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Dispersion of SiC nanoparticles in cellulose for study of tensile, thermal and oxygen barrier properties

Sudhir K. Kisku, Satyabrata Dash, Sarat K. Swain*

Department of Chemistry, Veer Surendra Sai University of Technology, Burla, Sambalpur 768018, India

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ABSTRACT

Cellulose/silicon carbide (cellulose/SiC) nanobiocomposites were prepared by solution technique. The interaction of SiC nanoparticles with cellulose were confirmed by Fourier transformed infrared (FTIR) spectroscopy. The structure of cellulose/SiC nanobiocomposites was investigated by X-ray diffraction (XRD), and transmission electron microscopy (TEM). The tensile properties of the nanobiocomposites were improved as compared with virgin cellulose. Thermal stabilities of cellulose/SiC nanobiocomposites were studied by thermogravimetric analysis (TGA). The cellulose/SiC nanobiocomposites were thermally more stable than the raw cellulose. It may be due to the delamination of SiC with cellulose matrix. The oxygen barrier properties of cellulose composites were measured using gas permeameter. A substantial reduction in oxygen permeability was obtained with increase in silicon carbide concentrations. The thermally resistant and oxygen barrier properties of the prepared nanobiocomposites may enable the materials for the packaging applications.

1. Introduction

Nanoreinforcement of biobased polymer with nano clay (Dash, Kisku, & Swain, 2012), carbon nanotubes (Swain, Pradhan, & Sahu, 2013), boron nitride (Dash & Swain, 2013a; Kisku & Swain, 2012; Swain, Dash, Behera, Kisku, & Behera, 2013) and other nanofillers offer potential to increase the utilization of biopolymers due to the improvement in their properties such as thermal properties, mechanical properties, fire retardant and gas barrier properties. Renewable resource based on biodegradable polymers like cellulose, has the potential to be used as an alternative to petroleum-based polymers. Cellulose, the most abundant biopolymer in nature are obtained from various plants and living organisms (Nakagaito, Iwamoto, & Yano, 2005). Cellulose is light, environment friendly and biodegradable. It has low cost, specific strength and modulus. It is a polysaccharide consisting of liner chain of several hundred to over ten thousand β (1→4) linked D-glucose units, is an almost inexhaustible biopolymer with outstanding properties (Klemm, Heublen, Fink, & Bohn, 2005; Nishiyama, Langan, & Chanzy, 2002; Updegraff, 1969). For many years it has been an important raw material used in the form of intact wood for construction purpose, natural textile fibres and papers. Cellulose derivatives have been used as optical films, coating, controlled released systems, biodegradable plastics, biomedical materials and composites (Bledzki & Gassan, 1999).

More recently, attention was devoted to nanocomposites based on cellulose (Fahmy, Mobarak, Fahmy, Fadl, & EI-Sakhawy, 2006; Fahmy, 2007a, 2007b; Fahmy & Mobarak, 2008a, 2009, 2011, 2013) and inorganic nano particle such as SiO_2 (Pinto, Marques, Barros Timmons, Trindade, & Pascoal Neto, 2008), TiO_2 (Marques, Trindade, & Pascoal Neto, 2006) and kaolin particle (Fahmy & Mobarak, 2008b). These materials normally show improved mechanical, optical and thermal properties due to the combination of inorganic and organic components in the final materials. Park, Liang, Mohanty, Mishra, and Drzal (2004) prepared the biodegradable cellulose acetate/organoclay nanocomposites and reported the improvement of mechanical properties due to good exfoliation and dispersion of clay in the cellulose acetate matrix. The cellulose-montmorillonite nanocomposites were prepared and the increase in thermo-oxidative stability as compared to the cellulose was reported (Cerruti et al., 2008). He, Chang, Peng, and Zhang (2012) prepared the cellulose/hydroxyaptite nanocomposites films and reported the improvement of thermal stability and mechanical strength. The cellulose/calcium carbonate nanocomposites having improved mechanical properties as compared to the virgin cellulose were synthesized and characterized (Vilela, Freire, Marques, Trindade, & Pascoal Neto, 2010). Pranger and Tannenbaum (2008) synthesized the biobased nanocomposites of furfuryl alcohol with cellulose whisker and montmorillonite clay and reported their high thermal resistance. Cellulose acetate/calcium carbonate hybrid nanocomposites were prepared and better thermal stability than pristine cellulose acetate was observed (John, Chen, & Kim, 2012). Seydibeyoglu and Oksman (2008) prepared the novel nanocomposites based on polyurethane and micro fibrillated cellulose by

* Corresponding author. Tel.: +91 09937082348; fax: +91 0663 2430204.
E-mail addresses: swainsk2@yahoo.co.in, swainsk2@rediffmail.com (S.K. Swain).

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compression moulding and reported the improvement of mechanical and thermal properties of polyurethane due to the addition of nano cellulose. The use of nano fibres in composite materials has recently been reviewed (Azizi Samir, Alloin, & Dufresne, 2005). Boldizar, Klason, Kubat, Naslund, and Saha (1987) reported that cellulose nanocomposites based on poly (vinyl acetate) matrix showed significantly improved mechanical properties as compared to the base material. Bacterial cellulose-poly (vinyl alcohol) nanocomposites were prepared by in situ process and reduction in Young's modulus and increase in toughness was observed (Gea, Billotti, Reynolds, Soykeabkeaw, & Peijs, 2010).

Among different nano fillers, silicon carbide is an important and non-oxide ceramic material having diversified industrial applications due to its exclusive properties such as, high melting point, oxidation resistance, high erosion resistance, high hardness, strength, chemical and thermal stability (Saravanan, Subramanian, Vishnu Kumar, & Tharanathan, 2006). Silicon carbide can occur in more than 250 crystalline forms called polytypes and has attracted much attention these days as it has a good match of chemical, mechanical and thermal properties (Guo et al., 2008). The effect of nanometre SiC filler on Polyetheretherketone (PEEK) was studied for improvement of tribological behaviours of PEEK composites (Wang, Xue, Liu, & Chen, 2000). There are a lot of works on cellulose based composites, but the work related to nano SiC reinforced cellulose nanobiocomposites is scanty in the literature.

In the present study environmental friendly SiC reinforced nanobiocomposites were synthesized from biodegradable cellulose. The nanobiocomposites were characterized by XRD and TEM to verify the silicon carbide contents in the composites. Tensile, gas barrier and thermal property of the composites were measured. A significant improvement of thermal property with reduction in gas barrier property was noticed in order to enable the materials for packaging applications.

2. Experimental

2.1. Materials

Cellulose powder used in this work was purchased from HiMedia Laboratories Pvt. Ltd., Mumbai, India and used as matrix material. Silicon carbide nano powder was obtained from Sisco Research Laboratories Pvt. Ltd., Mumbai, India and used as filler. The average diameter of SiC nanoparticle is 70 nm. The other chemicals used were of analytical grade and used as such. All solutions were prepared using double distilled water.

2.2. Preparation of cellulose/SiC nanobiocomposites

Cellulose/SiC nanobiocomposites were successfully prepared by solution method using variable percentage of silicon carbide concentrations. Cellulose was dispersed in distilled water by stirred for 30 min at 60 °C and sonicated for 30 min by using ultrasonic cleaner (120 W/80 kHz.). The different wt% solutions of silicon carbide were prepared in distilled water by stirring for 30 min at 60 °C and sonication for 30 min. The resulting solutions of cellulose and silicon carbide were mixed and continued stirring for 3 h at 60 °C to get a viscous solution. It was filtered and dried in an oven for 24 h at 50 °C. The series of cellulose/SiC nanobiocomposites of 1, 2, 3, 4, 5, 8 and 10 wt% silicon carbide were prepared by this method.

2.3. Characterization of cellulose/SiC nanobiocomposites

FTIR spectra of the samples (in form of KBR pallets) were recorded by using a Shimadzu IR Affinity-1 Fourier infrared spectrophotometer in the range of 4000–400 cm^{-1}. X-ray diffraction patterns were obtained using a Rigaku X-ray machine operating at 40 kV and 150 mA. The transmission electron microscope (TEM), Tec-nai 12, Philips operating at 120 kV was used to study the dispersion of SiC in nanobiocomposites. An ultra cut low temperature sectioning system equipped with diamond knife was used to cut ultra thin specimens of 75 nm by cryoultra microtome below the glass transition temperature of the sample. The specimens were transferred to a copper grid. Tensile properties of cellulose/SiC nanocomposites were measured with ASTM D-638-00 using an instron testing machine model 5567 and the test was performed at the rate of 50 mm/min with a load of 0.5 tonne. Reported data are the average value of five strips of same sample.

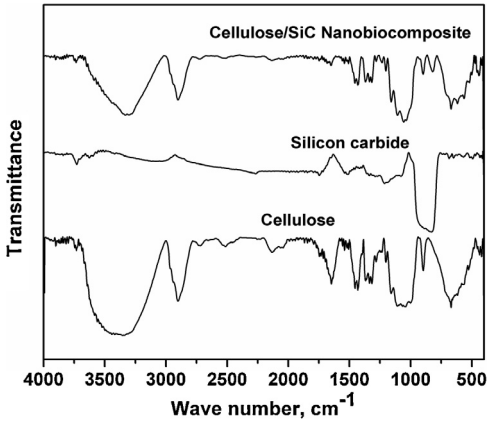

Fig. 1. FTIR analysis of SiC, cellulose and cellulose/SiC nanobiocomposites with 8% SiC loading.

The thermogravimetric analysis (TGA) was performed for the sample under nitrogen purge at a heating rate of 10 °C/min using a TGA apparatus (model DTG-60, Shimadzu Corporation, Japan). Oxygen permeability of the nanobiocomposites was measured with ASTM F 316-86 by using Oxygen Permeation Analyzer (PMI instrument, model GP-201-A, NY, USA). For testing, the synthesized powder nanobiocomposites were converted into films of 0.5 mm thickness with the help of a polymer press at a pressure of 9 tonnes and a temperature of 200 °C. The results were recorded as average of the values obtained from five same samples. The biodegradability of the cellulose/SiC nanobiocomposites was studied for 180 days using sludge water.

3. Results and discussion

3.1. FTIR analysis of cellulose/SiC nanobiocomposites

The FTIR analysis was carried out to observe the interaction between the cellulose and the silicon carbide. The spectra of the cellulose, silicon carbide and cellulose/SiC nanobiocomposites are compared as shown in Fig. 1. The peaks of cellulose at 3335 cm^{-1} and 2920 cm^{-1} are due to —OH and C—H vibrations respectively. The absorption peak at 1670 cm^{-1} corresponds to the >C=O stretching mode of ester of cellulose. The symmetric $-CH_2$ or asymmetric $-CH_3$ stretching mode band were observed at 1440 cm^{-1}. The broad peak at 1065 cm^{-1} is due to pyranose ring C—O—C and asymmetric ring stretching mode. In case of nano silicon carbide the strong peak at 864 cm^{-1} is due to Si—C stretching vibration. In the cellulose/SiC nanobiocomposites, the presence of Si—C stretching peak at 864 cm^{-1} along with the peaks of cellulose gives evidence for the formation of composites.

3.2. XRD analysis of cellulose/SiC nanobiocomposites

The XRD patterns of cellulose, SiC and cellulose/SiC nanobiocomposites are shown in Fig. 2. The pure SiC shows the crystalline

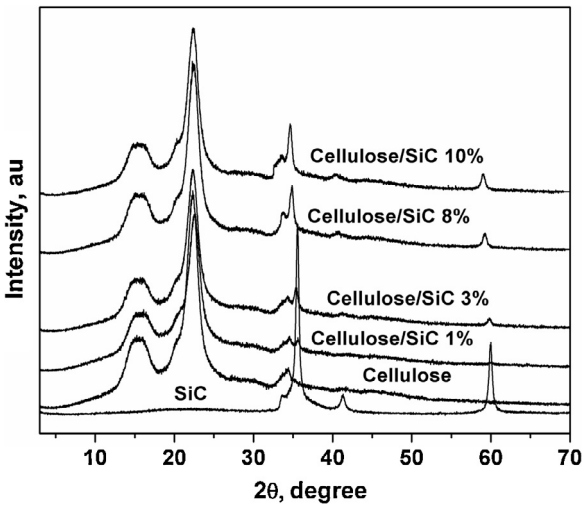

Fig. 2. XRD patterns of SiC, cellulose and cellulose/SiC nanobiocomposites.

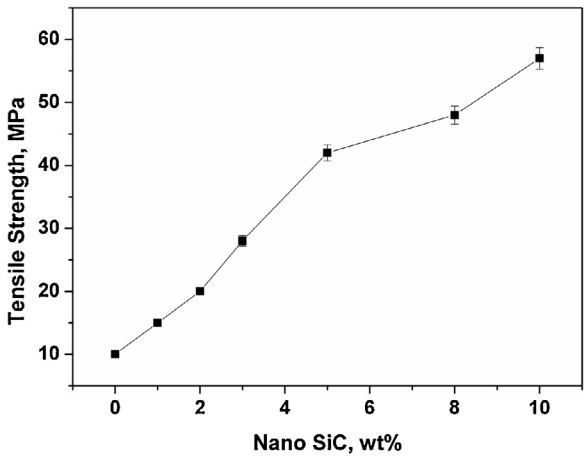

Fig. 4. Tensile properties of cellulose/SiC nanobiocomposites.

peaks at 2θ values of 37°, 45° and 60° due to different phases of SiC. Cellulose shows the three crystal peaks at 2θ values 17.7°, 22° and 35° are assigned to crystal plane of cellulose. In case of all nanobiocomposites the peaks of silicon carbide are less intense than the pure silicon carbide. This may be due to the interaction between cellulose and silicon carbide. The crystal peaks of both cellulose and silicon carbide are present at about same positions supporting the evidence for the formation of nanobiocomposites. The nanobiocomposites of 8% and 10% silicon carbide loading, the crystalline peaks at 2θ values of 37° of silicon carbide is shifted to a lower angle unlike the composites at lower percentage of SiC. It may be due to different extent of interaction of SiC with cellulose at different concentrations.

3.3. TEM analysis of cellulose/SiC nanobiocomposites

The dispersion of SiC nanopowder within cellulose matrix is represented in Fig. 3. Silicon carbide nanopowder with average diameter of 70 nm is dispersed with less interconnecting distribution (Fig. 3a). It is noticed that at higher SiC loading some local agglomeration of SiC occurs (Fig. 3b). It may be due to lack of interlocking of excess SiC with the cellulose matrix. In Fig. 3b, the black patches are due to the agglomeration of SiC at composites of 8 wt%. However, the local agglomeration of SiC in composites did not affect the physical properties of cellulose/SiC nanobiocomposites.

3.4. Tensile properties of cellulose/SiC nanobiocomposites

The tensile strength of cellulose/SiC nanobiocomposites as a function of SiC content are presented in Fig. 4. It was found that tensile strength of the composites is approximately five times more as compared to that of the pure cellulose. Further, it is observed that the tensile strength of composite materials increased with increase in SiC content. It may be due to the presence of rigid SiC nano particles which increases the tensile modulus of cellulose matrix. From the result of tensile property, it is evidenced that nano SiC act as good reinforcing agent for the cellulose matrix.

3.5. TGA analysis of cellulose/SiC nanobiocomposites

Thermal properties of cellulose, nano SiC and cellulose/SiC nanobiocomposites are compared by thermogravimetric analysis (TGA) in the temperature range of 35–800 °C as shown in Fig. 5. The thermal decomposition of cellulose occurred in three steps, first step was the initial weight loss from 50 °C to 100 °C due to the loss of water on the surface of cellulose, second step was due to decomposition of cellulose and third step was due to oxidation of partially decomposed cellulose at 350 °C and then charring. From TGA analysis it is observed that cellulose decomposes completely where as about 2%, 10% and 30% of residue left in the case of cellulose/SiC nanobiocomposites with 1, 5 and 8 wt% of SiC

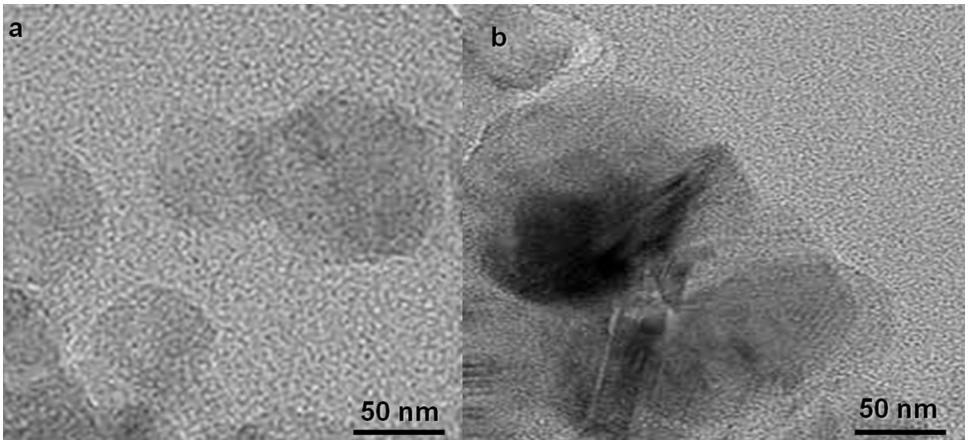

Fig. 3. TEM image of cellulose/SiC nanobiocomposites of (a) 2 wt% and (b) 8 wt%.

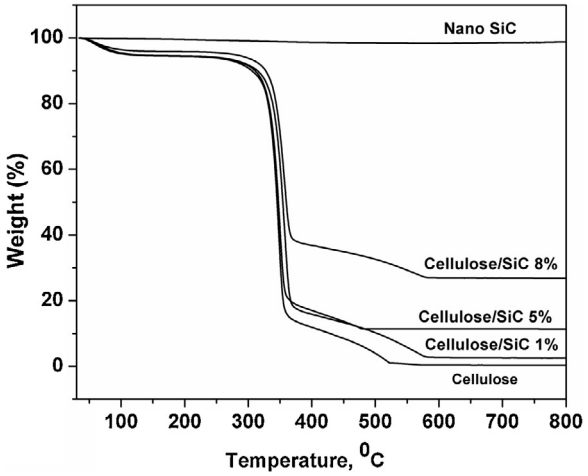

Fig. 5. TG curves of virgin cellulose, SiC and cellulose/SiC nanobiocomposites of 1, 5 and 8 wt% SiC concentrations.

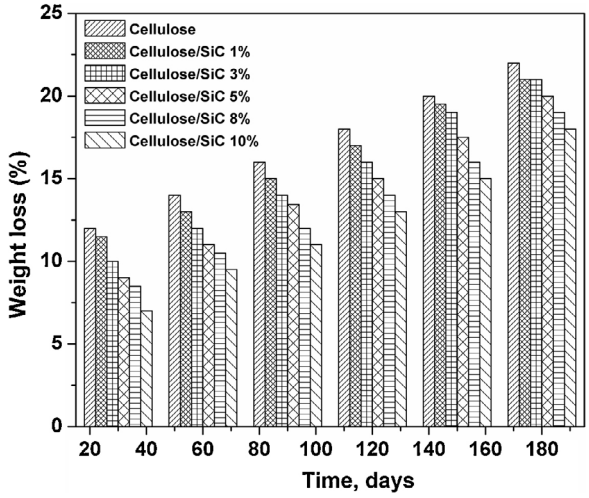

Fig. 7. Biodegradation property of cellulose/SiC nanobiocomposites.

loading respectively. In our earlier study (Dash & Swain, 2013b) of starch/SiC bionanocomposites the residue left after degradation was 2%, 20% and 30% for 1, 5 and 8 wt% of nano SiC loading. Starch/SiC and cellulose/SiC composites show similar results at low and high content of nano SiC whereas at 5 wt% loading, starch/SiC bionanocomposites show more thermal stability than the cellulose/SiC nanobiocomposites. Further it was noticed that the silicon carbide was stable and there was no weight loss up to the temperature of 800 °C. The interaction of thermally stable silicon carbide with the cellulose increases the thermal resistance of the composites and consequently the thermal decomposition temperature.

3.6. Oxygen barrier properties of cellulose/SiC nanobiocomposites

In Fig. 6, the oxygen permeability of virgin cellulose and cellulose/SiC nanobiocomposites are compared in order to study the effect of SiC loading. Oxygen permeability was found to be reduced proportionately with increasing concentration of the nano SiC. With 10% SiC loading, the oxygen permeability of cellulose/SiC nanocomposite was reduced by three times as compared to virgin cellulose. The reduction in oxygen permeability is because of the fact that silicon carbide nanoparticles are present within the voids of cellulose creating the restrictive path for oxygen penetration. Further, it was observed that oxygen flow rate through all the nanobiocomposites was less as compared to the virgin cellulose at a constant pressure of 2 psi. The flow rate was decreased with increase in percentage of SiC loading. In our earlier report (Dash & Swain, 2013b) on starch/SiC bionanocomposites, the permeability was reduced by four times whereas in the present study of cellulose/SiC nanobiocomposites, reduction is three times than the corresponding virgin matrix at 10 wt% loading. The substantial reduction in oxygen permeability may be due to the dispersion of silicon carbide within cellulose. In order to compare the accuracy of experimental data a model graph was prepared by using 3rd order regression polynomial equation (R^2 = 0.9997).

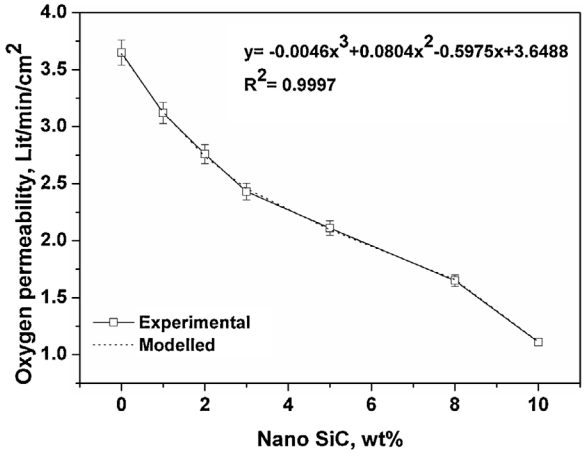

Fig. 6. Oxygen permeability of cellulose/SiC nanobiocomposites as a function of wt% of SiC at constant pressure of 2 psi.

3.7. Biodegradable properties of cellulose/SiC nanobiocomposites

The biodegradation of cellulose/SiC nanobiocomposites were compared with virgin cellulose for 180 days in an interval of 30 days. The degradation was studied in order to calculate percentage weight loss of materials in activated sludge water (Fig. 7). It was noticed that the percentage weight loss in nanobiocomposites was decreased with increase in percentage of silicon carbide loading. However biodegradation of nanobiocomposites as well as virgin cellulose was increased with increase in time duration with the sludge water. This is similar to our earlier report with starch/SiC bionanocomposites (Dash & Swain, 2013b). The weight loss of cellulose after 30 days was found to be double to the weight loss of nanobiocomposites after 180 days. The decrease in biodegradation of composites as compared to the virgin cellulose may be due to the active participation and well dispersion of SiC with cellulose matrix. However, the less biodegradable properties of nanobiocomposites have been compromised with the properties of thermal resistance and gas permeability.

4. Conclusion

The cellulose/SiC nanobiocomposites were prepared and formation of composite was evidenced by FTIR. The X-ray diffraction pattern was utilized in order to study the structure of cellulose/SiC nanobiocomposites. In thermogravimetric analysis, the thermal stability of the cellulose/SiC nanobiocomposites was enhanced as compared to the virgin cellulose. The improvement of thermal stability and tensile properties of cellulose was achieved due to uniform distribution of silicon carbide. The thermally stable nanobiocomposites with substantial reduction in oxygen permeability may enable the material for packaging applications.

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