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The effect of preparation conditions and biopolymer dispersants on the properties of SWNT buckypapers

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The effect of preparation conditions and biopolymer dispersants on the properties of SWNT buckypapers

Abstract

The effect of varying preparation conditions on the properties of buckypapers made using single-walled carbon nanotubes (SWNTs) was systematically investigated. Changing the sonication time, final dispersion volume or membrane filter used to prepare SWNT-Triton X-100 buckypapers all generally had only a small influence on the density, thickness, contact angle and electrical conductivity of the resulting material. More significant changes were noted when the effects of variations in the above preparation conditions on the mechanical properties and surface morphology of SWNT-Triton X-100 buckypapers were investigated. However, the largest changes in properties were found when various biopolymers (bovine serum albumin, lysozyme, gellan gum and chitosan) were used instead of Triton X-100 to prepare dispersions from which the buckypapers were made. It was observed that the electrical conductivity of the buckypapers decreased as the molecular mass of the biopolymer dispersant used increased, whereas the mechanical properties of the materials showed significant improvements.

Keywords

effect, swnt, preparation, buckypapers, conditions, biopolymer, dispersants, properties

Disciplines

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The effect of preparation conditions and biopolymer dispersants on the properties of SWNT buckypapers†

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The effect of varying preparation conditions on the properties of buckypapers made using single-walled carbon nanotubes (SWNTs) was systematically investigated. Changing the sonication time, final dispersion volume or membrane filter used to prepare SWNT–Triton X-100 buckypapers all generally had only a small influence on the density, thickness, contact angle and electrical conductivity of the resulting material. More significant changes were noted when the effects of variations in the above preparation conditions on the mechanical properties and surface morphology of SWNT–Triton X-100 buckypapers were investigated. However, the largest changes in properties were found when various biopolymers (bovine serum albumin, lysozyme, gellan gum and chitosan) were used instead of Triton X-100 to prepare dispersions from which the buckypapers were made. It was observed that the electrical conductivity of the buckypapers decreased as the molecular mass of the biopolymer dispersant used increased, whereas the mechanical properties of the materials showed significant improvements.

1. Introduction

There continues to be increasing interest in carbon nanotubes (CNTs), as more and more scientists become aware of their unique properties, which make them attractive for a wide range of applications.^{1–3} Before the potential of CNTs can be utilized, however, it is necessary to overcome their lack of solubility in most common solvents, which is a consequence of the natural tendency of materials as hydrophobic as CNTs to occur in aggregates.^{2,4–7} Two principal methods now exist for overcoming this inherent lack of processability. These involve covalent functionalization of the CNTs,^{2,3,8,9} or sonicating them in the presence of a dispersant.^{2,10–12} Covalent functionalization usually involves oxidation using a concentrated acid. This approach produces hydrophilic carboxylic acid residues on the surfaces of the CNTs, but can create defect structures that compromise their electronic and mechanical properties.¹³

The alternative approach uses ultrasonic energy to break up large bundles of CNTs. This produces many individual tubes, which are stabilized by non-covalent interactions with an added amphiphilic molecule (the dispersant).^{10,14} A useful feature of the sonication method is that it is possible to filter the resulting dispersions to obtain free-standing CNT films,¹⁵ also known as “buckypapers”. The non-ionic surfactant octylphenolethoxylate (more commonly referred to as Triton X-100) has been found to be very effective for dispersing both single-walled carbon nanotubes (SWNTs) and multi-walled carbon nanotubes (MWNTs),

and for producing buckypapers.⁷ It is thought that the aromatic rings of Triton X-100 π -stack onto the external graphene lattice of the CNTs leading to stable dispersions being formed.¹⁶

A buckypaper generally consists of a tangled network of CNT ropes and bundles, as well as some individual tubes, held together by non-covalent interactions. Since buckypapers have been proposed for a number of applications, it is of considerable interest to understand how their properties are affected by changing the conditions employed during preparation. For example, Smajda and co-workers investigated the effects of changing the solvent, sonication time and amount of CNTs used on the morphological properties of MWNT buckypapers and concluded that altering these parameters had little effect.¹⁷ In a second study by the same group it was shown that the lengths of MWNTs used to manufacture buckypapers have a significant effect on the diameter of the pores present in the final material.¹⁸ To the best of our knowledge there do not appear to have been any similar investigations conducted using SWNTs, or which systematically examine the effect of varying the dispersant on the properties of buckypapers.

There has been recent interest in using buckypapers for bionanotechnology applications, such as a supporting medium for the growth of cells^{19–21} and bacteriocidal filtration membranes.^{22,23} In order for buckypapers to be suitable for these applications they must be biocompatible with whatever tissue or media they come into contact with. It has been shown that Triton X-100, which is widely used as a dispersant for preparing buckypapers, can cause adverse biological effects making it essential to remove it prior to preparing buckypapers for biological applications.²⁴

Another method for preparing biocompatible buckypapers may be to prepare them from dispersions made by sonicating CNTs in the presence of biomolecules such as carbohydrates, proteins and nucleic acids. This method has the added attraction of potentially producing buckypapers that also exhibit the

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properties of the biomolecular dispersant, which can become wrapped around the CNTs during the formation of dispersions. A number of groups have recently explored the ability of proteins, carbohydrates and nucleic acids to disperse SWNTs and MWNTs.^{25–30} For example, Iijima's group examined the ability of a range of proteins, including egg white lysozyme, bovine serum albumin (BSA), papain and pepsin to disperse SWNTs in aqueous solution, and found that only the former two proteins were suitable.²⁵ This was attributed to favourable interactions between SWNTs and hydrophobic regions of these proteins. Later work by Nepal and Geckeler suggested that proteins containing a high proportion of basic residues were very suitable for dispersing CNTs, but this was also influenced by solution pH.²⁶ These studies, and others, demonstrate how proteins can be used to prepare CNT dispersions. However, there do not appear to have been any prior reports of the properties of buckypapers made from such dispersions.

A number of carbohydrate polymers have also been investigated for their ability to disperse CNTs.^{27–30} One of these is chitosan, which can separate SWNTs based on differences in their diameter, with only smaller diameter tubes being preferentially wrapped around by the carbohydrate and dispersed in an aqueous medium.²⁸ Another polysaccharide shown to disperse both SWNTs and MWNTs is gellan gum. This carbohydrate can disperse CNTs when present at concentrations as low as 0.0001% (w/v).²⁹ CNT dispersions prepared using gellan gum have been used to form optically transparent, self-supporting films that display high sensitivity towards water vapour.³⁰ However, there have not been any reports of buckypapers made with this dispersant.

The principal aim of the current work was to examine the effect of varying preparation conditions on the electrical and mechanical properties of buckypapers prepared using SWNTs. The preparation conditions examined included sonication time, final dispersion volume, and choice of dispersant. All buckypapers were prepared by vacuum filtration of dispersions through a synthetic membrane filter, which was also systematically varied, and were characterized by optical and scanning electron microscopy, and through measurement of their thickness, density, contact angle, electrical conductivity and mechanical properties.

2. Experimental

2.1 Reagents

The HiPco SWNTs used in this study were obtained from Unidym™ (lot number P0348) and were used without further purification. Triton X-100 (molecular mass 625 Da), chicken egg white lysozyme (molecular mass 14 300 Da), bovine serum albumin (molecular mass 69 300 Da), and low molecular mass fraction chitosan (75–85% deacetylated; molecular mass 42 200 Da) were obtained from Aldrich and used as-received. CP Kelco provided food grade gellan gum (Kelcogel®, batch number 7C9228A and molecular mass 250 000 Da). All solutions were prepared in Milli-Q water (18 MΩ cm). In the case of chitosan, 1% (v/v) acetic acid (AR grade, Ajax Chemicals) was added to solubilize the dispersant. Hydrophilic 0.2 μm cellulose nitrate (CN) and 0.45 μm nylon membrane filters were obtained

from Micro Filtration Systems, while hydrophobic 5.0 μm polytetrafluoroethylene (PTFE) and 0.45 μm polyvinylidene fluoride (PVDF) membrane filters were purchased from Millipore.

2.2 Preparation of dispersions

All dispersions were prepared using a SWNT concentration of 0.1% (w/v). The concentration of Triton X-100 in solutions to be sonicated was always 1% (w/v), while a series of absorption spectrophotometric experiments were performed to determine the optimal concentration of biopolymers for forming dispersions to be used for making buckypapers. In a typical experiment, 15 mg of SWNTs were dispersed in 15 mL of dispersant solution using a Branson 450 (400 W, Ultrasonics Corp.) digital sonicator horn with a probe diameter of 10 mm. The conditions used were an amplitude of 30%, pulse duration of 0.5 s and pulse delay of 0.5 s. During sonication, the sample vial was placed inside an ice–water bath to minimize increases in temperature. The only exception to this was when gellan gum was used. In these cases the sample vial was placed in a warm water bath (~70 °C) to prevent gelation from occurring. Two of the above dispersions were prepared and added to a further 50 mL of dispersant solution (e.g. 1% (w/v) Triton X-100) before being subjected to further treatment in an ultrasonic bath (Unisonics, power transducer output = 50 W) for 3 min. The resulting homogeneous 80 mL dispersions containing 30 mg of SWNTs were then diluted, when required, to their final volume with Milli-Q water, prior to processing into buckypapers.

2.3 Preparation of buckypapers

Circular buckypapers measuring approximately 37 mm in diameter were prepared by vacuum filtration of dispersions through commercial membrane filters housed in an Aldrich glass filtration unit, and using a Vacuubrand CVC2 pump that typically operated between 30 and 40 mbar. The tops of the filtration units were covered with film to prevent evaporative losses during the filtration process, which typically took approximately 1 day. Once a dispersion had been filtered, the resulting buckypaper was washed with 250 mL of Milli-Q water followed by 10 mL of methanol (99.8%, Merck) whilst still in the filtration unit. After washing, the damp buckypaper was placed between absorbent paper sheets and allowed to dry further overnight. The dry buckypaper was then carefully peeled away from the underlying commercial membrane filter.

2.4 Characterization techniques

Absorption spectra of all dispersions were obtained between 300 and 1000 nm using a Cary 500 UV-vis-NIR spectrophotometer and quartz cuvettes. The dispersions were first diluted with Milli-Q water to ensure that the measured absorbances were within the optimal range of the instrument.

Raman spectra of dispersions and buckypapers were obtained using a Jobin Yvon Horiba HR800 Raman spectrometer. The spectra were measured between 150 and 2000 cm⁻¹ using a 638.5 nm laser source with a 950-line diffraction grating. Small samples of buckypaper were placed directly under the Raman laser source, while single drops of dispersion were first placed on

a glass microscope slide and covered with a glass cover slip to prevent the sample from drying out.

A Leica Z16 APO microscope equipped with a digital camera was used to perform preliminary assessments of dispersions and to investigate the surface morphology of buckypapers. The porous structure of buckypapers was further examined using a Hitachi S-900 field emission scanning electron microscope located within the Electron Microscope Unit at the University of New South Wales, Sydney, Australia. The microscope was operated using a voltage of 4 kV for all samples, which had sufficient electrical conductivity to be imaged without prior sputter coating.

The thicknesses of buckypapers were measured using a Mitutoyo IP65 digital micrometer. Measurements were made at 10 separate points on each buckypaper and averaged to provide a mean thickness. In order to measure the density of buckypapers, three rectangular strips were cut and their lengths and widths measured using Pro-Max S225 digital calipers in order to allow their volumes to be determined. The weights of the strips were then measured using a Mettler Toledo AB204-S balance, and the density subsequently calculated. The densities reported are the average of three separate values.

The contact angles of buckypapers were determined using the sessile drop method and a Data Physics SCA20 goniometer fitted with a digital camera. The contact angles of 2 μ L Milli-Q water droplets on the surfaces of the buckypapers were calculated using the accompanying Data Physics software (SCA20.1). The mean contact angle was calculated using measurements performed on at least 10 water droplets.

Current–voltage (I – V) measurements were conducted under controlled conditions in air (21 $^{\circ}$ C, 45% relative humidity) with a waveform generator (Agilent 33220A) and a digital multimeter (Agilent 34410A). For conductivity measurements films were cut into strips of 3 mm width and lengths of up to 3 cm. These were then connected at each end to pieces of copper tape (3 M) on a glass microscope slide using high purity conducting silver paint. I – V measurements were made as a function of film length by cutting the end off the strip, and then reconnecting to pieces of copper tape on the microscope slide using silver paint.

The mechanical properties of all buckypapers were evaluated using an Instron 5566 tensile tester. The rectangular samples were tested under ambient conditions using a gauge length of approximately 15 mm and a strain rate of 0.1 mm min $^{-1}$. The tensile strength of each sample was determined as the maximum stress measured, while the ductility was the percentage elongation (% EL) at breaking point. The Young's modulus and sample toughness were also determined.

3. Results and discussion

3.1 Effect of sonication time on SWNT–Triton X-100 dispersions

The effect of sonication time on the degree of dispersion of SWNTs in a 1% (w/v) Triton X-100 solution was investigated using absorption spectrophotometry. This experiment was performed to determine the minimum amount of time required to effectively disperse the SWNTs, as excess sonication can shorten or cause defects to form in the tubes and thereby diminish their

properties.^{7,11,31} Absorption spectrophotometry has been widely used by researchers for this purpose, as aggregated SWNTs do not show distinct features in the region between 300 and 1000 nm, while the absorption spectra of well dispersed and de-aggregated tubes show a number of bands in this region related to the van Hove singularities.^{32–34} Since these bands become more pronounced with increasing de-aggregation, the absorption spectrum can be used to gauge the extent of dispersion of the SWNTs.³⁴

Fig. 1 shows the effect of increasing sonication time on the appearance of the absorption spectrum of a sample containing 0.1% (w/v) SWNTs and 1% (w/v) Triton X-100. As Triton X-100 absorbs minimally in the region shown, it can be assumed that the absorption bands observed are due solely to the presence of de-aggregated SWNTs. Increasing the sonication time resulted in both greater overall absorbance, and the bands assigned to van Hove singularities becoming more pronounced. Both effects may be attributed to greater de-aggregation of the SWNTs. In order to obtain more detailed information about the effect of the length of sonication on the extent of de-aggregation the absorbance of the sample at 660 nm was plotted as a function of sonication time. This particular wavelength was selected as it corresponds to the maxima of an absorption band arising from the van Hove singularities. The above plot is shown as the inset in Fig. 1, and clearly shows that initially the absorbance rapidly grew as the length of sonication was increased. However, after approximately 10–12 min the absorbance increased at a slower rate and approached a limiting value. Based on the data shown in the inset of Fig. 1, a sonication time of 24 min was selected as being optimal for ensuring that the SWNTs were well dispersed, but had not been subjected to such prolonged exposure to ultrasonic energy that defects and tube shortening were likely.

Optical microscopy was used to show that there were no large aggregates present in a SWNT–Triton X-100 dispersion that had been subjected to 24 min sonication. Furthermore, dispersions prepared in this manner were found to be very stable, as they showed little change after 6 months standing under ambient conditions. The dispersion obtained after 24 min sonication time was also evaluated by comparing its Raman spectrum to that of the pristine SWNTs, and that of a dispersion prepared using just 1 min of sonication (see ESI† for Raman spectra). The radial

Fig. 1 Effect of increasing sonication time on the UV-visible absorption spectrum of a dispersion containing 0.1% (w/v) SWNT and 1% (w/v) Triton X-100. The inset shows how the absorbance at 660 nm varied in response to changes in sonication time. All samples were measured after being diluted 25 $\times$ using Milli-Q water.

breathing mode peaks present in the spectra of both dispersions were shifted by approximately 5 cm^{-1} to higher energy compared to the corresponding peaks in the spectrum of the pristine SWNTs. These shifts are most likely caused by increases in vibrational radial energies of the SWNTs after they had become coated with Triton X-100 molecules during formation of the dispersion.³⁵ The ratio of the intensities of the G-band and D-band peaks in the Raman spectrum also provides information about SWNT dispersions, with a larger G/D ratio indicating greater retention of de-aggregated SWNTs in the dispersion compared to amorphous carbon impurities. The G/D ratio for the as-received SWNTs used throughout this study was 20, while for a dispersion prepared using a sonication time of 1 min it was 21. For the dispersion prepared using 24 min sonication time the G/D ratio was 23, indicating that there had been further de-aggregation of the SWNTs.

3.2 SWNT dispersions containing biopolymers

In order to compare the effectiveness of different biopolymers for dispersing SWNTs with each other, and with Triton X-100, the following series of experiments were performed. Samples containing the same amount of SWNTs, but different amounts of biopolymers, were sonicated for pre-determined periods of time and their absorption spectra measured. The spectra were then used to obtain the absorbance at 660 nm, which was subsequently plotted as a function of sonication time for each concentration investigated. At this wavelength none of the biopolymers absorbed significantly. Using the above approach resulted in a series of plots of absorbance vs. sonication time similar to that shown in the inset of Fig. 1. Increasing the concentration of each biopolymer was found to result in an increase in absorbance at 660 nm at each sonication time examined. Fig. 2 shows plots of absorbance at 660 nm against sonication time for selected concentrations of lysozyme, bovine serum albumin, chitosan and gellan gum. The concentrations chosen for the first three biopolymers were those that resulted in SWNT dispersions with almost identical absorbances after 24 min sonication time, as well as similar absorbances at all other sonication times examined. Furthermore, the absorbance at

Fig. 2 Effect of increasing sonication time on the absorbance at 660 nm of SWNT dispersions containing selected concentrations of biopolymer dispersants or Triton X-100. $\blacklozenge$ 1.0% (w/v) Triton X-100; $\blacksquare$ 0.6% (w/v) lysozyme; $\triangle$ 0.5% (w/v) chitosan; $\bullet$ 0.6% (w/v) bovine serum albumin; and $\times$ 1.0% (w/v) gellan gum.

660 nm for each of these three biopolymer dispersions was almost identical to that of a 1% (v/v) Triton X-100 dispersion.

Since the magnitude of the absorbance at 660 nm reflects the degree of dispersion of the SWNTs, the data shown in Fig. 2 provide a method for ranking the biopolymers in terms of their ability to act as dispersants. This was accomplished by comparing the concentrations required to obtain the same absorbance at 660 nm. Applying this approach, bovine serum albumin, chitosan and lysozyme all appear to be better dispersants for SWNTs than Triton X-100, since lower concentrations of the biopolymers were required to obtain the same absorbance at 660 nm, and therefore dispersions of similar quality. In contrast, gellan gum was found to be a poorer dispersant than Triton X-100, since the absorbances of dispersions obtained using a 1% (w/v) gellan gum concentration at different sonication times were always considerably less than that of dispersions prepared using the same concentration of organic surfactant. It was in fact not possible to obtain a similar absorbance–sonication time profile for gellan gum dispersions as those prepared using Triton X-100, as solutions containing higher concentrations of the former dispersant could not be used as a result of gelation occurring at room temperature.

The quality of the dispersions produced using the biopolymers was also investigated using Raman spectroscopy. The dispersions studied were those whose absorbance–sonication time profiles are shown in Fig. 2. The G/D ratio obtained from the Raman spectrum of a dispersion produced using gellan gum was found to be 20, which is identical to that of the as-received SWNTs from which it was produced. Each of the other three biopolymer dispersions gave G/D ratios of either 22 or 23, which may be compared to the value of 23 obtained for an optimized Triton X-100 dispersion. This indicates that there were similar ratios of SWNTs and amorphous carbon impurities present in BSA, lysozyme and chitosan dispersions.

3.3 Preparation of buckypapers—experimental design

Although many researchers have noted the potentially useful properties of buckypapers, there have been few systematic studies into how these are altered by changing the conditions used during their preparation.^{17,18} Such information is clearly of interest, as the properties which a buckypaper must possess differ from one potential application to another. We chose to examine the effect of varying four key aspects of the preparation process on several properties of buckypapers, including their electrical conductivity, morphology and mechanical properties. These aspects were the final volume of the dispersion prior to filtration, the length of time the sample was sonicated to make the initial dispersion, the identity of the dispersant used and the membrane filter used during the filtration process.

Sonication time was shown above to have an impact on the extent to which SWNTs are dispersed, and was therefore also expected to have a significant effect on the properties of buckypapers prepared from those dispersions. Our standard procedure consisted of sonicating a 15 mL sample containing a pre-determined quantity of SWNTs and the selected dispersant, after which the sample was further diluted with dispersant solution to 80 mL. In many instances these were then further diluted with Milli-Q water to a final dispersion volume of 250, 500 or 1000 mL

prior to vacuum filtration. This was done because it was expected that further diluting the sample may enhance the degree of dispersion of the CNTs, and therefore affect how they are deposited whilst being processed to form a buckypaper. The effect of filtering identical SWNT dispersions through membrane filters with different surface properties and pore sizes was also investigated using two hydrophobic (5.0 μm polytetrafluoroethylene (PTFE) and 0.45 μm polyvinylidene fluoride (PVDF)) and two hydrophilic (0.45 μm nylon and 0.20 μm cellulose nitrate (CN)) membrane filters.

The final preparation condition examined was the dispersant used. Many previous studies have used the organic surfactant Triton X-100 to produce dispersions for processing into buckypapers. In contrast, there have been very few examples of buckypapers prepared using other types of dispersants. Here we compare the properties of buckypapers made from SWNT dispersions containing one of four biopolymer dispersants or Triton X-100. The biopolymers investigated included both proteins and carbohydrates in order to examine whether this class of molecules can be generally used for preparing buckypapers, and also to provide the resulting materials with the widest possible range of properties.

The properties of all buckypapers prepared in this study are presented in the ESI† (Supplementary Tables S1 and S2), while in the following sections the effects of varying the preparation conditions on these properties are described.

3.4 Effect of sonication time on the properties of buckypapers

All buckypapers displayed densities in the range 1050–1400 kg m^{-3} , and thicknesses between ~ 10 and 120 μm . The differences in density did not show a correlation with any of the four preparation conditions examined. In addition, increasing the sonication time used to prepare a SWNT dispersion or its final volume did not produce any identifiable trends in buckypaper thickness. However, Fig. 3A shows that the wettability of buckypaper membranes prepared using Triton X-100, lysozyme and bovine serum albumin was generally enhanced when the amount of sonication used to prepare the initial dispersion was increased. For example, the contact angle of SWNT–BSA buckypapers decreased from 99.3° to 59.0° when the sonication time was increased from 12 to 48 min. SWNT–lysozyme buckypapers also displayed an improvement in wettability over the entire range of sonication times examined, as did SWNT–Triton X-100 buckypapers when the sonication time was increased from 24 to 48 min. Surprisingly, the contact angle of SWNT–Triton X-100 buckypapers made from 3 separate dispersions prepared using a sonication time of 12 min all exhibited a contact angle of 0.0°. We are unable at this point to explain this result, however, further experiments are planned to see if similar SWNT–Triton X-100 dispersions filtered through other membrane filters also yield buckypapers that are superhydrophilic.

Conventional optical microscopy showed that the surfaces of each of the buckypapers prepared in this study were homogeneous. Furthermore, microscopic images showed that the surfaces of Triton X-100 buckypapers were generally smooth and shiny, and similar in appearance to Triton X-100 buckypapers reported in previous studies.^{36,37} Changing the final dispersion volume, sonication time used to prepare the dispersion, or

Fig. 3 Effects of: (a) increasing sonication time during formation of dispersions, and (b) increasing final dispersion volume, on the contact angle of buckypapers. All buckypapers in part (a) were made by filtering 1 L of dispersion through 5.0 μm PTFE (Triton X-100), 0.45 μm nylon (BSA) or 0.2 μm cellulose nitrate (lysozyme) membrane filters. All buckypapers in part (b) were made by filtering dispersions prepared using 24 min. sonication time and 5.0 μm PTFE (Triton X-100), 0.45 μm nylon (BSA) or 0.2 μm cellulose nitrate (lysozyme) membrane filters. ♦ Triton X-100; ■ lysozyme; and ▲ BSA.

membrane filter used to deposit the buckypaper on, all had no significant effect on the appearance of surfaces of Triton X-100 buckypapers.

Scanning electron microscopic (SEM) images of SWNT–Triton X-100 buckypapers made from dispersions prepared using different conditions are shown in Fig. 4. Changing the sonication time used to prepare the initial dispersion was found to have a significant effect on surface morphology. When the optimum sonication time of 24 min was used, the SEM image of the resulting buckypaper (Fig. 4A) showed that the SWNTs were de-aggregated to the greatest extent. Decreasing the sonication time to 12 min resulted in a buckypaper with surface features consistent with significant aggregation of SWNTs (Fig. 4B). Surprisingly, the surface of a buckypaper prepared from a SWNT–Triton X-100 dispersion made with a sonication time of 48 min also appeared to contain similar structural features, perhaps as a result of degradation of the SWNTs caused by the additional energy they were subjected to.

One of the most important characteristics of a CNT-based material is the ease with which it transmits an electrical current. Linear I – V relationships were obtained for all lengths of the different buckypapers examined using the method outlined in Section 2.4, indicating that Ohmic behaviour was being

Fig. 4 Scanning electron microscope images of SWNT-Triton X-100 buckypapers made from dispersions prepared using: (A) 24 min sonication time, 1 L total dispersion volume, 5.0 μm PTFE membrane filter; (B) 12 min sonication time, 1 L total dispersion volume, 5.0 μm PTFE membrane filter; (C) 24 min sonication time, 80 mL total dispersion volume, 5.0 μm PTFE membrane filter; and (D) 24 min sonication time, 1 L total dispersion volume, 0.45 μm PVDF membrane filter.

exhibited. The slope of these plots, which are inversely related to the total resistance of the buckypaper, increased as the piece of buckypaper was lengthened. The total resistance (R_T) of a buckypaper is related to its length in cm (l) by eqn (1), where σ is the bulk conductivity of the sample (in S cm^{-1}), A is the cross-sectional area (in cm^2) and R_C is the contact resistance (in Ω). By plotting the total resistance of buckypapers as a function of their length it was possible to derive the bulk conductivities presented in Supplementary Table S1†. An example of a plot of total resistance vs. buckypaper length is shown in Fig. 5A.

$$R_T = \frac{1}{\sigma A} l + R_C \quad (1)$$

While the conductivities of the Triton X-100 buckypapers showed significant variations that reflect differences in the conditions used during their preparation, they were all still comparable to the value of $\sim 100 \text{ S cm}^{-1}$ reported previously by Blighe *et al.*, who used a similar 2-point probe technique.³⁸ Fig. 5B shows that increasing the sonication time had a similar effect on the electrical conductivity of buckypapers made using Triton X-100, BSA or lysozyme. In each case the highest value was obtained using a sonication time of 24 min, and increasing or decreasing the sonication time resulted in significant reductions in conductivity. Use of longer sonication times, in particular, might be expected to reduce electrical conductivity as a result of decreasing the average lengths of the SWNTs present in the buckypaper.

The mechanical properties of a buckypaper are amongst its most important characteristics, and play an important role in

Fig. 5 (A) Effect of changing the length of a strip of buckypaper prepared from a Triton X-100 dispersion on its total resistance. (B) Effect of increasing sonication time during formation of dispersions on the electrical conductivity of buckypapers. Buckypapers were prepared by filtering 1 L of dispersion through 5.0 μm PTFE (Triton X-100), 0.45 μm nylon (BSA) or 0.2 μm cellulose nitrate (lysozyme) membrane filters. ♦ Triton X-100; ■ lysozyme; and ▲ BSA.

determining the possible applications that it may be used for. For example, buckypapers must be mechanically robust if they are to be used for filtration applications where they may be exposed to fluids and gasses under a wide range of temperatures, pressures and flow rates. Typical stress-strain curves for the buckypapers prepared as part of this work are shown in Fig. 6, while the results obtained from tensile tests were used to determine the

Fig. 6 Stress-strain curves for SWNT buckypapers made from dispersions prepared using Triton X-100 or different biopolymers. All dispersions were prepared using a sonication time of 24 min and had a total volume of 80 mL, prior to filtration through 0.45 μm nylon membrane filters.

mechanical properties presented in Supplementary Table S2†. Varying the sonication time was found to affect the Young's modulus, tensile strength, ductility and toughness of SWNT buckypapers containing Triton X-100, BSA or lysozyme. In most cases it appeared that the best performing buckypapers were prepared from dispersions made using sonication time of either 24 or 36 min. However, the errors associated with some measurements prevent a definitive conclusion from being reached. More significant effects on mechanical properties were observed when the dispersant used to prepare the buckypaper was varied or, in some instances, the identity of the membrane filter used (see below).

3.5 Effect of dispersion volume on the properties of buckypapers

Varying the dispersion volume used to produce buckypapers was found to have a consistent effect on the wettability of the resulting materials. For SWNT buckypapers produced using Triton X-100, BSA and lysozyme the maximum contact angle was in each case observed when the dispersion volume employed was 500 mL (Fig. 3B). Increasing the dispersion volume to 1 L produced relatively small decreases in contact angle, which were within experimental error of the values obtained for the optimum materials. In contrast, decreasing the dispersion volume produced much larger decreases in contact angle, indicating that the buckypapers were becoming significantly more hydrophilic.

Comparison of Fig. 4A and C shows that decreasing the final dispersion volume from 1 L to 80 mL had a pronounced effect on the surface morphology of SWNT–Triton X-100 buckypapers, with evidence of considerable aggregation of SWNTs evident. The most likely explanation for this observation is that using a lower dispersion volume reduces the ability of the dispersant to maintain the SWNTs in a de-aggregated state.

Fig. 7 shows that increasing dispersion volume resulted in significant improvements in electrical conductivity for buckypapers made using Triton X-100, lysozyme or BSA. Most of the conductivities obtained using materials made with the first two

Fig. 7 Effect of increasing final dispersion volume on the electrical conductivity of buckypapers. All buckypapers were made from dispersions prepared by sonicating for 24 min. The resulting dispersions were then filtered through 5.0 μ m PTFE (Triton X-100), 0.45 μ m nylon (BSA) or 0.2 μ m cellulose nitrate (lysozyme) membrane filters. ◆ Triton X-100; ■ lysozyme; and ▲ BSA.

dispersants were similar to values previously obtained by the 2-point probe method.³⁸ However, the conductivities obtained for SWNT–BSA buckypapers were considerably lower than that of the other materials. This observation did not depend on the sonication time or dispersion volume employed during preparation of the SWNT–BSA buckypaper, and suggests that the identity of the dispersant used can have a major impact on the electrical properties of SWNT buckypapers.

The Young's modulus, tensile strength, ductility and toughness of buckypapers made using Triton X-100, BSA or lysozyme did not show any systematic dependence on the dispersion volume used. Furthermore, the values observed were similar to those obtained for other buckypapers prepared using the same dispersant, but with different sonication times or membrane filters (see Supplementary Table S2†).

3.6 Effect of membrane filter on the properties of buckypapers

The last step in forming a buckypaper involves filtering a CNT dispersion through a membrane filter either under vacuum or positive pressure. A large range of commercial membrane filters can be used for this purpose, that differ in their ability to repel or attract water as well as the size of the pores present. It was anticipated that both factors could affect the rate of filtration of dispersions, and the manner with which the dispersed tubes and dispersant pack to form a buckypaper on the membrane filter. We therefore set out to systematically examine the effect of changing the membrane filter on selected SWNT dispersions.

Varying the membrane filter was found to have limited effect on the amount of time it took to filter SWNT dispersions to form buckypapers. For example, it took typically approximately 50 h to filter 1 L of SWNT–Triton X-100 dispersion. However, changing the composition of the dispersion did have a significant effect on the length of the filtration process. This is shown by the fact that all SWNT–BSA dispersions took typically less than 10 h to filter to form a buckypaper regardless of how they were prepared, including the final dispersion volume.

Overall it was found that changing the membrane filter had no significant effect on the thickness, contact angle or conductivity of SWNT buckypapers produced using Triton X-100, lysozyme, chitosan or gellan gum as the dispersant. Comparison of Fig. 4A and 4D shows that the surface morphology of Triton X-100 buckypapers prepared using PTFE and PVDF membrane filters, and otherwise identical conditions, was very similar with no significant differences in the size of SWNT aggregates. Changing the membrane filter often altered the mechanical properties of buckypapers, although no consistent trends were found when buckypapers prepared using different dispersants were considered. These conclusions are supported by a consideration of the mechanical properties of buckypapers produced using chitosan or gellan gum as the dispersant. When either chitosan or gellan gum was the dispersant used, both the Young's modulus and the tensile strength of buckypapers were considerably greater when nylon was used as the membrane filter instead of cellulose nitrate. However, when Triton X-100 was the dispersant, filtration through cellulose nitrate gave buckypapers with superior Young's modulus, tensile strength, ductility and toughness to those prepared using any of the other three membrane filters examined.

3.7 Effect of dispersant on the properties of buckypapers

The thicknesses of SWNT–Triton X-100 buckypapers prepared as part of this study varied between ~10 and 40 μm . Varying the sonication time, dispersion volume or membrane filter failed to exert a significant or systematic effect on buckypaper thickness. However, changing the identity of the dispersant used did have a major impact on this property, with the thicknesses of buckypapers prepared using biopolymer dispersants being significantly greater than the upper limit of the above range of values. Furthermore, the thickness of the buckypapers was found to increase as the molecular mass of the dispersant used during its preparation increased (Fig. 8A). This resulted in the buckypaper made using the highest molecular mass dispersant, gellan gum, having a thickness of $114.0 \pm 6.0 \mu\text{m}$, which was approximately twice that of the thickest Triton X-100 buckypaper produced. One possible explanation for this observation is that during filtration the larger biopolymers were not able to become as compacted during the drying process.³⁹

Fig. 8 Effect of average molecular mass on the properties of SWNT buckypapers made by filtering 80 mL dispersions prepared using a sonication time of 24 min: (A) effect on buckypaper thickness; (B) effect on electrical conductivity; and (C) effect on tensile strength.

While the identity of the dispersant used had no significant effect on the wettability of SWNT buckypapers, both the electrical conductivity and the mechanical properties of these materials were found to be clearly affected. Fig. 8B shows that the conductivity of buckypapers prepared using otherwise identical conditions decreased dramatically when the molecular mass of the dispersant was increased. This trend was observed for data obtained using buckypapers prepared using both 0.45 μm nylon and 0.20 μm cellulose nitrate membrane filters. One explanation for the observed data is that when the size of the dispersant molecule increases, they become more effective at coating the outside of the SWNTs, which would create a tunneling barrier that results in higher resistance throughout the sample.⁴⁰

While Fig. 8B shows that the conductivity of buckypapers decreased when the molecular mass of the dispersant used during their preparation was increased, Fig. 8C shows that the inverse was true when tensile strength was examined. The tensile strength of buckypapers is largely dependent on the formation of tube-to-tube junctions.¹⁷ During the formation of a buckypaper the dispersant molecules are adsorbed onto the surface of different SWNTs, and therefore become involved in effectively binding them together by overlapping with adjacent tubes and filling in pores and other defects present in buckypapers that may act as stress-concentration sites.³⁶ Larger biopolymer dispersants may be able to cover a greater portion of the SWNTs at tube-to-tube junctions. While this may result in greater resistance and therefore lower conductivities for buckypapers made using the higher molecular mass dispersants, it is likely that their mechanical properties would be improved.

Unlike tensile strength, the Young's modulus, ductility and toughness of SWNT buckypapers did not show a clear correlation with the molecular mass of the dispersant used. However, the values of ductility and toughness obtained for buckypapers made using the two polysaccharide dispersants were considerably larger than those obtained for any other buckypaper examined. The extent to which these particular properties were improved suggests that the two polysaccharides are probably retained in the final materials, and that the ductility and toughness of the resulting composites are a reflection of the ability of the higher order structures of these carbohydrate polymers to absorb imposed stresses.

Fig. 9 shows that significant variations in surface morphology were also found when SEM was used to image the surfaces of buckypapers prepared using different dispersants. Each of the buckypapers illustrated was prepared using a sonication time of 24 min, a final dispersion volume of only 80 mL, and by filtration through 0.45 μm nylon membrane filters. This was necessitated by the very long times required to filter some of the dispersions through other membrane filters, and the desire to keep all other experimental parameters constant. The SEM image of a Triton X-100 buckypaper prepared under these conditions (Fig. 9A) shows some regions that appear to contain aggregates of SWNTs similar to those described earlier, and resembles more closely other Triton X-100 buckypapers prepared using small final dispersion volumes (Fig. 4C) or long sonication times, rather than the material prepared using optimized conditions (Fig. 4A).

The SEM image of a chitosan buckypaper (Fig. 9C) most closely resembles that obtained using Triton X-100 in overall appearance. However, the former buckypaper contains many

Fig. 9 Scanning electron microscope images of SWNT buckypapers made from dispersions containing: (A) Triton X-100; (B) lysozyme; (C) chitosan; (D) bovine serum albumin and (E) gellan gum. All dispersions were prepared using a sonication time of 24 min, had a final volume of 80 mL, and were filtered through 0.45 μm nylon membrane filters.

large bundles of tubes or other structures possibly resembling CNT ropes, and there appears to be a smaller number of pore openings on the surface. Using lysozyme as the dispersant resulted in a buckypaper that contained even fewer pores and a greater percentage of its total surface area covered in aggregates or bundles of SWNTs (Fig. 9B). In this respect it resembled closely that obtained using Triton X-100 and a very short sonication time (Fig. 4B). More dramatic differences were found with buckypapers prepared using the two remaining biopolymer dispersants. The surface of a bovine serum albumin buckypaper (Fig. 9D) shows several prominent ridges with large spaces between them. In addition, it is apparent that there are many large aggregates of SWNTs present. The SEM image of a gellan gum buckypaper (Fig. 9E) contains many extremely large pore openings measuring up to several hundred nanometres in diameter. In contrast the average pore size present in the other buckypapers was generally of the order of 40–50 nm in diameter.

4. Conclusions

The work presented here illustrates that many of the properties of SWNT buckypapers can be significantly affected by altering any one of a number of conditions used during their preparation. For example, the electrical conductivity of buckypapers prepared from dispersions made using the surfactant Triton X-100 varied from $64 \pm 3 \text{ S cm}^{-1}$ to $142 \pm 8 \text{ S cm}^{-1}$, depending on the length of time that the SWNTs were initially sonicated for, the final volume that the dispersion was diluted to prior to filtration, and the choice of membrane filter used. The four SWNT–Triton X-100 buckypapers with the highest electrical conductivities were all prepared from dispersions made using a sonication time of

24 min and a final volume of 1 L, and differed only in the identity of the membrane filter employed during the filtration process. This sonication time was determined to be optimal for preparing SWNT–Triton X-100 dispersions in a separate absorption spectrophotometric study, suggesting that the latter method may be useful as a guide for determining the best conditions to be used for preparing dispersions to be processed into buckypapers. Buckypapers prepared using the above preparation conditions also displayed some of the best mechanical properties. However, reducing the final volume of the SWNT dispersion, or increasing or decreasing the sonication time employed for making the dispersion, both generally resulted in buckypapers with poorer electrical and mechanical properties. Scanning electron microscopy showed that use of very short sonication times (*e.g.* 12 min), or a very low final dispersion volume (*e.g.* 80 mL), resulted in buckypapers whose surface morphology was suggested by SEM to contain a smaller number of pore openings as a result of greater aggregation of SWNTs. This suggests that buckypapers made under these conditions would possess poorer electrical and mechanical properties and also be less suitable for applications such as microfiltration and ultrafiltration, which require materials with high surface area and/or porosity.

To the best of our knowledge this study shows for the first time that buckypapers can be prepared from SWNT dispersions made using the biopolymers lysozyme, bovine serum albumin and gellan gum. During the course of investigations into the properties of these materials, and that of buckypapers prepared from dispersions made using SWNTs and chitosan, several trends became apparent. The first of these is that the electrical conductivity of the buckypapers decreased as the molecular mass of the biopolymer dispersant used was increased. This meant that

for buckypapers prepared using the biopolymer with the highest molecular mass, gellan gum, a conductivity of only $4 \pm 1 \text{ S cm}^{-1}$ was measured, which is much less than values of over 100 S cm^{-1} determined for SWNT–Triton X-100 buckypapers prepared from dispersions made under optimized conditions.

Use of a low final dispersion volume was shown to adversely affect the conductivity of several of the buckypapers examined here. For example, changing the final volume of a dispersion from 1 L to 80 mL, and keeping both the sonication time and the membrane filter used constant, resulted in the electrical conductivity of Triton X-100 buckypapers decreasing from $127 \pm 11 \text{ S cm}^{-1}$ to $80 \pm 6 \text{ S cm}^{-1}$. Therefore the low conductivities displayed by the biopolymer buckypapers may arise due to a combination of formation of an effective outer insulating coating for the SWNTs by the protein and carbohydrate molecules, and aggregation of nanotubes caused by the low final dispersion volume.

In contrast to what was observed with electrical conductivity, the mechanical properties of SWNT buckypapers were generally enhanced through the use of a biopolymer dispersant of increasing molecular weight. For example, the ductility and toughness of buckypapers prepared from dispersions made using either of the two carbohydrate biopolymers were considerably greater than that of all other materials examined here. In addition, the tensile strength of buckypapers prepared using each of the biopolymer dispersants was significantly greater than that of a Triton X-100 buckypaper made under identical conditions. In fact the toughness of the buckypaper produced using gellan gum was even greater than that of a Triton X-100 buckypaper produced from an optimized dispersion. This suggests that it might be possible to produce even more robust materials if a suitable method can be found to enable faster production of gellan gum and other biopolymer buckypapers from more dilute dispersions. Whilst these materials may lack the electrical conductivity of buckypapers produced using Triton X-100, their superior mechanical properties, together with the unusual surface morphologies revealed by scanning electron microscopy, suggest that they may prove useful for applications which place a premium on these characteristics, such as ultrafiltration or microfiltration.

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Title: The effect of preparation conditions and biopolymer dispersants on the properties of SWNT buckypapers.

Single-walled carbon nanotube buckypapers produced using protein and polysaccharide dispersants display a range of electrical, morphological and mechanical properties. These materials may be suitable for a variety of applications including ultra and microfiltration.

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