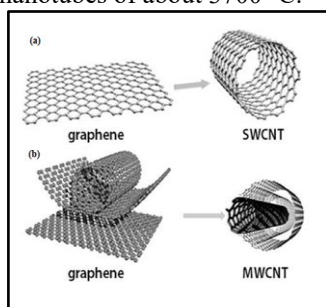


Figure 3: Carbon Nanotubes, (a) single wall carbon nanotube (SWCNT), (b) multiwall carbon nanotube (MWCNT) structures, taken from (20). As carbon nanotubes (CNTs) consist of rolled-up graphene sheets, they have the same sp<sup>2</sup>-hybridized hexagonal structure, while their centre is hollow. Generally, it has been found that when a graphene

sheet consists of a relatively small number of carbon atoms, it is more thermodynamically stable if it closes onto itself forming a tubular architecture, in order to eliminate all hanging open bonds at the edges (22). However, the rolling can occur at several manners, leading to various orientations of the lattice. Hence, with respect to the tube axis, the hexagonal arrays of carbon atoms follow a helical pattern on the tube surface. With respect to their helicity, CNTs can have a zigzag pattern, an armchair pattern, or a random helical one. The armchair CNTs have a metallic character with respect to their conductivity, while the others can be either metallic or semiconducting. Moreover, regarding the number of graphene sheets building their structure, CNTs can be either single-walled (SWNT) or multiwalled (MWNT). SWNTs consist of a single graphene sheet rolled up, and they have a diameter of 1-2 nm. MWNTs consist of more than one parallel graphene sheets rolled up as concentric cylinders and their diameter is usually in the range of 2–25 nm while their length can reach several micrometers (23).

CNTs carry an array of fascinating properties comparable to graphene, such as very high in plane thermal conductivity and Young's modulus. Nevertheless, their electronic structure depends on their lattice helicity, and their density is smaller than that of graphite. The surface area of MWNTs has been measured to be around 10–20 m<sup>2</sup>/g, while the surface area of SWNTs is around an order of magnitude larger (24). These values are higher than those for graphite but smaller compared to those of activated porous carbon.

Furthermore, CNTs are insoluble to all organic solvents and aqueous solutions, but their surface can be modified or functionalized to alter their physicochemical properties. This modification usually belongs to one of the three following categories; (1) attachment of molecules through covalent bonds on the surface of the CNT, (2) noncovalent adsorption of functional molecules on the CNT, and (3) filling of their hollow cavity. In the case where the surface of CNTs is modified through covalent bonding with functional groups, a variety of reactions such as halogenation, hydrogenation, cycloadditions, addition of radicals or inorganic compounds, ozonolysis, grafting of polymers, esterification or amidation at their edges, and attachment of biomolecules have been reported. Similarly, in the case of noncovalent bonding, it has been reported that the CNT surface can be covered by polymers, aromatic compounds, surfactants, or biomolecules through van der Waals forces or  $\pi$ - $\pi$  stacking, in a way that their electronic network is not affected (25). Finally, examples of filling their hollow cavity include the storage of liquid fuels or the encapsulation of fullerene derivatives, inorganic species, or biomolecules (26). Therefore, CNTs are characterized by the susceptibility to have their

surface or cavity adjusted in various manners related to a specialized application.

#### **4. Carbon nanotube applications**

It was mentioned that carbon nanotubes consist of rolled-up graphene sheets; hence, their surface structure and their sp<sup>2</sup>-hybridized hexagonal configuration are the same as graphene. Consequently, the utilization of CNTs in adsorptive desulphurization bears similarities to that of graphene, in that they are involved in  $\pi$ - $\pi$  interactions, they can be functionalized, and they can be doped with nanoparticles to increase their adsorption capacity (27).

In adsorption desulphurization, the pore volume and surface area of the adsorbents are important for organosulphur removal from the fuels. It is reported that the pore volume of the adsorbents and the organosulphur compounds is significant to obtain high adsorption capacity, which gives strong attachment and enhances trapping the molecules. Recently, researchers have focused on the graphene sheets and CNTs as adsorbents and found higher adsorption of sulphur compounds besides their chemical and thermal stability. In addition, CNTs were used as catalyst in oxidative desulphurization for 100% removal of organosulphur compounds at atmospheric pressure and low temperature (150 °C) (28, 29). The adsorption capacity of commercial MWNTs was compared with graphene oxide (GO) and activated carbon for the removal of thiophene and dibenzothiophene (DBT) from a model diesel fuel (n-hexane). In all three adsorbents, DBT was more favorably adsorbed than thiophene. According to the author, the reason is the difference in the dipole moment between the two molecules; thiophene has 0.55D, while DBT has 0.95D, so DBT develops stronger dipole-dipole interactions. The adsorption capacity of MWNTs for DBT was measured 23.42 mgDBT/g, close to the one of GO (22.73 mgDBT/g), but much lower than that of activated carbon (41.49 mgDBT/g). It seems that activated carbon outperformed the other two adsorbents because of the large differences in their specific surface area (882 m<sup>2</sup>/g for activated carbon, 217 m<sup>2</sup>/g for MWNTs and 10.8m<sup>2</sup>/g for GO) and the average pore width (14.5 Å for activated carbon, 73.8 Å for MWNTs, and 68.5 Å for GO). It is important that the adsorbent should have a small average pore width comparable to the size of DBT (<7 Å), so that the adsorbate will be trapped more efficiently. Here, activated carbon contained smaller pores on average, while its total micropore volume was much larger (0.487 ml/g compared to 0.286 ml/g for MWNTs and 0.021 ml/g for GO). Therefore, the differences in surface area, average pore width, and pore volume were crucial for the efficient removal of DBT in this case (30). Vu et al., 2012, Composite of MWNTs and titanium oxide (TiO<sub>2</sub>) were prepared by a heterogeneous gelation method. The activities of the MWNTs/TiO<sub>2</sub>

composites were evaluated by photocatalytic oxidative desulphurization using dibenzothiophene (DBT), 4,6-dimethyl dibenzothiophene (4,6-DMDBT), n-tetradecane, and commercial diesel under irradiation using a high-pressure Hg lamp. They found that more than 98% of sulphur compounds in commercial diesel were oxidized and removed by the use of the MWNTs/TiO<sub>2</sub> composite as a photocatalyst. (32) has been reported the development of novel nanomaterials of multiwalled carbon nanotubes doped with titania (CNT/TiO<sub>2</sub>) for the adsorptive desulphurization of model fuel oils. Their initial results indicated the effectiveness of the prepared CNT/TiO<sub>2</sub> nanomaterials in removing sulphur compounds from model fuel oil. They performed batch mode system to study the adsorption of dibenzothiophene, benzothiophene, and thiophene from model fuel onto the derived sorbents. These CNT/TiO<sub>2</sub> nanomaterials initially afforded approximately 45% removal of DBT, 55% BT, and more than 65% thiophene compounds from model fuels. They evaluated and compared the desulphurization activity of CNT, titania, mechanical mixture of titania and CNTs (MM), and the prepared CNT/TiO<sub>2</sub> nanomaterials for the adsorption of sulphur compounds. They claimed that prepared CNT/TiO<sub>2</sub> nanomaterials have better desulphurization activity than other tested materials. (33) investigated the adsorption of various organic molecules, namely, benzene (aromatic), thiophene (heterocyclic), and cyclohexane (nonaromatic) on SWNTs of two different diameters (12 and 16.8 Å). The thinner nanotubes had a surface area of 961 m<sup>2</sup>/g while the thicker ones had 322 m<sup>2</sup>/g, which reflects the difference in the synthesis and purification methods. Their results showed that narrower tubes adsorb larger amounts of adsorbates than the thicker ones, for all three organic compounds. They claimed that the adsorbate molecules in thinner SWNTs are exposed to a larger interaction potential from the delocalized electrons of the tubes. Moreover, the authors suggest that adsorption mainly occurs inside the nanotubes and to a smaller degree on their external surface or in the grooves among the nanotubes. Regarding the different adsorbates, there is no clear selectivity between the adsorption of benzene and thiophene. However, based on the measured heats of adsorption, thiophene was adsorbed more strongly on SWNTs. Both are adsorbed through  $\pi$ -interactions based on their aromaticity, while the nonaromatic cyclohexane is adsorbed to a smaller degree more weakly. The researchers also investigated the batch adsorption of thiophene from a model fuel (benzene) and also the desulphurization of a commercial diesel fuel (containing approximately 20% aromatics and 80% aliphatics, while the majority of sulphur compounds were various types of alkyl-dibenzothiophenes). In the model fuel, selectivity of thiophene over benzene

was confirmed, which depends on the purification treatment of CNTs with HNO<sub>3</sub> that anchored some oxygen functional groups; the adsorption capacity was 0.86mg thiophene/g. In the commercial diesel, the adsorption capacity was generally lower due to competitive adsorption. Comparing to activated carbon, they found that heat-treated SWNTs performed better for the removal of heavier alkyl-dibenzothiophenes (34).

(35) compared the adsorption efficiency between commercial CNTs and synthesized GO for the removal of DBT from heptane. Their strategy, however, was different in that they doped these carbon nanomaterials with silver sulfide (Ag<sub>2</sub>S) nanoparticles (10 wt.%). Ag, just like other transition metals such as Cu and Zn, can have a positive impact on adsorptive desulphurization. The results revealed that CNT/Ag<sub>2</sub>S exhibited the highest adsorption capacity (52 mgDBT/g), slightly larger than GO/Ag<sub>2</sub>S (49.6mgDBT/g). However, compared to the capacities of raw CNTs and GO, which were both close to 23 mgDBT/g, it is obvious that the presence of Ag<sub>2</sub>S almost doubled the removal efficiency of the adsorbents. The authors state that Ag<sup>+</sup> acts as a Lewis acid; its empty d-orbital is available to interact with the lone-pair sulphur electrons available in DBT (Lewis base). Probably, there was also a sulfur-sulfur interaction between the sulfur atom of DBT with the sulfur atom of Ag<sub>2</sub>S (36).

(37) doped CNTs with N atoms and tested them for the removal of tertiary butyl mercaptan (TBM) from n-heptane. Doping was achieved by adding urea during CNT synthesis. N atoms are incorporated into the CNT structure through pyridinic and pyrrolic N bonds, and their presence promotes the release of electrons; therefore, they can assist the adsorption of molecules through polar or acid-base interactions. The results revealed that the adsorption capacity of N-doped CNTs with 1.38 wt.% N-loading was 63.5 mgTBM/g, which was around 2.5 times higher than that of pure CNTs. As the electron density is increased locally on the surface of CNTs, these sites act as electron donors (Lewis base) and interact with the S atoms in TBM which are Lewis acids (electron acceptors). Moreover, the doped N-CNTs had a specific surface area of 145.9 m<sup>2</sup>/g, larger than that of undoped CNTs (74.4 m<sup>2</sup>/g). Based on the adsorption kinetics, the authors suggested that the adsorption proceeds through two stages; a rapid diffusion on the external surface, followed by a slow pore diffusion (38).

## 5. Conclusions

The main goal of this report was to demonstrate and highlight the use of carbon nanomaterials in the petroleum industry. The investigations were on the desirable inherent properties of an adsorbent, specifically carbon nanotubes, that will enhance the efficiency of an adsorptive desulphurization process. These properties include, their high surface

area and total pore volume, selectivity over aromatic or aliphatic hydrocarbons, regenerability, presence of transition metals or metal oxides, thermal stability when the process is conducted at high temperature, and their low cost. In general, these adsorbents possess most of these properties and the additional advantage that they can relatively easily be functionalized or modified to improve their performance. Their main disadvantage is probably the selectivity against aromatic components, while their regeneration is also an important factor to be considered. Nevertheless, they can provide an excellent substrate for the development of hybrids by combining them with other types of adsorbents to boost their performance.

Generally, comparing the adsorption capacities of various adsorbents from literature values is not a reliable task, unless they are measured together for a specific system. The reason is that the adsorption capacity depends on various factors, such as the initial S concentration, the solvent or the type of fuel used, the type of sulphur compound, the presence of other competitive adsorbates such as aromatics, and so on. However, carbon nanomaterials seem to have the potential of industrial applications either alone or in combination with other adsorbents in a multistage process or even combining the adsorptive desulphurization with other processes such as oxidative desulphurization. From the stated findings we can conclude that carbon nanotubes as adsorbents will find useful applications in petroleum industry because of their operational simplicity, high efficiency, and high capacity.

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## مراجعة علمية: استخدام الأنابيب النانوية الكربونية متعددة الجدران في إزالة الكبريت من الوقود

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إن الطلب العالمي المتزايد على الطاقة، إلى جانب الحاجة إلى وقود أكثر نظافة في ظل التشريعات الحديثة الصارمة الخاصة بمواصفات الوقود التجاري، قد دفع الباحثين إلى تطوير طرائق بديلة لتحسين تقنيات إزالة الكبريت الصناعية المستخدمة حالياً. وقد برزت إزالة الكبريت بالامتزاز (Adsorptive Desulfurization)، والتي تعتمد على إزالة المركبات الكبريتية المقاومة باستخدام مواد مازة انتقائية مصممة خصيصاً، كأحد أكثر البدائل الواعدة خلال السنوات الأخيرة. وتُظهر المواد النانوية الكربونية، ولا سيما الأنابيب النانوية الكربونية (Carbon Nanotubes, CNTs)، إمكانات كبيرة بوصفها مواد مازة لإزالة الكبريت. ويعود ذلك إلى ما تتميز به من مساحة سطحية عالية، وبنية مسامية متطورة، وقابليتها للتعديل الوظيفي بسهولة، فضلاً عن ملائمتها كدعامات لأنواع مختلفة من المواد المازة، مما يجعلها مرشحة واعدة لهذا الغرض.