Journal of Applied Sciences Research, 9(6): 3411-3416, 2013
ISSN 1819-544X
This is a refereed journal and all articles are professionally screened and reviewed

ORIGINAL ARTICLES

The Effect of Chemical Solutions (Isopropyl Alcohol, Dichloromethane, Acetone and Triton X-100) on the Dispersion of Single-Walled Carbon Nanotubes

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ABSTRACT

The effect of chemical solutions on the dispersion of single-walled carbon nantubes (SWCNTs) was studied. The SWCNTs were dispersed using several chemical solutions such as isopropyl alcohol (IPA), dichloromethane (DCM), acetone and triton X-100 (Triton-X) under ultrasonically process. The results show that the types of chemical solutions greatly affect the dispersion of SWCNTs. The IPA solution is found to be the best solution to disperse SWCNTs using ultrasonication process due to the difficultly of evaporation in room temperature to get a transparent solution.

Key words: Carbon nanotubes, ultrasonically, dispersion.

Introduction

Carbon nanotubes (CNTs) have become the subject of intense investigation since 1991 because of their marvelous electrical, mechanical, electromechanical and chemical properties (Ijima, S., 1991; Saito, R., *et al*., 1998). There are two different types of CNTs: single-walled carbon nanotubes (SWCNTs) and multi-walled carbon nanotubes (MWCNTs).Without any chemical functionalization, CNTs are hard to be dispersed in any liquids due to the van der Waals force interactions that tend to cause agglomeration of CNTs and prevent them to be used in any application.Thus, debundling and individual dispersion of CNTs in an aqueous solution is highly important to use them in related research and various applications (Syamsul, N., *et al*., 2010; Wei-Wen Liu, U. Hashim, Sharma Rao, 2012; Adam, T., *et al*., 2013; Adam, T., U. Hashim, K.L. Foo, 2013; Adam, T., *et al*., 2013; Adam, T., *et al*., 2013; Adam, T., *et al*., 2013).

There is a growing body of literature reporting on issues of dispersibility of CNTs in aqueous media which demonstrating an increasing interest in this field. Depending on the application, different methods were used to debundle CNTs homogenously. CNTs can be dispersed in solvents which involve the chemical treatment to individualize while simultaneously creating interactions between the CNTs surface and solvent. When debundling CNTs in aqueous media mainly two methods are used: Firstly, the CNTs can be chemically functionalized by adding functionalized groups to disperse CNTs homogenously in aqueous environments (Wei-Wen Liu, U. Hashim, Sharma Rao, 2012; Adam, T., U. Hashim, 2012). However, this can destroy the structure of CNTs and affects the mechanical and electrical properties of CNTs (Jenny Hilding, *et al*., 2003; Adam, T., U. Hashim, 2012; Adam, T., U. Hashim, 2012; Adam, T., U. Hashim, 2012). Alternatively, the non-covalent physical adsorption of surfactants and polymers is widely used to enhance the dispersibility of the CNTs without damage the structure of CNTs. In this case, the dispersed CNTs are stabilized by the electrostatic repulsion formed around them (Vaisman, L., H.D. Wagner, G. Marom, 2006).

Recently, absorption of surfactants onto the sidewall structure and dispersion of CNTs into individual CNT in an unzipping fashion was reported (Strano, M.S., *et al*., 2003; Adam, T., U. Hashim, 2012). Several surfactants have been demonstrated to disperse CNTs such as sodium 4-dodecylbenzenesulfonate (NaDDBS), hexadecyl (trimethyl) azanium bromide (CTAB), Triton® X-100, and sodium dodecane-1-sulfonate (SDS) (Islam, M.F., *et al*., 2003; Adam, T., *et al*., 2012). Contradictory results were often obtained from the CNT's dispersion studies. For example, (Vaisman *et al*. 2006; Hashim, U., *et al*., 2012) reported that non-ionic surfactants are suitable to disperse CNTs in organic solvents while ionic surfactants are better for dispersing CNTs in aqueous solution. However, the non-ionic surfactant Triton® X-100 was found to debundle CNTs better than the anionic surfactant SDS, which was due to the p–p stacking capability of the former (Islam, M.F., *et al*., 2003; Al-Mufti, W.M., U. Hashim, T. Adam, 2012). In the present study, we examined the aqueous dispersions of functionalized single-walled carbon nanotubes (SWCNTs) using four different types ofsurfactants: isopropyl alcohol (IPA), dichloromethane (DCM), acetone and Triton® X-100 (Triton-X).The dispersed functionalized SWCNTs were investigated using scanning electron microscopy (SEM) and fourier transform infrared

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J. Appl. Sci. Res., 9(6): 3411-3416, 2013

spectroscopy (FTIR) to find the best solvent to debundle SWCNTs (Nazwa, T., U. Hashim, A. Saifullah, 2011; Th S Dhahi, U. *et al*., 2011; Adam, T., U. Hashim, 2012; Adam, T., *et al*., 2012; Adam, T., U. Hashim, 2012).

Materials And Method

Materials:

Functionalized SWCNTs-COOH (>97% carbon purity and <3% metal oxides impurities), IPA, DCM, acetone and Triton-X were purchased from Sigma Aldrich, Maalysia. All materials were used as-received.

Preparation of dispersion of SWCNTs in various surfactants:

The dispersion process was conducted in two set of experiments. In the first set of experiment, 10mg of functionalized SWCNTs were mixed with each solution at 100ml, namely the acetone, IPA, DCM and Triton X. The purchased SWCNTs were readily functionalized with the carboxyl group through the chemical modification with the hydrochloric acids (HCl). After that, all mixture of functionalized SWCNTs and surfactants were sonicated for 30mins.

In the second set of experiments, 10μl was extracted from each of the sonicated mixtures for a total of 10 times. The droplets were then mixed with 10ml of new surfactants (acetone, IPA, DCM and Triton X) and were sonicated again for 30mins. Next, all the mixtures were left aside for 14 days and no sonicationwere carried out to determine the best surfactants to disperse functionalized SWCNTs. The two sets of experiment for functionalized SWCNTs dispersion are illustrated in Fig. 1.The experimental setup for ultrasonic process is shown in Fig. 2.

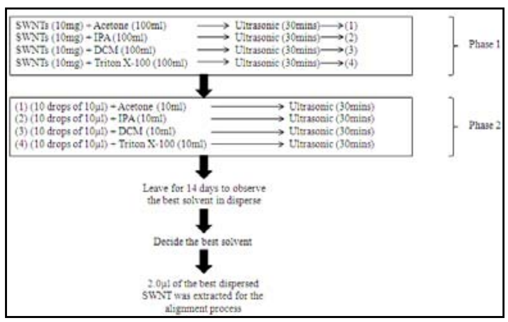


Fig. 1: Functionalized SWCNTs dispersion process flow

Fig. 2: Experimental setup for ultrasonic process

Characterization of dispersion:

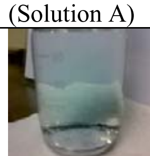
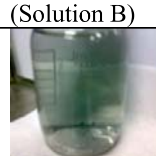
The dispersion of functionalized SWCNTs in surfactants were characterized using FTIR operating between 800-4000cm^{-1} to check the presence of reactive groups on SWCNTs surface. All pictures of functionalized SWCNTs dispersion in surfactants in beakers were captured using a flash compact digital camera for the 1st and 14th day. The morphology of dispersion was examined by SEM.

Results And Discussion

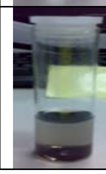
Comparison of dispersion:

For this dispersion study, it is necessary to conduct ultrasonic process to ensure that functionalized SWCNTs were fully dispersed in the surfactants. In others words, the purpose of ultrasonication was todebundle the functionalized SWCNTs in the surfactants to obtain a transparent solution by utilizing the ultrasonic waves. Fig.3 shows the compact camera and SEM images for the two sets of experiment. From the compact camera images, we can clearly see that black particles (functionalized SWCNTs) were distributed at the bottom of beakers in solution A for all surfactants which suggests the functionalized SWCNTs were not well dispersed without the sonication. For solution B, black particles (functionalized SWCNTs) were clearly observed in acetone surfactant whereas IPA, DCM and Triton X dispersed them in much better manner. Due to the IPA surfactant was turned into dark color, it is believed that IPA was the best surfactant to disperse functionalized SWCNTs. It is noticeable that transparent solutions C were obtained by adding 10 droplets of solution B into IPA and Triton X. However, black particles (functionalized SWCNTs) were observed in the DCM and acetone surfactant which indicates that unsuccessful of dispersion (Fig. 3).

The results show that IPA surfactant can disperse the functionalized SWCNTs with a most transparent solution after 14 days as compared to other surfactants (Fig.3). It is because some black particles (functionalized SWCNTs) were observed clearly at the bottom of bottles for acetone, DCM and Triton X-100. This is in agreement with the SEM images that IPA able to fully disperse the SWCNTs bundle into individual SWCNT (Fig. 3).

Solutions	Before Ultrasonic (Day1) (Solution A)	After Ultrasonic (Day1) (Solution B)
Acetone		
IPA		
DCM		
Triton X-100		

Solutions	10 droplets of 10µl (from Solution B) + 10ml of new solvent + ultrasonic (Solution C)	Days 14 observation (solution D)
Acetone		
IPA		

DCM		
Triton X-100		

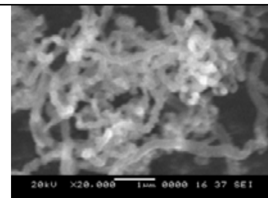
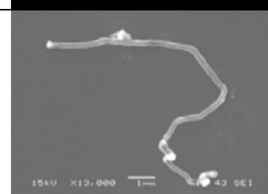
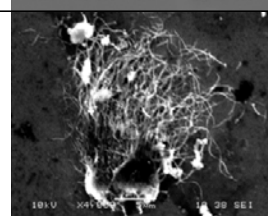
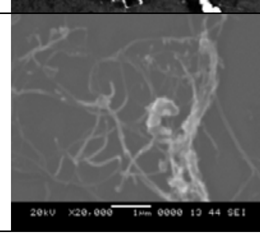
Solutions	SEM Results
Acetone	
IPA	
DCM	
Triton X-100	

Fig. 3: Dispersion Process and Results

FTIR characterization:

For FTIR, the absorbance (A) peak intensity is directly proportional to the amount of functional group (CM^{-1}) existed in the sample (Fig. 5). Figure 5 shows the FTIR spectrum of acetone, IPA, DCM and Triton X surfactants.In dispersion, the functionalized SWCNTs are dispersed in these four types of solvents, the HCl modified SWCNTs contained carboxyl group, –COOH and able to react with solvents. For SWCNTs mixed in IPA solvent (Red line), it is clearly shown that at wavelength 3400 cm^{-1}, 2950 cm^{-1}, 1150 cm^{-1}, and 960 cm^{-1} was contained alkenes, =CH stretch, alkanes, C-H stretch, carboxylic acids C-O and O-H bend respectively, which Acetone, DCM, and Triton-X do not have the similar group in IPA solvents. While only for SWCNT mixed with DCM solvent (green line), it contained alkanes, CH_2 bend at 1250 cm^{-1}, and acid chlorides, C-Cl stretch at 730 cm^{-1}. On the other hand, only SWCNTs mixed with Acetone solvent (Blue line) have sharp peak at 1700 cm^{-1} which contained alkenes, C=C stretch, at 1350 cm^{-1} and 1216 cm^{-1}contained ketones, C-O stretch, and carboxylic acids, C-O stretch, O-H bend. While for SWCNTs mixed in Triton-X solvent (purple line), it have sharp peak at 2840 cm^{-1}, 1600 cm^{-1}, 1500 cm^{-1}, 1240 cm^{-1}, and 1100 cm^{-1} which contained carboxylic acids, O-H stretch, alkenes, C=C stretch, alkanes, CH_2 bend alcohols, C-O stretch respectively.

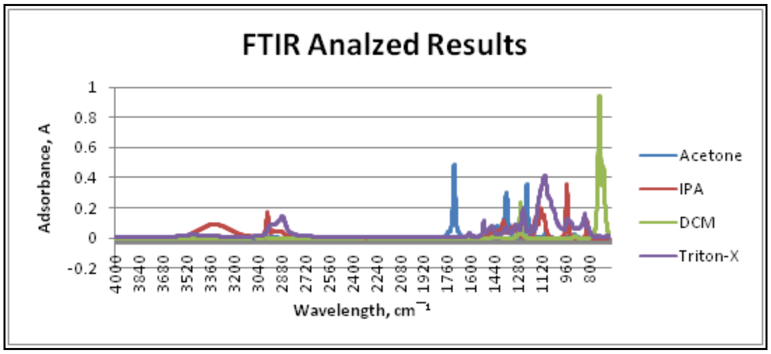


Fig. 5: FTIR Analyzed Results

Conclusions:

In conclusion we present this dispersion study to get flexible way and can straight proceed our work in order to drop selected solvent apply for our fabricated device. As a summary for dispersion study, SWCNTs in IPA solvent is the best choice for dispersion process. It is because stability of SWCNTs mixed with IPA solvent is very good. It was still able to remain debundled after some period of time compared with other solvents. After investigated the 4 solvents for dispersion process, the SWCNTs mixed with DCM solvent is the fastest evaporate to the air, followed by acetone and IPA while the slowest evaporate to the air is Triton-X. The SWCNTs mixed with DCM solvent is not suitable as alignment due to it evaporates the fastest thus lowering the possibility for SWCNT alignment. Whereas, SWCNTs mixed with Triton-X may not be chosen for alignment due to it evaporates into air the slowest. This also lowers the possibility for alignment due to too many strands of SWCNTs will be aligned thus causing instability.

Acknowledgment

The authors wish to thank Universiti Malaysia Perlis (UniMAP), Geran MOA Sciencefund 050-01-15-SF1009 and FRGS for giving the opportunities to do this research in the Micro & Nano Fabrication Clean room. The appreciation also goes to all the team members in the Institute of Nano Electronic Engineering (INEE) especially Nano Biochip Research Group members.

References

Adam, T., U. Hashim, 2012. Additivity ensures stability of design: Role of orthogonal arrays for process optimization through additive model *ICSSBE 2012 - Proceedings, 2012 International Conference on Statistics in Science, Business and Engineering: "Empowering Decision Making with Statistical Sciences"*, art. no. 6396572, pp: 277-280.

Adam, T., U. Hashim, 2012. Low resistance electrical layer formation: A simulation study of diffusive rapid thermal process on implanted dopant species for electronics active devices *Proceedings of International Conference on Computational Intelligence, Modelling and Simulation*, art. no. 6338116, pp: 428-430.

Adam, T., U. Hashim, 2012. Simulation study of non ionic implantation process: Thinner electrical interfacial semiductor junction formation using ionic diffusion process *Proceedings of International Conference on Computational Intelligence, Modelling and Simulation*, art. no. 6338117, pp: 431-433.

Adam, T., U. Hashim, 2012. Statistical parameter evaluation for swing curves for the 1.2 μm and 1.8 μm resist thickness in CMOS photolithography process technology *ICSSBE 2012 - Proceedings, 2012 International Conference on Statistics in Science, Business and Engineering: "Empowering Decision Making with Statistical Sciences"*, art. no. 6396571, pp: 273-276.

Adam, T., U. Hashim, 2012. Taguchi's method of statistical design to form an ultra thin silicon dioxide *Journal of Applied Sciences Research*, 8(8): 4249-4253.

Adam, T., U. Hashim, 2012. The effect of exposure time and development time on photoresist thin film in Micro/Nano structure formation *10th IEEE International Conference on Semiconductor Electronics, ICSE 2012 - Proceedings*, art.no. 6417102, pp: 107-110.

Adam, T., U. Hashim, 2012. Ultra thinpolysilicon layer formation: Statistical process optimization by Taguchi's technique *ICSSBE 2012 - Proceedings, 2012 International Conference on Statistics in Science, Business and Engineering: "Empowering Decision Making with Statistical Sciences"*, art. no. 6396546, pp: 155-157.

Adam, T., U. Hashim, D. Isa, C.Y. Yee, 2012. An Electric Double-Layer Capacitor (EDLC) Production for optimum energy driven communication system using taguch technique *Proceedings of International Conference on Computational Intelligence, Modelling and Simulation*, art.no. 6338112, pp: 405-409.

Adam, T., U. Hashim, K.L. Foo, 2013. Microfluidics design and fabrication for life sciences application *Advanced Science Letters*, 19(1): 48-53.

Adam, T., U. Hashim, K.L. Foo, T.S. Dhahi, T. Nazwa, 2013. Technology development for nano structure formation: Fabrication and characterization (2013) *Advanced Science Letters*, 19(1): 132-137.

Adam, T., U. Hashim, M.E. Ali, P.L. Leow, The electroosmosis mechanism for fluid delivery in PDMS multi-layer microchannel (2013) *Advanced Science Letters*, 19(1): 12-15.

Adam, T., U. Hashim, P.L. Leow, 2012. Design and fabrication of passive fluid driven microchamber for fast reaction assays in nano lab-on-chip domain *Journal of Applied Sciences Research*, 8(8): 4254-4261.

Adam, T., U. Hashim, P.L. Leow, K.L. Foo, P.S. Chee, 2013. Selection of optimal parameters in fabrication of poly(dimethylsiloxane) microfluidics using taguchi method *Advanced Science Letters*, 19(1): 32-36.

Adam, T., U. Hashim, P.L. Leow, Q.H. D, 2013. Fabrication of nanowire using ash trimming technique *Advanced Materials Research*, 626: 1042-1047.

Al-Mufti, W.M., U. Hashim, T. Adam, 2012. Current trend in simulation: Review nanostructures using comsolmultiphysics *Journal of Applied Sciences Research*, 8(12): 5579-5582.

Hashim, U., T. Adam, P.N.A. Diyana, S.T. Ten, 2012. Computational micro fluid dynamics using COMSOL multiphysics for sample delivery in sensing domain *IEEE-EMBS Conference on Biomedical Engineering and Sciences, IECBES 2012*, art. no. 6498208, pp: 969-973.

Ijima, S., 1991. Nature 354: 56.

Islam, M.F., E. Rojas, D.M. Bergey, A.T. Johnson, A.G. Yodh, 2003. Nano Lett., 3: 269.

Jenny Hilding, Eric A. Grulke, Z. George Zhang, Fran Lockwood, 2003. "Dispersion of nanotube in Liquid", Journal of Dispersion Science and Technology, 24(1): 1-41.

Nazwa, T., U. Hashim, A. Saifullah, 2011. Th. S. Dhahi, "PolysiliconNanogap capacitive biosensors for the pH detection", Micro and Nanoelectronics (RSM), 2011 IEEE Regional Symposium, pp: 250-252.

Saito, R., G. Dresselhaus, M.S. Dresselhaus, 1998. Physical Properties of Carbon Nanotubes, Imperial College Press, London.

Strano, M.S., V.C. Moore, M.K. Miller, M.J. Allen, E.H. Haroz, C. Kittrell, R.H. Hauge, R.E. Smalley, J. Nanosci, 2003. Nanotechnol., 3: 81.

Syamsul, N., M.N.M. Nuzaihan, U. Hashim, 2010. "Development of carbon nanotube based biosensor fabrication for medical diagnostics application", Enabling Science and Nanotechnology (ESciNano), 2010 International Conference, pp: 1-2.

Th S Dhahi, U. Hashim, N.M. Ahmed, MdEaqub Ali, T Nazwa, 2011. "Electrical characterization of in-house fabricated polysilicon micro-gap for yeast concentration measurement", Journal of Engineering and Technology Research, 3(8): 246-254.

Vaisman, L., H.D. Wagner, G. Marom, 2006. The role of surfactants in dispersion of carbon nanotubes.Adv Colloid Interface Sci., 128-130: 37-46.

Wei-Wen Liu, U. Hashim, Sharma Rao, 2012. "Carbon nanotubes-based electrochemical biosensors", Biomedical Engineering and Sciences (IECBES), 2012 IEEE EMBS Conference, pp: 392-397.