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Cellulose nanofibril (CNF) reinforced starch insulating foams

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Abstract In this study, biodegradable foams were produced using cellulose nanofibrils (CNFs) and starch (S). The availability of high volumes of CNFs at lower costs is rapidly progressing with advances in pilot-scale and commercial facilities. The foams were produced using a freeze-drying process with CNF/S water suspensions ranging from 1 to 7.5 wt% solids content. Microscopic evaluation showed that the foams have a microcellular structure and that the foam walls are covered with CNF's. The CNF's had diameters ranging from 30 to 100 nm. Pore sizes within the foam walls ranged from 20 to 100 nm. The materials' densities ranging from 0.012 to 0.082 g/cm^3 with corresponding porosities between 93.46 and 99.10 %. Thermal conductivity ranged from 0.041 to 0.054 W/m-K. The mechanical performance of the foams produced from the starch control was extremely low and the material was very friable. The addition of CNF's to starch was required to produce foams, which exhibited structural integrity. The mechanical properties of materials were positively correlated with solids content and CNF/S ratios. The mechanical and thermal properties for the foams produced in this study appear promising for applications such as insulation and packaging.

Keywords Cellulose nanofibril (CNF) · Starch · Foam · Atomic force microscope (AFM) · Thermal conductivity

Introduction

In recent years, there has been increasing interest in development of nanocomposites based on nanocellulosic materials (Siro and Plackett 2010). In this study cellulose nanofibril and starch insulation foams were produced and characterized. The main reason for using cellulose is that it is an abundant material, which can be obtained from renewable sources including a broad range of plants and sea animals (Moon et al. 2010). Starch is another abundant natural polymer, which is a

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promising raw material for the development of novel materials (Martins et al. 2009). It is a widely available biopolymer with a price half that of polyethylene and polystyrene. Annually, millions of metric tons of starch are used as non-food products in the paper and textile industries (Glenn et al. 2011). Starch is mostly water soluble, difficult to process and has low mechanical properties. It was found that reinforcing starch with cellulose microfibrils increases the mechanical properties significantly (Dufresne and Vignon 1998). Glenn and Irving produced microcellular starch foams with different drying techniques and investigated the mechanical and thermal properties. They showed that mechanical properties of microcellular foams are positively correlated with density. They found that corn starch foams exhibited greater compressive strength (0.19–1.14 MPa) and density (0.12–0.31 g/cm^3) than the wheat starch and high amylose cornstarch foams. (Tatarka and Cunningham 1996) showed most starch-based foams have similar compressive strength (0.0927 MPa) with EPS foams. (Chen et al. 2004) studied starch graft poly (methyl acrylate) loose-fill foams and they found that starch graft poly (methyl acrylate) foams (S-g-PMA foams) have 0.07 ± 0.01 MPa compressive strength with 0.0086 ± 0.00021 g/cm^3 density. (Nabar et al. 2005) showed that starch based foams have compressive strength between 12.5 and 13.1 Pa with the densities changes from 0.003 to 0.0035 (g/cm^3). Svagan et al. (2011) investigated the mechanical properties of amylopectin-based foams with varying microfibrillated cellulose (MFC) contents and they showed increasing the MFC content produces higher mechanical properties, however, maximizing the MFC content doesn't mean having the highest mechanical properties. Svagan et al. (2008) obtained the optimum mechanical properties from the 40 % MFC reinforced foams when compared to 0, 10 and 70 % MFC reinforcements. On the other hand, (Glenn et al. 2007) found that, adding soft wood fibers to starch foams increases the thermal degradation temperature from 270 °C to over 300 °C. Dispersed cellulose fibers significantly increase the mechanical and thermal properties of starch foams. Glenn and Irving 1995a, b produced corn starch foams have thermal conductivity values ranging from 0.037 to 0.040 W/m-K.

The higher specific properties of NFC compared to the previously used forms of wood reinforcement was judged to offer an opportunity for additional improvements. This exploratory study aimed to determine the impact of solids content and CNF/S ratio on the morphology, physical, and mechanical properties of insulation foams with the intent to evaluate their suitability for application as structural insulation foam or other market opportunities.

Table 1 Experimental design of produced foams

Sample	Number of trays (30.6 cm × 61 cm × 3.5 cm)
0.5 % CNF+ 0.5 %starch	4
1 % CNF	4
1.5 % CNF+ 3 %starch	4
1.5 % CNF+ 6 %starch	4
7.5 % starch	4

Materials and methods

Nano fibrillated cellulose (CNF) used in this study was produced by the University of Maine Process Development Center. The CNF was prepared mechanically using a pilot-scale double disk refiner to fibrillate a bleached softwood Kraft pulp. The materials are typically 20–50 nm in diameter and have a length of several micrometers. Five different thermal insulation foams were prepared from aqueous suspensions (tap water + material) with the following solid contents; 1 % CNF, 0.5 % CNF+0.5 % starch, 1.5 % CNF+3 % starch, 1.5 % CNF+6 % starch and 7.5 % starch (Table 1).

The CNF/water suspensions were obtained at 3 wt% solids content. The CNF suspensions were reduced to 1 wt% by adding water into the suspension. The suspension was placed in a 20 L capacity container. A high shear mixer was used to disperse the CNF in suspension (1700 RPM for 20 min). For creating the starch foams, industrial corn starch (Tate&Lyle) was used and cooked at 87.8 °C (190 °F) and mixed at 500 RPM for 1 h. Starch solutions were cooled down to the room temperature (23 ± 2 °C). The final solids content was determined by oven drying 30–35 g suspension samples. The suspensions had a high consistency and were gel-like in appearance. Starch solution and CNF suspension were put into high shear mixer and dispersed (1700 RPM for 20 min). The dispersed suspensions were poured into trays (30.6 cm × 61 cm) to a depth of

3.5 cm and placed in a freeze dryer. Suspensions were freeze-dried using a Millrock Technology Max53 freeze dryer utilizing the Opti-Dry 2009 control system. T-type thermocouples were placed in the material to monitor temperature during the freeze-drying process. At first, partial vacuum was pulled then he chamber temperature was lowered from 20 to −45 °C in 1 h and maintained at that temperature for 250 min. The chamber was then evacuated to a pressure of 100 mTorr. The chamber temperature was maintained at −45 °C for 30 min, ramped to 0 °C over 2 h, ramped to 20 °C in 4 h and then maintained until average thermocouple reading in the materials was 20 °C for 4 h.

Morphology

Representative CNF/starch foam samples were imaged using scanning electron microscopy (SEM) and atomic force microscopy (AFM). For the SEM measurements, 3 mm × 3 mm × 2 mm samples were prepared from the freeze dried foams using a razor blade, placed on double-sided carbon paper and pasted onto stubs. SEM micrographs were obtained with a Zeiss Nvision 40 FIB-SEM at an acceleration voltage of 5.0 kV after gold–palladium (Au:Pd) sputter coating (~15 nm thickness) was applied to the specimens. Surface topography was measured with an MFP-3D AFM (Asylum Research). Specimens (foam powders) were pasted onto stubs using an epoxy adhesive and imaged 24 h later. Images were obtained a chamber temperature of 25 °C using tapping mode (AC Mode) and an Asylum Research AC240TS-10 cantilever tip with a 9 ± 2 nm radius with spring constant, k (N/m) = 2 (0.5–4.4).

Physical properties

Density and relative density

Density measurements of the foams were performed according to ASTM C303-10 by measuring six 150 mm x 150 mm x 25.4 mm (6 in × 6 in × 1 in) specimens from each group. The measured mass (g) was divided by the measured volume (cm^3) to calculate the density. Relative density is a significant property for the cellular materials which is the ratio between bulk density (P_{bulk}) and particle density (P_{particle}) (Gibson and Ashby 1997).

Porosity

The void fraction, which is called porosity, the ratio of pore volume to its total volume of foams, was calculated using liquid porosimetry method (Gibson and Ashby 1997) (Eq. 1).

$$\Phi = 100 \times \left(1 - \frac{\rho_{\text{bulk}}}{\rho_{\text{particle}}}\right) \tag{1}$$

where Φ porosity, P_{bulk} density of the foam, P_{bulk} density of the foam (1.50 g/cm^3).

Volume fractions

Fiber volume fraction which is the volume fraction of the fibers in the composition is calculated using Eq. 2(Reuss 1929).

$$V_f = V_f/(V_f + V_m) \tag{2}$$

where V_f fiber volume fraction, and V_m matrix volume fraction.

Mechanical properties

Flexural testing

Six (6) samples with 300 mm × 100 mm × 25.4 mm (12 in × 4 in × 1 in) dimensions from each sample group were tested by using a three-point bending test method according to ASTM C203-12. The crosshead displacement rate was 6 mm per min. Specimen displacement was obtained from the crosshead displacement (Instron 5966, with 100KN maximum load). Flexural tests were applied under laboratory conditions (25 ± 2 °C and 50 % relative humidity). The flexural modulus of the foams was obtained from the linear initial part of the force-deflection curves (Fig. 1).

Compression testing

Six (6) samples with 150 mm × 150 mm × 25.4 mm (6 in × 6 in × 1 in) dimensions from each sample group were tested according to ASTM C165-07. Each specimen was compressed at a rate of 6 mm per min

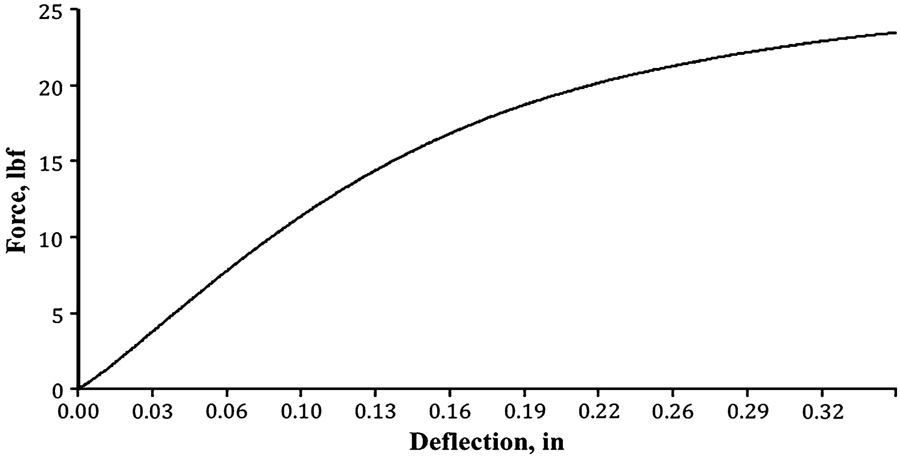

Fig. 1 Typical force–deflection curve for flexural tests of foams

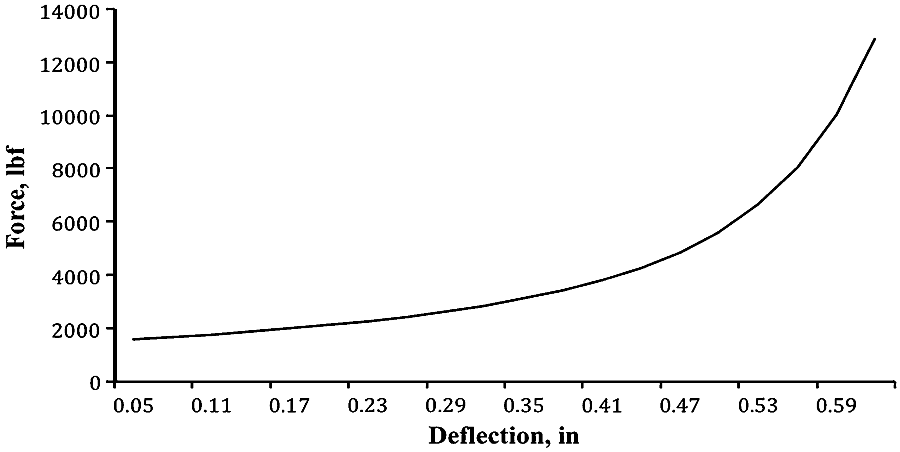

Fig. 2 Typical force–deflection curve for compression tests of foams

and the specimen displacement was obtained from the crosshead displacement (Instron 5966, with 100KN maximum load). Compression tests were conducted under laboratory conditions, 25 ± 2 °C temperature and 50 % relative humidity and the specimens were conditioned for 1 day prior to testing. The compression modulus was obtained from the linear initial part of the force-deflection curves (Fig. 2).

Thermal properties

Thermal conductivity measurements

Six (6) specimens (300 mm × 300 mm × 25.4 mm) were prepared from each group, two specimens from each tray. The steady-state thermal transmission was measured according to ASTM C518-10 using a heat flow meter (NETZSCH Lambda 2000 heat flow meter).

Thermogravimetric analysis (TGA)

Six (6) specimens from each group, which weighed from 6 to 10 g, were prepared as a powder and placed into crucibles according to ASTM E113-08. The thermogravimetric analysis was conducted from 25 to 800 with 10 °C increase per min using a Mettler Toledo Thermogravimetric Analyzer (TGA/SDTA851^e).

Statistical analysis

The density, compression, flexure strength and modulus, thermal conductivity, thermal resistivity, and

Table 2 Physical properties of foams

Sample	Solid–water content (%)	Fiber volume fraction (%)	Density (g/cm^3)	Relative density	Porosity (%)
0.5 % CNF + 0.5 %starch	1–99	50	0.013 (0.0010) D	0.00867	99.10 (0.081) A
1 % CNF	1–99	100	0.014 (0.0016) D	0.00933	99.10 (0.118) A
1.5 % CNF + 3 %starch	4.5–95.5	33.3	0.053 (0.0011) C	0.03533	96.50 (0.877) B
1.5 % CNF + 6 %starch	7.5–92.5	20	0.076 (0.0020) B	0.05067	94.95 (0.131) C
7.5 % starch	7.5–92.5	0	0.098 (0.0036) A	0.06533	93.46 (0.231) D

Parentheses indicate the standard deviation. A, B, C and D letters indicates the significant differences between the treatments

thermogravimetric analysis data were compared by conducting a one-way Means/ANOVA to check if there was a significant overall difference (significance level (alpha) = 0.01). Significant different between groups were evaluated by use of a Tukey–Kramer Honestly Significant Differences (HSD) test with alpha = 0.05. A sample size of six (n = 6) was used for all statistical analysis.

Results and discussion

The large range in sample density (0.013–0.098 g/cm^3) resulted in all statistical analysis having significant overall differences. There was a significant effect of solids content on the porosity, density and relative density (Table 2). As expected, including less solids content in the suspension produced a more porous structure. The reason for the inverse proportion between solid content and porosity can be explained by the increased bonds with increase solid content between starch and CNF in the same volume. This indicates that the production process (suspension-dispersion) and foam preparation method (freeze-drying) can be manipulated to produce foams of varying density and porosity.

A representative set of SEM images and fiber size measurements of the foams are given in Fig. 3. It is shown that there is variety in fibril diameters due to production process (freeze-drying) and raw material (mechanically produced CNF). The selected fibrils show the average fibril diameters, whereas there are fibrils with couple hundred nanometers diameters and there are other fibrils with <10 nm diameters in the structure as well. Some damage (cracking) was evident because of sample preparation process (razor blade cutting). The structure of the foam wall material was evaluated (Fig. 3b). The foam wall structure is a plate of CNF material embedded within a starch matrix. Further investigation of the foam cell wall material (Fig. 3c) illustrates the nanoscale fibril structure of the CNF with diameters ranging mostly from 30 to 100 nm.

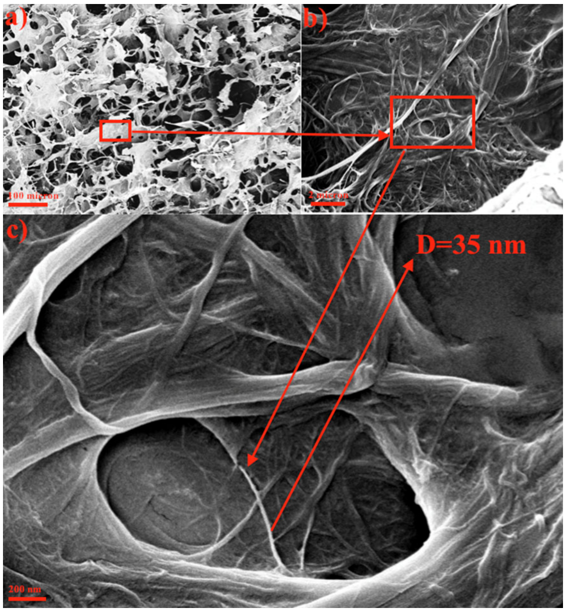

Fig. 3 Representative SEM images of foams (1.5 %CNF + 6 %S), (**a**) cellular structure, (**b**) distribution of nanofibrils in 2 micron *scale bar* and (**c**) fibril diameter measurements

Atomic force microscopy was used to measure the diameter of nano pores in the cellular wall material. It was determined that the diameter of the pores (Fig. 4) ranged from 20 to 100 nm. The difference between the SEM and AFM images can be explained by the difference in imaging principles and different regions and field of view of the images.

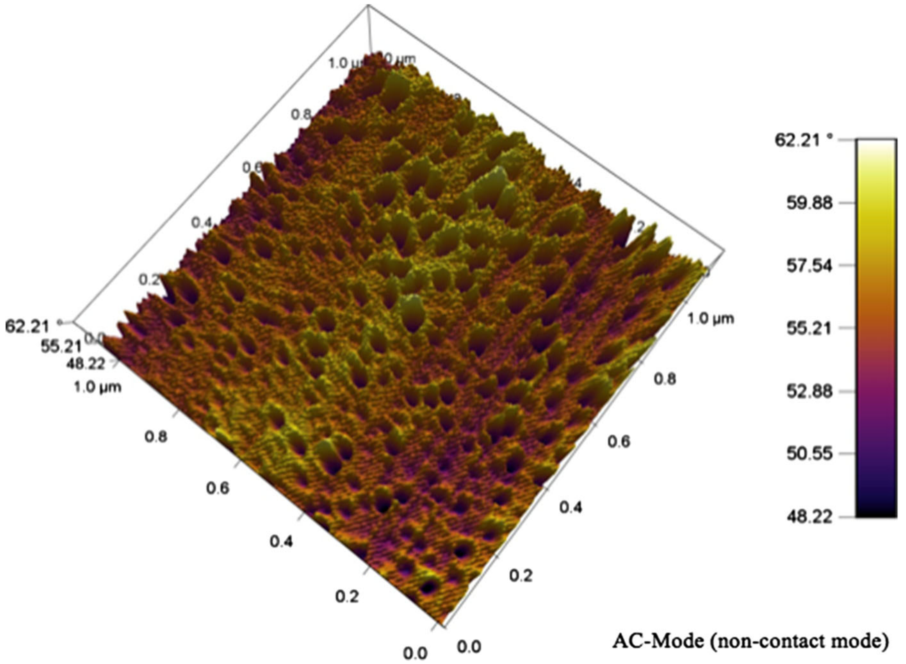

Fig. 4 Representative AFM images of foams (1.5 %CNF + 6 %S)

Table 3 Flexural properties of foams

Sample	Density (g/cm^3)	Elastic modulus (kPa)	Modulus of rupture (kPa)
0.5 % CNF+ 0.5 %starch	0.013 (0.0007) D	2.30 (1.10) C	40.00 (10.0) C
1 % CNF	0.014 (0.0012) D	14.0 (8.00) C	140.0 (40.0) C
1.5 % CNF+ 3 %starch	0.053 (0.0011) C	610 (80.0) B	2,140 (300) B
1.5 % CNF+ 6 %starch	0.082 (0.0049) B	2,530 (670) A	6,590 (1,110) A
7.5 % starch	0.098 (0.0,036) A	N/A	N/A

Parentheses indicate the standard deviation. A, B, C and D letters indicates the significant differences between the treatments. The extreme friability of the 7.5 % starch specimens did not allow for determination of the mechanical performance

The density, flexural modulus and flexural strength (MOR) of the foams are summarized in Table 3. Density of the foams ranged between 0.013 and 0.082 g/cm^3. The increase in the solids content increased the total mass of the foam structure, which increased the density. The elastic modulus (MOE) and modulus of rupture (MOR) plotted against density are shown in Fig. 5.

Increasing the density was correlated with increases in modulus of elasticity (MOE) and MOR and a curvilinear relationship was indicated between density and flexural properties. However, pure starch foams with even higher solid content (7.5 %) and density exhibited very low mechanical properties. Reinforcement of the starch foams using CNF produced significant increases in flexural properties for all groups. However, when the 1 % CNF (blue sign in Fig. 5) and 0.5 % CNF + 0.5 % S (yellow sign in Fig. 5) combinations were compared, it seems there is no effect of adding CNF because of the given scale in “y” direction. The CNF addition produced an 84 % increase in MOE and 71 % increase in MOR (Table 3). In addition to this, when 3 % starch or 6 % starch was reinforced with 1.5 % cellulose nanofibrils, flexural properties increased significantly.

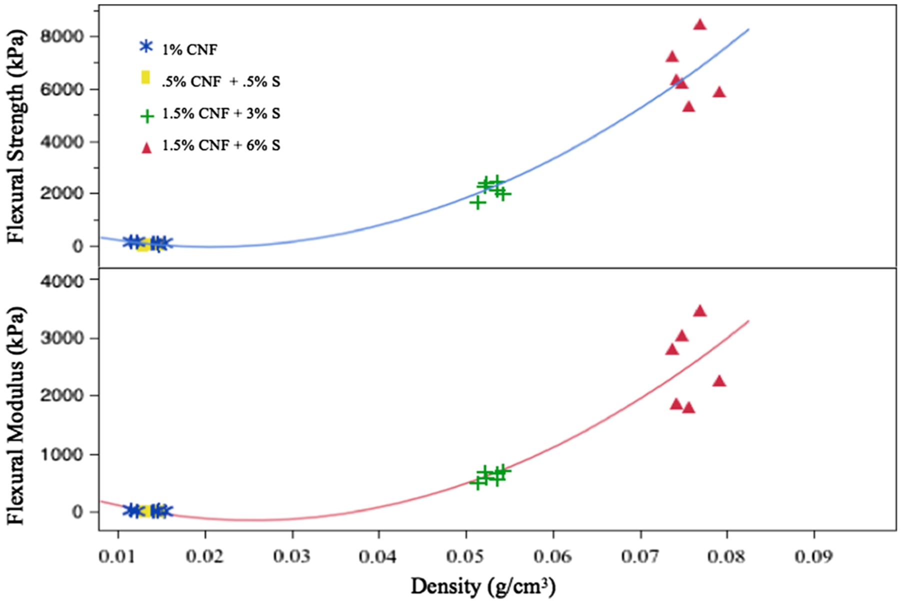

Fig. 5 The flexural modulus and flexural strength of foams plotted against density

Table 4 Compression properties of foams and comparisons with other studies

Sample	Density (g/cm^3)	Compression modulus (kPa)	Compressive resistance (kPa)	Fmax at 10 % (kg)
0.5 % CNF+ 0.5 %Starch	0.014 (0.0010) C	15.0 (2.0) C	144 (44.0) C	326.8 (98.80) D
1 % CNF	0.014 (0.0016) C	30.0 (7.0) C	359 (63.0) C	792.8 (108.90) C
1.5 % CNF+ 3 %starch	0.053 (0.0011) B	496 (82) B	2,753 (203) B	2,897.3 (213.6) B
1.5 % CNF+ 6 %Starch	0.076 (0.0020) A	907 (171) A	3,330 (249) A	7,195.7 (429.0) A
7.5 % starch	N/A	N/A	N/A	N/A
Carbon foam (Wang et al. 2006)	0.73	–	6,100	–
Gypsum board (Gypsum Association 2010)	–	–	2,750	–
CNF aerogel (Sehaqui and Berglund 2011)	0.014	34.9	3.20	–
CNF aerogel (Sehaqui and Berglund 2011)	0.029	199	24.4	–
CNF aerogel (Sehaqui and Berglund 2011)	0.050	1,030	69	–
CNF aerogel (Sehaqui et al. 2011)	0.105	2,800	238	–
CNF foam (Ali and Gibson 2013)	0.016	62	–	–
CNF foam (Ali and Gibson 2013)	0.027	249	–	–
CNF foam (Ali and Gibson 2013)	0.063	1,760	–	–

Parentheses indicate the standard deviation. A, B, C and D letters indicates the significant differences between the treatments

Shey et al. (2006) indicated that the commercial polystyrene structural foam (Styrofoam^{TM}) has a 1,610 kPa stress at yield. Gypsum board has 4,600 kPa MOR in the machine direction and 1,500 kPa MOR in cross direction (Gypsum Association 2010). The performance of the 1.5 % CNF/6% starch formulation exceeded both levels of performance.

The compression performance of the foams (Table 4) showed a trend consistent with that found

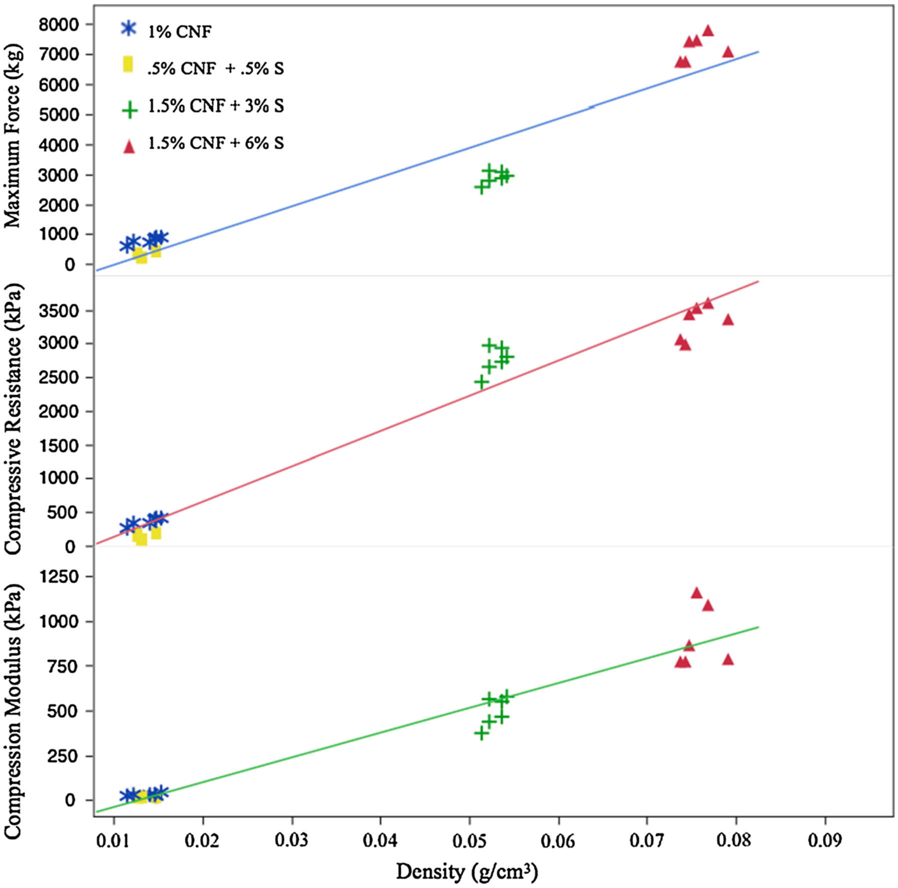

Fig. 6 Maximum force applied when the foams were compressed up to 10 % of original thickness, compression modulus and compression resistance plotted against density

for flexural behaviour. Density was positively correlated with compression modulus, compressive resistance and the maximum force when the specimens were compressed to 10 % of specimen thickness (Fig. 6).

The role of starch and CNF was directly evaluated at 1 % solids content (1 % CNF vs. 0.5 % CNF + 0.5 % Starch). The foam made with half starch resulted in significant decreases in compression modulus (50 %), compressive resistance (60 %) and maximum force (59 %). For a constant solids content (1.5 %) of CNF, increasing the starch content in the structure from 3 % to 6 % increase the compression modulus (83 %), compressive resistance (21 %), and maximum force (148 %).

The comparison of foam compression properties (Table 4) showed that the compression results for the foams produced in this study appear promising for applications such as insulation and packaging. Shey et al. (2006) reported that mechanical properties of CNF aerogel were positively correlated with density. The reinforced starch foams, which were produced in this study, have similar, comparable compression properties with pure CNF foams, aerogels and some other insulation foams. Ali and Gibson Ali and Gibson (2013) found that, the 2 wt% CNF foams have 1,760 kPa compression modulus with 0.063 g/cm^3 density, whereas foams produced in this study (1.5 % CNF + 6 % S) have 907 kPa compression modulus with 0.076 g/cm^3 density. Including 6 % starch, and less CNF content in the foam structure produced lower mechanical properties.

Thermal conductivity and resistivity results (Table 5) indicated no statistically significant differences among the foam combinations, which have 4.5 % or higher solid content in its structure. The pure CNF with 1 % solid content showed higher thermal

Table 5 Thermal conductivity and thermal resistivity properties of foams

Sample	Density (g/cm^3)	Thermal Conductivity (W/m-K)	R value (°F.h.ft^2/BTU)
0.5 % CNF+ 0.5 %starch	0.013 (0.0007) D	0.048 (0.003) B	3.03 (0.34) B
1 % CNF	0.014 (0.0012) D	0.054 (0.004) C	2.87 (0.37) B
1.5 % CNF+ 3 %starch	0.053 (0.0011) C	0.047 (0.002) AB	3.60 (0.16) A
1.5 % CNF+ 6 %starch	0.082 (0.0049) B	0.042 (0.001) A	4.02 (0.06) A
7.5 % starch	0.098 (0.0036) A	0.041 (0.002) A	4.14 (0.18) A
Gypsum Board (Gypsum Association 2010)	–	–	0.83
Silica Aerogels(Hong et al. 2013)	–	0.041–0.098	–
Fiberglass-urethane(Mahlia et al. 2007)	–	0.021	–
Fiberglass-rigid (Mahlia et al. 2007)	–	0.330	–
Urethane-rigid (Mahlia et al. 2007)	–	0.024	–
Perlite (Mahlia et al. 2007)	–	0.054	–
Extruded polystyrene (Mahlia et al. 2007)	–	0.029	–
Urethane (roof-deck) (Mahlia et al. 2007)	–	0.024	–
Granular starch (Hsu and Heldman 2004)	–	0.490	–
Gelayinized starch (Hsu and Heldman 2004)	–	0.470	–
Freeze-dried corn starch (Glenn and Irving 1995a)	–	0.040	–

Parentheses indicate the standard deviation. A, B and C letters indicates the significant differences between the treatments

Table 6 TGA and DTGA results for foams

Sample	T, weight loss 10 %, °C	T, weight loss 50 %, °C	DTGA temp. °C	Mass loss (%)	Residue (%)
0.5 % CNF+ 0.5 %starch	276 (1.08) AB	320 (2.35) B	303 D	33.2 (1.74) D	14.4 (2.73) A
1 % CNF	277 (6.16) A	335 (0.41) A	339 A	55.3 (0.69) A	15.3 (1.32) A
1.5 % CNF + 3 %starch	260 (3.68) ABC	308 (1.17) C	304 C	45.5 (0.71) B	8.70 (1.56) C
1.5 % CNF+ 6 %starch	255 (12.0) C	318 (1.09) B	310 B	40.9 (1.40) C	11.6 (1.53) B
7.5 % starch	259 (1.91) BC	300 (0.35) D	295 E	43.3 (0.58) BC	14.9 (0.49) A

Parentheses indicate the standard deviation. A, B and C letters indicates the significant differences between the treatments

conductivity when compared the other combinations where the lower thermal conductivity means better insulation.

The comparison of thermal insulation properties (Table 5) showed that the thermal conductivity and thermal resistivity results for the foams produced in this study appear promising like the compression properties. Foams produced in this study have 3–4 times better thermal resistivity properties when compared to gypsum board that has 0.83 °F.h.ft^2/BTU R-value (thermal resistivity) for 25.4 mm thickness (Gypsum Association 2010) and the foams have similar thermal conductivity with nanoporous silica aerogel impregnated highly porous zirconia ceramics have thermal conductivity from 0.041 W/m-K to 0.098 W/m-K (Hong et al. 2013). Mahlia et al. showed that, thermal conductivities of some insulation materials as follows: fiberglass-urethane 0.021 W/m-K, fiberglass-rigid 0.33 W/m-K, urethane-rigid 0.024 W/m-K, perlite 0.054 W/m-K, extruded polystyrene 0.029 W/m–K and the urethane (roof deck) 0.021 W/m–K (Mahlia et al. 2007). Hsu and Heldman showed that, granular starch has 0.49 W/m–K thermal conductivity and gelatinized starch have 0.47 W/m-K thermal conductivity (Hsu and Heldman 2004), which are so conductive when compared the CNF reinforced starch foams produced in this study. Glenn and Irving showed that, corn freeze-dried starch has a 0.040 W/

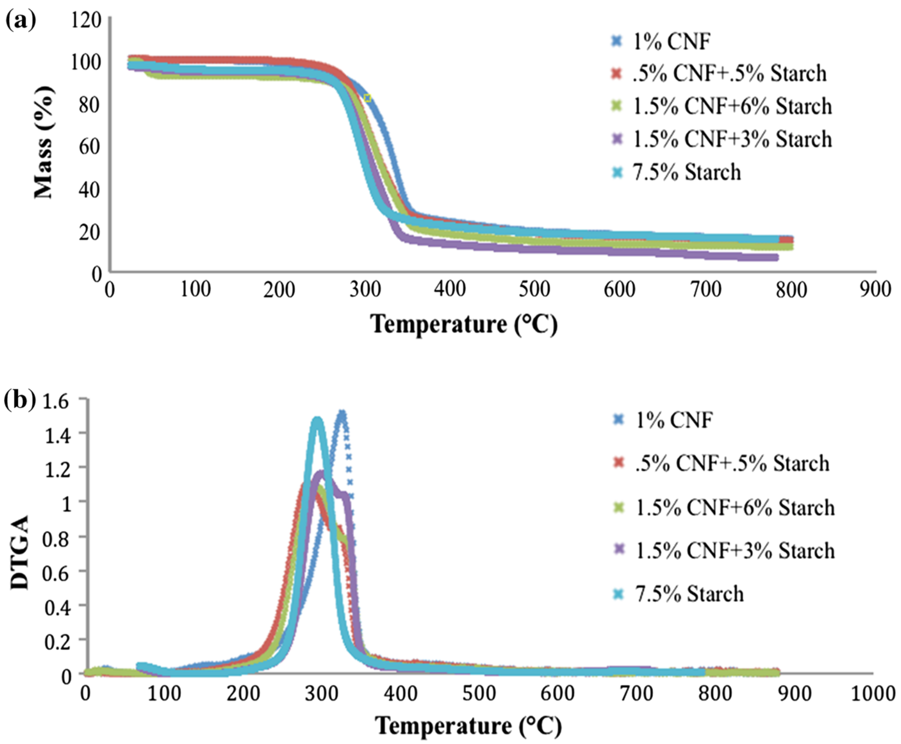

Fig. 7 TGA and DTGA curves of foams, (**a**) TGA curves, (**b**) DTGA curves

m-K thermal conductivity (Glenn and Irving 1995a), which is almost same when compared to our study due to similar production method and cellular structure of materials.

As a result of thermal analyses (Table 6), it was found 1 % CNF foams have a higher thermal degradation point of (onset temperature) 277 °C when compared to the other combinations tested. (Ptersson et al. 2007) indicated that the onset temperature of cellulose nanoparticles is between 200 and 300 (°C). The addition of starch consistently decreased onset temperature with reduction from 277 to 260 °C and 277 to 255 °C as initial starch concentration increased to 3 and 6 %. Switching the 0.5 % CNF to 0.5 % starch or adding more starch to the foam, decreased the onset temperature.

The DTGA (derivative TGA) temperature (decomposition temperature) of cellulose was found between 315 and 400 °C by (Yang et al. 2007) and it was indicated in another study that cellulose has a sharp weight loss starting at 305 °C (Moran et al. 2008). In this study it was determined to be 338.6 °C for 1 % CNF and when the starch was added to the structure, two peaks were evident in the DTGA curves, one for starch and one for CNF (Fig. 7b). DTGA temperatures were determined statistically different for CNF + starch combinations, whereas they showed similar results that changes between 303 and 310 °C (Table 6).

Conclusions

Biodegradable and renewable foams produced in this study have highly porous microcellular structure includes foam walls of CNF material embedded within a starch matrix. Due to starch‘s low mechanical properties cellulose nanofibrils were used as a reinforce material which provided superior performance to starch foams. Increasing the CNF amount and increasing the density produced higher mechanical properties. The optimum properties were obtained from 6 % Starch + 1.5 % CNF combinations, which has promising mechanical and thermal properties when compared with previous studies. This study was an exploratory and that it has found promising results indicating excellent potential to use the CNF reinforced starch foams as a potential structural insulation material. Future work will include investigating the effect of CNF reinforcement for higher total loadings, creating micromechanic model for different volume fractions and quantifying the hygroscopicity of the material and the effect of that on performance.

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