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COMPLETE SPECIFICATION

SECTION 10

MANUFACTURE OF SINGLE WALLED CARBON NANOTUBES PROVIDING VISIBLE EMISSION FOR IMAGING OF NANOSTRUCTURES

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THE FOLLOWING SPECIFICATION PATICULARLY DESCRIBES THE NATURE OF THIS INVENTION AND THE MANNER IN WHICH IT IS TO BE PERFORMED:

This invention relates to the manufacture of single walled carbon nanotubes providing visible emission for imaging of narestructures.

Ever since the advent of carbon nanotubes, there has been an enormous interest in exploring newer phenomena and applications based on this material Their unique properties, especially related to electronics and optoelectronics, have made them ideal candidates for use in a wide range of applications like miniaturized sensors, molecular probes; optics, quantum cryptography and so on Our invention is directed towards light emission from nanotube structures in the visible region of the electromagnetic spectrum.

The method herein proposes the fabrication of a composite film of silver nanopartides and carbon nanotubes at a liquid-liquid interface and nonstructural based on this film. Such a film shows light emission in the visible region when irradiated with light of appropriate wavelength. This technology is believed to have far-reaching applications in quantum optics, sensors, waveguides and in probing biological entities and processes at the molecular level, it is possible *to* obtain visible emission from single walled *carbon* nanotubes using a variety of other nanopartides of gold, silver and, metals of various shapes and sizes

The nanopartides of silver, stabilized by citrate groups (Ag@citrate) were synthesized by a procedure reported in the literature. The typical size of the particles synthesized by this method is

60-70 nm in diameter

The method, according to this invention, for the manufacture of single wailed nanotubes providing visible emission for imaging of nfanostructures comprises the steps of fabricating a composite film having nanopartides and single walled carbon nanotubes (SWNTs) as the components, the said composite film being formed at the liquid-liquid interface; purifying single walled carbon nanotubes, obtained from a commercial source by repeated sanitations and centrifugation in N.N-dimefhyf forrnamide (DMF) preparing gold and silver nanopartides, using the citrate reduction route the aqueous phase (liquid phase 1) while diethyl ether formed the organic phase (liquid phase 2);adding to this biphasic system, the dispersion of SWNTs in DMF; transferring the film formed at the liquid-liquid interface to a glass substrate; and allowed to dry in ambience

The film was irradiated with a 514.5 nm Ar ion laser, 40 mW maximum power through a 100X microscope objective and the light obtained from the sample were collected by the same objective and sent to a spectrometer through a multimode fiber. A super-notch filter placed in the path of the signal effectively cuts off the excitation radiation. The signal was then dispersed using a 150 grooves/mm grating and the dispersed light was collected by a fast cooled charge coupled device (CCD) Spectra in the desired window were acquired from an area, point by point to construct a spectral image. Typical signal acquisition time, at a point of the image spectrum, was 0.1 s. Single spectra were also acquired using the same grating but with larger integration times. For improved resolution and to ascertain peak positions, a grating with 1800 grooves/mm was also used while acquiring single spectra. Images were constructed using the spectral data collected over 10x10 micron area.

On irradiating the sample with the excitation laser source, a red visible emission between 600 - 700 nm was obtained   Typical signatures of single walled carbon nanotubes, namely the radial breathing mode (RBM) and the D and G bands were found to be superimposed on the background of this emission, confirming that *the* emission was an *inherent* property of the carbon nanotubes. In order to verify the authenticity of the phenomenon, a similar film was formed at the biphasic liquid interface, with the aqueous phase being contributed by triply distilled water. On observing this film under a confocal Raman spectrometer, the regular Raman features of carbon nanotubes were seen; while the visible emission was absent. Therefore, it was confirmed that the emission was occurring only due to the nanoparticle-nanotube composite film.

The visible emission and Raman bands of nanotubes were used to image the nanotubes All the images suggest that the emission was from the nanotube structure Separate transmission electron microscopic experiments have shown that nanotube-nanopartide composites are present in the film.

The same emission features were found with another excitation source, namely 532 nm confirming that these are fluorescence in character

Example: Experiment with silver nanoparticles

As-synthesized citrate reduced silver nanoparticles of 60-70 nm mean diameter in aqueous medium and diethyl ether were taken in equal volumes to form the aqueous-organic liquid-liquid interface To this biphasic system a dispersion of single walled carbon nanotubes in DMF was added. This was immediately followed by the formation of a film with metallic appearance at the interface This film was transferred *onto* a 0.2 mm cover-glass substrate and dried in ambience. This sample was mounted on a sample stage of a confocal Raman spectrometer, the details of the instrument being provided in the earlier section.

On irradiating (he sample with the excitation laser source, a red visible emission between 600 - 700 *nm was* obtained Typical signatures of single walled carbon nanotubes, namely the radial breathing mode and the D and G bands were found to be superimposed on the background of this emission, confirming that *the emission* is an inherent property of the carbon nanotubes;

Control experiment

In order to verify the authenticity of the phenomenon, a similar film was formed at the biphasic liquid interface, with the aqueous phase being contributed by triply distilled water On observing this film under the confocai Raman spectrometer the regular features of carbon nanotubes were seen, while the visible emission was absent Therefore, it was confirmed that the emission was occurring only because of the nanoparticle-nanotube composite film

The same phenomenon was observed with gold nanoparticles of 15 nm mean diameter. Other shapes such as nanorods also show the same phenomenon when made in the form of a composite film with nanotubes.

The salient features of this invention are;

1. Emission in the visible region of the electromagnetic spectrum from carbon nanotube
2. The use of visible emission for imaging of carbon nanotubes and nanotube composites.
3. The imaging method used here may be applied for chemical, physical and biological purposes.
4. Such *an* emission can be achieved by the method of consisting of the steps of dispersing the single walled carbon nanotubes in DMF, purifying it through repeated sonication and centrifugation cycles, synthesizing the nanoparticles of silver, stabilized by citrate groups, forming the film at an aqueous-organic liquid interface, transferring the composite film onto a substrate and subsequently drying it to obtain the film.
5. The above mentioned method can also be done with other nanomateriais having various other shapes and sizes such as rods, triangles prisms, sheets, etc.
6 The above mentioned method can be achieved with nanomateriais formed by other elements and substances such as gold, platinum, rhodium, palladium, etc. or their alloys.
7. The above mentioned method wherein the procedure for purifying the single walled carbon nanotube can be any other suitable method or a modified version of what is set out here..
8. The above mentioned method wherein other chemicals and reagents are used to prepare the nanoparticles and the composite film

9 T he above mentioned method wherein the substrate used for transferring the film can be made of any other suitable material.
10 The above mentioned method wherein the film is formed at other interfaces or in single phase or m media other than liquids.
11 The above mentioned method which makes it possible to observe visible emission from single walled carbon nanotubes, assisted by any other nanoparticle of any other shape or size.
12 The above mentioned method of obtaining visible emission from nanotubes or nanotube composites and use of emission in applications such as detection of anaiytes as well as chemical. physical,biological processes
13. A method of utilizing the fluorescence from single walled carbon nanotubes using any other instrument or excitation light source

The objective of making a nanoparticle-nanotube composite is to cover the nanotube structure with nanoparticles such that there is electronic interaction between the too. This nanoparticle cover can be achieved by various means such as spraying a nanopartide dispersion over nanotubes, evaporating a nanopartide dispersion over a nanotube covered substrate, mixing nanoparticles and nanotubes intimately by a physical method such as grinding, etc. Any method which covers the nanopartides on the nanotubes may be used to meet the desired objective. In order to improve the interaction between the nanopartides and nanotubes, either *or* both of them may be chemically modified.

The terms and expressions herein are of description and not of limitation having regard to the scope and ambit of this invention

We Claim;

1. A method for the manufacture of single walled nanotubes providing visible emission for imaging

of nonstructural comprising the steps of fabricating a composite film having nanopartides and single walled carbon nanotubes (SWNTs) as the components, the said composite film being formed at the liquid-liquid interface; purifying single walled carbon nanotubes, obtained from a commercial source by repeated sonication and centrifugation in N,N-diethyl formaldehyde (DMF);preparing gold and silver nanoparticles, the citrate reduction route the aqueous phase (liquid phase 1) while diethyl ether formed the organic phase (liquid phase 2);

adding to this biphasic system, the dispersion of SWNTs in DMF; transferring the film formed at the liquid-liquid interface to a glass substrate; and allowed to dry in ambience.

2. A method of manufacture of single walled nanotubes providing visible emission for imaging of nanostructures substantially as herein described with reference to, and as illustrated by, the Examples.

3. Single walled nanotubes providing visible emission for imaging of no structures when manufactured by a method as claimed in any one of the preceding Claims.

4. *Single walled nanotubes providing visible emission for imaging of nanostructures substantially as herein described.*

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INDIAN INSTITUTE OF TECHNOLOGY

By Their Attorney

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