Thermal Reactions of Small Loblolly Pine Cubes Heated on One Face in an Air Atmosphere

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ABSTRACT. When 1-, 2-, and 3-mm cubes of wood were placed on the sample pan of a differential scanning calorimeter and the pan heated at a constant rate from 150 to 513°C. in an air atmosphere, all 144 specimens formed an endothermic peak (all-specimen average 345°C.) at which the rate of heat dissipation exceeded the rate of heat absorption; the peak corresponded to the evolution of volatiles and cube charring. On further heating, most latewood cubes evolved heat until glowing ignition occurred (498 to 513°C.). With earlywood, a secondary reaction of unknown nature stopped the exothermic reaction, so that the exotherm changed at 434°C. to an endotherm and a second endothermic peak occurred at 480°C.; the second peak in turn yielded to a renewed exothermic reaction followed by glowing ignition at 490 to 513°C. or by degradation to ash without ignition. These values are overall averages for scan rates of 10 and 20°C./min. and for wet and dry wood both extracted and unextracted. A 20°C./min. scan rate (compared to a 10°C. rate) delayed endothermic and exothermic peaks from 14 to 19°C.; saturation of cubes delayed peak temperatures 5 to 15°C.; extraction increased the temperature of the first endotherm by 4°C. and the temperature of the second earlywood endotherm by 7°C. The first exothermic peak temperature (434°C.) for earlywood was positively correlated with cube size. Because many specimens decomposed without glowing, conclusions about glow initiation temperatures and glow duration during dynamic tests are hard to frame. Ignition temperature was lowest in 3-mm, initially dry earlywood cubes (494°C. for extracted and unextracted specimens heated at 10°C./min.). In dynamic tests, glow duration was shortest in 1-mm cubes (6 sec.); generally speaking, small earlywood cubes failed to ignite at all. Glow duration was longest for 3-mm latewood cubes (106 sec.). In static tests at 513°C., elapsed time before glow initiation was shortest with 1-mm, initially dry, extractive-free cubes of earlywood (average 6 sec.); elapsed time was longest with 3-mm, initially saturated, unextracted cubes of latewood (average 189 sec.). Glow time was maximum for 3-mm, initially dry, extractive-free cubes of latewood (average 112 sec.); it was minimum for 1-mm, initially saturated cubes of earlywood (average 3.5 sec.).

THE DESIGN of the present study was influenced by two factors. First was the absence of information on the thermal behavior of southern pine earlywood and latewood at temperatures approaching the ignition point. Second was the availability of a scanning calorimeter permitting evaluation of very small specimens (1/2 to 100 mg) at precise temperatures.

Loblolly pine (*Pinus taeda* L.) was studied because it is the principal commercial southern pine. The research was exploratory and the sample limited to wood from a single tree.

The specific objective was to determine thermal reaction temperatures and times — as affected by specimen size, wood moisture content, and extractive content — as follows:

1) In dynamic tests in an air atmosphere with the wood subjected to a temperature increasing at constant rate (10 or 20°C./min.), to determine the average ignition temperature (temperature at which glowing occurs), and average

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glow time of small cubes of latewood and earlywood.

2) In dynamic tests in an air atmosphere to determine the average temperatures of thermal transitions — both endothermic and exothermic — below the ignition temperature.

3) In static tests to measure the average time to ignition and the average glow time for cubes of earlywood and latewood heated at a constant temperature of 513°C. in an air atmosphere.

Review of the Literature

There is voluminous literature on thermal reactions of wood. Some papers having a direct bearing on ignition temperatures include those of Graf (1949), Fons (1950), Browne (1958), Matson *et al.* (1959), Browne and Tang (1962), Stamm (1964, p. 291), Beall (1968), and Kollmann and Cote (1968, p. 151). A review of the literature on thermal degradation of wood components (74 citations) has been provided by Beall and Eickner (1970).

In brief, the literature contains three distinct definitions of ignition temperature:

1) The exothermic reaction point, *i.e.,* the temperature at which the rate of heat dissipation exceeds the rate of heat absorption — resulting in eventual flaming or glowing in an atmosphere that will support combustion. In the present study, this point is termed an endothermic peak.

2) The temperature at which a glowing wire or pilot light will cause flaming ignition of the combustible gases evolving from heated wood.

3) The auto-ignition point, at which the test specimen ignites and burns. Auto-ignition takes place at temperatures above either the flame point or the exothermic reaction point.

From the literature, a list of possible variables affecting thermal reactions would include:

1) The specimen properties of specific gravity, permeability, specific heat, thermal conductivity, overall size, thickness, moisture content, volatile content, wood species and proportion of latewood, natural defects (knots, checks), form (solid or ground), and surface condition (smooth or rough).

2) The heating variables of heat source, exposure time, rate of heating, initial temperature, atmosphere, and rate of atmospheric flow.

Procedure

In a series of preliminary trials cubes were: tested in air and in nitrogen flowing at 20, 30, and 40 cc/min.; heated at 5, 10, 20, and 40°C./min.; heated from starting temperatures of 50, 100, 150, and 200°C. Bark as well as ground wood and varying sizes of cubes were tested at 10 percent moisture content, saturated, and ovendry. Attempts were also made to ignite the combustible gases evolved from heated wood, but both spark gaps and glowing wires were unsuccessful.

Bark was eliminated from the study because it was difficult to ignite. For wood, ignition was most frequent and predictable in an air atmosphere flowing at 40 ml/min. Starting temperature was set at 150°C. Study variables were as follows:

- Cell type: earlywood and latewood
- Cube size: 1, 2, and 3 mm on a side
- Extractive content: unextracted and extractive-free
- Moisture content: saturated and 10 percent of ovendry weight
- Replications of cubes: three

Heating modes were:

- Dynamic, scanning from 150°C. at 10°C./min. to ignition
- Dynamic, scanning from 150°C. at 20°C./min. to ignition
- Static (513°C.)

The entire experiment required 216 specimens. The dynamic tests called for 144: (two cell types) (three cube sizes) (two extractive contents) (two moisture contents) (two scan rates) (three replications). The static evaluation was done at the single temperature of 513°C. and therefore required only 72 specimens.

Specimens were prepared from the 66th growth ring of a 16-inch-diameter butt log of a loblolly pine tree cut near Alexandria, Louisiana. Earlywood and latewood bands from the chosen ring were separated and 1-, 2-, and 3-mm cubes cut and conditioned to 10 percent moisture content.

Cubes were weighed to the nearest 0.01 mg. Within a cube size and cell type class, standard deviation of weight was not in excess of 15 percent of the average for the class.

Cube size	Average weight at 10 percent moisture content	
	Earlywood	Latewood
(mm)	(mg)	
1	0.45	1.12
2	2.21	7.22
3	7.42	20.77

The cubes were cut with a razor blade and not to a degree of accuracy that would permit calculation of specific gravities from the foregoing data.

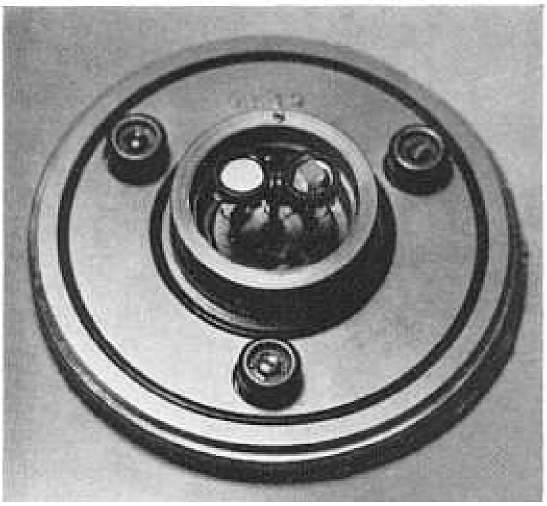

Figure 1. — Sample holder on differential scanning calorimeter. The right-hand pan contains a 3-mm latewood cube.

The 3-mm latewood cubes probably contained some transition wood; the 1-mm latewood cubes were taken near the end of growth increments, and hence were perhaps more dense than normal.

Half the specimens were extracted by TAPPI Standard Method T 12m-59 and conditioned to 10 percent moisture content. One or 2 days before evaluation, cubes to be saturated were boiled in distilled water for 1 hour, then cooled and subjected to a slight vacuum for an additional hour.

Specific gravity of earlywood specimens was 0.38; latewood specific gravity was 0.79 (based on ovendry weight and green volume). Extractive content of the specimens was generally less than 7 percent of the extractive-free weight; earlywood averaged 4.2 percent, while latewood averaged only 3.1 percent.

Scanning Calorimeter

The instrument employed for evaluation of thermal behavior was the Perkin-Elmer DSC-1B differential scanning calorimeter.[1] In this calorimeter, the two matched sample holders (Fig. 1) are part of a controlled electrical circuit. A test specimen is placed in one holder; the specimen and the empty reference holder are subjected to increasing temperature at a closely controlled rate. A monitoring system detects any difference between specimen and reference temperature and simultaneously changes the amount of heat supplied to either specimen or reference in order to maintain both at the same temperature.

The instrument's actual measurement is not the difference in temperature between specimen and reference; this always remains very small. Instead, the measurement is the difference in electrical power required to maintain specimen and reference at the desired temperature. Thermal lags are minimized by use of very small specimens.

Because the recorder is arranged with differential energy as the ordinate and time (hence temperature when programmed to a specific rate of temperature rise) as the abscissa, the ordinate readout is directly proportional to dH/dt, where H is heat energy and t is time.

[1]For a more detailed explanation of the DSC specifications and applications, see O'Neill (1964), Watson *et al.* (1964), and Koch (1969).

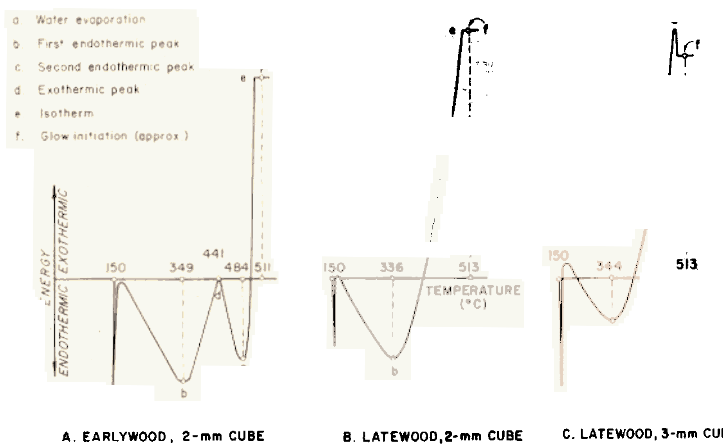

Figure 2. — Typical calorimeter plots of loblolly pine wood heated from 150 to 513°C. at 20°C./min. in a dynamic air flow of 40 ml/min. Full-scale chart deflection: 16 millicalories for A and B, and 32 millicalories for C.

In this study, the thermal property of interest is not the quantitative amount of heat absorbed in an endothermic process, or given off in an exothermic reaction. Rather, the temperatures at which the rate of heat dissipation exceeds the rate of heat absorption (and vice versa) are observable from reversals in the trend of the plot of differential energy (Fig. 2).

Temperature was calibrated daily. Between 150 and 450°C., observed temperatures are probably accurate within ±1°C.; recordings in the range 450 to 513°C. may be in error by ± 3°C.

During both dynamic and static tests a glass top was placed over the sample holders, and air at atmospheric pressure flowed under this cover (at 40 ml/min.) to purge decomposition products and maintain a uniform atmosphere. While the specimen holders were heated to 513°C., the surrounding air remained near room temperature (volatile decomposition products did increase the temperature from ambient to approximately 50°C. during a run). The recorded temperatures were those of the sample holder; these temperatures were not necessarily equal to average cube temperature.

Dynamic Tests

After calibration, the starting temperature of the calorimeter was set at 150°C., and a suitable chart range selected. Weight-matched aluminum foil pans approximately 6.3 mm in diameter were placed in each sample-pan holder. Pans were tamped to insure uniform contact between the heated surface and the pan, and the cover of the assembly put in place. Through an aperture in the cover, a specimen was then placed in the right sample holder — end grain down. The scan was immediately started, with the temperature increasing linearly at 10 or 20°C./min. to either the top of the scanning range (513°C.) or to the temperature at which the cube ignited. The recording pen traced endothermic and exothermic peaks (Fig. 2). Ignition temperatures were visually observed and the glow duration timed to the nearest second with a stopwatch. The 144 specimens were run in random order.

Static Tests

Static tests were run at 513°C., the highest temperature achievable on the calorimeter. A test cube of wood was placed end grain down in the right-hand holder, and the time to ignition and duration of glow measured to the nearest second with stopwatches. The 72 specimens were evaluated in random order.

Results

Dynamic Tests

Distinct thermal zones were apparent from scans of both earlywood and latewood (Fig. 2).

150 to 310°C. — All cubes heated from 150 to 310°C. absorbed heat and changed color from light to medium to dark brown. Below 200°C., there was an initial sharp endothermic peak caused by moisture loss; it occurred at a higher temperature with saturated specimens than with 10 percent specimens.

310 to 400°C. — Within this temperature range, the first thermal transition occurred as cubes began to char and evolve a stream of gaseous decomposition products in an exothermic reaction; an endothermic peak was formed. By 400°C., charring was complete; the charcoal retained the specimen's original cubic structure.

390 to 480°C. — A significant difference between earlywood and latewood behavior was apparent in this temperature range. In earlywood the exothermic evolution of gaseous byproducts stopped (forming an exothermic peak about tangent to the baseline), and was followed by absorption of heat by the charcoal in a second endotherm. Most latewood cubes, however, continued to evolve gas and lose heat in an exothermic reaction throughout the range.

460 to 513°C. — Earlywood continued to absorb heat, then — in a sudden exotherm — formed a second endothermic peak in the range 460 to 490°C. One to 3 sec. before ignition (in the range 490 to 513°C.), earlywood reached an isothermal condition, after which the charcoal either ignited and glowed bright orange or gradually decomposed to ash without combustion (Fig. 2A). While all earlywood cubes formed two endothermic peaks, less than half (31 of 72) ignited and glowed.

Latewood cubes in the temperature range 460 to 513°C. followed one of three reaction paths: 1) some small cubes (1 to 2 mm) continued to evolve gas and were exothermic until just prior to ignition, when they reached an isothermal condition before glowing (Fig. 2B); 2) other small cubes formed a second endotherm similar to the earlywood cubes (Fig. 2A); 3) in contrast, the 3-mm latewood cubes — shortly before glowing ignition — reached a temperature at which the reaction changed from exothermic to endothermic (Fig. 2C). The second endotherm observed with some

Table 1. — RANGE OF THERMAL TRANSITION TEMPERATURES AND FREQUENCY OF OCCURRENCE FOR LOBLOLLY PINE EARLYWOOD AND LATEWOOD CUBES HEATED IN AIR.[1]

Transition	Earlywood		Latewood	
	Frequency	Range	Frequency	Range
		°C.		°C.
First endothermic peak	72/72	313-380	72/72	320-382
Exotherm change to second endotherm	72/72	391-486	39/72	394-512
Second endothermic peak	72/72	436-513	18/72	460-511
Glowing ignition				
1-mm cube	1/24	506	17/24	507-513[2]
2-mm cube	9/24	508-513[2]	24/24	501-513[2]
3-mm cube	21/24	490-513	24/24	498-513[2]

[1]Includes values for extracted and unextracted specimens at both scan rates.

[2]Ignition of some cubes took place only after a time delay at 513°C.

latewood cubes usually did not occur until the temperature range 490 to 510°C.; unlike earlywood, many latewood cubes ignited and glowed before forming a second endothermic peak. All latewood cubes formed a first endothermic peak; 39 of the 72 specimens absorbed heat in a second endothermic region, while only 18 of 72 formed a second endothermic peak. Sixty-five of a possible 72 latewood specimens ignited, although some not until after a time delay at the top (513°C.) of the scan range.

Transition temperatures are summarized in Table 1.

Because some cubes failed to form a second endotherm and others failed to ignite, a fully factorial analysis of variance could be completed only for the first endothermic peak. All factors except cell type (earlywood and latewood) significantly affected the temperature of the first endothermic peak (Table 2). The grand mean temperature for the first endothermic peak was 345°C.; it was observed that this temperature corresponded to the evolution of visible gases. The peak came at lowest temperature with 10° scan rate on unextracted 3-mm cubes at 10-percent moisture content.

For all earlywood specimens analysis was complete for first endothermic, first exothermic, and second endothermic peaks; average transition temperatures (Table 3) were:

Peak	°C.
First endothermic	344
Exothermic	434
Second endothermic	480

Saturated cubes had transition temperatures 5 to 15° higher than wood at 10 percent moisture content.

For earlywood, the 3-mm cubes had the lowest first endothermic peak; temperatures at the exothermic and second endothermic peaks were positively correlated with cube size. Both first and second endothermic peaks were higher for extractive-free earlywood than for unextracted earlywood. For earlywood, temperatures for all three peaks were 18 or 19°C. higher at the 20° scan rate than at the 10° rate.

For 3-mm cubes only, analysis was complete for the first endothermic peak and the first exothermic peak. Scan rate, moisture content, and cell type significantly affected these peaks (Table 4). Latewood reached the change from exothermic to endothermic reaction at a temperature more than 40°C. higher than earlywood; there was only a 4° difference in the temperature of the initial endothermic peak.

Table 2. — EFFECT OF PRIMARY VARIABLES ON THE FIRST ENDOTHERMIC PEAK TEMPERATURE OF LOBLOLLY PINE WOOD WHEN HEATED DYNAMICALLY.

Factor	First endothermic peak[1]
	°C.
Cell Type	
Earlywood	344
Latewood	346
Cube size (mm)	*
1	345
2	349
3	342
Moisture content	*
10 percent	339
Saturated	352
Extractive content	*
Unextracted	343
Extractive-free	347
Scan Rate (°C./min.)	*
10	336
20	355

[1]All factors were tested by variance analysis at the 0.05 level; significant differences are indicated by an asterisk.

Table 3. — EFFECT OF PRIMARY VARIABLES ON TRANSITION TEMPERATURES OF EARLYWOOD WHEN HEATED DYNAMICALLY.[1]

Factor	First endo-thermic peak	First exo-thermic peak	Second endo-thermic peak
		°C.	
Cube size (mm)	*	*	*
1	344	408	475
2	349	435	482
3	340	458	481
Moisture content	*	*	*
10 percent	337	431	475
Saturated	351	436	484
Extractive content	*		*
Unextracted	342	433	476
Extractive-free	346	434	483
Scan rate (°C./min.)	*	*	*
10	335	424	470
20	353	443	489

[1]All factors were tested by variance analysis at the 0.05 level; significant differences are indicated by an asterisk.

Grand mean temperatures for these large cubes were 342°C. for the first endothermic peak and 480°C. for the first exothermic peak. Extractive content did not affect these transition temperatures in the 3-mm cubes (Table 4).

Glowing ignition and glow duration, dynamic. — Because all the cubes did not ignite and glow, analysis was difficult. Some portions of data for the five-factor design were complete for two or three factors; by analysis of variance these limited data supply some clues about the effects of all factors.

Only one block of data (18 observations) permitted the *size effect* of all three cubes to be analyzed. Size was negatively correlated with temperature of glow initiation and positively correlated with duration of glow; with extracted and unextracted latewood of 10 percent moisture content heated at 10°C./min., values were as follows:

Cube size	Glowing ignition temperature	Glow duration
(mm)	(°C.)	(sec.)
1	512	6
2	506	39
3	503	106

Latewood cubes tested saturated, dry, extracted, and unextracted all showed the same trend in glow duration when heated at 10°C./min.; 3-mm cubes averaged 99 sec., while 2-mm cubes averaged 37 sec.

In dynamic test, only 18 of all forty-eight 1-mm cubes glowed, while 45 out of the forty-eight 3-mm cubes glowed. Some glowed only after a time lapse at 513°C.

Only one block of 12 observations was complete to permit *scan rate* to be correlated with glow initiation and duration; with 3-mm extracted earlywood cubes, scan rate proved not to be correlated with ignition temperature of cubes at 10 percent moisture content or saturated. For the same wood, however, duration of glow was positively correlated with scan rate.

Scan rate	Duration of glow
	(sec.)
	23
	27

When wood reached the top of the scan (513°C.) without glowing, delayed ignition occurred more frequently at the higher scan rate.

The effect of *cell type* (earlywood or latewood) on glow initiation and duration was observable in two blocks of data each having 12 observations. For 3-mm extracted and unextracted cubes at 10 percent moisture content heated at 10°C./min., earlywood ignited before latewood but glowed a much shorter time.

Table 4. — THREE-MM CUBES ONLY. EFFECT OF PRIMARY VARIABLES ON THE FIRST ENDOTHERMIC AND EXOTHERMIC PEAK TEMPERATURES OF DYNAMICALLY HEATED LOBLOLLY PINE WOOD.[1]

Factor	First endothermic peak	First exothermic peak
	- - - - °	- - -
Cell Type	*	*
Earlywood	340	458
Latewood	344	502
Moisture content	*	*
10 percent	335	477
Saturated	350	483
Extractive content		
Unextracted	342	480
Extractive-free	342	480
Scan rate (°C./min.)	*	*
10	335	473
20	349	487

[1]All factors were tested by variance analysis at the 0.05 level; significant differences are indicated by an asterisk.

Cell type	Glow initiation	Glow duration
	(°C.)	(sec.)
Earlywood	494	27
Latewood	503	106

With both wet and dry 3-mm cubes (extracted) heated at the same rate, earlywood glowed only 23 sec., while latewood glowed 100 sec.; temperatures of ignition were not significantly different, however.

The effects of *moisture content* were seen in four blocks of data. One block of 24 observations consisted of 2- and 3-mm cubes of latewood — both extracted and unextracted — heated at 10°C./min. Here temperatures of glow initiation did not vary significantly with initial moisture content; but on the average, the initially saturated cubes glowed longer (72.5 sec.) than the dry ones (64.3 sec.).

In three smaller blocks, each of 12 observations on 3-mm cubes, initially dry wood ignited at lower temperatures than initially saturated cubes. The data below seem to indicate that initially dry earlywood glows longest. In the data for earlywood and latewood combined, saturated wood glowed longest; possibly glow of the latewood component was lengthened by saturation.

Description of 3-mm cubes	Glow initiation		Glow duration	
	10 percent	Saturated	10 percent	Saturated
	(°C.)		(sec.)	
Earlywood and latewood, extracted, scan 10°C./min.	498	508	56	66
Earlywood, extracted and unextracted, scan 20°C./min.	498	510	30	25
Earlywood, extracted, scan rates of 10 and 20°C./min.	498	509	28	22

The effects of *extractive content* were shown by three blocks of data, each with 12 observations, and one block with 24 observations. In none of these did extractive content significantly affect duration of glow.

Extractive content was the only variable significantly affecting ignition temperature of 2- and 3-mm latewood cubes (initially both wet and dry) heated at 10°C./min. Extraction lowered the temperature an average of 3.5°C. (from 507.5 to 504°C.).

With 3-mm dry cubes heated at 10°C./min., extraction lowered the ignition temperature of latewood 6°C. (from 506 to 500°C.). By contrast, extraction of earlywood raised the temperature of glow initiation an average of 6°C. (from 491 to 497°C.).

Table 5. — RANGES OBSERVED FOR TIME LAPSE BEFORE IGNITION, AND DURATION OF GLOW, FOR CUBES OF LOBLOLLY PINE EARLYWOOD AND LATEWOOD HEATED AT 513°C. IN AIR.

Cube size	Earlywood		Latewood	
	Time lapse	Length of glow	Time lapse	Length of glow
mm	Sec.			
1	5-15	2-5	11-44	7-13
2	12-39	12-24	32-64	47-62
3	20-110	31-52	146-196	77-128

Static Tests

Reaction of cubes heated at a constant temperature of 513°C. were similar to those observed in dynamic tests, but occurred more rapidly. Blocks darkened, evolved gas, charred, and burned, all within a 6-min. interval or less. All 72 specimens glowed in static testing, whereas 48 of the 144 cubes heated dynamically decomposed without igniting. Static test results are listed in Tables 5 (range) and 6 (averages).

Charring occurred first at the cube face in contact with the heat source, then progressed to

Table 6. — EFFECT OF PRIMARY VARIABLES ON TIME LAPSE BEFORE IGNITION AND DURATION OF GLOW FOR CUBES OF LOBLOLLY PINE EARLYWOOD AND LATEWOOD HEATED STATICALLY AT 513°C. IN AIR.[1]

Factor	Time to glow	Glow duration
	Sec.	
Cell type	*	*
Earlywood	30	20
Latewood	80	54
Cube size (mm)	*	*
1	15	7
2	37	36
3	113	68
Moisture Content	*	*
10 percent	44	40
Saturated	66	34
Extractive content	*	*
Unextracted	57	35
Extractive-free	53	39

[1]All factors were tested by variance analysis at the 0.05 level; significant differences are indicated by an asterisk.

the opposite face. Glowing followed the opposite direction, starting at the face exposed to the air, then progressing toward the center of the cube, and finally becoming very bright at the center just before ceasing.

As in the dynamic tests, duration of glow ranged from less than a second to longer than 2 min. In general, small saturated cubes of unextracted earlywood glowed the shortest time, while large dry cubes of extractive-free latewood glowed longest. Cubes either burned completely or left a very small amount of gray ash in the sample pan.

All factors tested were significant at the 0.05 level as determined from analysis of variance (Table 6). Interactions did not alter primary effects observed to be significant. Time to glow and glow duration increased with increasing cube size. Latewood cubes took nearly three times as long to reach ignition as earlywood cubes, and glowed nearly three times longer when ignited. Saturated wood took an average of 22 sec. longer to glow than wood at 10 percent moisture content, but duration of glow was shorter. Extractive-free wood ignited 4 sec. earlier than unextracted cubes and glowed 4 sec. longer.

Discussion

In all earlywood and in some latewood samples an exothermic reaction that defined the first endothermic peak (all-specimen average 345°C.) was stopped by a secondary reaction that led to a second endothermic peak (average 480°C. for earlywood). The literature indicates that the interruption of the exotherm may be caused by adsorption of gases by the charcoal or because evolving gases block oxygen from the charcoal surfaces. In most latewood a second endothermic peak did not appear; *i.e.,* the initial exotherm led directly to ignition.

Gases first became visible at the first endothermic peak (345°C.), and the cubes began to char. Evolution of visible gases ceased when the exothermic reaction yielded to the second endotherm (average temperature 434°C. for earlywood — see Figure 2A for typical plot). In these cases, the ensuing endotherm progressed until sufficient heat was absorbed to initiate a second and final exotherm.

The fact that 48 of the 144 specimens heated dynamically degraded to ash without glowing combustion supports Graf's (1949) finding that a minimum sample mass is required for ignition when heating is slow. However, because even the small earlywood specimens did display two endothermic peaks and one exothermic peak, a minimum mass was evidently not required for thermal transitions below glowing ignition.

A 20°C./min. scan rate (compared to a 10° rate) delayed endothermic and exothermic peaks from 14 to 19°C.; saturation of cubes further delayed these peak temperatures 5 to 15°C. Since specimens lost all residual moisture during testing and became ovendried before reaching 200°C., the increase in peak temperatures may mean that saturation removed some water-soluble substances that contribute to early thermal degradation.

Extraction was not a significant factor in all cases. It did increase the temperature of the first endotherm (by 4°C.) and the temperature of the second endotherm for earlywood (by 6°C.). This result corresponds to Beall's (1968) finding that extraction of southern pine did not significantly change thermal degradation reactions.

The average temperature at the first endothermic peak was not a strong function of cube size (range 342 to 349°C., see Table 2) or of cell type. It seems, therefore, that the temperature at which gases started to evolve was not clearly related to sample size or density.

The first exothermic peak temperature for earlywood, however, was strongly correlated with cube size (see Table 3). Therefore, it appeared that the temperature at which gas evolution ceased in earlywood was a function of sample mass.

Temperatures here observed for glowing ignition (490 to 513°C.) are higher than many reported in the literature (260 to 300°C.). Others have heated wood specimens in a furnace, thus bringing heated air into contact with all surfaces. The scanning calorimeter applied heat to a single cube face; the surrounding air remained near room temperature. Internal temperature of the cube was thus partially determined by the thermal conductivity of the wood and rate of dissipation into the cooler air. It was observed that the faster heating rate increased transition temperatures. All 72 specimens heated statically ignited and glowed; in contrast, only 96 out of 144 cubes subjected to the dynamic heating ignited and glowed. This would support the belief that slow heating may cause gradual dissipation of heat and mass.

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