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ENGINEERING AND GINNING

Physical Processing and Emission Characteristics of Firelogs from Cotton Ginning Byproducts

Dilpreet S. Bajwa*, Sreekala G. Bajwa, Tom C. Wedegaertner, and Greg A. Holt

ABSTRACT

Cotton burr with stems (CBS), a byproduct from cotton ginning operations was evaluated for its suitability in firelogs. Firelog burn quality was examined by varying the CBS to paraffin wax ratio, and firelog processing conditions. The firelogs were burned in a wood stove to quantify the burn quality, burn time, product durability, and gaseous emissions. Firelog composition had no effect on the burn temperatures. Carbon monoxide (CO), ammonium (NH_4), hydrogen (H_2), and nitrous oxide (NO) gases were not detected in the gaseous emissions. Air emission samples from combustion showed the presence of n-alkanes, n-alkenes, oxygenated volatile organic compounds, polycyclic aromatic hydrocarbons (PAH), halogenated alkane, cyclic terpenes, benzene derivatives and naphthalene. Overall this study showed the potential of CBS as an alternative material for manufacturing firelogs.

Air emissions from residential wood combustion have been identified as impacting indoor air quality. In most homes air emissions are primarily from two appliances: wood stoves and fireplaces. Over the years manufactured metal fireplaces have become very popular and economical. About 50% of all single-family homes have a fireplace and unlike wood stoves, fireplaces are not regulated or certified for emissions by the United States Environmental Protection Agency (EPA) (Houck et al., 2000; Li and Rosenthal, 2006). Metal fireplaces may represent a growing source of indoor air pollution.

The use of densified bioenergy products in wood stoves and manufactured metal fireplace burrs has increased significantly over the last few years due to environmental concerns and fluctuations in the price of petroleum derivatives (Roy and Corscadden, 2012). Most of the densified solid biofuels use a combination of high-energy solid fuel, biomass, and a binder. The ratio of solid fuel can vary from 2% to 80% based on the final products. Firelogs and fire starters contain 50% or more paraffin wax (De Hoop et al., 2005). It is estimated that over 100 million firelogs are burned in fireplaces by United States (U.S.) consumers annually (Houck et al., 2000). Firelogs were created in the 1960's to meet the need to recycle wood waste coming out of a California wood products plant (Duraflame Report, 2008). The firelogs are composed mainly of wax and biomass from wood chips or sawdust in equal proportions by weight basis. Wax is the primary binder and source of heat in a firelog. Petroleum based paraffin wax is the most common wax used in firelogs. In the U.S. 50% of fireplaces in the larger metropolitan areas have used a manufactured firelog in the last year (Gopu, 2010). Currently Duraflame® is the leader in the market with 41.3% market share followed by Pine Mountain® at 19.8% (Grocery Headquarters, n.d).

Customer demand for eco-friendly and sustainable products and federal initiatives for sustainable bioenergy has encouraged firelog manufacturers to look into sustainable alternative raw materials that can be used to make a 100 percent natural product (Environment, 2012). Some of the cellulosic materials that have been incorporated into firelogs include agricultural crop residue, corrugated board, and coffee grounds (Lovgren, 2004; Resource Recycling, 2010). Soybean wax and palm wax have been used as an analog to paraffin wax (De Hoop et al., 2005). However, the market for vegetable oil-based waxes has been limited due to strong demand for vegetable oil by the food industry. In the U.S. paraffin wax is generally preferred in firelogs due to its economics and availability. One major issue with paraffin wax-based firelogs is air emissions and impact on indoor air quality.

Gaseous emission from the combustion of any type of biomass fuel depends directly on the chemical composition of the fuel and combustion conditions. During combustion, the cellulosic materials

D.S. Bajwa*, Department of Mechanical Engineering, North Dakota State University, Fargo, ND 58108; S.G. Bajwa, Agricultural and Biosystems Engineering, North Dakota State University, Fargo, ND 58108; T.C. Wedegaertner, Cotton Inc., Cary, NC 27513; and G.A. Holt, USDA-ARS, Cotton Production & Processing Research Unit, Lubbock, TX 79403
*Corresponding author: Dilpreet.bajwa@ndsu.edu

start to hydrolyze, oxidize, dehydrate and pyrolyze as the temperature increases, forming combustible volatiles, tarry substances, and highly reactive carbonaceous char (Eriksson et al., 2014; Shafizadeh, 1984). The paraffin wax combustion shows an n-alkane emission pattern with carbon analogs ranging from C19 to maximum C38 that is comparable to that typically found in the crude oils (Khanna et al., 2014). The n-alkane emissions from paraffin wax logs can exceed more than 500 mg/kg burned (Wolfgang et al., 1998). Also found are n-alkenes from C19 to C40. Another petroleum related component found in logs is cyclohexyalkanes with carbon variance from C21 to C40. Additional compounds found in paraffin wax include n-alkanals (C9–C32) an oxidation product of n-alkenes, released? due to slow combustion and pyrolysis. The presence of polycyclic aromatic compounds (PAH) in the gaseous emissions of synthetic wax logs has been widely reported. The smoke from synthetic logs contained the highest total PAH as well as a diverse suite of PAHs (Woflfgang et al., 1998).

Several studies have listed the pollutants released during the burning of paraffin wax. A joint study published by Canadian and U.S. EPA showed high levels of particulate and chemical emissions such as PAH's (Eriksson et al., 2014; Houck et al., 2000). Additional research suggested that burning wood or synthetic logs are both indoor PAH exposure sources, but positive associations are only noted when burning synthetic logs, under long exposures (White et al., 2014). Indoor use of paraffin-based products also results in higher levels of sulphur dioxide (SO_2) and CO (Bailie et al., 1999). Mass emission rates of approximately 200 organic compounds associated with burning of paraffin wax-based fire logs and wood were reported (Wolfgang et al., 1998). Two recent studies on household candles comparing the emissions from paraffin and soy wax showed that paraffin wax emitted 50-60 fold higher concentrations of PM 2.5 and toxic chemicals like toluene, PAH etc., possibly responsible for causing allergic or asthmatic responses (Derudi et al., 2014; Massoudi and Hamidi, 2009). In a previous study, cotton gin byproducts have been evaluated as a source of pellet fuel, since the disposal of these byproducts costs the cotton gin approximately $ 1.65 (U.S.) per Mg (Holt et al., 2006).

Considering the typical cost of semi-dried wood fiber, between 0.5-0.15 cent per pound, cotton by-products can help farmers to turn a liability into potential revenue. The goal of this project was to evaluate the suitability of cotton ginning by-product, cotton burr and stems (CBS), as a raw material in manufactured firelogs. The specific project objectives were to (1) evaluate the effect of manufacturing conditions and product composition on product quality and performance, and (2) characterize the air emissions of CBS based fire logs during combustion.

MATERIALS AND METHODS

Materials. CBS fiber size 250 – 841 micron used in this study was supplied by United States Department of Agriculture - Agricultural Research Service (USDA-ARS), Cotton Production and Processing Research Unit, Lubbock Texas, US (Figure 1). The major fractions of the CBS consisted of clean lint (5-12%), hulls (16-48%), seeds (6-24%), motes (16-24%), and leaves (14-30%). The CBS composition is reported to include ash (7.9-14.6%), acid-insoluble material (18-26%), xylan (4-15%), and cellulose (20-38%) (Aglbevor et al., 2003). The oak wood fiber (250-841 micron) was supplied by Southern Wood Services, (Macon, GA, US). The chemical composition of oak wood fibers contained 44% cellulose, 24% hemicellulose, 24% lignin, 5.4% extractives and 1% ash. The bulk density of the CBS fibers was 249 Kg/m^3 and oak wood flour 348 Kg/m^3. The fully refined paraffin wax Parvan 1540 with a melting point of 67.2°C was used as binder and fuel in the firelogs (Exxon Mobil, Baytown, TX, US). It is derived from petroleum via a carefully controlled refining process and is primarily comprised of straight chain normal paraffin hydrocarbons, which impart excellent gloss and water repellent properties.

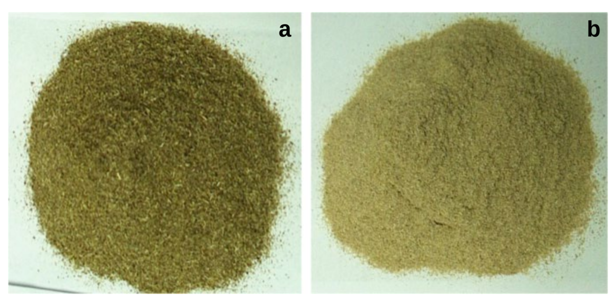

Figure 1. Biofibers used in firelogs. (a) Cotton burr with stems (b) Oak wood fiber

Firelog manufacturing. Firelog samples were manufactured with three fiber compositions, oak fiber 50% (control), CBS fibers 50%, and CBS at 60% loading, the remaining percentage being parfaffin wax. The firelog mixtures were subjected to three compression loads of 4, 6, and 8 tons. The manufacturing methodology was a three-step process. First, the natural fibers were dried in a convection oven to 1% or less moisture content on dry basis. In the second step the dried fibers were mixed with melted paraffin wax (73°C) at the required proportion in an automatic rotary mixer (Figure 2). The fiber-wax mix was mechanically tumbled for 15 minutes in a mixer to coat all the fibers with wax and obtain a free flowing mass, which was allowed to cool to ambient temperature. In the third step the fiber-wax mixture was compressed into firelogs using a cylindrical mold with 10 cm diameter in a 35 T hydraulic press. The resulting firelogs weighed approximately 1.4 kg similar to commercial firelogs. Ten samples were manufactured for each of the treatment combinations and conditioned under ambient temperature (23.5°C) for four weeks before burn testing.

Figure 2. Rotary mixer for blending paraffin wax with CBS and wood fibers

Firelog burn test. The conditioned fire logs were subjected to a burn test to evaluate their physical combustion characteristics in a Pleasant Hearth wood stove Model No. WS-2720, an EPA certified wood stove, with an 82% efficiency rating and emission rate of 4.4 grams per hour. It is a commonly used residential wood stove sold in the market. The firelog burn tests were conducted following wood stove manufacturer guidelines and simulation of customer use in their homes. The schematic diagram of the experimental setup is shown in figure 3. The highest combustion temperature of each sample was measured at three locations using Raytek infrared thermal gun model Rayngerm® ST^{TM}ST60 PRO PLUS (Santa Cruz, CA) a non-contact thermometer with an accuracy of 1%/1 °C. For each location three temperature readings were taken after 30, 60 and 90 minute intervals after combustion started (Figure 3). The distance between the thermal gun and firelog was approximately 900 mm. The smoke characteristics were recorded visually. The flame height was measured from a distance of 900 mm using a measuring tape. Smoke rating was based on the amount of smoke produced by firelogs during combustion. A light smoke means no smoke built up below the firelog grate and flame is clearly visible whereas moderate smoke refers to presence of smoke below the firelog grate and partial flame visibility. During the burn tests, the exhaust gas emission samples were collected at 30 min intervals through the vent hole located 45 cm above the stove using vacuum sealed 6.0 L Suma air sampling canisters in accordance with EPA SOP #1704. The vacuum pressure of Suma canisters was -4.20 psi/g. Three exhaust air samples were collected for each firelog at 15, 45 and 75 minute periods after combustion started. The air sampling canisters were carefully sealed, tagged, packed in tamper proof boxes supplied by the testing laboratory and sent overnight to the ALS Environmental Laboratory (Simi Valley, California USA) for gaseous emission characteristic quantification.

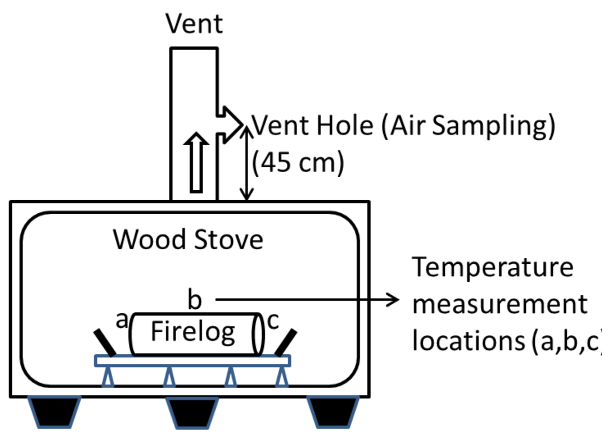

Figure 3. Schematic diagram of experimental setup

Gaseous emission analysis. The air sample canisters were opened by the testing laboratory for analysis within 24 h after sample collection. All the gaseous emission analyses were performed according to EPA Method 3C Modified by the ALS laboratory's NELAP and DoD-ELAP-approved quality assurance program (EPA and DoD-ELAP, 2014). The Summa canisters were cleaned, prior to sampling, down to the method reporting limits (MRL) of 0.18%, v/v, the minimum quantity of a target analyte that can be confidently determined.

Fixed gases and NO analysis. The firelog air emission samples were analyzed for fixed gases (hydrogen, oxygen/argon, nitrogen, carbon monoxide, methane and carbon dioxide) and nitrous oxide according to modified EPA Method 3C (single injection) using a Hewlett Packard Model 5890 gas chromatograph equipped with a thermal conductivity detector. The MRL limit for fixed gases was 0.18%, v/v. This procedure is described in laboratory SOP VOA-EPA3C (EPA, 2013)

Volatile Organic compound analysis (VOC). Gaseous emission samples were also analyzed for volatile organic compounds in accordance with EPA Method TO-15 from the Compendium of Methods for the Determination of Toxic Organic Compounds in Ambient Air, Second Edition (EPA/625/R-96/010b) (EPA, 1999). Three replicates of each firelog composition were tested for VOC's. The analytical system gas chromatography and mass spectroscopy (GC/MS), Agilent Model 6890N/5973 NMSD / Tekmar Autocan comprised of a gas chromatograph /mass spectrometer interfaced to a whole-air pre-concentrator. The method was modified to include the use of helium as a diluent gas in place of zero-grade air for canister pressurization. Analytical sample volumes were adjusted by a correction factor for canisters pressurized with helium as recommended by EPA standard SOP VOE-TO15.

Experimental design and analysis. Firelogs containing either oak wood fiber or CBS blended in paraffin wax were manufactured in the laboratory. The air emission samples from burn tests were collected following a completely randomized scheme. The treatments evaluated were: Fiber type and loadings (3), and Compression loads (3). Each treatment combination was replicated 10 times. The data was statistically analyzed using JMP software (Version 11.0.2, SAS Institute, Cary, NC) and one-way analysis of variance using paired and unpaired tests (Student's T test). Significance was accepted at $P < 0.05$.

RESULTS AND DISCUSSION

Impact of composition on physical characteristics. The physical characteristics of fire logs manufactured by varying the proportion of two of CBS and oak wood fibers and different processing conditions are shown in Table 1. The one-way ANOVA for means comparison using Student's T test showed that density and average burn time were significantly different within different treatments (Figure 4 and 5).

The target weight of the firelogs was fixed at 1.4 kg to match the weight of commercial firelogs used in the previous study (Houck et al., 2000). The length of the logs varied from 18 cm - 22.4 cm and the density ranged between 841.9 kg/m3 – 1006.1 kg/m3 depending on compression loading. The control formulation containing oak fibers had a higher density compared with two CBS based formulations (Figure 4). This was attributed to higher bulk density of oak wood fibers compared to CBS fibers (Bajwa

Table 1. The physical characteristics of various formulations of firelogs manufactured under different processing conditions

Treatment Number	Formulation	Weight (gm)	Compression load (ton)	Height (cm)	Density (kg/m3)	Compaction Ratio	Av. Burn Temp (°C)
1	50%Oak, 50% Wax	1409.3 (9.0)*	4	19.3 (0.5)	874.5 (2.6)^{F**}	2.23:1	270.2 (5.4)^{D}
2	50%Oak, 50% Wax	1412.1 (6.8)	6	18.5 (0.2)	952.7 (3.5)^{C}	2.44:1	269.4 (3.5)^{D}
3	50%Oak, 50% Wax	1408.3 (9.6)	8	18.1 (0.3)	1004.1 (4.2)^{A}	2.51:1	273.8 (4.8)^{C}
4	50%CBS, 50% Wax	1412.0 (6.2)	4	21.0 (0.3)	841.9 (2.9)^{G}	2.15:1	288.3 (6.6)^{A}
5	50%CBS, 50% Wax	1412.2 (10.1)	6	19.1 (0.2)	937.8 (2.4)^{D}	2.30:1	281.7 (3.3)^{B}
6	50%CBS, 50% Wax	1412.4 (7.4)	8	18.5 (0.4)	971.3 (3.1)^{B}	2.50:1	290.3 (5.1)^{A}
7	60%CBS, 40% Wax	1414.5 (7.4)	4	22.3 (0.3)	791.4 (3.1)^{H}	2.00:1	261.1 (4.9)^{F}
8	60%CBS, 40% Wax	1414.0 (5.2)	6	20.1 (0.2)	873.8 (1.2)^{F}	2.22:1	257.3 (7.8)^{G}
9	60%CBS, 40% Wax	1410.7 (4.7)	8	19.1 (0.2)	932.6 (2.3)^{E}	2.36:1	265.6 (7.4)^{E}

***Standard deviation value is shown in parenthesis. **Treatments not connected by same letter are significantly different at 0.95% confidence level.**

et al., 2011). An increase in compression load from 4T to 6T resulted in 9%-11% increase in density whereas density changed from 3%-6% when compression increased from 6 T to 8 T. The compaction ratio followed a similar trend as demonstrated in the compression ratio and density relationship. The compaction ratio was directly proportional to the compression load. Within the three main formulations, firelogs containing oak fibers had slightly higher compaction ratios under the same compression load than CBS fiber firelogs.

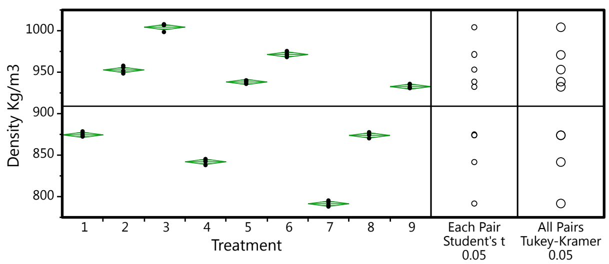

Figure 4. Density comparison of different firelog treatments (One-way ANOVA)

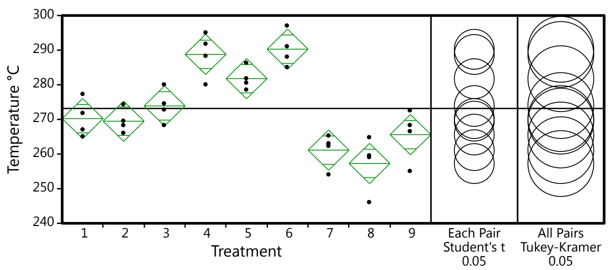

Figure 5. Burn temperature comparison of different firelog treatments (One-way ANOVA)

Burn characteristics of firelogs. The burn characteristics of firelogs are important from qualitative and quantitative performance measures such as ease of ignition, odor, and identifying the nature of chemicals released from exhaust air emissions. The qualitative factors include ease of ignition, smoke generation, flame height, total burn time and ash content. Pollutants include aromatic polycyclic hydrocarbons and gases, which can be detrimental to indoor air quality (Christian et al., 2003). Overall firelogs based on oak and CBS fibers showed similar burning characteristics. Petrichor scent, a slight earthy odor, was observed when CBS fiber based firelogs were burn tested. This can be attributed to combustion and oxidation of terpenes and other oxygenated hydrocarbons present in biomass (Wolfgang et al., 1998). All three formulations exhibited similar ignition characteristics. The firelog samples sustained flame in 45-60 s of exposure to butane lighter flame on both ends. The flame travelled from the outer edges to the center. The entire firelog was covered with flame after 15-20 minutes exposure to ignition. From the visual appearance of the flame it appeared that density of the firelogs had an impact on the flame characteristics. The lower density firelogs disintegrated or collapsed after 60-75 minutes of exposure to flame whereas the highest density (8 T) firelogs retained their integrity and burned slightly over two h. A typical burn time for a 1362 g (three lbs) commercial firelog is two h. The variation in the burn characteristics can be explained by the density of materials (fibers), as high-density materials tend to have a high compaction ratio as compared to low-density materials. Flame height, ranging from 15-25 cm, was noticed for lower density firelogs. The firelogs containing 50% oak or 50% CBS fibers compressed under an eight T load exhibited a medium flame height of 12-16 cm and showed no signs of disintegration during the entire burn test. The two factors that influenced firelog burn characteristics the most were compaction ratio and density as noticed by total burn time. The high density and high compaction ratio resulted in improved flame quality, product integrity and longer burn time. In this study no substantial difference was observed in the average combustion temperatures between the three material compositions (Table 1). The highest combustion temperature, 291.1°C, was recorded for firelogs with 50% CBS and 50% wax and the lowest temperature 259.4°C, was observed in the formulation with 60% CBS and 40% wax. The compression load and compaction ratio had some influence on the combustion temperature of the firelogs (Table 1). Firelogs with 50% oak fibers and 50% wax exhibited minimal variation (3.4°C) in the combustion temperature in response to compression load. CBS based firelogs showed ash content of 3.9% as compared to 1.4% for oak fiber based firelogs that is similar to an earlier reported study (Holt et al., 2006).

Gaseous emission analysis. Table 2 indicates detectable emissions of fixed gases from the three different formulations of firelogs. None of the three formulations indicated any detectable levels of principal gases (CO, NH_4, H, N_2O, and CH_4). The amount of nitrogen gas was consistent in all three formulations with concentration roughly at 78 µg/m^3. Argon gas levels ranged from 20.8 to 21.4 % v/v in all formulations. The list of volatile organic compounds detected in firelogs is shown in Table 3. The smoke emitted from combustion included aliphatic and cyclic hydrocarbons. All three formulations showed varying amounts of unbranched straight chain n-alkanes (C4 – C36) which is consistent with previous studies and a signature of crude oils (Wolfgang et al., 1998, Houck et al., 2000). The alkanes detected included hexane, heptane, octane and n-nonane. The firelogs with 50% CBS –paraffin wax exhibited a higher concentration of n-alkanes. The lowest level alkene (1,3-Butadiene) was found in oak-paraffin wax firelogs. Haloalkane chloromethane was found in all firelog formulations. Chloromethane level was higher for CBS logs as compared to oak fiber logs, it may be due pesticide residue on plant material (Koinecke et al., 1994).

Table 2. Fixed gas air emissions from paraffin based fire logs with different composition during burning

Elements (μg/m^3)	50% Oak + 50% Wax	50% CBS + 50% Wax	60% CBS + 40% Wax
Hydrogen	ND	ND	ND
Oxygen + Argon	20.8	21.2	21.3
Nitrogen	78.3	78.2	78.1
Carbon monoxide	ND	ND	ND
Methane	ND	ND	ND
Carbon dioxide	0.9	0.7	0.6

ND = Compound was analyzed for, but not detected above the laboratory reporting limit.

Table 3. Volatile organic compound air emissions from paraffin based fire logs

Elements (μg/m^3)	50% Oak + 50% Wax	50% CBS + 50% Wax	60% CBS + 40% Wax
Propene	596.7	2950.0	2940.0
Dichlorodifluoromethane (CFC 12)	2.0	ND	ND
Chloromethane	46.8	140.5	254.0
1,3-Butadiene	154.8	525.0	436.0
Ethanol	24.3	65.7	109.5
Acetonitrile	62.0	345.0	328.0
Acrolein	211.5	960.0	1332.0
Acetone	127.8	860.8	1350.0
Acrylonitrile	5.7	50.5	53.6
Vinyl Acetate	59.7	110.0	129.3
2-Butanone (MEK)	35.9	257.5	270.0
N Hexane	18.9	53.8	33.8
Tetrahydrofuran	28.9	44.8	50.8
Benzene	224.0	952.5	718.0
n-Heptane	20.4	48.8	29.4
Toulene	32.7	417.5	288.0
2-Hexanone	13.2	49.0	53.0
n-Octane	17.3	42.3	26.6
Ethylbenzene	9.3	71.0	47.0
m.p-Xylene	8.5	97.3	65.4
Styrene	21.6	78.5	43.8
o-Xylene	6.9	48.0	25.6
n-Nonane	20.1	39.8	23.6
n-Propylbenzene	6.2	20.5	12.3
4-Ethyltoulene	ND	16.0	9.9
1,2,4-Trimethylbenzene	5.3	21.2	14.7
D-Limonene	7.2	7.3	14.1
Napthalene	21.2	104.5	61.8

ND = Compound was analyzed for, but not detected above the laboratory reporting limit

The oxygenated, volatile organic compounds ethanol, acetone, ether, butanone, vinyl acetate, tetrahydrofuran and hexanone were found in all the firelog formulations. This is consistent with a previous study that cited the presence of these compounds in plant biomass burning emissions (Christian et al., 2003). Another major group of compounds detected in the smoke emissions included polycyclic aromatic hydrocarbons (PAH) typically associated with crude oil byproducts. The mono substituted benzene derivatives dominated this category consisting of benzene, 1,2,4 -Trimethyl benzene, n-propylbenzene, ethylbenzene, xylene, toluene, 4-Ethyltoluene and styrene. Some of these compounds are produced by catalytic reforming called reformate. D-Limonene a cyclic terpene with a lemonish smell was found in all three firelog formulations. The formulation with 60% biomass had a higher level of D-Limonene. Firelogs made from oak had detectable levels of dichlorodifluoromethane (CFC 12), bromomethane, and trichlorofluoromethane whereas none of those compounds were observed in firelogs made with CBS. The presence of high amount of extractives, hydrolysable tannins (*ellagitannins*), in oak wood fiber may have contributed to these compounds. In an activated state oxygen is able to oxidize alcohols into acetaldehydes which may further react with paraffin wax to generate these compounds. Although there are some minor differences in the emissions from the firelogs made with CBS versus oak fiber, there is no standardized definition of fireplace emissions tests or maximum limits both types of firelog should perform equally well (Li and Rosenthal, 2006).

Gaseous emission from the combustion of any type of biomass fuel depends directly on the chemical composition of the fuel and combustion conditions. During combustion the cellulosic materials start to hydrolyze, oxidize, dehydrate and pyrolyze as temperatures increase forming combustible volatiles, tarry substances and highly reactive carbonaceous char (Shafizadeh, 1984). When reaching the ignition temperature of the volatiles, tarry substances, and hydrocarbons, the exothermic reactions typical of combustion, begins. The heat generated during initial combustion first provides the energy required for gasification of the cellulosic substrate and propagation of fire. It also helps in the evaporation of the free cellulosic moisture available in the cell cavities followed by the vaporization of bound water, extractives such as resinous compounds and decomposition of cellulose, hemicelluloses and lignin are vaporized via partial or complete combustion (Rowell and Dietenberger, 2013). During flaming combustion, char formation continues until the flux of combustible volatile substances drops below a level required for propagation of flaming combustion. This leads to a smoldering process where enough heat is produced to propagate the charring process and release of additional volatile products.

CONCLUSIONS

Firelogs were produced from CBS and oak wood fibers using petroleum based paraffin wax. Based on the firelog processing characteristics and air emissions, CBS fiber was found to be an acceptable material for manufacturing firelogs. Fiber type had minimal impact on the firelog physical properties, whereas bulk density of fibers, compression load and compaction ratio had a significant influence on the overall burn quality of firelogs. Higher density and compaction ratio of firelogs resulted in improved flame height, durability and uniform burn quality. None of the fire logs indicated any detectable levels of CO, NH_4, H_2, or NO. Firelogs made from oak fibers had detectable levels of dichlorodifluoromethane (CFC 12), bromomethane, and trichlorofluoromethane whereas none were observed in the case of firelogs made with CBS. The study shows that cotton burr with stem fibers has the potential to serve as an alternative biomass material for the commercial firelog industry.

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