Development of laboratory studies on the off-gassing of wood pellets

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Tumuluru, J.S., X. Kuang, S. Sokhansanj, C.J. Lim, T. Bi, and S. Melin. 2010. **Development of laboratory studies on the off-gassing of wood pellets**. Canadian Biosystems Engineering/Le génie des biosystèmes au Canada. **52**: 8.1–8.9. In the present study three sealed containers (304.8 mm inside diameter and 609.6 mm height) were developed to investigate the concentration of off-gases accumulated in the headspace as well as changes in some of the physical properties of wood pellets during storage. Pellets occupied 75% of the container volume leaving 25% headspace. The outside wall of the steel containers was wrapped with electric heating tapes and fiber glass insulation. The storage studies were carried out at room temperature of about 22°C and at elevated temperatures of 30, 40 and 50°C. The off-gases were collected and analyzed using micro gas chromatography. The accumulations of CO (5000 ppmv) and CO_2 (10000 ppmv) were relatively high at room temperature of about 22°C for a storage period of 24 days. These accumulations increased sharply at storage temperatures >30°C. At 50°C, the maximum measured concentration of CO, CO_2 and CH_4 was about 17,000, 70,000 and 3,000 ppmv, respectively. Storage temperature had a significant effect ($P<0.01$) on all of the pellet properties, including pellet durability, which dropped by about 20% at the end of 60 days of storage. **Keywords:** Wood pellets, off-gas, gas chromatography, moisture content, durability, pellet density, bulk density, storage temperature.

Trois contenants hermétiques (304,8 mm de diamètre intérieur et 609,6 mm de hauteur) ont été développés aux fins de la présente étude dans le but mesurer la concentration ainsi que les propriétés physiques des gaz émis par des granules de bois dans l'espace libre à l'intérieur des contenants durant l'entreposage. Les granules occupaient 75% du volume du contenant laissant un espace libre de 25%. Les parois extérieures des contenants en acier inoxydable étaient recouvertes avec du ruban électrique chauffant et de l'isolation en fibre de verre. Les études d'entreposage ont été réalisées à une température ambiante d'environ 22°C de même qu'à des températures plus élevées de 30, 40 et 50°C. Les gaz émis ont été recueillis et analysés à l'aide d'un micro chromatographe analyseur de gaz. Les accumulations de CO (5 000 ppmv) et de CO_2 (10 000 ppmv) étaient relativement élevées dans le cas de granules entreposées à la température ambiante pour une période de 24 jours. Ces accumulations augmentaient de manière importante à des températures d'entreposage supérieures à 30°C. À 50°C, les concentrations maximales mesurées de CO, CO_2 et CH_4 étaient d'environ 17 000, 70 000 et 3 000 ppmv respectivement. La température d'entreposage avait un effet significatif ($P<0,01$) sur toutes les propriétés des granules incluant la durabilité des granules qui diminuait d'environ 20% après une période d'entreposage de 60 jours. **Motsclés:** granules de bois, gaz émis, chromatographie en phase gazeuse, teneur en eau, durabilité, densité des granules, température d'entreposage.

INTRODUCTION

The use of wood pellets as solid biofuel is increasing as the cost of fossil fuels rises and as environmental concerns regarding greenhouse gas emissions are growing. The raw materials used in wood pellets vary depending upon the logging residues and source of sawdust and shavings from saw mills. In Europe, Scots pine and Norway spruce (Lehtikangas 2000) and in North America both softwood and hardwood species are ground to make pellets (Zaini et al. 2009).

Currently, in Canada, most wood pellets are made from relatively dry planer shavings or sawdust. As part of the pelletization process, sawdust is dried at temperatures ranging from 100 to 400°C to a moisture content of less than 10%, and hammer-milled to fractions less than 2 mm. During the drying process, monoterpenes and other volatile compounds are released (McGraw et al. 1999; Banerjee 2001). The dried sawdust is pressed through holes in a die to form pellets. The high pressure in the die develops a temperature sufficient to soften the wood lignin to make pellets self-binding (Mani and Sokhansanj 2008). Therefore, no binders are used in Canadian wood pellets. The wood pellets are 6.4 mm in diameter with a length varying from 6 to 18 mm. The warm pellets exiting from the pellet press are cooled to room temperature and stored in steel bins or warehouses until shipping.

Levitt et al. (1995) observed that organic matters, when stored at room temperature, particularly in the presence of air and light, emit small amounts of CO, and the emission rate increases at elevated temperature. During storage, the extractives in the wood pellets are reduced and the

composition is modified by microbiological and auto-oxidative chemical reactions (Hemingway et al. 1971; Martinez-Inigo et al. 1999). Unsaturated fatty acids under certain conditions of storage are oxidized to volatile aldehydes such as hexanal and pentanal compounds, which cause odors. Svedberg et al. (2004) reported that CO and volatile organic compounds (VOCs) are emitted from wood pellets during storage. More recently Svedbeg et al. (2008) measured elevated concentration of CO, CO_2, and depletion of O_2 in ship holds filled with wood pellets en-route to Sweden. They recorded O_2 depletion from the headspace down to 4% and a concentration of CO and CO_2 significantly higher than the threshold levels. Kuang et al. (2008, 2009a, 2009b) have characterized the concentration of CO_2, CO and CH_4 from wood pellets in sealed containers that would be described in this paper. They developed kinetic models for predicting emission rate factors (grams of gas per killogram of pellets) at different storage temperatures. They also investigated the effects the volume of headspace and humidity levels have on accumulation of CO_2, CO, CH_4, and depletion of O_2.

Table 1 lists the safe level of CO concentration at 35 ppm for an average of 8-h exposure (U.S. Department of Labor Occupational Safety and Health Administration). Exposure to CO concentration above the threshold level can lead to dizziness and headaches, and concentrations greater than 800 ppm can result in fatal accidents. In addition to asphyxiation by displacing oxygen, accumulation of high concentrations of CH_4 in the storage bins could lead to explosions and fires. Springer and Hajny (1970), Kuber et al. (1985) and Kuber (1982) found that auto oxidation of unsaturated fatty acids and other extractives leads to spontaneous heating of wood chips and sawdust. Reuss and Pratt (2001) observed CO of about 300–400 ppm in the air above 7000 tons of rapeseed stored in a sealed warehouse and the calculated specific emission rate was estimated to be 200 mg/ton/day. Whittle et al. (1994) also found CO emissions in a wheat grain warehouse with a calculated specific emission rate of 9 mg/ton/day.

Knowledge of the properties of wood pellets, such as moisture content, durability, pellet density, bulk density and percent fines during storage are important in order to design storage silos and ensure safe handling of the product. Bulk density affects transport efficiency and storage capacity. Durability is a measure of the mechanical strength of wood pellets. Durable pellets are more resistance to breakage. Estimation of fines in the wood pellets, which accumulate during storage and transportation, is important in understanding gas emissions and dust explosions that frequently happen in large commercial wood pellet operations (Lehtikangas 2001).

There is a lack of data on the effect of storage temperature on moisture content, durability and density of wood pellets and the accumulation of off-gases during extended periods of storage. The objectives of this research were (1) to develop an experimental sealed container to store and maintain wood pellets at a constant temperature, (2) to test the container for measuring the concentration of major gases CO, CO_2, and CH_4 during storage, and (3) to quantify changes in moisture content, durability, and density of stored wood pellets.

MATERIALS and METHODS

Development of storage containers

Three medium sized cylindrical containers, denoted C_1, C_2 and C_3 with inner dimensions of 304.8 mm diameter and

Table 1. Threshold levels of CO, CO_2, and CH_4.

Gas	Threshold levels	Effects	Source
CO	35 ppm for 8 h	Maximum allowable exposure limit by OSHA in the workplace over an 8-h period.	US Department of Labor Occupational Safety and Health Administration
	800 ppm for 45 min	Dizziness, nausea and convulsions. Unconscious within 2 h. Death within 2–3 h	
	3200 ppm for 5–10 min	Headache, dizziness and nausea. Death within 1 h	
	6400 ppm for 1–2 min	Headache, dizziness and nausea. Death within 25–30 min	
	12,800 for ppm 1–3 min	Death	
CO_2	5,000 ppm as an 8-h time weighted average	At very high levels, 30,000 ppm (short-term exposure level) and above, CO_2 can cause asphyxiation as it replaces oxygen in the blood, and results in loss of judgment, dizziness, drowsiness, and rapid breathing	American Conference of Governmental Industrial Hygienists (ACGIH)
CH_4	500,000 ppm- for 8 h	Could asphyxiate by displacing oxygen at this concentration. CH_4 is one of the main constituants of natural gas, which can result in explosions.	Ministry of Agriculture Food and Rural Affairs, Ontario, Canada

609.6 mm in height were made of mild steel (Fig. 1). The size of a container was selected to ensure accumulation of adequate measurable gases (Shankar et al. 2007). The containers were coated inside with PTFE (Crown 6075 Dry Film Lubricant, 6075 G, Aervoe Industries Inc.), which is an inert epoxy and can withstand temperatures above 100°C. This coating prevents rusting; it prevents the metal wall from interaction with the wood pellets. Each container was wrapped with silicone rubber extruded heating tapes (SRT-101-200L SE, Omega, Canada). The heating tapes could reach up to 250°C. Containers were further insulated with 50 mm thick glass wool with external surface covered with aluminum foil to minimize heat losses. K-type surface mounted thermocouples (SA1XL-K-SRTC, Omega, Canada) were used for the measurement of the container wall temperature. The set temperature (T_2 shown in Fig. 1) on each container was maintained using the temperature controller (K-02110-64, Cole Parmer, Canada) to set and maintain the containers (C_1, C_2 and C_3) wall temperature at desired levels. Each container was initially kept at room temperature and was further heated to 30, 40 and 50°C on days 9, 11 and 26 of storage. Once at the target, the container temperature remained unchanged during the experiment.

Two more thermocouples were further pushed inside the bulk of the wood pellets to record the temperatures at two different positions within the bulk wood pellets in each container (T_0 and T_1 shown in Fig. 1). Ports with 6.25-mm fittings were provided on the side of a container for gas sampling. Each container was equipped with a pressure transducer (PX143-01BD5V, ± 1 Pa, Omega, Canada) to record gas pressures developed during storage. The sensed pressure and temperature were logged into a personal computer running on the LABTECH software using a data acquisition board (PCI-DAS08, Techmatron Instruments Inc, Canada).

Materials

The wood pellets were procured from Fibreco Export Inc., North Vancouver, BC, Canada. This material-handling facility receives annually more than 600,000 metric tons (t) of wood pellets produced in interior British Columbia. The wood pellets arrive in rail cars, transferred temporarily to large steel bins where the pellets are kept for up to a month before being loaded onto ocean vessels for transport to Europe. The wood pellets used in our experiments were those from rail cars arriving at the facility. The elapsed time from the date of their production site and the day of sampling in Vancouver was about 30 days. Once in the lab, wood pellets were subjected to the following measurement: moisture content, pellet density, bulk density, tapped density, durability and percent fines.

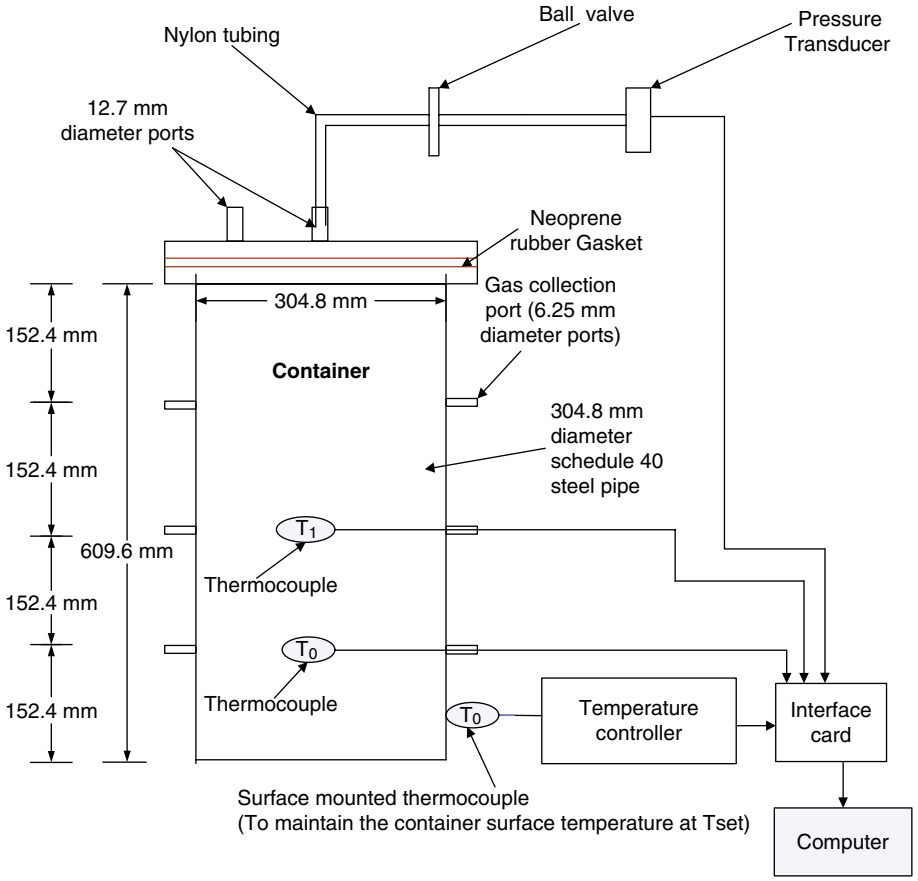

Fig. 1. Experimental setup for off-gassing studies.

Moisture content

The moisture content of the pellets was measured by the oven method (AOAC 1990) at 105°C for 24 h and was expressed in wet basis. Measurements were repeated three times.

Pellet density

The two ends of a pellet were smoothed using sand paper. The volume of individual pellets was calculated based on the measurements of length and diameter using an electronic vernier. The mass of each pellet was measured using a balance to 0.01 g precision. Pellet density was calculated by dividing the weight of a pellet by its volume. The reported values are an average of measurements on 20 pellets (Shankar et al. 2007, 2008).

Bulk density

A cylindrical container measuring 152 mm inside diameter and 122 mm height was used to determine the bulk density of wood pellets. The samples were poured slowly into the bucket from a 500 mm high from the bottom of the container until the container was overflowing. The excess material was removed by striking with a wooden bar. The mass of the material with the bucket was recorded to the second decimal place. For dense fill density, the loosely filled container was tapped on the laboratory bench 5 times. Filling and tapping was repeated until the container was overflowing. The filled container was weighed to 0.01 g precision. Bulk density was calculated by dividing the net mass of the material over the filled volume of the container (Lam et al. 2007; Shankar et al. 2007). Measurements were repeated on 5 different batches of pellets.

Durability

The durability of wood pellets was measured using a DURAL tester developed at the University of Saskatchewan for testing alfalfa cubes and pellets (Sokhansanj and Crerar 1999). Initially, pellets were screened using 3.35 mm screen to remove fines. Next, 100 g of pellets was weighed and put into the DURAL tester. The rotation speed was set to 1600 rpm for 30 s. The treated samples were passed through the 3.35 mm screen. The mass of the pellet retained on the screen after testing over the initial sample was recorded as durability. These tests were repeated 5 times each time using a new batch of pellets.

Percent fines

The fines were defined as the mass fraction of the pellet passed through the 3.35 mm sieve and collected in the pan. The duration of sieving was 5 minutes. The recorded values are an average of measurements on five batches.

Off-gas measurements

Pellets received from Fibreco were sieved using a 3.35 mm sieve. About 24 kg of wood pellets filled a container leaving 25% of volume as headspace. The containers were capped and sealed using a silicone sealant to prevent gas leakage. A GC-14A (Shimadzu, Kyoto, Japan) was used to identify and quantify CO, CO_2 and CH_4 concentrations. The GC was specifically equipped with a molecular sieve column to trap and separate CO and CH_4 at low concentrations of 30 ppmv for CO. The standard gas composition used was for CO: 1000 ppmv, for CO_2: 5000 ppmv and for CH4: 1000 ppmv. An algorithm was developed to convert the readings from GC to the calibrated volumetric gas concentration in parts per million on a volume basis (ppmv). A known quantity of 1 mL gas mixture was collected using an airtight syringe (A-85137-20 mL SGE Gas-Tight Syringe, Luer-Lock, Mandel Scientific, Canada). The syringe had a Luer lock facility to collect a known quantity of gas samples up to 20 mL drew gas through the gas port (Fig. 1).

Initially the three containers designated C_1, C_2 and C_3 were filled with pellets and the off-gas concentrations were measured at room temperature of about 22°C for 9, 11 and 26 days, respectively. The same containers were then heated to 30, 40 and 50°C and maintained at these temperatures up to 60 days. The sampling of the off-gases was initially carried out every day to the point there was not much daily change in concentrations. Frequency of sampling decreased to once every four or five days until the end of the 60 days of storage.

RESULTS and DISCUSSION

The two thermocouples (T_0 and T_1) placed in each container (Fig. 1) recorded similar values for temperature reading during the entire storage period. The size of the containers selected in the present study was found to maintain the temperature of the bulk wood pellets uniformly during the entire storage period. Figure 2 shows the average temperatures of the bulk wood pellets (T_{avg}, which is the average of T_0 and T_1) inside the three containers (C_1, C_2 and C_3), initially at room temperature and when heated to 30 and 40, and 50°C on 9, 11 and 26 days of storage. From the time of heating of each container it took about 1 day for the bulk pellets in each container to reach the set temperature of 30, 40 and 50°C. There was very little change in container pressure at room temperature. The maximum pressure of 6 kPa was registered at 50°C and the pressures for the other two temperatures of 30 and 40°C were <6 kPa.

Pellet properties

Table 2 lists the initial and final pellet properties at different temperatures for a storage period of 60 days. There was a slight drop in the initially low moisture pellets at 4.7 to 4.3% when heated to 50°C. The density of individual pellets decreased from 1168 (SD$=\pm 34$) kg/m^3 to 1119 (SD$=\pm 33$) kg/m^3. Loose and tapped bulk density decreased from 804 to 79841 kg/m^3 and from 880 to 794 kg/m^3, respectively. The pellet durability measured on the DURAL decreased from 58.1 to 39.2%. During storage at 50°C, the durability of wood pellets decreased by about 20% and the corresponding percent fines increased to 1% (SD$=0.1$%). The weight of wood pellets, 24 kg, used in each container for off-gassing studies remained the same at the end of 60 days of storage. A slight decrease in moisture content might have reduced the binding characteristics of the wood pellets and in turn increased the

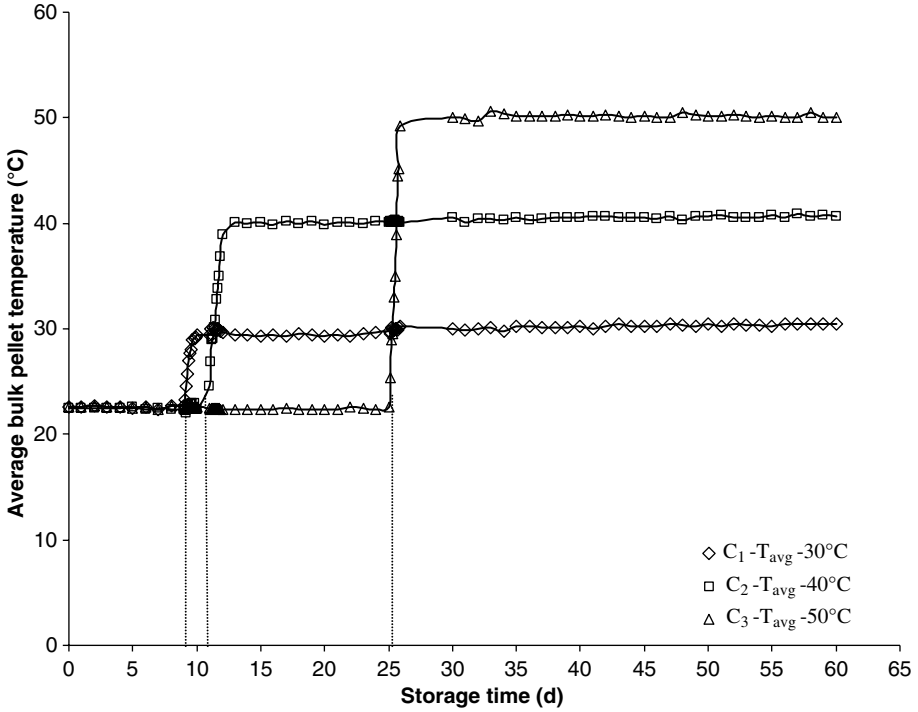

Fig. 2. Temperature profile of the bulk wood pellets initially at room temperature and when containers (C_1, C_2 and C_3) were heated to 30, 40 and 50°C on days 9, 11 and 26 of storage. Note: (1) T_{avg} is the average reading of the two thermocouples placed in each container (T_0 and T_1 shown in Fig. 1). (2) Maximum and minimum standard deviation values at 30, 40 and 50°C are between 0.3 and 0.9°C.

percent fines by 1% and decreased the durability by about 20%. The decrease in moisture content might have increased the air-filled cavities in the wood pellets, which lowered the bulk and tapped density (Lehtikangas 2000).

Gas concentrations

The off-gassing experiment was initially carried out at room temperature (22°C) using three containers C_1, C_2, C_3 for different storage periods of 9, 11 and 26 days, respectively. For the first 9 days, when the storage conditions were identical, the accumulation of three gases, CO, CO_2, and CH_4, in the three containers were identical as well. Figures 3 and 4, and 5 show that the slope of the concentration vs. storage time was largest for CO, followed by CO_2 and CH_4. The CO and CO_2 concentrations were about 2000 ppmv and 5000 ppmv, respectively, and the concentration of CH_4 was less than 150 ppmv after 9 days of storage.

Figures 3 and 4, 5 show the concentration of CO, CO_2, and CH_4 following a step change in temperature when the container's outer wall was heated to 30, 40 and 50°C. The accumulation of CO, CO_2, and CH_4 increased sharply following an increase in the container temperature. The maximum concentrations of CO_2, CO and CH_4 observed at 50°C were about 70,000, 17,000 and 3,000 ppmv, respectively, at the end of the storage time. A greater temperature increase caused a faster rate of increase in gas emissions from pellets.

The concentration of CO, CO_2, and CH_4 at different storage temperatures can be due to auto-oxidation of fatty acids present in the wood. According to Hoell and Piezconka (1978) and Piispanen and Saranpaa (2002) dry

Table 2. Pellet properties at the beginning and end of the storage period of 60 days.

Pellet property categories	Initial pellet property values	30°C	40°C	50°C	Number of measurements
Moisture content (% ± SD)	4.7 ± 0.1	4.6 ± 0.0	4.4 ± 0.0	4.3 ± 0.1	3
Durability (% ± SD)	58.1 ± 0.3	50.9 ± 1.9	45.4 ± 1.0	39.2 ± 2.3	5
Bulk density (kg/m^3 ± SD)	804 ± 6	772 ± 3	771 ± 8	741 ± 4	5
Tapped density (kg/m^3 ± SD)	880 ± 5	823 ± 3	833 ± 9	794 ± 5	5
Pellet density (kg/m^3 ± SD)	1168 ± 34	1161 ± 31	1136 ± 36	1119 ± 33	20
Percent fines (% ± SD)	0	0.3 ± 0.1	0.4 ± 0.1	1.0 ± 0.1	5

Note: Storage temperature was significant at $P < 0.01$ for all the pellet properties. SD = standard deviation.

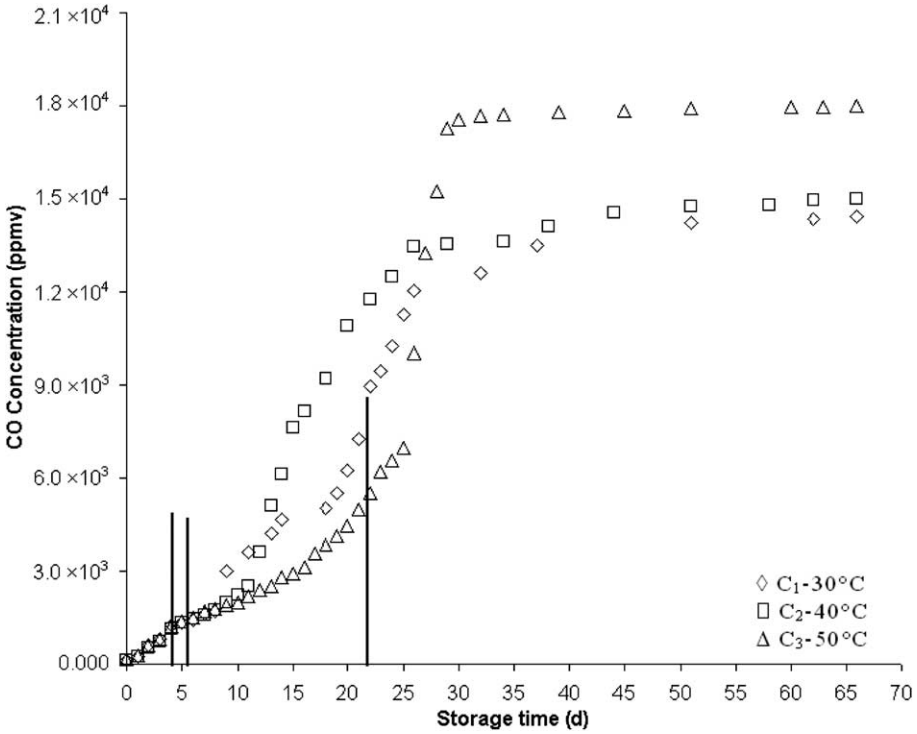

Fig. 3. CO concentrations at room temperature followed by elevated temperatures. Note: Containers C_1, C_2 and C_3 were heated on days 9, 12 and 26 of storage. Vertical lines indicate the onset heating of C_1, C_2 and C_3 to designated temperatures of 30, 40, and 50°C respectively.

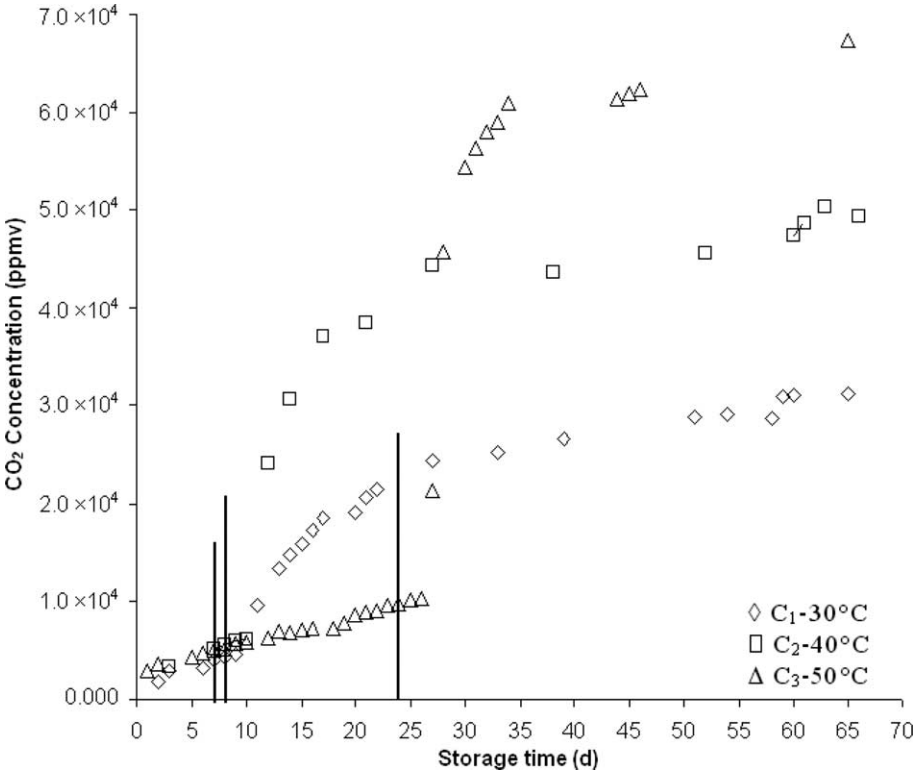

Fig. 4. CO_2 concentrations at room temperature followed by elevated temperatures. Note: Containers C_1, C_2 and C_3 were heated on days 9, 12 and 26 of storage. Vertical lines indicate the onset heating C_1, C_2 and C_3 to designated temperatures of 30, 40, and 50°C respectively.

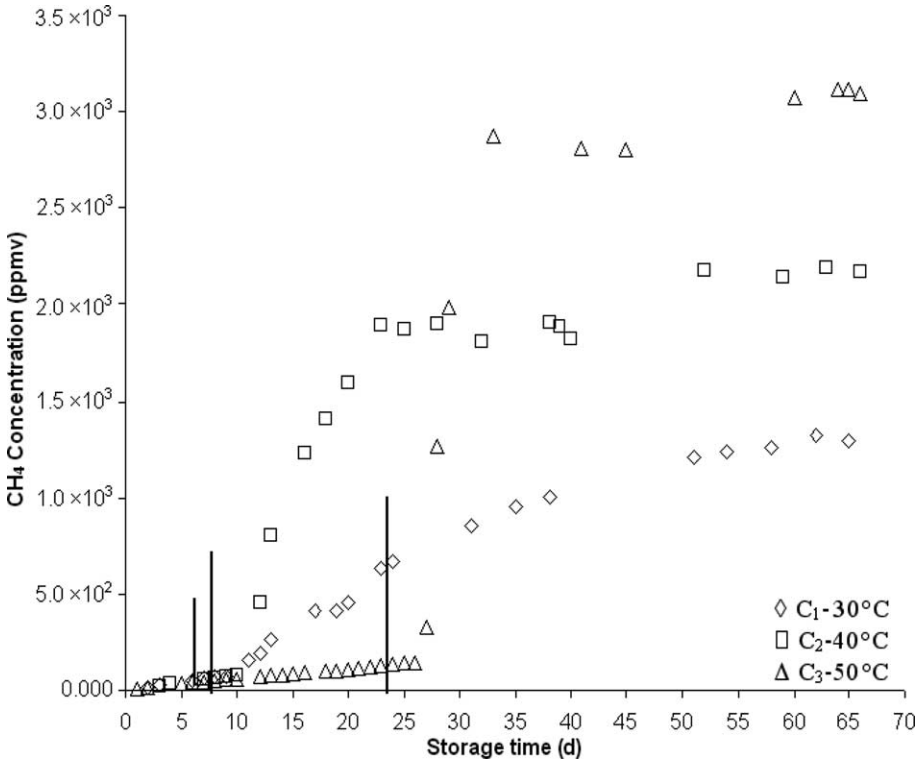

Fig. 5. CH_4 concentrations at room temperature followed by elevated temperatures. Note: Containers C_1, C_2 and C_3 were heated on days 9, 12 and 26 of storage. Vertical lines indicate the onset heating C_1, C_2 and C_3 to designated temperatures of 30, 40, and 50°C respectively.

woods of Scots pine and Norway spruce contain 3–5% triglycerides and free fatty acids. Polyunsaturated acid (linoleic acid) is the major constituent in the mixture of free fatty acids and triglycerides. Svedberg et al. (2004) also attributed emissions from stored wood pellets to auto-oxidation of fatty acids. Shankar et al. (2010) found that the unsaturated fatty acids in extruded fish based products were converted to free fatty acids during the prolonged storage period. They reasoned that the formation of peroxides and free fatty acids during storage was due to oxidation.

Temperature data taken from the pellet-filled holds of an ocean vessel transporting pellets from the Port of Vancouver, Canada, to the Port of Helsingborg, Sweden, indicated that temperatures reached 35°C (Melin 2007). Considerable accumulations of gases occurred during these shipments, indicating potentially toxic conditions for the ship's crew and for those handling the pellets at the port of destination. In addition to gas accumulation, there was also noticeable moisture condensation beneath the hatch and the top cover in the hold. This moisture accumulation resulted in mold development on the top layer of the pellets. These mild temperatures measured during the 40-day journey in January 2007 could be higher when the journey occurs during the warmer months (Sokhansanj et al. 2003).

The research reported in this paper was conducted to describe the test set up specially the suitability of the containers, the heat tapes, insulation, placement of the thermocouples and temperature controller on the outer wall of the container, recording the bulk pellets temperature, development of calibration for GC to record the off-gas concentrations. We observed that the equipment design, the temperature controller and gas sampling procedure worked reasonably well. The physical properties of pellets were measured to assess the properties of pellet stored in heated sealed containers. We noted statistically significant reductions in durability and density of pellets as a result of prolonged storage. However, a larger pellet sample of different origin and quality is needed in order to develop a rigorous functional relation between pellet properties and storage conditions. Increasing the frequency of quality testing during the storage experiment would improve the robustness of the data.

CONCLUSIONS

1. The sealed container developed, its heating control and the instrumentation for measuring gas concentrations appeared to be working well and are suitable for future studies on wood pellet storage.
2. The durability and bulk density of the wood pellets decreased by about 20% and 60 kg/m^3 when the storage temperature was increased from 22 to 50°C.
3. The emission of CO and CO_2 from the wood pellets stored at room temperature (22°C) for a period of 9 days reached levels of 2000 and 5000 ppm, respectively, levels toxic to humans.

4. The maximum concentrations of CO_2, CO and CH_4 observed at 50°C were about 70,000, 17,000 and 3,000 ppmv.
5. The rate of increase in off-gas accumulation increased sharply with an increase in storage temperature (>30°C) and was highest for CO, followed by CO_2 and CH_4.

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