Mechanical, Optical, and Electrical Properties of Cellulosic Semiconductor Nanocomposites

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ABSTRACT: Photoluminescent cellulose fibers were prepared by impregnation of bleached bagasse pulp with the different cadmium and zinc sulfides nanostructures, namely, CdS, ZnS, CdS/ZnS, ZnS/CdS (Core/shell nanostructures), CdS/ZnS/CdS and ZnS/CdS/ZnS multilayered nanostructures. The prepared cellulosic nanocomposites were characterized regarding strength properties (tensile strength, tensile energy absorption, and burst resistance), optical properties (fluorescence emission), thermal stability, and dielectric properties. The strength properties of paper sheets nanocomposites impregnated with different nanoparticles solutions were noticeably decreased in spite of the very low loading of the fibers with the semiconductor nanoparticles. The decrease in tensile index, tensile energy absorption, and burst index of paper sheets nanocomposites are found to be ranged from 12-27, 13-36, and 11-38 %,

INTRODUCTION

Semiconductor nanoparticles or quantum dots (QDs), particularly Group II-IV semiconductor nanoparticles have been attracting increasing attention because of their unique electronic and optical properties resulting from the quantum size effect.^{1,2} Cadmium sulfide (CdS) and zinc sulfides (ZnS) nanoparticles are important semiconducting Group II-IV materials. Cadmium sulfide has a typical wide band gap of 2.42 eV at room temperature. Because of unique and excellent optical, photoluminescence, and electroluminescence properties, CdS nanoparticles find wide applications in modern technology such as light emitting diodes, solar cells, and optical devices based on the non linear optical properties.^{3,4} Zinc sulfide semiconductor has band gap energy of 3.7 eV. Zinc sulfide nanoparticles have received much attention because of their unique properties such as the large band gap energy, direct recombinarespectively. The different paper sheets nanocomposites showed fluorescence emission different from the as prepared polyethyleneimine-stabilized nanoparticles. Thermogravimetric analysis results showed that: the semiconductor nanoparticles did not affect the onset degradation temperature of cellulosic fibers but it caused faster termination of the main degradation step. The dielectric loss and the dc-conductivity of cellulosic fibers increased as a result of impregnating the fibers. The nature of the nanoparticles and the properties of the interphases strongly influenced the dielectric properties of the cellulosic/semiconductors nanocomposites. © 2009 Wiley Periodicals, Inc. J Appl Polym Sci 115: 2847–2854, 2010

Key words: zinc sulfide; cadmium sulfide; nanocrystal; cellulose; nanocomposite; paper sheet; dielectric properties

tion, resistance to high electric field, photoluminescence, and electrolumenices properties. Zinc sulfide nanoparticles also have potential applications in modern technology such as in solar cells,⁵ infrared windows,^{6,7} sensors and displays,⁸ bioimaging, and catalysis.⁹ Preparation of the complex semiconductor nanoheterostructures such as core-shell structures^{10–12} and quantum dot quantum well structures^{13–15} has also drawn considerable interest of researchers because of the enhanced optical and electrical properties of these complex structures compared with the single phase semiconductor nanoparticles.

The area of incorporating of semiconductor nanoparticles within a polymer matrix is of significant interest because of the possibility of producing polymer-nanoparticles based devices that could be easily processed in different geometrics, morphologies, and composition with consequent control over different electro-optical properties.¹⁶ Dispersion of nanoparticles within the polymer could be accomplished by both *ex situ* and *in situ* methodologies.^{17–22}

Inorganic-cellulosic nanocomposites (or so-called inorganic-cellulose hybrids) are of increasing interest because of the unlimited possibility to tailor natural polymer-based composites that have the advantages of the constituting materials and making a new

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material with new properties and suitable for different applications. Presence of semiconductor nanoparticles inside the fibrous structure of lignocelluloic materials could impart the fibers interesting properties concerning the optical and electrical properties.

For the best of our knowledge, little work has been published in the open literature in the area of semiconductor-cellulose nanocomposites. Hassan et al.²³ studied the preparation of new dendronized cellulose derivatives, which were used in the preparation of cadmium sulfide quantum dots/cellulose nanocomposites.²⁴ Ruan et al.²⁵ studied preparation and properties of CdS nanocrystals/regenerated cellulose nanocomposites by using *in situ* synthesizing method during cellulose dissolution in NaOH/thiourea system. Small et al.²⁶ studied the preparation of ZnS doped nanocrystals and mechanical and optical properties of cellulose–ZnS: Cu and cellulose-ZnS: Mn nanocomposites.

In a previous work²⁷ CdS, ZnS, CdS/ZnS, ZnS/ CdS (core/shell nanostructures), CdS/ZnS/CdS, ZnS/CdS/ZnS multilayered nanostructures have been prepared at low temperature using cadmium chloride, zinc chloride, and sodium sulfide in the presence of polyethyleneimne hyperbranched (PEI) polymer. The aim of this work is to use the previously prepared PEI-stabilized nanoparticles to prepared functional cellulosic materials that could be used in different optical and electrical applications.

EXPERIMENTAL

Materials

Reagent grade cadmium chloride (CdCl₂.3H₂O), zinc chloride, sodium sulfide (Na₂S), and polyethylene imine (M. Wt. 25,000–50,000) were reagent-grade chemicals and purchased from Sigma-Aldrich and used without further purification unless indicated. Bleached bagasse pulp was used for making of nano-composites, it was kindly supplied by Qena Company for pulp and paper, Qena, Egypt. The pulp had the following composition: 21% hemicelluloses, 65% alpha cellulose, 1.3% ash content, 0.2% lignin.

Synthesis of cadmium sulfide and zinc sulfide nanocrystals, core/shell, and multilayered structures

The synthesis cadmium sulfide and zinc sulfide nanocrystals, core/shell, and multilayered structures was performed using 0.1M cadmium or zinc chloride in the presence of 1% (wt/wt) PEI as described earlier.²⁷

Preparation of cellulosic semiconductor nanocomposites

Bleached bagasse pulp was immersed in the as-prepared sulfides solutions (0.1*M* solutions) for half an hour with continuous stirring and without further additives. The mixture was drained and paper hand sheets were made using hand sheets paper machine according to the SCAN-C 26 : 67. The sheets were dried at 105°C for 4 h then left at 25°C and 65% relative humidity before mechanical and optical testing. The retained amount of sulfide nanoparticles in the paper sheets (concentration of nanoparticles in the fibers) was determined thermogravimetrically by heating loaded fibers at 400°C for 30 min and at 800°C for 45 min. Correction was made by subtracting the ash content of the blank fibers from that of the loaded fibers.

Characterization of cellulose-semiconductor nanocomposites

Strength properties of the cellulose-semiconductor paper sheets were studied using tensile strength and burst resistance testing according to the known standard methods TAPPI T494 om-02 and TAPPI T403 om-02, respectively.²⁸ For tensile testing, Lloyd universal testing machine was used at crosshead speed of 5 cm/min and 10 cm gap, the width of the samples was 1.5 cm. For burst testing, Mullen type burst tester was used. The fluorescence properties of the cellulose-semiconductor paper sheets were studied using Jasco EP-777 spectrofluorometer at room temperature. Thermal stability of cellulose-semiconductor paper sheets was studied by thermogravimetric analysis (TGA) using a Perkins Elmer thermogravimetric analyzer under nitrogen at a temperature range from 25 to 500°C at a heating rate of 10°C/ min, the rate of nitrogen flow was 40 mL/min. The dielectric permittivity ε' , the dielectric loss, and ac resistance $R_{\rm ac}$ of cellulose-semiconductor paper sheets were measured over a temperature range from 30 to 90°C and at frequencies 100 mHz-1MHz using an impedance analyzer (Schlumberger Solartron 1260), an electrometer, amplifier, and measuring cell as described before. 29 The error in ϵ' tand amounts to \pm 1%, 1 and \pm 3%, respectively. The temperature of the samples was controlled by a temperature regulator with Pt 100 sensor. The error in temperature measurements amounts \pm 0.5°C. To avoid moisture, the samples were stored in desiccators in the presence of silica gel. Thereafter the sample was transferred to the measuring cell and left with P_2O_5 until the measurements were carried out.

RESULTS AND DISCUSSION

Cellulose fibers-semiconductor nanocomposites

In this work, the prepared PEI-stabilized semiconductor nanoparticles mentioned above were used to make cellulosic nanocomposites by soaking bagasse pulp in the as-prepared nanoparticles solutions. In a



Figure 1 Bagasse fibers loaded with CdS nanocrystals.

previous work, the prepared semiconductor nanoparticles were characterized using X-ray diffraction, transmission electron microscopy, fluorescence, and UV spectroscopy.²⁷ The size of the prepared semiconductor nanoparticles was in the range from 7-15 nm, 4-13 nm, 19-24 nm, 23-35 nm, 108-185 nm, an 100-130 nm for CdS, ZnS, CdS/ZnS, ZnS/CdS CdS/

ZnS/CdS, and ZnS/CdS/ZnS, respectively, as seen in the TEM pictures (particle size distribution was not measured).²⁷ The fibrous structure of bagasse, like all lignocellulosic materials, has porous structure and the size of pores and voids that are much larger than the size of the prepared nanoparticles. As the nanoparticles in the aqueous solution can strongly bind to the PEI polymer,³⁰ they could be retained in the fibrous structure during paper sheet making. PEI is one of the well-know coagulants that have the ability to collect very small particles and is expected to be able to stick to the fibers.

Figure 1 shows the TEM micrograph of bagasse pulp fibers loaded with CdS nanoparticles. As shown, the fibers are highly loaded inside the lumen of the fibers. Table I shows the ash content of the paper sheets prepared after their loading with the semiconductor nanoparticles. A slightly higher as content, i.e., fiber loading, of the single phase CdS and ZnS nanocrystals was found compared with the fiber loading with core/shell and multilayered nanostructures. This could be due to smaller size of CdS and ZnS nanoparticles than the other nanostructures. The results of ash content also show that fiber loading with the semiconductor nanoparticles was very low. This is expected because only the lumen of the fibers is loaded with the semiconductor nanopartilces, and excess nanoparticles were drained and washed out during paper sheet formation.

Strength properties of paper-semiconductor nanocomposites

The effect of the loaded semiconductor nanoparticles on the tensile strength, tensile energy absorption, and burst resistance of paper sheet/semiconductor nanoparticles was studied and the results are shown in Table I. In spite of very low loading of the semiconductor nanoparticles inside the fibers the effect on the strength properties was significant compared

TABLE I Nanoparticles Retention and Strength Properties of Semiconductor-paper Sheets Nanocomposites

	I.			
Sample	Retention of sulfides nanoparticles in paper sheet (%)	Tensile index (kgf/g m ²)	Tensile energy absorption	Burst index (g/cm ² g m ²)
Blank paper sheets (P)	_	4.47 (0.31)	1249 (85.4)	16.14 (0.48)
CdS-P	0.86	3.19 (0.32)	826 (73.9)	11.18 (0.43)
ZnS-P	0.87	3.06 (0.28)	801 (77.4)	10.00 (0.58)
CdS-ZnS-P	0.66	3.40 (0.24)	841 (65.1)	12.36 (0.80)
ZnS-CdS-P	0.75	3.37 (0.29)	833 (80.4)	11.95 (0.64)
CdS-ZnS-CdS-P ZnS-CdS-ZnS-P	0.70 0.79	3.52 (0.28) 3.95 (0.34)	925 (60.3) 1034 (73.8)	13.20 (0.55) 14.27 (0.67)

Values between brackets are the standard deviation.

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with the blank paper sheets. The tensile index was decreased by about 12 up to 27%. This significant loss in tensile strength at the very low fiber loading could be due to the nanosize nature of the semiconductor nanoparticles, i.e., very high surface area, that could corrupt hydrogen bonding between the fibers and reduce the strength properties of paper sheet. Generally, the highest decrease in tensile strength was recorded for ZnS-paper nanocomposite, i.e., the composites with the smallest size nanoparticles. The results generally show that the tensile index decreases as the size the loaded nanoparticles decreases.

The tensile energy absorption of paper sheets, which correlates the tensile breaking load with the extension at break, also decreased as a result of fiber loading with semiconductor nanocomposites. The decrease was about from 13 to 36%, and the highest loss in tensile energy absorption was for the ZnS-paper nanocomposites.

The burst strength of the paper sheets also decreased as a result of the loading with the semiconductor nanoparticles. The decrease ranged from about 11% up to 38%. The results show that the highest loss in burst strength was also recorded for the ZnS-loaded paper sheets and the smaller the size of the loaded nanoparticles the lower the burst index of the paper nanocomposites.

Optical properties of cellulose fibers-semiconductor nanocomposites

The fluorescence properties of cellulose fibers-semiconductor nanocomposites were studied and their emission spectra are shown in Figure 2. The obtained emission spectra of the semiconductor nanoparticles inside the fiber were different from those found in case of the fluorescence of the as-prepared PEI-stabilized nanoparticles in solution (not shown). The CdS nanocrystals in solution exhibit a band with two unresolved fluorescence peaks at about 452 nm and 470 nm (excitation wave length 270 nm) while the emission spectra of ZnS nanocrystals in solution exhibit asymmetric curve with a pronounced unresolved peaks at 400-420 nm (excitation wavelength 220 nm). The CdS/ZnS core/shell and CdS/ZnS/CdS multilayered structures in solution exhibit similar fluorescence main peaks at 442-445 and 442 nm (excitation wave length 220 nm). On the other hand, fluorescence emission of all cellulosic/ semiconductor nanocomposites showed a strong broad band centered at about 420 nm in addition to smaller ones at about 463, 469, 474, 483, 493 nm (excitation wave length 220 nm). This means that a blue shift of the band edge took place in case of all semiconductor nanoparticles except for in case of paper-ZnS nanocomposite where a red shift (~20 nm) in the band edge emission took place. These results

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Figure 2 Fluorescence spectra of paper sheets loaded with different sulfide nanoparticles.

indicate a strong interaction between the PEI- stabilized nanoparticles and the cellulosic fibers with the semiconductor nanoparticles is not suitable for utilizing the different optical properties of the embedded semiconductor nanoparticles in applications that utilize the modulating optical properties of the prepared nanoparticles. However, the high intensity of fluorescence emission is clear in spite of the very low semiconductor fiber loading.

Thermal stability of paper-semiconductor nanocomposites

To investigate the effect of loading of cellulosic fibers with sulfides nanoparticles on their thermal



Figure 3 TG and DTG curves of bagasse fibers, ZnS-, CdS- loaded fiber.

stability, TGA was carried out on paper sheets with cadmium sulfide and zinc sulfide nanoparticles at temperature range from room temperature up to 500°C. Figure 3 shows the TGA and deferential (DTGA) curves of these samples. As it is clear from the curves, loading of the fibers with nanoparticles did not affect significantly the thermal degradation of the fibers. The onset degradation temperature which is believed to be due to the evolution of noncombustible gases such as carbon dioxide, carbon monoxide, formic acid, and acetic acid,³⁰ was about 270°C. However, as seen in the deferential thermogravimetric curves (DTGA) the maximum weight loss temperature of this stage of degradation were at 360, 352, and 343°C for blank, CdS and ZnS-loaded paper nanocomposites samples. This means that loading the fibers with CdS or ZnS nanoparticles increased termination of the main degradation step once started. Also, fibers loaded with ZnS nanoparticles which have lower size than CdS, have lower maximum weight loss temperature. The mechanism of how the CdS or ZnS nanoparticles faster termination of the main degradation step of the cellulosic fibers need further detailed investigation.

Electrical properties cellulose fibers-semiconductor nanocomposites

The permittivity ε' and the dielectric loss ε'' of the cellulose fibers-semiconductor nanocomposites were

measured in the frequency range from 100 mHz to 1 MHz at different temperatures. Figures 4 shows ε' and ε'' of the blank paper sheet in the frequency range 100 mHz to 1 MHz at room temperature; the paper sheets were conditioned at 50% relative humidity for 48 h. From the figures, it is noticed that at low frequencies both ϵ' and ϵ'' increased with decreasing frequency according to a power-law behavior with an exponent close to unity.³¹ This behavior is characteristic for dc-like imperfect charge transport.³² The dielectric spectra in Figure 4 are broader than Debye curves,33 indicating that more than one relaxation process are present. Fitting the data is preformed by a computer program using Cole-Cole³⁴ shows different relaxation processes denoted MWS_{-rel} , β_{wet} , β_{-rel} . The low frequency MWS-rel process may be due to interfacial polarization found in heterogeneous samples. 35 β_{wet} process has been observed earlier $^{36-38}$ and is related to collective motions of water-cellulose mixed phase.39 The high-frequency relaxation process, referred to β



Figure 4 Cole-Cole relaxation of never-dried soft-dried cellulose fiber (at 65°C under vacuum).

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Figure 5 The permittivity ε' and dielectric loss ε'' versus the frequency *f* of paper sheets loaded with different amounts of CdS and ZnS nanoparticles. H: ~0.86%, M: 0.7%, and L: 0.5% (based on oven-dry weight of fibers).

 $_{\rm rel}$ could be related to the segmental motion of the polymer chain in the fibers. Drying the blank sample under vacuum at 65°C produces sample with water of about 0.7%. This low water content creates c $\beta_{\rm wet}$ and is denoted as $\delta_{\rm -rel}.^{40}$ as in Figure 4.

On the other hand, the data obtained for ε' and ε'' vs. frequency, for the paper sheets impregnated with different amount of CdS and ZnS nanoparticles are illustrated graphically in Figure 5. As shown in the figure ϵ' and ϵ'' CdS- impregnated fibers both ϵ' and ε'' are higher than those of ZnS- impregnated especially in the lower frequency side. This may be due to the fact that the band gap of CdS is lower than that of ZnS. Accordingly, the ion paths through the sample along the electric field would be shorter this in turn would reduce the low frequency losses especially if these are due to Maxwell Wagner-Sillars polarization. The conductivity results presented in Figure 6 also show higher conductivity of CdS-fiber nanocomposite that ZnS-fiber at 30°C at the different levels of nanoparticles loading.

In the case of core/shell paper nanocomposites, the CdS/ZnS paper nanocomposite showed lower ϵ'

and ε'' values than those of ZnS/CdS paper nanocomposite (Fig. 7). In case multilayered nanoparticles paper nanocomposites, the ZnS/CdS/ZnS paper



Figure 6 DC conductivity of paper sheets loaded with different amounts of CdS and ZnS nanoparticles (H: \sim 0.86%, M: 0.7%, and L: 0.5% based on oven-dry weight of fibers) and the other core/shell and multilayered nanostructures at 30°C and 90°C.



Figure 7 The permittivity ε' and dielectric loss ε'' versus the frequency *f* of paper sheets loaded with core/shell and multilayered nanoparticles.

nanocomposite showed lower ϵ' and ϵ'' values than those of CdS/ZnS/CdS paper nanocomposite as shown in the same figure. These results indicate that the electrical properties of the different paper nanocomposites depend on the structure of the outer layer of the nanocomposite as all samples with ZnS in the outer layer had lower ϵ' and ϵ'' values than those with CdS in the outer layer. The different nanoparticles could act as interlayers with different permittivity and conductivity between the fibers and hinder free-charge carriers in their motion between the two phases by causing potential barriers. As it is clear from Figure 7, the ZnS/CdS/ZnS and CdS/ ZnS/CdS have higher ε' and ε'' values than the core/shell paper nanocomposite. This could be attributed to the Maxwell Wagner-Sillars polarization mechanism at the boundaries between different phases of the nanoparticles, because of difference in the permittivity and conductivity of the components. It could be also attributed to the larger particle size of the multilayered nanoparticles than the core/shell ones.

The effect of temperature on the permittivity of different fiber/semiconductors nanocomposites is

also studied and the results are shown in Figure 8. From the figure it is noticed that the permittivity ε' increased with the increase of temperature and this increase is much pronounced at low frequencies.



Figure 8 Permittivity of the different cellulosic semiconductor nanocomposites at frequency 0.1 Hz and different temperatures.

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The core/shell ZnS/CdS shows the highest conductivity accompanied with a reduction of $\Delta\epsilon$ of δ -relaxation. The reduction of $\Delta\epsilon$ varied from sample to sample depending on the nature of impregnated particles structure until reaching its lowest value was for CdS/ZnS/CdS nanocomposite paper sample. On the other hand the $\Delta\epsilon$ of the MWS relaxation for all nanocomposite paper samples was higher than that of blank paper sample.

CONCLUSIONS

Cellulose fibers/semiconductor nanocomposites made by impregnating bagasse pulp fibers and the prepared semiconductor nanostructures showed lower strength properties than blank cellulose fibers sheets in spite of the very low fiber loading with the nanoparticles. The optical properties of the different cellulose/semiconductor nanocomposites were close to each other. Loading of cellulosic fiber with the prepared nanostructured semiconductors does not affect the degradation onset temperature but increases the rate of thermal degradation. The kind of nanocomposite and the properties of the interphases strongly influence the dielectric properties and interfacial polarization of cellulose fibers/semiconductors nanocomposites.

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