

**DIFFERENTIAL SCANNING CALORIMETRY OF HYDROGEN PEROXIDE AND HYDROGEN PEROXIDE-TREATED LIGNOCELLULOSE. I. AMBIENT PRESSURE CONDITIONS \***

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**ABSTRACT**

The decomposition reaction of aqueous solutions of hydrogen peroxide was examined by DSC at ambient pressure, and the heat of evaporation of  $H_2O_2$  was determined. The reaction parameters for the oxidation reaction of lignocellulose (wood powder) with hydrogen peroxide were also examined. The pans made from gold and alodined (pyrophosphate/fluoride treated) aluminum were unsuitable for the work due to surface catalysis, in contrast to pure aluminum which proved to be acceptable within the temperature range examined. In pans made from pure aluminum, the reaction between lignocellulose and hydrogen peroxide takes place after the latter evaporates, i.e. it represents a gas—solid reaction.

**INTRODUCTION**

The study of molecular surface constitution and its modification toward a more optimum substrate for bonding is of practical importance for the production of glued products. This is particularly true for oxidative bonding methodology based as it is on the formation of covalent chemical bonds between the surfaces to be bonded.

Research at the University of California Forest Products Laboratory has been devoted for many years to the development of oxidative bonding systems for wood and derived lignocellulosic materials. Fortunately, lignocellulosic materials represent some of the most complex natural polymers, containing many chemical groupings, such as phenolic and alcoholic hydroxyls, carbonyl groups, etc., capable of being readily "activated" by physical and

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\* Content in part from Ph.D. dissertation of Dr. T. Nguyen. The work was performed at the Forest Products Laboratory.

chemical methods and providing the required active sites of acceptable longevity. These active sites can then react under pressure either directly with the active sites from the other surface to produce the desired covalent bonds holding the two surfaces together or indirectly in presence of added polymerizable monomers by forming polymer bridges between the two surfaces. Promising preliminary results have been reported by the University of California group [1-8]. According to the recommended procedures, lignocellulose (wood particles) was treated with specific oxidative activators and pressed in the presence or absence of a polymerizable crosslinking agent, such as furfuryl alcohol. The activators which produced products with the best physical and mechanical properties included hydrogen peroxide,  $H_2O_2$ , nitric acid,  $HNO_3$ , and chlorates,  $ClO_3^-$ .

The present series of studies represents an effort to clarify the chemistry of surface activation of lignocellulosic materials by hydrogen peroxide; hopefully this will be followed by research involving other types of activators. The clarification of the chemical transformations on the surface should be of help in the formulation of practical procedures for the manufacture of glued lignocellulose products.

Due to several advantages such as simplicity of operation, small sample weight, fast and objective results, versatility, etc., thermal analysis has recently attained a position of prominence, particularly in the applications to fibers, plastics and other polymeric materials. One of the most commonly used techniques is differential scanning calorimetry, DSC, yielding curves that are unique for a particular chemical composition or configuration which show small changes in chemical structure associated with changes in enthalpy. Understanding thermal behavior of hydrogen peroxide and hydrogen peroxide-treated lignocellulose under the conditions of thermal analysis could thus provide valuable information on the behavior of these materials under the hot pressing conditions used for forming the composite product.

Due to their explosive characteristics, hydrogen peroxide and derivatives have been used as propellants. Quantitative and qualitative studies of other propellants by differential thermal analysis, DTA, and differential scanning calorimetry, DSC, have been extensively made [9-14] and thermal analysis of organic peroxides has also received some attention [15,16]. In fact, Mair and Hall [17] recommended the use of thermal analysis for quantitative analysis, characterization and thermal decomposition studies of peroxides. To the best of our knowledge, there have been no studies of hydrogen peroxide itself using thermal analysis. This may be attributed to the sensitivity of hydrogen peroxide to the surface nature of sample holders and to impurities, resulting in irreproducible results. Still another reason could be connected with the volatility of water, making difficult the quantitative studies of the decomposition of aqueous hydrogen peroxide solutions at ambient pressure as the evaporation endotherm of water obscures the decomposition exotherm of hydrogen peroxide.

In this paper, we report on the differential scanning calorimetry analysis at ambient temperature of hydrogen peroxide and of mixtures of hydrogen peroxide and lignocellulosic materials.

## EXPERIMENTAL

### *Materials*

Hydrogen peroxide solution used was ACS reagent grade of 50 wt.% concentration ("Perone 50")\*. The lignocellulose used represented alcohol and hot water extracted white fir (*Abies concolor*) 60–80 mesh wood flour, extracted for 12 h in 95% ethanol and 12 h in boiling water. Before use, the wood was immersed overnight in 5% ethylenediamine tetraacetic acid disodium salt (EDTA) solution to remove catalytically active metal ions either pre-existing or resulting from machining and grinding of the wood. The EDTA-treated wood was washed well with distilled water, filtered and dried under vacuum at 75°C.

### *Sample preparation*

One part of 50% H<sub>2</sub>O<sub>2</sub> was mixed with one part of lignocellulosic material (oven-dry weight) in a pyrex weighing container and stored under closed conditions for 8 h to prevent evaporation and to attain thorough penetration of the aqueous H<sub>2</sub>O<sub>2</sub> into the lignocellulose particles. In order to eliminate the potential catalytic effect of glass on H<sub>2</sub>O<sub>2</sub> decomposition and possibly on the reaction between lignocellulose and H<sub>2</sub>O<sub>2</sub>, and to produce a consistent glass surface, the weighing container was treated as follows: (1) soaked in boiling 15% HNO<sub>3</sub> for 15 min, (2) washed with distilled water, (3) immersed in boiling 50% H<sub>2</sub>O<sub>2</sub> for 30 min, (4) washed well with boiling distilled water, and (5) vacuum dried until use. The glass capillary tubes used to transfer the H<sub>2</sub>O<sub>2</sub> solution from the container to the sample holder were treated in the same manner.

### *Thermal analysis*

A DuPont standard DSC cell attached to a cell base connected directly to the DuPont 990 Thermal Analyzer was used for this study. The theory and operational characteristics of DuPont standard DSC cell have been described by Barter [18].

Lightly pressed samples weighing between 1.2 and 3.0 mg were hermetically sealed by a DuPont sample encapsulating press. The equilibration of H<sub>2</sub>O<sub>2</sub> liquid and vapor phases was obtained by providing a pin hole on the top of the sample holder. Pin holes of constant size between 0.05 and 0.065 mm were produced in the sample holder lids by a punching device. For a DuPont DSC unit, three encapsulating types of sample holder are currently available. They are hermetic pure aluminum, hermetic alodined aluminum (aluminum etched with pyrophosphate/fluoride), and hermetic gold pans. Each kind was used in this study. The cell was purged with N<sub>2</sub> gas at a flow rate of 50 ml min<sup>-1</sup>; the heating rate was 20°C min<sup>-1</sup>. An empty sample holder was used as a reference.

\* Courtesy of DuPont de Nemours and Co.

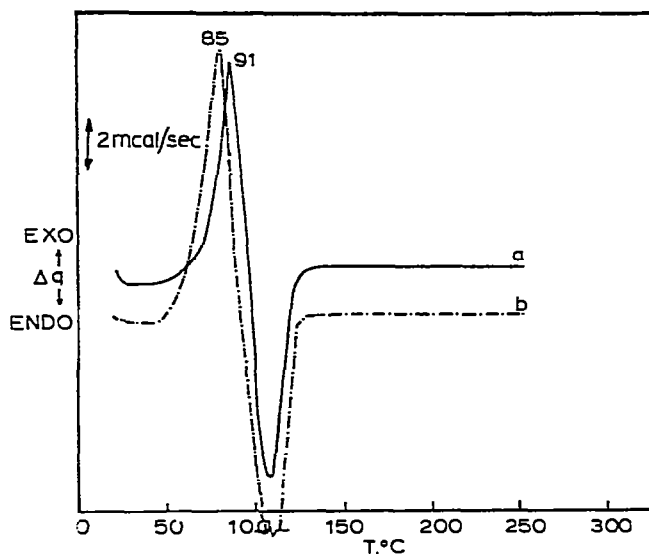


Fig. 1. DSC curves of  $\text{H}_2\text{O}_2$  50 wt.% in different types of sample pan. Heating rate:  $20^\circ\text{C min}^{-1}$ ; atmosphere:  $\text{N}_2$  at ambient pressure; sample size: 1.76 mg. a, Pin hole alodined aluminum pan (pyrophosphate/fluoride etched); b, pin hole gold pan.

## RESULTS AND DISCUSSION

### *DSC of hydrogen peroxide solution in different types of sample holder*

DSC curves of 50 wt.% hydrogen peroxide in three different sample holders are shown in Figs. 1 and 2. Drastically different thermal behaviors were observed. DSC curves of  $\text{H}_2\text{O}_2$  in pin hole alodined aluminum (Fig. 1a) and gold (Fig. 1b) sample holders \* follow the same pattern and exhibit an exotherm followed by an endotherm. The exotherm is believed to be catalytic decomposition of 50%  $\text{H}_2\text{O}_2$  while the endotherm is the evaporation of water. The catalytic effect due to the nature of the sample holder on the decomposition of  $\text{H}_2\text{O}_2$  can be quantitatively characterized by the respective decomposition onset temperature,  $T_0$ , (point of intersection of the baseline with the tangent at the inflection point) and the peak temperature,  $T_1$ . Both  $T_0$  and  $T_1$  of  $\text{H}_2\text{O}_2$  in the gold pan ( $57^\circ\text{C}$  and  $85^\circ\text{C}$ ) are somewhat lower than those of  $\text{H}_2\text{O}_2$  run in the alodined pan ( $65^\circ\text{C}$  and  $91^\circ\text{C}$ ). Gold has been known for some time to act as a heterogeneous catalyst for  $\text{H}_2\text{O}_2$  decomposition as do other noble metals such as platinum, palladium, etc. [19].

The DSC curve of 50 wt.%  $\text{H}_2\text{O}_2$  in the pin hole plain aluminum pan (aluminum without etching with pyrophosphate/fluoride) (Fig. 2a) shows only one endotherm, with onset at  $113.5^\circ\text{C}$  and peak at  $135^\circ\text{C}$ . The endotherm corresponds to the boiling point of hydrogen peroxide at 50% solution. The boiling point which corresponds to the onset temperature ( $T_0$ ) [20],  $113.5^\circ\text{C}$ , is in excellent agreement with the  $114.0^\circ\text{C}$  value obtained by

\* Hermetic sample holders which were sealed and provided with a pin hole on the cover.

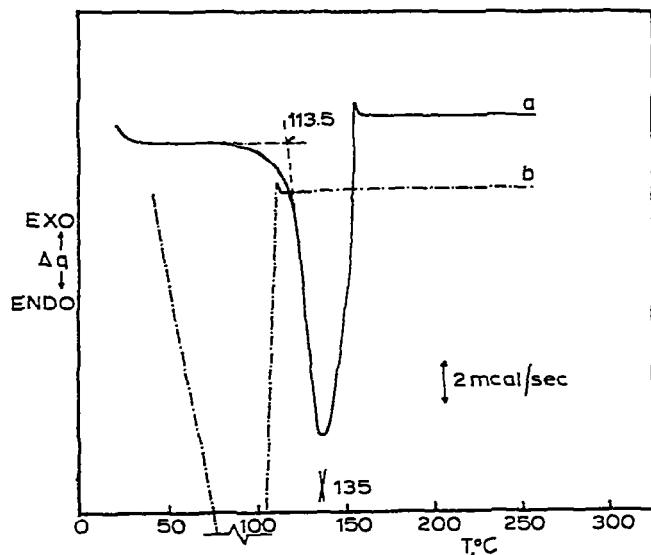


Fig. 2. DSC curves of  $\text{H}_2\text{O}_2$  50 wt.% in hermetic, plain aluminum pan. Heating rate:  $20^\circ\text{C min}^{-1}$ ; atmosphere:  $\text{N}_2$  at ambient pressure; sample size: 1.80 mg. a, Sealed, with a pin hole on top (0.055–0.065 mm); b, open.

interpolation of vapor pressure–composition data for various temperatures [21] and the  $113.9^\circ\text{C}$  value reported by Manufacturing Chemists Association [22].

Based on the data in Fig. 3, a heat of evaporation ( $\Delta H_v$ ) of  $440.6 \text{ cal g}^{-1}$

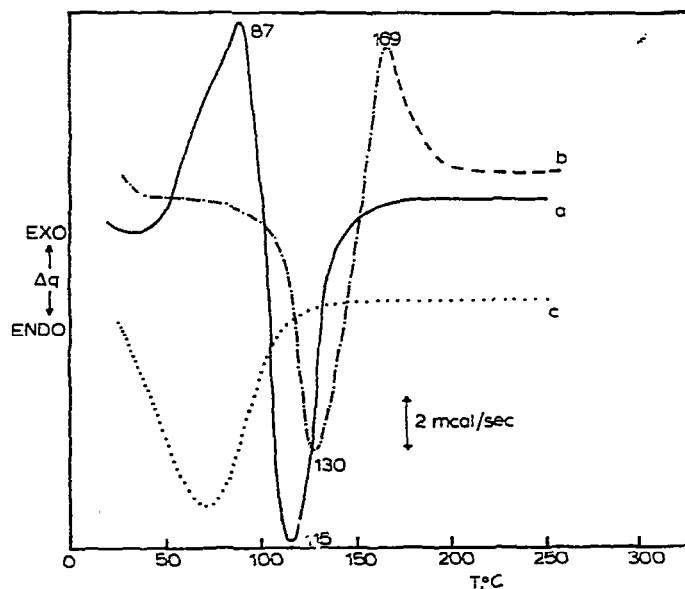


Fig. 3. DSC curves of  $\text{H}_2\text{O}_2$ -treated wood. Heating rate:  $20^\circ\text{C min}^{-1}$ ; atmosphere:  $\text{N}_2$  at ambient pressure; sample size: 3 mg. a, Pin hole alodined aluminum pan; b, pin hole plain aluminum pan; c, open plain aluminum pan.

for 50% H<sub>2</sub>O<sub>2</sub> in H<sub>2</sub>O was calculated from the equation [23]

$$\Delta H_v = \frac{A}{m} K$$

where  $A$  is peak area (cm<sup>2</sup>),  $m$  the sample mass (mg),  $K$  the calibration coefficient and  $\Delta H_v$  is expressed in mcal mg<sup>-1</sup>.

For the DuPont DSC cell [24]

$$K = \frac{60 BE \Delta qs}{6.452}$$

where  $B$  is the time base setting (min/cm.),  $E$  the cell calibration coefficient at the temperature of the experiment and  $\Delta qs = Y$ -axis range (mcal sec<sup>-1</sup> cm<sup>-1</sup>).

The above  $\Delta H_v$  value for 50% H<sub>2</sub>O<sub>2</sub> in H<sub>2</sub>O was the average of two values and was calculated using  $B = 47.2$  sec cm<sup>-1</sup>,  $\Delta qs = 0.787$  mcal sec<sup>-1</sup> cm<sup>-1</sup>, and  $E = 1.01$  (for indium). This  $\Delta H_v$  value is in a good agreement with the 444.5 cal g<sup>-1</sup> value obtained from expression of Giguère et al. [25], viz.

$$\Delta_m H_{vap} (T^\circ C) = \Delta_m H_{vap} (26.9^\circ C) - D_m C_p \Delta T$$

where  $D_m C_p = N_w C_p$  (H<sub>2</sub>O, gas) +  $N_p C_p$  (H<sub>2</sub>O<sub>2</sub>, gas) -  $C_p$  (solution),  $D_m C_p$  (50wt.% H<sub>2</sub>O<sub>2</sub>) = 9.9 cal deg<sup>-1</sup> mole<sup>-1</sup> (solution),  $C_p$  is the heat capacity in cal deg<sup>-1</sup> mole<sup>-1</sup> (solution),  $N_w$  and  $N_p$  are the mole fractions of water and hydrogen peroxide, respectively, and  $\Delta H_{vap} (26.9^\circ C) = 11.332$  kcal mole<sup>-1</sup> (50 wt.% H<sub>2</sub>O<sub>2</sub>) [25].

It should be noted that 50 wt.% H<sub>2</sub>O<sub>2</sub> is equivalent to 0.3462 mole fraction of H<sub>2</sub>O<sub>2</sub> according to the expression [26]

$$\frac{M_w}{M_h} = \frac{(1-w)(X_h)}{(1-X_h)(w)}$$

where  $M_w$ ,  $M_h$  are the molecular weights of H<sub>2</sub>O and H<sub>2</sub>O<sub>2</sub>, respectively and  $X_h$  and  $w$  are the mole and weight fractions of H<sub>2</sub>O<sub>2</sub>, respectively.

The difference in shapes of the DSC endotherms of H<sub>2</sub>O<sub>2</sub> solution during evaporation from pin hole plain aluminum and open plain aluminum pans (Fig. 2a and b) indicates that the rate of evaporation of aqueous H<sub>2</sub>O<sub>2</sub> is controlled by the size of the pin hole through which the vapor escapes to the surroundings. The best results were obtained by determining the boiling point and heat of evaporation of H<sub>2</sub>O<sub>2</sub> by DSC in the hermetically sealed plain aluminum pan with a 0.05–0.065 mm pin hole. A sealed pan with a pin hole was previously recommended by Barrall [20] to determine the boiling point and heat of evaporation of a liquid at various pressures. Obviously, in the case of H<sub>2</sub>O<sub>2</sub>, both the hermetic gold and alodined aluminum pans are unsuitable for that purpose due to their catalytic effect on the decomposition of aqueous H<sub>2</sub>O<sub>2</sub> with the resulting endotherm arising from the evaporation of water rather than hydrogen peroxide solution.

The disappearance of the decomposition exotherms in the DSC curves for H<sub>2</sub>O<sub>2</sub> in pin hole plain aluminum pans (Fig. 2a and b) indicates that the boiling point of aqueous H<sub>2</sub>O<sub>2</sub> is reached before the decomposition takes place and points to the particularly inert surface of the plain aluminum.

In fact, Schumb et al. [27] determined that hydrogen peroxide can withstand long-time contact with 99.6% or higher purity aluminum alloys.

*DSC of aqueous hydrogen peroxide-treated wood in different types of sample holder*

DSC curves of white fir flour treated with 50 wt.%  $H_2O_2$  in pin hole alodined aluminum, plain aluminum and unencapsulated plain aluminum (open) sample holders (all are "hermetic type") are shown in Fig. 3a, b and c, respectively. The thermal behavior of  $H_2O_2$ -treated wood in pin hole alodined aluminum pan follows the same pattern as that observed for  $H_2O_2$  alone, an exotherm followed by an endotherm. The exotherm is probably due to the simultaneous reactions of heterogeneous catalytic decomposition of  $H_2O_2$  and catalytically induced oxidation of wood by  $H_2O_2$ . The DSC of  $H_2O_2$ -treated wood in the pin hole plain aluminum pan (Fig. 3b) shows first an endotherm corresponding to the evaporation of hydrogen peroxide followed by a major exotherm peaking at 167°C. Thus, there is a marked difference between the DSC curves for  $H_2O_2$ -treated wood and  $H_2O_2$  alone under the same conditions, as in the latter case the DSC curve of  $H_2O_2$  exhibited no exotherm following the endotherm (Fig. 2a). In the case of an open pan (Fig. 3c), the DSC pattern of  $H_2O_2$ -treated wood is very similar to that of  $H_2O_2$  alone, both exhibiting only one endotherm. These results suggest that, in the sealed pan with a pin hole, the  $H_2O_2$  vapor remains largely within the pan escaping very slowly through the pin hole, and reacts exothermically with the wood substance, while in the open system, the  $H_2O_2$  vapor evaporates from the wood surface and escapes completely. In the open system, therefore, either no reaction between wood and  $H_2O_2$  takes place, or the amount of the residual  $H_2O_2$  is so small that its exotherm is completely overlapped by the strong endotherm due to evaporation of  $H_2O_2$ .

From the standpoint of oxidative bonding, the observation that  $H_2O_2$  reacts with wood in a closed, but not in an open system and in a vapor state is important and could explain why the edge areas of a particle board produced by using hydrogen peroxide as an oxidant always show poor bonding strength and water resistance properties [6]. While the interior particles react to the fullest possible extent with  $H_2O_2$  vapors, the particles in the edge areas receive much less hydrogen peroxide vapor due to its escape. The above observation may also explain why water resistance properties of low-density  $H_2O_2$  oxidatively bonded particle board ( $0.65 \text{ g cm}^{-3}$  or lower) are generally poorer than those of nitric acid oxidatively bonded and phenolformaldehyde glue bonded particle boards [8]. Low density implies greater void volume which allows a greater amount of  $H_2O_2$  vapor to escape than in the case of the higher density board. The great variation in properties of bonded laminates, using peracetic acid as an oxidant, reported by Johns and Nguyen [3] may also be explained by the above observation. In these types of board, the surface unevenness of veneers or laminates can enhance the loss of hydrogen peroxide vapor. The DSC observation that  $H_2O_2$  reacts with wood in the vapor phase also suggests the mode whereby hydrogen peroxide reacts with wood in the press. Wood particles apparently do not react with liquid

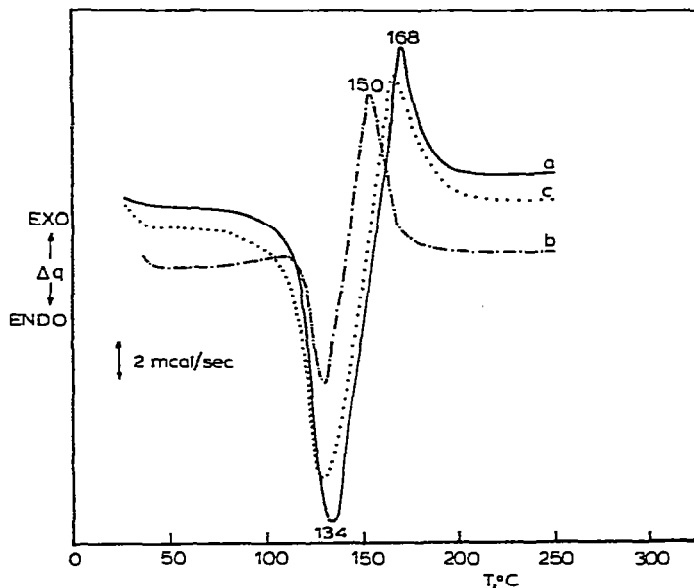


Fig. 4. DSC curves of  $\text{H}_2\text{O}_2$ -treated wood in pin hole aluminum pans. Heating rate:  $20^\circ\text{C min}^{-1}$ ; atmosphere:  $\text{N}_2$  at ambient pressure; sample size: 1.76 mg. a, Alodined, treated with boiling 15%  $\text{HNO}_3$  and 50 wt.%  $\text{H}_2\text{O}_2$ ; b, alodined, interior surface covered with aluminum foil; c, plain aluminum, no treatment.

$\text{H}_2\text{O}_2$  attached to them during spraying, but react with the  $\text{H}_2\text{O}_2$  vapor produced from liquid  $\text{H}_2\text{O}_2$  during hot pressing. Finally, the  $\text{H}_2\text{O}_2$  vapor contains more energy than its liquid counterpart and for that reason should be more reactive towards wood than liquid hydrogen peroxide. For all these

TABLE 1

DSC characteristics of  $\text{H}_2\text{O}_2$  50 wt.% and  $\text{H}_2\text{O}_2$ -treated wood at different conditions and sample holders at ambient pressure.  
Heating rate:  $20^\circ\text{C min}^{-1}$ ; gas:  $\text{N}_2$

Sample holder type and conditions	Exotherm peak temp. ( $T_1$ °C)	Endotherm peak temp. (°C)
<i>H<sub>2</sub>O<sub>2</sub></i>		
Pin hole hermetic gold pan	85	104 ( $\text{H}_2\text{O}$ )
Pin hole hermetic alodined aluminium pan	91	105 ( $\text{H}_2\text{O}$ )
Pin hole hermetic plain aluminium pan		135 ( $\text{H}_2\text{O}_2 + \text{H}_2\text{O}$ )
Open hermetic plain aluminium pan		85 ( $\text{H}_2\text{O}_2 + \text{H}_2\text{O}$ )
<i>H<sub>2</sub>O<sub>2</sub>-treated wood</i>		
Pin hole hermetic alodined aluminium pan	87	115 ( $\text{H}_2\text{O}$ )
Pin hole hermetic plain aluminium pan	169	130 ( $\text{H}_2\text{O}_2 + \text{H}_2\text{O}$ )
Open hermetic plain aluminium pan		70 ( $\text{H}_2\text{O}_2 + \text{H}_2\text{O}$ )
Pin hole hermetic alodined aluminium pan (a) covered with aluminium foil	150	125 ( $\text{H}_2\text{O}_2 + \text{H}_2\text{O}$ )
(b) treated with boiling 15% $\text{HNO}_3$ and 50% $\text{H}_2\text{O}_2$	168	134 ( $\text{H}_2\text{O}_2 + \text{H}_2\text{O}$ )



reasons the vapor—solid interaction is likely to represent the predominant pathway for the reaction between hydrogen peroxide and wood particles in a hot press.

Figure 4b represents a DSC curve of H<sub>2</sub>O<sub>2</sub>-treated wood in a pin hole alodined pan with its interior covered with aluminum foil, while Fig. 4a shows the DSC curve of the same sample pan treated first by immersing it in boiling 15% HNO<sub>3</sub> for 30 min, soaking it in boiling 50% H<sub>2</sub>O<sub>2</sub> for 1 h and finally washing it well with boiling distilled water. This method of treatment did not seem to affect the DSC behavior of H<sub>2</sub>O<sub>2</sub>-treated wood in a pin hole plain aluminum pan (compare Fig. 4d with Fig. 4c). The DSC of H<sub>2</sub>O<sub>2</sub>-treated wood in a crimple-sealed aluminum pan also gave about the same pattern, although the endotherm started earlier and the exotherm peaked at a lower temperature. This supports the earlier suggestion that the alodine process using pyrophosphate/fluoride tends to catalyze the hydrogen peroxide decomposition and oxidation reactions. The DSC characteristics of H<sub>2</sub>O<sub>2</sub> and H<sub>2</sub>O<sub>2</sub>-treated wood at different sample holder conditions at ambient pressure are summarized in Table 1.

## CONCLUSIONS

Pure aluminum represents an acceptable pan material for thermal analytical studies of transformations of hydrogen peroxide aqueous solutions, alone or mixed with lignocellulosic materials. Both alodined aluminum (aluminum treated with pyrophosphate/fluoride) and gold exert a strong catalytic influence on the decomposition of hydrogen peroxide and are unsuitable for the work with this chemical. A hermetically sealed pan provided with a small pin hole in the cover proved to be the optimal container for studying the decomposition and oxidation reactions of hydrogen peroxide. The study indicated that, in strongly catalytic alodined aluminum or gold pans, hydrogen peroxide decomposes or reacts with admixed lignocellulose in the liquid phase, but reacts in the vapor phase in pure aluminum pans.

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