Fabrication of color conductive inks by introducing SWCNT/Ag and various dyes into polymeric solutions for potential applications in disposable, cheap, and flexible electronics

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The multifaceted field of conductive inks is moving from a preliminary to an advanced stage. In this study, cellulose filter paper was used as a popular, renewable, and inexpensive material, with very interesting flexible characteristics. The novelty of this work was to use a single-walled carbon nanotube/silver (SWCNT/Ag) nanopowder in a color polymeric matrix for preparing highly conductive color inks resistant to washing. An investigation comparing three inks colored separately with different anionic and cationic dyes was performed to examine possible changes in electrical resistivity of the papers. The results obtained from FT-IR spectroscopy showed the presence of carboxylic groups in acid-treated SWCNTs and revealed Ag-containing bonds. XRD results confirmed functionalization of SWCNTs and obtaining SWCNT/Ag powder with Ag nanoparticles (NPs). Thermal stability and degradation of specimens were studied using TGA analysis to measure the percentage of Ag NPs in the SWCNTs network. The TEM micrographs were consistent with the Scherrer results. Finally, different color inks were synthesized with/without SWCNT/Ag nanopowder, and the four-point probe technique was utilized to measure the electrical resistivity of each colored paper. Consequently, preparation of color conductive inks by using ultra-narrow SWCNTs was done successfully.

Keywords: *carbon nanotubes; silver nanoparticles; polymer; color conductive ink*

1. Introduction

Engineered nanomaterials-enabled blends are replacing the conventional ones [1]. One of the extraordinary types of nanomaterials are one-dimensional cylindrical carbon nanotubes (CNTs) made from carbon atoms, having excellent electrical, mechanical, and thermal stability, suitable for a wide number of applications [2]. Due to sp^2 hybridization of carbon nanotubes causing out-of-plane delocalization of three s orbitals along with the p orbital, modification of mechanical, electrical, chemical, and biological properties of the CNTs takes place. Conductive inks have recently attracted increased researchers' attention for various applications in the fields of disposable sensors and biosensors, recyclable electronic products, photovoltaics, RFID tags,

smart labels, touch screens and switches, antennas, printed heaters, and so on [3–8]. Notable increase in thermal strength of CNT-filled polymers has been observed in different thermal-measuring systems, such as TGA [9]. Due to significant van der Waals attraction between CNTs causing them to aggregate, three strategies have been pursued by the researchers to address this drawback. One of them is carboxylic functionalization of CNTs, another solution is sonication, and the third but not least, is using anionic surfactant SDS as a surface functionalizer to over-disperse the CNTs [10–12].

A facile transfer stamping technique was developed by Chan et al. [13] to engineer organic micro-electrodes applicable for flexible electrodes. PEDOT:PSS and conductive silver paste were used, and it was demonstrated that enhancing the mold thickness decreases the gap sagging phenomenon. The obtained results showed a better transfer of PEDOT:PSS, but higher electrical conductivity

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of silver paste electrode. Pekkanen et al. [14] printed metal NP-based inks on the polymer dielectrics. Statistical methods were used to study the process performance, suggesting significant error sources and optimizing process factors applicable in practice. Staudinger et al. [15] reported that sonication can generate cavitation which breaks the CNTs agglomerations, depending on the time interval, frequency and CNTs concentration in the polymeric solution matrix. Gaspar et al. [16] printed different papers and polymer substrates by silver-containing ink via thermal and IR sintering inkjet printing. It was decided that IR sintering was more beneficial, absorbing two-times more bulk silver particles than the thermal route, even in a short time.

Ternary composites comprising polymer, CNTs, and metal NPs have been deeply studied and manufactured by researchers for many applications. For example, Choi et al. [17] synthesized the polyvinyl acetate/SWCNT/gold nanocomposite with impressive electrical conductivity and thermopower. In other work, Sharma et al. [18] reported a PVDF film which was electrospun from DFM solution. Carboxylic functionalized MWCNs and MWCNT/Ag nanofillers enhanced significantly the piezoelectric coefficient of the electrospun fibers, which latter showed further benefits due to the presence of Ag NPs. Also, Wang et al. [19] worked on the ternary Cu NPs/chitosan/SWCNT nanocomposites deposited on the glassy carbon electrode (GCE) for glucose biosensing applications, and observed an exquisite sensitivity, rapid response time, broader linear range, and perfect selectivity of the modified biosensors.

The studies on screen-offset printing [20], low-temperature metallization mechanism [21], and hand-drawn circuits on paper have been carried out by researchers recently [22]. Printed electronics and flexible electronics are the most common applications of the conductive inks. Presently, using complex and expensive technologies to manufacture printed circuit boards (PCBs) encouraged researchers towards solvent-based conductive inks decreasing the mass production costs. One of the best choices within this new technology can

be inexpensive silver, especially in the novel nanoscale form, because the corrosion-resistant silver is an abundant material used in a broad field of applications capable of resisting the moisture. Silver as a strong oxidant is supposed to be resonant, moldable, and ductile, and benefit from the high electrical and thermal conductivity [23]. In addition, the production cost of the high-quality nanocomposites containing silver is relatively low.

In this study, we investigated incorporation of Ag into SWCNTs for preparation of polymeric color inks and different color conductive inks and their use in real applications. As the main goal of this work, several cellulose papers have been colored with the new color conductive inks with high electrical conductivity, even after the washing process.

2. Experimental

2.1. Chemicals

SWCNTs (outer diameter: 1 nm to 2 nm, inner diameter: 0.8 nm to 1.6 nm, length: 15 μm to 50 μm) with purity of >95 % were obtained from US Research Nanomaterials, Inc., USA. Sunset yellow ($\text{C}_{16}\text{H}_{10}\text{N}_2\text{Na}_2\text{O}_7\text{S}_2$, 90 %) azo dye was obtained from Sigma-Aldrich, while methylene blue ($\text{C}_{16}\text{H}_{18}\text{ClN}_3\text{S}$, C.I. 52015) and phenol red ($(\text{C}_6\text{H}_4\text{OH})_2\text{C}_7\text{H}_4\text{SO}_3$) dyes were of Merck brand. Sulfuric acid (95 % to 98 %), nitric acid (≥ 65 %), silver nitrate (AgNO_3 , 99.99 %), and polyvinylpyrrolidone known as PVP (hydrophilic polymer, $(\text{C}_6\text{H}_9\text{NO})_n$) were supplied by Sigma-Aldrich. Sodium dodecyl sulfate (SDS, $\text{NaC}_{12}\text{H}_{25}\text{SO}_4$, Sigma-Aldrich) was used as an anionic surfactant. Other chemicals used in this work were of analytical grade and used without further purification.

2.2. Characterization

A BRUKER TENSOR 27 spectrometer having a DTGS-KBr detector was used in ambient conditions to perform the Fourier transform infrared (FT-IR) spectroscopy investigation, in mid-infrared region (4000 cm^{-1} to 400 cm^{-1}) with a resolution of 2 cm^{-1} . The powder specimens were compressed

into pellets under a high pressure. X-ray diffraction (XRD) patterns of powder samples were obtained with a PW1800 diffractometer, Philips, the Netherlands, using a monochromatic $\text{CuK}\alpha$ radiation ($\lambda = 0.154$), in the diffraction angle range of 4° to 90° , at room temperature. The machine operated at 40 kV and 30 mA, while the step width was set at 0.04° with a scan speed of $1^\circ/\text{min}$. The quantity of Ag NPs decorated on the surface of SWCNTs was probed by thermogravimetric analysis (TGA), using a TGA Q500 machine, in an inert nitrogen environment to prevent any oxidation or unwanted reaction. The temperature was set between 20°C and 800°C , while the ramp rate was adjusted to $10^\circ\text{C}/\text{min}$. The amount of 2 mg of each powder sample was used and held in a suspended platinum pan. Morphology, uniformity, and composition of pristine SWCNTs and SWCNT/Ag nanopowder were studied using a transmission electron microscope (TEM) model Philips CM30 at 300 kV. The prepared powder SWCNT-containing samples were analyzed also by a two-probe electrical measurement, for which a simple circuit containing a $10\text{ k}\Omega$ resistor and one sample as the second resistor was designed, as it was reported in our previous work [24]. A four-point probe configuration was set up to measure sheet resistance of the colored papers, before and after the washing stage.

2.3. Carboxylic functionalization of SWCNTs

Chemical acid-treatment route was applied for covalent functionalization of SWCNTs using a reflux procedure in an oil bath (temperature 60°C), explained in detail in our previous publications [24, 25]. Concentrated sulfuric (30 mL) and nitric (10 mL) acids, were used in the ratio of 3:1, while an optimum 3-hour time interval was considered. Cooling, dilution, filtration, and washing were performed stepwise to obtain a black precipitated powder, followed by drying at 60°C for 12 h. Acid-treatment was performed to both purify and functionalize the SWCNTs with COOH groups [26–28].

2.4. Synthesis of SWCNT/Ag nanopowder

Once SWCNTs were functionalized, the synthesis process of SWCNT/Ag nanomaterial was carried out. The Ag NPs deposition in the functionalized SWCNTs (f-SWCNTs) procedure began with the preparation of 0.1 M AgNO_3 solution by mixing 0.425 g of solid AgNO_3 in an appropriate volume of deionized water. Next, 0.175 g of SWCNTs was dispersed in 25 mL of 0.1 M AgNO_3 by ultrasound treatment for 30 min, followed by a rigorous stirring at 60°C for 24 h. Since SWCNTs were functionalized, reducing DMF solvent was not necessary and simply deionized water was used as the reaction medium, due to the increase of adsorption power by COOH functionalization, so that no high-temperature annealing step was needed at the final stage. Even metallic Ag NPs couldn't be captured with untreated (pristine) carbon nanotubes and it was reported that surface-functionalized SWCNTs due to their stronger efficiency were capable of producing larger Ag NPs than surface-functionalized MWCNTs [29]. The obtained precipitated slurry was rinsed with distilled water and ethanol, successively filtered by a filter paper, and finally dried in an oven at 60°C overnight.

2.5. Preparation of polymeric conductive inks in different colors

In a typical experiment, aqueous sunset yellow (anionic dye) solution (5 mL, 1000 ppm) as a solvent, SWCNT/Ag powder (0.015 g) as a nanofiller, PVP (0.01 g) as a polymeric binder, and SDS (0.01 g) as a dispersant were used. First, SDS was magnetically stirred in the sunset yellow solution for 1 h, followed by the addition of SWCNT/Ag nanopowder. The mixture was stirred for more 2 h, and then PVP was added. The stirring process continued for 24 h, so that a homogenous dark yellow solution was obtained. In other experiments, a similar work was performed, but instead of sunset yellow dye, 1000 ppm of methylene blue (cationic dye) and phenol red (anionic dye) were used, separately. All the above mentioned inks were repeated without adding SWCNT/Ag nanopowder, so that six color inks were obtained, three of which

without SWCNT/Ag nanopowder, and the other three with SWCNT/Ag nanopowder.

2.6. Surface treatment of printable paper pieces

After preparing homogenous color inks, six square-cut small-pore cellulose filter papers (D: 2 cm) were separately immersed into the prepared baths of the six inks, and left at ambient conditions for 24 h. The colored membranes were rinsed in deionized water to remove unattached materials, and were gently dried at room temperature overnight. Because of reproducibility of the experiments, new colored papers were prepared when necessary. Fig. 1 shows a typical photograph of insulating (Fig. 1a to Fig. 1c) and conductive (Fig. 1d to Fig. 1f) colored cellulose papers.

Fig. 1. Photograph of cellulose filter papers colored with (a) to (c) pure polymeric inks, and (d) to (f) SWCNT/Ag-containing conductive inks.

3. Results and discussion

3.1. FT-IR spectroscopy

In the interaction between an infrared radiation and a material, either stretching, contracting, or bending of the chemical bonds happen, resulting in the radiation absorption via the functional groups present in the molecules structure. The presence of functionalized groups in f-SWCNTs was confirmed by FT-IR spectra demonstrated in Fig. 2. As it can be seen in Fig. 2, a broad

absorption band around 3432 cm^{-1} , corresponding to the stretching vibrations of OH bonds, is present in all curves [30]. It is worth mentioning that this signal is stronger in the case of functionalized and decorated SWCNTs in comparison with the pristine ones, due to some reasons: first, the addition of -COOH functional groups containing hydroxyl bonds, and second, humid nature of the treated materials. Peaks appearing at 1632 cm^{-1} and 1463 cm^{-1} in Fig. 2a to Fig. 2c are related to the basic $\text{C}=\text{C}$ stretching of graphitic structures in aromatic rings. At curve b, two sharp peaks assigned to the C-O and C=O stretching vibrations from carboxylic groups, respectively can be found at ca. 1728 cm^{-1} and 1280 cm^{-1} . These peaks also confirm the success of functionalization process, and are apparent at curve c, with weaker intensity. The results of FT-IR spectroscopy suggest that acid-treatment of pristine SWCNTs causes the carbons on deficient sites or end caps to be oxidized into -COOH groups [31]. The respective carboxylic groups guarantee further and more homogeneous attachment of metal NPs on the surface of SWCNTs due to their chemical activity. Finally, the peak near 581 cm^{-1} can be attributed to the existence of Ag NPs on the walls of SWCNTs.

Fig. 2. FT-IR spectra of (a) pristine SWCNTs, (b) f-SWCNTs, and (c) SWCNT/Ag nanopowder.

3.2. XRD analysis

XRD is a non-destructive, multipurpose, analytical method revealing crystalline nature of samples, their chemical composition, and physical

characteristics. XRD is a simple, fast, and computer-based technique with a comprehensive database for materials identification. CNTs' XRD results resemble graphite, having a (0 0 2) reflection peak near $2\theta = 26^\circ$. Another peak with lower intensity may arise at higher angles owing to the honeycomb graphitic walls typical of CNTs. Crystalline nature of all three specimens is confirmed in XRD patterns in Fig. 3. In all three patterns in Fig. 3, two signals at around 26° and 43° correspond to the (0 0 2) and (1 0 0) crystallographic planes of SWCNTs, respectively, as per JCPDS Card No. 75-1621. In the patterns of f-SWCNTs, the intensity of these peaks has been weakened slightly, due to the strong purification of unbound carbons and defects created in the CNTs walls. The uppermost curve in Fig. 3 belongs to the SWCNT/Ag nanopowder, in which four noticeable peaks at 2θ values of 38° , 44° , 65° , and 77° relating, respectively, to the (1 1 1), (2 0 0), (2 2 0), and (3 1 1) Miller indices, confirm the attachment of FCC structural phase of Ag NPs to the CNTs surface, in accordance with silver file number JCPDS Card No. 04-0783 [32]. The average crystallite size of the cubic Ag NPs calculated by Scherrer equation was found to be 18 nm [33].

Fig. 3. XRD patterns of pristine SWCNTs, f-SWCNTs, and SWCNT/Ag nanopowder.

3.3. TEM micrographs

The TEM pictures of pristine SWCNTs and SWCNT/Ag nanopowders have been shown in Fig. 4a to Fig. 4c, respectively. The uniform

lattice of SWCNTs is evident in Fig. 4a, while Fig. 4b and Fig. 4c indicate the Ag NPs deposited onto the surface of SWCNTs. Furthermore, Fig. 4d outlines the size distribution histogram of Ag NPs present inside SWCNT/Ag nanopowder. Fig. 4 shows an acceptable dispersion of Ag NPs in the CNTs matrix. As it can be concluded, most of Ag particles are within the size range of 10 nm to 30 nm.

Fig. 4. TEM micrographs of (a) pristine SWCNTs, and (b) and (c) SWCNT/Ag nanopowder, and (d) size distribution histogram of Ag NPs in SWCNT/Ag nanopowder.

3.4. TGA analysis

TGA is a method of analysis that analyses the sample weight changes in function of temperature, benefiting from significant consistency and accuracy in weight and temperature measurement. Besides investigating the thermal stability of the powder samples, the value of metal NPs was obtained from the thermal graphs. Fig. 5 shows the TGA results for pristine and functionalized SWCNTs in comparison with SWCNT/Ag nanopowder. As it can be inferred from the thermographs, the main drop in weight percentage happens in the range of 280°C to 600°C . In addition, approximately below 280°C , Ag NPs show a slightly less weight loss of CNTs probably due to the metal heat transfer properties. The results obtained for pristine and functionalized SWCNTs are alike to some extent

(samples residues at 800 °C, are 6 % and 8 %, respectively). According to the TGA records, about 35 % of the nanopowder ingredients maintained at 800 °C, are assigned mostly to the Ag NPs, so the initial content of silver in the prepared nanocomposite would be around one-third of the nanocomposite powders.

Fig. 5. TGA analysis of pristine, functionalized, and Ag-doped SWCNTs.

Fig. 6. Schematic circuit for electrical measurement by two-probe method.

3.5. Electrical measurements on the prepared nanopowders

In order to calculate the electrical conductivity of the prepared powder samples, a simple two-probe technique was used. A schematic diagram of the applied DC circuit has been outlined in Fig. 6. 10 kΩ series resistor has been employed in the circuit to prevent short circuit. By controlling

the total voltage in the circuit, the voltage measured between two ends of each sample was changed up to 7 V, whereas the values of electrical current were measured at selected voltages. It is worth mentioning that sample voltages higher than 7 V could make the materials very hot, causing it to burn. Incombustible capillary tubes with an inner diameter of 0.3 mm were used to hold the powder samples, while a conductive silver paste was used to guarantee a suitable connection between the sample and the circuit wires. The length of accumulated powder sample in the tube was set to be 5 mm, and each test was performed in triplicate. DC conductivity of pristine SWCNTs, f-SWCNTs, and SWCNT/Ag nanopowder at various sample voltages was calculated from the values of sample resistance (R), length (L), and cross-section (A). Fig. 7 shows the samples voltage graphs with respect to their electrical conductivity. All the calculated values of electrical conductivity for pristine, functionalized and Ag reinforced SWCNTs have been listed in Table 1. As it is evident, due to removing impurities, functionalization process has increased the conductivity of SWCNTs. As we know, the impurities in semiconductors can alter drastically their resistivity which is very important in solid state electronics. In addition, silver NPs have shown a great effect on the nanopowder electrical characteristics, so that the conductivity has increased by several orders of magnitude. The non-linear behavior of conductivity at low voltages can be attributed to the nanoscale state of the samples. This nonlinearity can be specifically observed in the case of Ag-deposited SWCNTs due to the higher quantum effect in the interaction between CNTs and Ag NPs. However, at higher voltages a linear relationship dominates in the behavior of electrical conductivity. The phenomenon of the strange behavior of nanoscale composite, especially in low voltages, can be assigned to different kinds of barriers created in the electron paths.

3.6. Electrical characterization of the colored papers

The electrical sheet resistance of the colored filter papers was measured using a well-known

Table 1. Powder samples electrical conductivity calculated at various samples voltages.

Voltage [V]	Conductivity [S/m] Pristine SWCNTs	Conductivity [S/m] f-SWCNTs	Conductivity [S/m] SWCNT/Ag nanopowder
0.1	21042±155	22826±226	75160±2493
0.2	18818±377	20867±271	58634±2503
0.5	13107±481	15486±645	46206±5966
1	11426±197	13652±173	33540±1127
2	10521±305	12032±212	29448±1855
3	10000±125	11134±243	24473±1194
4	9765±32	10906±19	21263±1054
5	9362±137	10751±99	19506±533
6	8287±147	10123±502	18251±283
7	8079±58	9374±134	17112±516

Fig. 7. Conductivity versus voltage diagram of prepared powder composites.

four-point probe method. In the schematic setup shown in Fig. 8, the film thickness (paper) is far less than the distance between the probes (S), so the relevant formula for the value of sheet resistance is expressed by:

$$R_S = (\pi / \ln 2) \times (V / I) \quad (1)$$

More details on the physical theory, instructions, and derivations can be found in reference [34]. The value of input I was set to 0.5 A, and each test was repeated 10 times to get close to the real value of sheet resistance. Table 2 collects the values of sheet resistance for different colored papers. Interestingly, SWCNT/Ag containing

colored papers show a good electrical conductivity, whereas nanopowder-free (SWCNT/Ag-free) colored papers reveal an insulating behavior. Washing tests were also performed to measure the papers sheet resistance values after being washed. For this aim, colored papers were immersed in a warm water bath (60 °C) for 30 min under continuous stirring. Then, the specimens were dried overnight at room temperature. The last column of Table 2 presents the re-measured sheet resistance values of colored papers. After washing procedure, the sheet resistance values of sunset yellow-, methylene blue-, and phenol red-containing conducting papers increased by almost 50 %, 53 %, and 62 %, respectively, which implies good conductivities.

4. Conclusion

In this work, SWCNTs were functionalized by acid treatment method and decorated by f-SWCNTs with Ag NPs through a chemical treatment for using in polymeric conductive inks. Instead of multiwalled CNTs, in the proposed conductive inks single-walled CNTs were utilized to exploit their unique intrinsic properties, such as narrower structure, higher flexibility, etc. It can be concluded that conductive inks in different colors have been successfully synthesized. The inks were tested on cellulose filter papers, and a spectacular effect on the electrical conductivity of the papers

Table 2. Measured values of sheet resistance for different colored papers prepared by immersing into color inks lacking/containing SWCNT/Ag nanopowder.

Ink details	Input current [A]	Primary calculated sheet resistance [$\Omega/\square$]	Calculated sheet resistance after first washing [$\Omega/\square$]
Yellow paper without SWCNT/Ag	0.5 A	∞	∞
Blue paper without SWCNT/Ag	0.5 A	∞	∞
Red paper without SWCNT/Ag	0.5 A	∞	∞
Yellow paper containing SWCNT/Ag	0.5 A	1.54 ± 0.27	2.31 ± 0.31
Blue paper containing SWCNT/Ag	0.5 A	1.09 ± 0.18	1.67 ± 0.25
Red paper containing SWCNT/Ag	0.5 A	1.36 ± 0.36	2.20 ± 0.34

Fig. 8. Schematic configuration for four-point probe electrical test.

covered with SWCNT/Ag containing color inks was observed even after washing with warm water. It can be stated that SWCNT/Ag is a high-end candidate for low-cost, flexible microelectronics.

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