



A computational analysis of the carbon-nanotube-based resonant-circuit sensors

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Abstract

Available values for the molecular polarizability and the dipole moment and the computed adsorption energies to single walled carbon nanotubes (SWCNTs) for a couple of polar (NH₃ and CO) and several non-polar (He, Ar, N₂ and O₂) gases are used to help establish a correlation between the adsorbed gas-induced changes in the dielectric constant of the SWCNTs (the sensing material) and the resulting reduction in the resonant frequency of the resonant circuit-based chemical gas sensors. It is found that simple weighting methods which neglect the effect of changes in the electronic structure of the carbon nanotubes during adsorption are generally incapable of predicting correctly the changes in the effective dielectric constant of the carbon nanotubes. Conversely, the use of adsorption-induced changes in the band gap of the carbon nanotubes and a relationship between the band gap and the dielectric constant is found to be a promising approach for assessing the adsorption-induced changes in the effective dielectric constant of the carbon nanotubes and for establishment of their effect on the resonant frequency of resonator-based chemical gas sensors.

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1. Introduction

Gas sensors play an important role in a variety of applications ranging from house gas and fire alarms to medical diagnostic applications and the control of manufacturing processes (e.g. [1]). The most commonly used sensing materials are semiconducting

metallic oxides (e.g. [2]), silicon-based materials (e.g. [3,4]), organic materials (e.g. [5,6]) and carbon black laden polymer-matrix conductive composites (e.g. [7]). Semiconducting metallic oxides-based sensors show a very high sensitivity but they must be operated at relatively high temperatures (500–700 K) to attain the necessary level of reactivity between the sensing material and the gas molecules whose presence is being detected [8,9]. Polymeric materials-based sensors are generally characterized by very short response and recovery times but the sensing material must be in the liquid state which makes them

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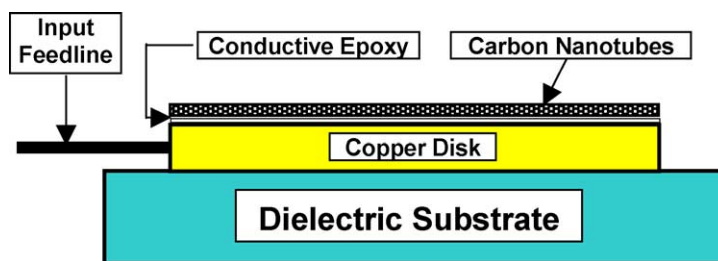


Fig. 1. A schematic of the front view of the circular-disk resonator-based chemical gas sensor developed by Rao and co-workers [12,13].

not very attractive in remote sensing applications [10]. Conducting composites-based sensors are generally found to suffer from a limited sensitivity, while the organic materials-based sensors have undesirably high electrical resistance [11].

Recently, Rao and co-workers [12,13] reported the development of a novel class of gas sensors based on a carbon-nanotubes coated circular-disk resonator. Upon exposure of the circular disk to various either polar (NH_3 and CO) or non-polar (He , Ar , N_2 and O_2) gases, the electrical resonant frequency of the sensor exhibits a substantial species-specific downward shift. In addition, this type of sensor is found to possess very attractive, relatively short response and recovery times (ca. 10 min).

A schematic of the disk resonator-based gas sensor used by Rao and co-workers [12,13] is shown in Fig. 1. In remote sensing applications, the sensor shown in Fig. 1 would be used as follows: an RF transmitter would send a microwave signal to a sensor located in the interrogation region (a region around the sensor which contains the gas to be detected). The interaction between the microwave signal and the sensor produces a return signal whose intensity peaks at the interrogated species-specific resonant frequency of the resonator. The resonant frequency of the circular disk, f_0 , is given by the following equation:

$$f_0 = \frac{1.84c}{2\pi a\sqrt{\varepsilon}} \quad (1)$$

where c is the speed of light in vacuum, a is the disk radius, and ε is the effective dielectric constant (i.e. relative permittivity) of the resonator. A typical response of the resonator sensor in vacuum (displayed as a return loss versus frequency curve) obtained by Rao and co-workers [12,13] is shown

in Fig. 2 (curve labeled “*Degassed*”). The minimum in this curve corresponds to the resonant frequency (~ 3.88936 GHz) of the sensor. When the sensor is exposed to 1500 ppm of helium (the curve labeled “1500 ppm He” in Fig. 2), the resonant frequency is shifted downward by ~ 0.8 MHz. The resonant frequency (~ 3.88936 GHz) is recovered upon re-degassing the sensor for approximately 10 min. Similar response and recovery behaviors of the sensor are observed when the sensor is exposed to other non-polar gases (such as Ar , N_2 and O_2) or to polar gases (such as NH_3 and CO), but the resonant frequency downshift is found to be a characteristic of the gas being detected. In addition, Rao and co-workers [12,13] observed that the downward shift in the resonant frequency is larger for polar than for non-polar gases. For the polar gases, the frequency shift is found to scale with the dipole moment of the gas, while for non-polar gases, Rao and co-workers [12,13] speculated that the resonant frequency downshift scales with the magnitude of the adsorption energy.

In this paper, an attempt is made to establish a relationship between the polarizability, the dipole moment and the adsorption energy to SWCNTs for NH_3 , CO , He , Ar , N_2 and O_2 and the resulting downward shift in the resonant frequency of the resonator-based gas sensors developed by Rao and co-workers [12,13].

The organization of the paper is as follows: a description of the computational method used in this work is overviewed in Section 2. The main results obtained in the present work are presented and discussed in Section 3, while the key conclusions resulted from the present study are summarized in Section 4.

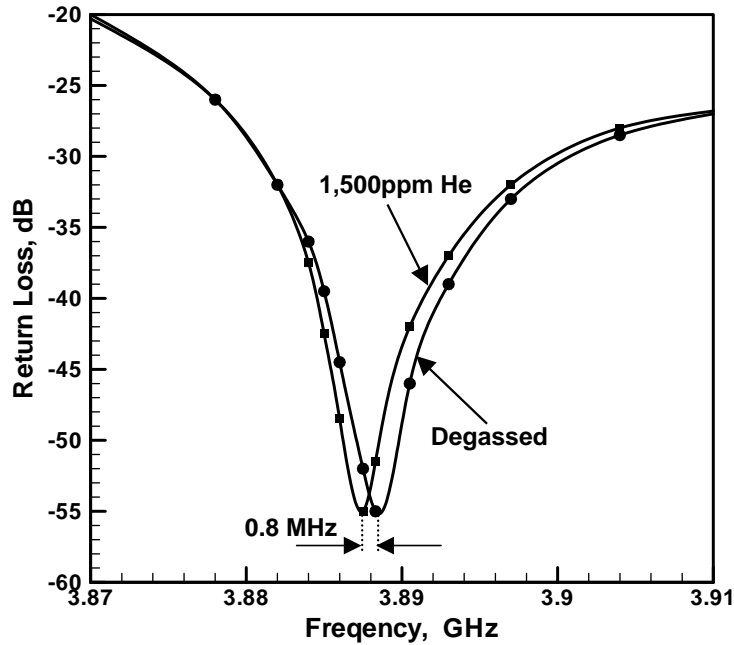


Fig. 2. A response of the chemical gas resonator-based sensor in vacuum (the curve labeled “Degassed”) and to 1500 ppm of helium (the curve labeled “1500 ppm He”) obtained by Rao and co-workers [13].

2. Computational procedure

2.1. Computation of the dielectric constant

Within classical electromagnetics, it is well established that the resonant frequency of a resonator such as the one shown in Fig. 1, depends on the effective dielectric constant of the resonator circuit. The latter is, on the other hand, affected by the dielectric constants of the substrate, the conductive epoxy used to bond carbon nanotubes to the disk, the nanotubes and the gas molecules adsorbed onto the nanotubes. Since the same sensor was used to interrogate different gases in the work of Rao and co-workers [12,13], the only component in the effective dielectric constant of the sensor which changes is the one associated with the gas adsorbed to the outer walls of carbon nanotubes. Therefore, one should expect that the (gas specific) downward shift in the resonant frequency is directly correlated with the dielectric constant of the gas being detected.

For a gas with the number density, N (number of molecule/volume) and the molecular polarizability, α ,

the polarization P_E in the direction of an applied electric field, E , can be expressed as

$$P_E = N(\alpha|E| + \langle \mu_E \rangle) \quad (2)$$

where boldfaced symbol in Eq. (2) is used to denote a vector quantity, $|\cdot|$ represents the magnitude of a vector, $\langle \cdot \rangle$ is the average quantity and μ_E is the component of the permanent molecular dipole moment along the direction of the applied field.

The $\langle \mu_E \rangle$ term in Eq. (2) can be expressed as

$$\langle \mu_E \rangle = |\mu_0| \langle \cos \theta \rangle \quad (3)$$

where θ is the angle between the permanent dipole moment, μ_0 , and the applied electrical field, E , and based on the Boltzmann statistics for the orientation of polar dipoles, an average value of its cosine, $\langle \cos \theta \rangle$, can be defined as

$$\begin{aligned} \langle \cos \theta \rangle &= \frac{\int_0^\pi \cos \theta \exp(-|\mu_0||E|\cos \theta/kT) \sin \theta d\theta}{\int_0^\pi \exp(-|\mu_0||E|\cos \theta/kT) \sin \theta d\theta} \\ &= \coth\left(\frac{|\mu_0||E|}{kT}\right) - \frac{kT}{|\mu_0||E|} \end{aligned} \quad (4)$$

where k is the Boltzmann's constant and T is the absolute temperature. Since generally $|\mu_0||E|/kT \ll 1$, Eq. (4) can be simplified as

$$\langle \cos \theta \rangle \approx \frac{|\mu_0||E|}{3kT} \quad (5)$$

Substitution of Eq. (5) into Eq. (3) and then in Eq. (2) yields

$$P_E = N \left(\alpha + \frac{|\mu_0|^2}{3kT} \right) |E| = \chi_E |E| \quad (6)$$

where χ_E is used to denote electrical susceptibility.

Since the dielectric constant is related to the electrical susceptibility as

$$\varepsilon = 1 + 4\pi\chi_E \quad (7)$$

the final expression for the dielectric constant, ε , of a gas is obtained by combining Eqs. (6) and (7) to yield

$$\varepsilon_{\text{ads}} = 1 + 4\pi N \left(\alpha + \frac{|\mu_0|^2}{3kT} \right) \quad (8)$$

Eq. (8) is commonly referred to as the Debye equation (e.g. [14,15]) and a subscript 'ads' is used in Eq. (8) to indicate that the dielectric constant pertains to the gas adsorbed to the outer walls of carbon nanotubes.

Since for non-polar molecules $|\mu_0| = 0$, the term within the parenthesis on the right-hand side of Eq. (8) is generally significantly larger for polar gases than for non-polar gases. On the other hand, if Eq. (8) is applied to a resonator-based gas sensor, the N term (which scales with the surface concentration of the gas molecules adsorbed to outer walls of the carbon nanotubes) increases with an increase in the gas adsorption energy to the carbon nanotubes and decreases with an increase in repulsion between the adsorbed molecules.

2.2. Computation of the adsorption energy and adsorbate–adsorbate interactions

The adsorption energy of a gas to carbon nanotubes is generally defined using the following procedure: the binding energy $E_{\text{binding}}(d)$ (energy/molecule) between a single gas molecule and the nanotube as a function of the gas–molecule/nanotube–surface separation, d , can be computed using the following expression:

$$E_{\text{binding}}(d) = E_{\text{nanotube+gas}}(d) - E_{\text{nanotube}} - E_{\text{gas}} \quad (9)$$

where $E_{\text{nanotube+gas}}(d)$, E_{nanotube} and E_{gas} are the total energies of the fully-relaxed structures of a nanotube containing a single gas adsorbate, an isolated nanotube and an isolated gas molecule, respectively. The E_{binding} versus d curve has a familiar shape, that is as d approaches zero E_{binding} becomes unbounded in the positive direction (due to repulsions of the atomic cores) and as d becomes very large, E_{binding} approaches zero from the negative side. The gas adsorption energy is then defined as a negative of the minimum value of the binding energy.

The procedure described above was combined with ab initio density functional theory (DFT) calculations of the interactions between selected semiconducting and metallic single-walled carbon nanotubes and oxygen molecules in our recent work [16,17] to compute the oxygen adsorption energy. The same approach is used in the present work to compute the adsorption energies for the remaining gas species analyzed by Rao and co-workers [12,13]. The results of these calculations are shown in Table 1. Also shown in Table 1 is a compilation of the available data for molecular polarizabilities and dipole moments of the gas species under consideration.

Following the procedure outlined in our recent work [18], interactions between the gas molecules adsorbed

Table 1

| Gas species | Resonant frequency shift, Δf (MHz) | Molecular polarizability, $\alpha \times 10^{-40}$ (C ² m/N) | Permanent dipole moment, $\mu_0 \times 10^{-30}$ (C m) | Dielectric constant, ε |
|-----------------|--|---|--|------------------------------------|
| He | 0.8 | 0.227689 | 0 | 1.147917 |
| Ar | 1.2 | 1.823324 | 0 | 1.425762 |
| N ₂ | 1.9 | 1.933464 | 0 | 1.879510 |
| O ₂ | 2.3 | 1.756647 | 0 | 1.807550 |
| CO | 3.4 | 2.155062 | 0.374 | 2.599109 |
| NH ₃ | 3.55 | 2.397074 | 4.904 | 3.398663 |

onto the nanotubes surfaces are modeled using the van der Waals, the Coulomb electrostatic, and the hydrogen bond terms in the COMPASS (condensed-phased optimized molecular potential for atomistic simulation studies), the first ab initio forcefield that enables an accurate and simultaneous prediction of various gas-phase and condensed-phase properties of organic and inorganic materials [19].

2.3. Computation of the N term in Eq. (8)

To determine the N term in Eq. (8), the following procedure is utilized: first it is assumed that the gas molecules adsorbed onto the outer walls of carbon nanotubes form a regular triangular lattice with a lattice parameter, d_{tria} . The surface concentration of the adsorbed gas molecules, N_S (molecules/area), can then be defined as

$$N_S = \frac{4}{(d_{\text{tria}}^2 \sqrt{3})} \quad (10)$$

N_S can be, in turn, converted into N using the following relation:

$$N \approx \frac{N_S^{\text{eq}}}{d^{\text{eq}} + 0.5d_{\text{gas}}} \quad (11)$$

where N_S^{eq} is the equilibrium surface concentration of the adsorbate, and d^{eq} and d_{gas} are the equilibrium adsorbed gas–molecule/nanotube–surface separation and the diameter of the adsorbed molecule, respectively. The values of d^{eq} are obtained during the adsorption energy calculation discussed earlier, while d_{gas} can be calculated from the volume of a gas molecule, V_{gas} , as $d_{\text{gas}} = 2(3V_{\text{gas}}/4\pi)^{1/3}$.

N_S^{eq} can be calculated using Eq. (10) and the equilibrium distance of the adsorbate, $d_{\text{tria}}^{\text{eq}}$. The latter is defined as a value of d_{tria} at which the adsorption energy (per unit area of the nanotube surface) given as

$$E_{\text{ads}}(d_{\text{tria}}) = -[E_{\text{gas-gas}}(d_{\text{tria}}) + E_{\text{binding}}(d^{\text{eq}})] \left(\frac{4}{d_{\text{tria}}^2 \sqrt{3}} \right) \quad (12)$$

experiences a minimum with $E_{\text{gas-gas}}$ and E_{binding} being the adsorbate–adsorbate interaction energy and the nanotube/adsorbate binding energy (both expressed per molecule of the adsorbate). The net effect of E_{binding} in Eq. (12) is to reduce the distance

between molecules of the adsorbates, i.e. to increase the surface concentration of the adsorbed molecules. In other words, the more negative E_{binding} , the larger is the N term in Eq. (8).

3. Results and discussion

Using the procedure for computation of the N term in Eq. (8) which was described in the previous section and the available data for the molecular polarizabilities and the dipole moments which are listed in Table 1, the dielectric constants of the species adsorbed onto the carbon nanotubes in the experimental work of Rao and co-workers [12,13] are computed. The results of this calculation are listed in the last column in Table 1. These results show that, as expected, the largest values of the dielectric constant occur in the case of polar-gas adsorbates, while in the case of non-polar gases, the largest values of the dielectric constant appear in the gases such as oxygen and nitrogen which are characterized with a relatively large adsorption energy (i.e. with a large surface concentration of their adsorbates).

Once the dielectric constants for gas adsorbates are computed, an attempt was made to determine how adsorption of the gas species alters the effective dielectric constant of the carbon nanotubes. To a first approximation, as suggested by Rao and co-workers [12,13], one can define the effective dielectric constant of the carbon nanotubes containing gas adsorbates as a weighted average of the dielectric constants of the pristine nanotubes and the adsorbates. The dielectric constant of single-walled carbon nanotubes is found to be a function of their chirality and diameter [20] and generally falls in a range $\epsilon_{\text{SWCNT}} = 4\text{--}6$.

The mole fraction of the adsorbates, ϕ_{ads} , in the case of nanotubes covered with adsorbates is proportional to N_S^{eq} and can be approximately defined as

$$\phi_{\text{ads}} = \frac{N_S^{\text{eq}}}{N_S^{\text{eq}} + N_C} \quad (13)$$

where N_C is the surface packing density of carbon atoms in a graphene sheet (a single close-packed (0 0 0 1) graphite plane) and is defined as

$$N_C = \frac{4\sqrt{3}}{3d_C^2} \quad (14)$$

where $d_C = 0.142$ nm is the carbon–carbon atomic distance in a graphene sheet. It should be noted that Eq. (13) defines an upper limit for ϕ_{ads} since it assumes that adsorption takes place on the entire outer surface of all carbon nanotubes. In practice, steric hindrance effects can render some portions of the outer nanotubes walls inaccessible to the gas molecules.

Finally, a change in the dielectric constant of carbon nanotubes induced by the gas adsorbates can be defined as

$$\begin{aligned}\Delta\varepsilon_{\text{SWCNT}} &= \phi_{\text{ads}}\varepsilon_{\text{ads}} + (1 - \phi_{\text{ads}})\varepsilon_{\text{SWCNT}} - \varepsilon_{\text{SWCNT}} \\ &= \phi_{\text{ads}}(\varepsilon_{\text{ads}} - \varepsilon_{\text{SWCNT}})\end{aligned}\quad (15)$$

Eq. (15) is used to compute $\Delta\varepsilon_{\text{SWCNT}}$ for the case of gas species (NH_3 , CO , He , Ar , O_2 , and N_2) analyzed by Rao and co-workers [12,13]. The results of this calculation are plotted against the associated reductions in the resonant frequency of the resonator-based gas sensor in Fig. 3a (the curve labeled “*Molar Weighting*”).

The results displayed in Fig. 3a show that the largest change in the sensor resonant frequency occurs in the case of polar gases which give rise to a lowest change in the effective dielectric constant of the carbon nanotubes. While this finding appears counterintuitive, it can be readily explained. The values of the dielectric constant (1.14–3.40) of the gas-adsorbates analyzed in the present work are significantly lower than the dielectric constant in carbon nanotubes (an average value of 5 is used in the present paper). Hence, the polar-gas adsorbates which have the largest values of the dielectric constant will give rise to the lowest change in the effective dielectric constant of the carbon nanotubes, as long as the latter quantity is calculated using Eq. (15) or any other similar equation based on a weighting approach. To demonstrate this point, the effective dielectric constant of the carbon nanotubes is recalculated using the self-consistent effective medium approach [21]. Within this approach, the two constituents (the pristine carbon nanotubes and the gas adsorbates) are modeled as spherical particles embedded in the effective homogeneous medium composed of the two phases. The effective dielectric constant, ε_{eff} , of such medium is next defined by the following non-linear algebraic equation:

$$f_{\text{ads}} \frac{\varepsilon_{\text{ads}} - \varepsilon_{\text{eff}}}{\varepsilon_{\text{SWCNT}} + 2\varepsilon_{\text{eff}}} + (1 - f_{\text{ads}}) \frac{\varepsilon_{\text{SWCNT}} - \varepsilon_{\text{eff}}}{\varepsilon_{\text{ads}} + 2\varepsilon_{\text{eff}}} = 0\quad (16)$$

where f_{ads} is the volume fraction of the adsorbate. Eq. (16) is used to determine the effective dielectric constant of the carbon nanotubes and, in turn, to determine the change in this constant due to adsorption of the gases. The results of this calculation are also displayed in Fig. 3a, the curve labeled “*Effective Medium*”. It is seen that again, the gases which give rise to the smallest change in the effective dielectric constant of carbon nanotubes cause the largest shift in the resonant frequency of the gas sensor.

There is an even more serious problem with the results displayed in Fig. 3a and the use of Eqs. (15) and (16). The experimental results of Rao and co-workers [12,13] show that the adsorption of various polar and non-polar gases leads to a reduction in the resonant frequency of the sensor. However, based on Eq. (1), a decrease in the dielectric constant of the resonator circuit caused by the adsorption of various gases (Fig. 3a), should give rise to an increase (not a decrease) in the resonant frequency. This finding suggests that the effective constant of carbon nanotubes cannot be obtained by simply weighting the dielectric constants of the pristine carbon nanotubes and the adsorbates as suggested by Rao and co-workers [12,13]. Instead, or in addition, one must take into account changes in the dielectric constant of the carbon nanotubes caused by the nanotubes/adsorbates interactions.

In our recent work [16,17], tight binding calculations were combined with a Green’s function method to quantify the effect of the adsorption of molecular oxygen and nitrogen on the electrical conductivity of semi-conducting single-walled carbon nanotubes. The results obtained show that oxygen and nitrogen gas adsorbates give rise to a reduction in the band gap in these types of carbon nanotubes, which, in turn, results in an increase in electrical conductivity. Recently, Margulis and Gaiduk [22] carried out a linear response analysis of the SWCNTs subjected to an electromagnetic field and showed that the electrical susceptibility, χ_E , scales inversely with a square in the valence/conduction band gap. Since both oxygen and nitrogen decrease the band gap in the semiconducting SWCNTs, the finding of Margulis and Gaiduk [22] suggests that the adsorption of these two gases would give rise to an increase in the dielectric constant of the semiconducting carbon nanotubes. To take into account this effect when computing the effective

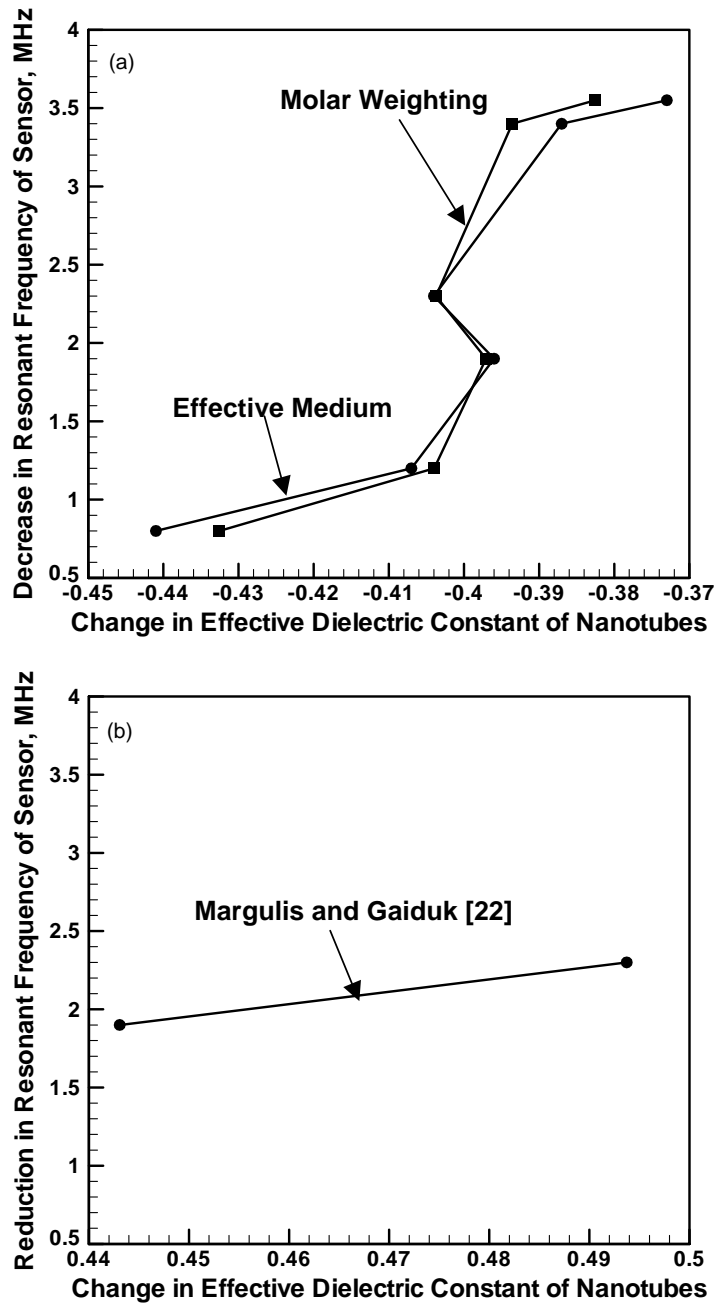


Fig. 3. Correlation between the adsorption-induced reduction in the resonant frequency of the sensor and the adsorption induced change in the effective dielectric constant of the carbon nanotubes (the sensing material). Please see text for details.

dielectric constant of the carbon nanotubes, a term $(1 - \phi_{\text{ads}})\Delta\epsilon_{\text{SWCNT}}^{\text{ads}}$ is added to Eq. (15) where $\Delta\epsilon_{\text{SWCNT}}^{\text{ads}}$ (=0.962630 and 1.027387 for nitrogen and oxygen, respectively) represents a change in the

dielectric constant of the carbon nanotubes associated with the adsorption-induced changes in their band gap. This term is assessed by taking the average value of the dielectric constant for the pristine carbon

nanotubes as $\epsilon_{\text{SWCNT}} = 5$ and adopting the finding of Margulis and Gaiduk [22] that the dielectric constant of carbon nanotubes scales inversely with a square of their band gap. The resulting changes in the effective dielectric constant of the carbon nanotubes are shown in Fig. 3b (the curve labeled “Margulis and Gaiduk [22]”). It is seen that, at least for oxygen and nitrogen, the adsorption causes an increase in the effective dielectric constant of the carbon nanotubes and that there is a positive correlation between such adsorbate-induced increase in the effective dielectric constant of the carbon nanotubes and the associated decrease in the resonant frequency of the gas sensor (as required by Eq. (1)). This finding is very encouraging and we are currently conducting a comprehensive tight-binding calculations of the effect of a number of gas species (including the ones analyzed by Rao and co-workers [12,13]) on the band structure of single walled carbon nanotubes of different chirality and diameter. The results of this investigation will be reported in a future communication.

4. Conclusions

Based on the results obtained in the present work, the following main conclusions can be drawn:

1. Gas adsorption-induced changes in the effective dielectric constant of the carbon nanotubes (act as a sensing material) appear to play a dominant role in the operation of resonator-based gas sensors.
2. Weighting methods which neglect the effect of changes in the electronic structure of the carbon nanotubes (and the adsorbate molecules) during adsorption are generally incapable of predicting correctly the changes in the effective dielectric constant of the carbon nanotubes.
3. The use of adsorption-induced changes in the band gap of the carbon nanotubes and relationships between the band gap and the dielectric constant (such as the one proposed by Margulis and Gaiduk [22]) as a way of assessing the adsorption-induced changes in the effective dielectric constant of the carbon nanotubes appears quite promising and should be pursued.

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