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Carbon Nanotubes with Magnetic Particles

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Abstract—Magnetic carbon nanotubes composites were obtained by filling carbon nanotubes with paramagnetic iron oxide particles. Detailed investigation of magnetic behaviour of resulting composites was done at different temperatures. Measurements indicate that these functionalized nanotubes are superparamagnetic at room temperature; however, no superparamagnetism was observed at 125 K and 80 K. The blocking temperature T_B was estimated at 145 K. These magnetic carbon nanotubes have the potential of being used in a wide range of applications, in particular, the production of nanofluids, which can be controlled and steered by appropriate magnetic fields.

Keywords—carbon nanotubes, magnetic nanoparticles, magnetization, nanofluids

I. INTRODUCTION

THE research field of carbon nanotubes (CNTs) has received a continuously growing interest since their discovery in 1991 [1] due to their unique and highly desirable electrical, thermal and mechanical properties [2]. Functionalizing CNTs with magnetic nanoparticles can combine the features of magnetic nanoparticles and CNTs, which may result in materials with novel chemical and physical properties, and thus promising applications. It has been reported that CNTs filled with Fe_3O_4 may be used as diffraction gratings, optical filters, and polarizers [3]. Other applications of these materials include cantilever tips in magnetic force microscopes, magnetic stirrers or magnetic valves in nanofluidic devices [3]–[5] as well as transporting drugs to specific locations in the body and for medical diagnosis without surgical invasive procedures [4]. Our motivation for creating a CNT/magnetite composite stems from the goal of creating a new generation of coolants, which have very high thermal conductivity and are amenable to be controlled by appropriate magnetic fields. These coolants are nanofluids, i.e. a suspension of nanoparticles, in the present case magnetic carbon nanotubes, in a basefluid [4], [5], and they have the potential of being used in micro-electronics cooling and they have the potential of not requiring a pumping system for their circulation. This can be achieved using appropriately placed and designed external magnets; therefore, this system is practically maintenance free, which makes it very attractive for cooling of micro-electronic devices in remote and/or inaccessible locations, which is the case of space applications.

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Recent reports described the attachment of various inorganic nanoparticles to the internal surface of the CNT cavity through several experimental methods. One of these methods based on spontaneous penetration of fluid into wettable capillaries by capillary action. This method of filling nanotubes was pioneered by Ajayan and Iijima [6], and it was also used to fill CNTs with different metal solutions [7]–[13].

The present study largely follows the work of Gogotsi's group (Drexel University, USA), Korneva *et al.* [12], whom demonstrated that it is possible to fill CNTs with a diameter of 300 nm using commercially available ferrofluid. These CNTs are prepared through a CVD (chemical vapour deposition) process with alumina membranes as templates. An aqueous suspension of magnetic nanoparticles is drawn by capillary suction into an open ended nanotube. Subsequent evaporation of the solvent resulted in nanotubes loaded with magnetic nanoparticles. Most of these processes involve physical deposition, with minimal molecular modification of the CNTs. To produce a nanofluid with magnetic CNTs in suspension, this method seems to have some potential, however, the level of CNTs filling, in terms of the ratio between the magnetic particles volume by that of the nanotube, is relatively low (approximately 11%).

Besides, it is technologically important to retain the desirable magnetic properties of the magnetic nanoparticles in CNTs through control over the particle size and uniformity of packed particles. Despite a number of previous studies, filling CNTs completely with monodisperse Fe_3O_4 nanoparticles has remained a challenging task. Moreover, it is essential to understand the magnetic interactions that are likely to influence the magnetic properties of CNTs filled with Fe_3O_4.

In this paper it is presented a modification of the procedure CNTs production proposed in [12], which can increase the nanotubes filling percentage to levels that are adequate for the production of nanofluids with magnetic CNTs as nanoparticles and detailed magnetic characterization of CNTs filled with ferroparticles.

II. EXPERIMENTAL

A. Carbon nanotubes

Carbon nanotubes were produced in the NRD laboratory (TEMA, University of Aveiro) by a non-catalytic chemical vapour deposition technique based on the pyrolysis of ethylene. The procedure was similar to that reported by Korneva *et al.* [12]. Before the growth of CNTs, the alumina template membranes (13 mm diameter, 60 μm thick, and 0.2 μm pore size purchased from Whatman® Inc., England) were placed vertically inside the quartz tube. The nanotubes were formed in straight cylindrical pores, which run through the membrane thickness. The pore diameter and thickness of the membrane determine the dimensions of the nanotubes. The microstructures of the alumina membrane and the CNTs samples inside the membrane were obtained with a Hitachi SU-70 scanning electron microscope (SEM) operated at 15 kV (University of Aveiro).

World Academy of Science, Engineering and Technology
International Journal of Materials and Metallurgical Engineering
Vol:6, No:7, 2012

Observation of the SEM images (Fig. 1) reveals the pores have different diameter within one membrane sample. The diameter of pore in our experimental results is in the range of 200 ± 15 nm (Fig. 2).
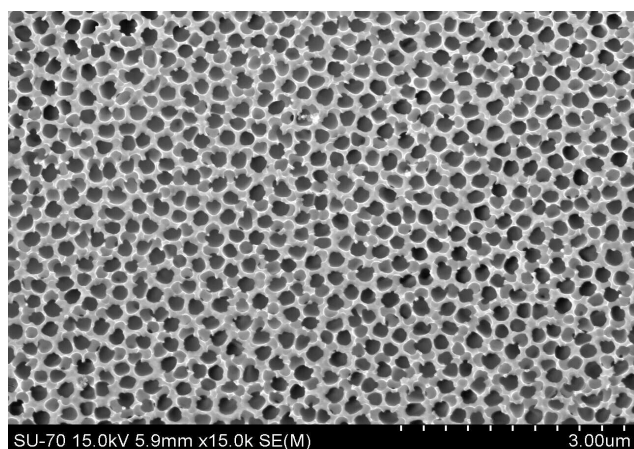

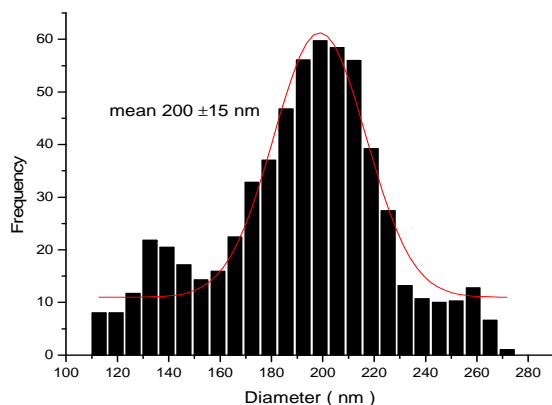Fig. 1 SEM micrograph of the alumina membrane

Fig. 2 Pore size distribution for the alumina membrane calculated from SEM image analysis

The resulting CNTs have at least one open end, and their walls are highly disordered and amorphous - this characteristic makes viable filling the nanotubes with both organic and water-based fluids, as noted in [12], [14]. The average length of the CNTs is 6 μm. The size of the membrane pores with CNTs was characterized using atomic force microscopy (AFM). The AFM measurements were conducted with a commercial AFM (Ntegra, NT-MDT, CICECO, University of Aveiro). Detailed analysis of the AFM images shows that the majority of carbon nanotubes have a diameter close to 124 nm [15].

B. CNTs filling procedure

The organic based ferrofluid EMG 911 (Ferrotec Corporation) was used in this experiment. This ferrofluid was the one that presented the best performance in terms of magnetization due to its Fe_3O_4 nanoparticles and allowed a clear demonstration about the filling of the nanotubes with a colloidal fluid (the surface tension of EMG 911 is 68 mN/m; the average diameter of Fe_3O_4 nanoparticles is 7.5 nm) [15].

To fill the carbon nanotubes sitting in the template with ferrofluid the procedure reported by Korneva *et al.* [12] and Pal *et al.* [13] was modified. In [12] and [13] the magnetic particles were incorporated inside the CNTs using a magnetically-assisted capillary action technique. As an alternative, in the present study, the loading was conducted by making multiple additions (up to 100 times by weight) of the ferrofluid to the system; no magnetic field was used in this filling procedure. The ferrofluid overflow enters the tubes at a relatively fast pace, and the flow was kept by making small additions of ferrofluid to the overflow. When it is apparent that no further ferrofluid enters the tubes, the system is left still for 24 hours to allow impregnation. At the end of this period, the membrane was repeatedly rinsed first with hexane and then with ethanol, then is air-dried and broken into small pieces and dipped into 4.0 M NaOH solution for dissolving the alumina template. After 24 hours, the dispersion was sonicated for 15 minutes and vacuum filtered; the filter with nanotubes was placed into a small vial with approximately 2-5 ml of isopropanol and sonicated for 2-3 minutes to release the nanotubes from the filter. After filtration, the sample was dried at room temperature. The nanoparticle-filled CNTs were characterized using scanning transmission electron microscopy (TEM) mode in the SEM Hitachi SU-70 operated at 30 kV. This scanning electron microscope is equipped with an energy-dispersive X-ray spectrometer (EDS) (University of Aveiro).

The magnetic properties of original carbon nanotubes and sample filled with Fe_3O_4 were measured using a vibrating sample magnetometer (VSM, Lake Shore Cryotronics, Inc., model 7404, Institute of Physics, Latvia). Measurements were done at several temperatures from 293 K to 80 K for fields up to 10 kOe. Magnetic characterization of ferrofluid EMG 911 was carried out using a vibrating sample magnetometer (Cryogenic, University of Aveiro) at 80 K and 5 K.

III. RESULTS AND DISCUSSION

A. Filling of carbon nanotubes with ferrofluid

A typical TEM image of CNTs with Fe_3O_4 nanoparticles from an organic-based ferrofluid is shown in Fig. 3.

In the image, it can be easily identified the nanotubes filled with the Fe_3O_4 nanoparticles. Fig. 3 (inset) results from the EDS measurements of the CNTs filled Fe_3O_4 nanoparticles; these measurements confirm the presence of atomic iron in the CNTs.

The filling of the CNTs can be explained in terms of capillary forces induced during the impregnating process. Capillarity occurs if γ_{SV}, the surface tension at the solid-vapour interface for a dry tube, is larger than γ_{SL}, the surface tension at the solid-liquid interface, for the same tube during impregnation. The impregnation parameter, I, is defined as: $I = \gamma_{SV} - \gamma_{SL}$ [7].

World Academy of Science, Engineering and Technology
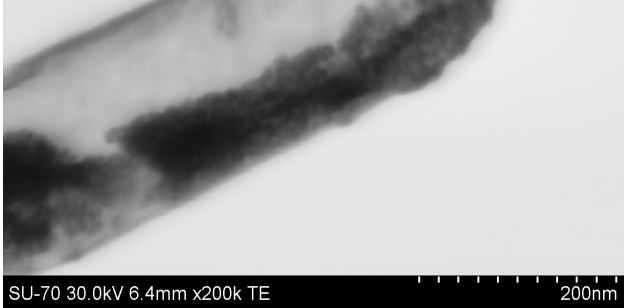International Journal of Materials and Metallurgical Engineering
Vol:6, No:7, 2012

Fig. 3 TEM image of CNTs filled with Fe_3O_4 nanoparticles. Inset: EDS results for CNTs filled with Fe_3O_4

According to Young's equation ($\gamma_{SV} - \gamma_{SL} = \gamma cos\theta_E$, γ: surface tension of liquid and θ_E: equilibrium contact angle), the impregnation criterion can be written as: $I = \gamma_{SV} - \gamma_{SL} = \gamma cos\theta_E > 0$.

Wetting, i.e. $\theta_E < 90°$, is necessary for capillarity action in CNTs [6-8]. Previous studies reported in the literature [6-8, 16, 17] have shown that elements and compounds with surface tension lower than 200 mN/m are suitable for wetting and filling the carbon nanotubes. The ferrofluid EMG 911 has a surface tension value of 68mN/m; therefore, as demonstrated, this ferrofluid is capable of strongly wetting and filling the nanotubes. However, it should be noted, there is some uncertainty about the driving forces of nanocapillarity, particularly in what concerns the filling by capillarity and the diameter of the tube. In [6], it is reported the capillarity was reduced when the inner diameter of the tube was reduced. Similarly, Ugarte et al. [8] note that the nanotubes with larger diameters are preferentially filled as compared to the tubes with smaller diameters; for the latter situation, the van der Waals repulsion forces are significantly higher than those of capillarity, and thus they may inhibit the penetration of the solution into the tube. In the present work, the relatively large inner diameter of the CNTs and the relatively small diameter of Fe_3O_4 nanoparticles of the ferrofluid associated with its low surface tension value corroborate the findings reported in the literature; therefore, our experimental results have a plausible explanation on the basis of the capillary forces.

B. Nanotubes magnetization

The original carbon nanotubes do not present any ferromagnetic properties. The magnetization curves of the CNTs samples after being filled with Fe_3O_4 nanoparticles are shown in Fig. 4.
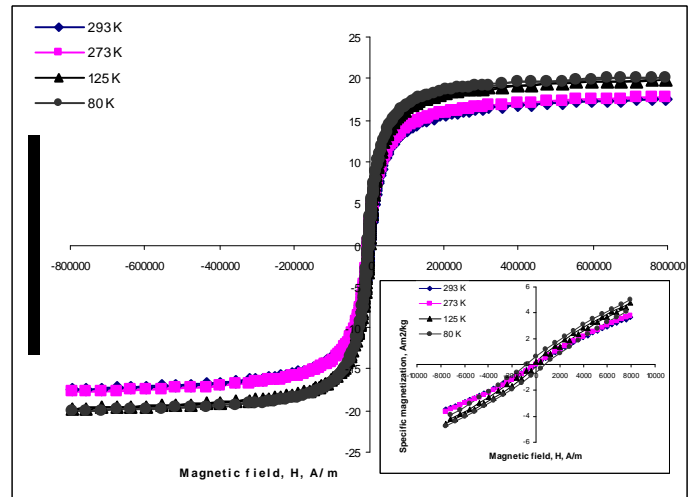

Fig. 4 Magnetization curves of CNTs filled with Fe_3O_4 at different temperatures. Inset: Enlargement of the region at low magnetic field

Fig. 4 indicates the magnetic CNTs show no hysteresis loop in the presence of a low magnetic field at room temperature and at 273 K, however, hysteresis is clearly observed at low temperature: at 125 K and 80 K (Fig. 5).
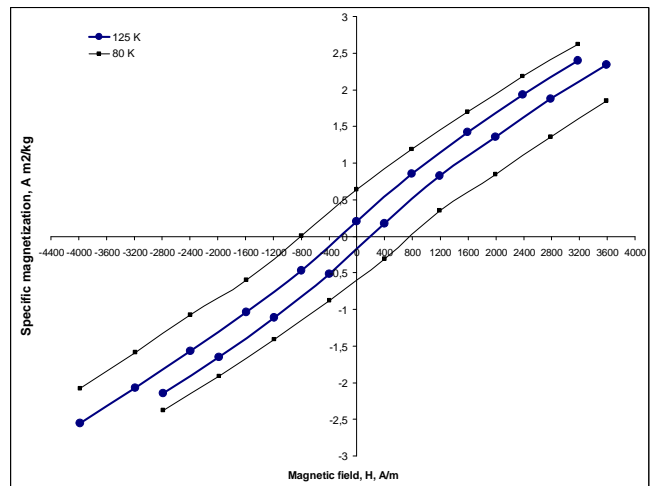

Fig. 5 Magnetization curves at low magnetic field of CNTs filled with Fe_3O_4 nanoparticles

This sample indicates that ferromagnetic properties do occur. The saturation magnetization depends on the temperature, and it varies from 17.53 Am^2/kg to 20.10 Am^2/kg; this range is in good agreement with the values reported in the literature [18].

Detailed analysis of the magnetic behaviour of CNTs with magnetic particles was done. The some parameters such saturation magnetization M_S, coercivity H_C, remanent magnetization M_R and ratio $R=M_R/Ms$ at different temperatures are presented in Table I.

TABLE I
SOME CHARACTERISTICS OF CNTs WITH MAGNETIC NANOPARTICLES

Temperature T, K	H_C, A/m	M_s, Am^2/kg	M_r, Am^2/kg	Ratio R =

World Academy of Science, Engineering and Technology
International Journal of Materials and Metallurgical Engineering
Vol:6, No:7, 2012

	(Oe)			M_r/M_s
293	0	17.53	0	0
273	0	17.78	0	0
175	0	19.26	0	0
125	217.7 (2.75)	19.78	0.2	0.01
80	794.4 (9.98)	20.10	0.63	0.03

At low temperatures the coercivity of a system of non-interacting and randomly oriented particles is expected to follow the relation [19]:

$$H_C(T) = H_{C0}\left[1 - \left(\frac{T}{T_B}\right)^{1/2}\right] \qquad (1)$$

For CNTs with Fe_3O_4 at low temperatures we obtained the following extrapolated value of H_{C0}: 3101.13 A/m (~ 40 Oe) at T= 125 K; and 800 A/m (~ 10 Oe) at T = 80 K.

Besides, the blocking temperature T_B was estimated. At temperatures well below T_B, the hysteresis appears and, consequently the superparamagnetism disappears, as the thermal energy is no longer sufficient to overcome the magnetic anisotropy energy. In our case the value of T_B is equal to 144.58 K (~ 145 K).

Alternatively, the magnetic properties of ferrofluid EMG 911 was estimated taking into account the ferrofluid shows the significant hysteresis in the presence of a low magnetic field at 80 K and 5 K. Detailed analysis for EMG 911 (similar to that described above for the CNTs filled with Fe_3O_4) yields the following parameters:
$H_{C0} \approx 21073$ A/m (~264 Oe) at T = 80K;
$H_{C0} \approx 180000 \pm 20000$ A/m (~2250 ± 250 Oe) at T = 5K.

For the ferrofluid EMG 911 the blocking temperature T_B is equal to 93±6 K.

This value is much higher than that for pure magnetite ($T_B \approx$ 31 K) as well for CNTs filled with Fe_3O_4 ($T_B \approx$ 58 K) is comparable with values reported in the literature [13]. This may infer that there is an increase in the strength of the interactions between magnetic nanoparticles and CNT walls [13], [20] as well as those among magnetic nanoparticles inside of carbon nanotubes. It is generally accepted that the magnetic interactions that dominate magnetic nanoparticle assemblies are dipole–dipole interparticle interactions (which are always present) and exchange interactions through the surfaces of particles that are in close contact [13], [20]. It is known [21] the exchange interaction among Fe_3O_4 particles might be minimal because the average edge–edge separation between particles is large due to the presence of the surfactant. To prevent the particles from sintering, they are coated with surfactant molecules whose polar head groups attach to the particle surfaces.

When the particles are magnetically collected and then transferred to a low boiling point solvent, solvent evaporation leads to the formation of ordered arrays. The surfactant coating remains as a barrier between adjacent nanoparticles [21].

However, the tight packing of Fe_3O_4 inside CNTs can reduce the average distance between Fe_3O_4 particles, thus enhancing the strength of the dipolar interparticle interaction. In the present case the filling level of carbon nanotubes with magnetic particles is about 46 %; this value is determined in section C below. This percentage corroborates our premise that there occurs a strong interaction among the magnetic particles inside the CNTs, which are the object of the present study.

The average magnetic properties of the CNTs with the magnetic particles were estimated based on calculations and from the data depicted in Fig. 4 and Fig. 5.

C. Estimation of the quantity of magnetic nanoparticles inside the nanotube

In the experiments, the number of tubes in a piece of alumina membrane of cross-section area S_t is estimated by

$$N_t = S_t/s_t \times p, \qquad (2)$$

where s_t is the area of one tube on the surface of the membrane of area S_t ($S_t = 0.33 \times 10^{-4}$ m^2) and p is the membrane porosity.

This area, s_t, can be approximately calculated by the formula:

$$s_t = \pi d_t^2/4, \qquad (3)$$

where d_t is the diameter of the tubes. The average diameter of the nanotubes, d_t, is 124 nm. Therefore, the cross-section of one nanotube, s_t, is 1.21×10^{-4} m^2. The membrane porosity, p, in Eq. 2, i.e., the area occupied by the nanotubes divided by the membrane area, it was found from the analysis of the AFM images to be equal to 0.12 [15]. The total number of nanotubes on the membrane, N_t, is 3.24×10^8.

The magnetic moment of a nanoparticle can be calculated taking into account the magnetic CNTs show no hysteresis in the presence of a low magnetic field at room temperature; therefore, the magnetite nanoparticles can be assumed to behave as superparamagnetic in the sample [22]. In this case, the magnetization curve of CNTs filled with Fe_3O_4 is described by eq.23.52 of Vonsovskij [22] with two parameters - the magnetic moment of a nanoparticle, μ_{np}, and the concentration the nanoparticles in the sample, n_{np}, namely:

$$M(H)= n_{np} \mu_{np} [\text{cth} (\mu_{np} H/k_b T) - k_b T/ \mu_{np} H] \qquad (4)$$

This dependence in a high intensity magnetic field can be approximated by a straight line (eq.23.54 of [22]) yielding:

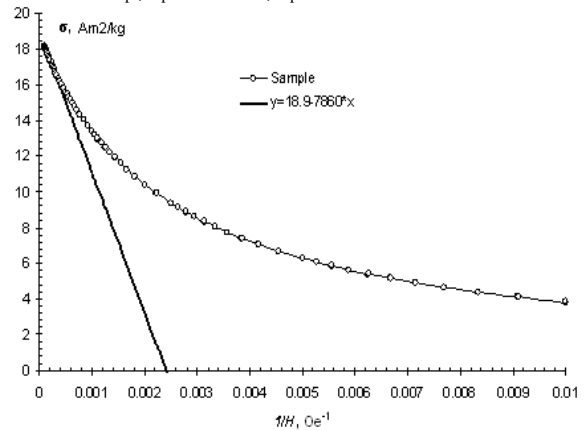$$M(H)= n_{np} \mu_{np} [1 - k_b T \mu_{np} H] \qquad (5)$$

Fig. 6 Magnetization curve of CNTs filled with Fe_3O_4 using the ordinates (M -1)-H

World Academy of Science, Engineering and Technology
International Journal of Materials and Metallurgical Engineering
Vol:6, No:7, 2012

The concentration of the nanoparticles in the sample n_{np} and magnetic moment of a nanoparticle μ_{np} were estimated after approximating the experimental dependence of the magnetization (y=M, x=1/H) by a straight line at high magnetic field (Fig. 6). In this case n_{np} is equal to 2×10^{17} l/g and μ_{np} is equal to 9.62×10^{-17} emu (9.62×10^{-20} Am^2).

Hence, the total concentration of nanotubes in the sample (m=2.46 mg), n_t is estimated to be: $n_t = N_t/ m_; n_t = 1.32 \times 10^{12}$ l/g. The total number of nanoparticles inside the nanotubes is estimated to be

$N_{npt} = n_{np}/n_t$ and $N_{npt} = 1.5 \times 10^5$.

Since, volume of single magnetic nanoparticle and volume of single nanotube are equal $V_{np} = 2.2 \times 10^{-25}$ m^3 and $V_t = 7.24 \times 10^{-20}$ m^3, respectively [15], the percentage (%) of filling of the nanotube volume by the magnetic particles can be estimated from:

$$X = N_{npt} \times V_{np} / V_t ;$$

as X = 0.46 or 46%.

This corresponds to 46 % filling by volume of the Fe_3O_4 particles in the nanotubes, which is a very promising result.

IV. CONCLUSION

It is proposed an *ad-hoc* modification of a published procedure for the fabrication of magnetic CNTs. The new procedure is based on capillarity effects using wetting fluids and it employs CNTs with a large diameter (more than 100 nm), which were loaded with magnetic Fe_3O_4 particles. The magnetic properties of CNTs filled with Fe_3O_4 particles were investigated at different temperatures. The resulting composite at room temperature shows excellent superparamagnetic properties in perfect consonance with other magnetic materials. However, the superparamagnetic properties disappear at low temperatures (125 K and 80 K). The blocking temperature T_B was estimated to be around 145 K. Overall, the proposed methodology opens the opportunity for the simplified fabrication of magnetic nanotubes aiming engineering applications and, in particular, the production of magnetic nanofluids with high thermal conductivity and capable of being steered by appropriate magnetic fields.

NOMENCLATURE

d_t	diameter of nanotubes (nm)
I	impregnation parameter
H	magnetic field strength (A/m)
H_C	coercivity (A/m)
m	mass of sample (mg)
M	magnetization (Am^2/kg)
M_R	remanent magnetization (Am^2/kg)
M_S	saturation magnetization (Am^2/kg)
n_{np}	total concentration of the nanoparticles (l/g)
n_t	total concentration of the nanotubes (l/g)
N_{npt}	total number of nanoparticles inside the nanotubes
N_t	number of nanotubes in a piece of alumina membrane
p	membrane porosity
R	ratio M_R/ M_S
s_t	area of one tube on the surface of the membrane
S_t	cross-section area of alumina membrane
γ	surface tension of of liquid (mN/m)
γ_{SL}	surface tension at the solid-vapour interface (mN/m)
γ_{SV}	surface tension at the solid-liquid interface (mN/m)
θ_E	equilibrium contact angle (degree)
μ_{np}	magnetic moment of nanoparticle (Am^2)

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World Academy of Science, Engineering and Technology
International Journal of Materials and Metallurgical Engineering
Vol:6, No:7, 2012

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