X-ray Photoelectron Spectroscopy for Characterization of Wood Surfaces in Adhesion Studies

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Abstract

X-ray photoelectron spectroscopy (XPS) is one of a set of tools that have been used to characterize wood surfaces. Among the advantages of XPS are surface sensitivity, identification of nearly all elements, and frequently, discrimination of bonding states. For these reasons, XPS seemed to be an appropriate tool to help explain the differences in bond strength under wet conditions for planed and unplaned acetylated wood bonded with epoxies. Some care needs to be taken to ensure correct analysis of the high-resolution XPS spectra. The XPS, in conjunction with labeling with trifluoroacetic anhydride, was used to characterize the extent of acetylation at cellulose and wood surfaces. These results led to the conclusion that the trifluoroacetic anhydride labeling was a useful method for characterizing reactable cellulosic hydroxyl groups and that planing the acetylated wood exposed additional unmodified hydroxyl groups.

Introduction

Understanding how adhesives interact with wood is limited by our understanding of these surfaces. Wood surfaces are very complex both physically and chemically (5). Wood surfaces to be bonded have much cellular debris on the surface from planing, but even a microtomed surface is complicated because of the complex cellular and ultrastructure of wood (6). Open cells and debris from surface planing limits the usefulness of some surface analysis techniques, especially on the sub-micrometer scale. The ultrastructure of the cell wall consists of four main cell wall layers and a middle lamella, and each of these is made of nanoscale domains of cellulose, hemicellulose, lignin, and other wood components. This complexity makes it difficult to understand the influence of wood's chemical and physical properties on bond performance.

Given the complexity of cellular and ultrastructure changes in comparing adhesion in different species, examining adhesion to unmodified and chemically modified wood is a way to alter the variables and enhance the chemical aspects of adhesion. Of the various ways that have been used to modify wood (10), acetylation is of interest for the following reasons:

- It does not greatly alter the bulk mechanical strength of wood and therefore should do little to alter the cell wall's physical construction.
- It alters surface characteristics by replacing most accessible hydroxyl groups with acetate groups and reducing the hydrogen bonding potential.
- The reaction can be controlled, as acetylation modifies a functional group but does not cause polymerization or crosslinking.

Although the adhesive bonding of acetylated wood is not new, many aspects are not well understood. Earlier studies indicated that poor adhesion of a phenol-formaldehyde (PF) adhesive was due to poor wetting (11), but two later studies did not support this concept. Youngquist et al. showed that improved wetting of PF adhesives did not provide improved bond strength (18). Vick and Rowell demonstrated that a waterborne PF adhesive pro-

vided durable bonds to acetylated wood (16). Vick and Rowell also showed that some waterborne adhesives gave bonds to acetylated wood of equal or better performance than to untreated wood, even though the overall trend was reduced adhesive bond strength upon acetylation. A recent study by Frihart et al. found that epoxies bonded better to unplaned acetylated wood than to planed acetylated and untreated wood (7). These data are contrary to the concept that acetylation should decrease adhesion to wood through reduced hydrogen bonding.

The current study has characterized specimens prepared for the Frihart et al. adhesion study (7). X-ray photoelectron spectroscopy (XPS) was used to quantify the free hydroxyl groups available at the wood surface. Because hydroxyl groups are difficult to quantify by XPS, the use of labeling with trifluroacetic anhydride was investigated.

When modeling adhesion, it is valuable to know what components become modified and, therefore, have altered interactions with the adhesives. The literature indicates that under typical wood acetylation conditions used for bonding experiments, lignin is modified most rapidly, with hemicellulose second in rate; cellulose was essentially not modified (9). The lack of any cellulose modification seemed surprising when the hot acetic anhydride reacts with the hemicellulose hydroxyl groups. To better understand if any modification was taking place (under these conditions), the reaction of both acetic anhydride and trifluoroacetic anhydride with cellulose filter paper using XPS analysis was examined.

XPS

XPS, also known as electron spectroscopy for chemical analysis (ESCA), involves irradiation of specimens with monochromatic x-rays that promote the removal of a core or valence electron. The escaping electron has a kinetic energy that is determined by the energy of the photon and the binding energy of the electron to an atom. The kinetic energy of the emitted electrons is measured by an electrostatic analyzer, and the numbers of electrons at each kinetic energy are counted.

XPS is an ultrahigh vacuum technique because any appreciable amount of gas would interact with either the x-ray irradiation or the emitted electrons. XPS is surface sensitive because only emitted electrons that do not suffer inelastic collisions contribute to the coherent spectrum. As a general rule, electrons that travel through more than 3 to 5 nm of solid material will lose energy through interactions with atoms in the solid. The inelastically scattered electrons contribute to the incoherent background of the detected signal.

The electron-binding energy depends on the element and its chemical state. For example, the binding energy for electrons originating from the 1s orbital of carbon is slightly different than that from the same orbital of carbons singly bonded to oxygen or carbons that are doubly bonded to oxygen. Survey spectra that use low-analyzer resolution are used to determine which elements are present, and high-resolution spectra are used to determine chemical state information.

XPS can be used to quantify the extent of acetylation near the wood surface. XPS cannot directly measure the free hydroxyl groups; however, vapor-state reaction of trifluoroacetic anhydride with hydroxyl groups can provide an indirect means of quantification. In both cases of chemical modification, the information can be extracted from the fine structure of the carbon 1s electron emission spectrum. The advantage of this method is that it is not necessary to know the relative sensitivity of oxygen or fluorine to that of carbon. Thus, differences in amounts of hydroxyl groups near the surface have been measured for the untreated wood and planed and unplaned acetylated wood samples.

Methods

Acetylation

Strips of pre-dried yellow-poplar (*Liriodendron tulipifera*) sapwood (0.6 by 3.2 by 20.3 cm) or Whatman #1 filter paper (1.3 by 7.6 cm) were placed in a glass reactor fitted with a reflux condenser. The reactor was filled with enough acetic anhydride to not only cover the strips at the initial filling but also to cover them after absorption of chemical. The acetic anhydride and wood was heated to boiling for 4 hours and then cooled. Strips were removed, washed for 4 hours in reversed osmosis water, air-dried overnight, and then ovendried for 24 hours at 105°C. Weight gain from acetylation was determined after ovendrying by calculation as a percent of the original ovendried weight.

Planing

Wood is often resurfaced just before bonding to remove any raised grain areas and any extractives. This seems logical for unmodified wood, but planing acetylated wood might not provide a fully acetylated surface in that the fracture of the cell walls may expose unmodified hydroxyl groups. Approximately 1.6 mm of the outer surface is normally removed by planing.

Trifluoroacetylation

Squares (~ 1.2 by 1.2 cm) were cut from strips of acetylated and untreated Whatman #1 filter paper. These were placed on aluminum foil in a 50-ml weighing bottle along with a 2-ml beaker containing 0.5 ml of trifluoroacetic anhydride (TFA). These were allowed to react for 1 hour at room temperature. The weighing bottle was partly opened in a fume hood to vent the volatiles. The treated and untreated paper were placed in folds of aluminum foil and dried by evacuating overnight at 10^{-5} Pa.

One of the advantages of using TFA to label hydroxyl groups by this method is that no exposure to liquids is necessary, and the wood is not saturated with excess reactants that would need to be removed (3). The TFA is very volatile, with a boiling of point 62°C. The reaction product trifluoroacetic acid (boiling point 72°C) is also volatile, compared with 139°C for acetic anhydride and 118°C for acetic acid. Thus, the excess materials can be removed without washing. Most of the specimens were reacted with TFA vapor; however, some acetylated paper specimens were treated by immersion in TFA liquid to determine if higher conversions were obtained. The same procedure was used except that the specimens were placed in the beaker containing the liquid.

Portions from the outer surface of the acetylated and untreated yellow-poplar specimens were isolated. These portions were about 1 mm thick and about 1 cm square to reduce the volume to facilitate drying. Some of these specimens were treated with TFA in the same way as the paper specimens.

XPS

X-ray photoelectron spectra were obtained using the Perkin Elmer 5400 ESCA spectrometer (Perkin Elmer, Wellesley, MA) at the University of Wisconsin's Material Science Center in Madison, Wisconsin. This instrument is equipped with a twin anode x-ray source and uses a hemispherical electron energy analyzer.

Small specimens (1 cm^2) about 1 mm in thickness were mounted on a stainless steel stub using double-coated conductive tape. The XPS is conducted in an ultra-high vacuum environment (approximately 10^{-6} Pa). It is necessary to outgas the specimens and tape before introducing them into the analytical chamber; this requires about 20 to 30 minutes. Previous experience has shown that the major outgassing component is water, but other volatile components may be removed. To reduce the instrument time used for outgassing, specimens were dried overnight in a vacuum chamber pumped by a turbomolecular vacuum pump. These specimens were stored and transported to the XPS spectrometer in a small desiccator. Specimens were protected from contamination by aluminum foil.

Spectra were obtained using a Mg K_a x-ray source energized to 300 watts and a pass energy of 35 eV for the electron analyzer. The x-ray source window was about 1 cm away from the specimen surface. Changes in source specimen distance, orientation of specimen with the grain parallel or perpendicular to the source, or source power showed no effect as determined by the emission curve on wood, epoxy, or paper. However, fluorocarbons are known and observed to degrade under this condition because of exposure to electrons generated by x-rays passing through the aluminum window of the x-ray gun (17); thus, the exposure times were kept short and the source power was low.

Curve Fitting

The data from each of the high-resolution spectrum were manipulated in the same way using AugerScan software (RBD Enterprises, Bend, OR). First, the contributions from the electron emission stimulated by the 3,4 components of the Mg K_a x-ray emission were subtracted. Points were manually selected to represent a baseline, and a non-linear background (14) was subtracted from each spectrum. These are all conventional practices (15).

Each of the high-resolution spectra was fitted with Gaussian-Lorentzian components to characterize the surface chemistry. It is important that the fitting process be guided by the relevant physics and chemistry or the information may lose objectivity. Peak width and peak shape are determined by the spectrometer and x-ray source characteristics (13). We used values determined empirically from fitting a spectrum of cellulose. The relative binding energies for components should be determined from spectra of appropriate materials and not just selected to give good fit to the experimental spectra. The parameters used to describe different carbon species were derived from high-resolution spectra using a Scienta ESCA300 spectrometer (Gammadata Scienta AB, Uppsala, Sweden), which has extraordinary resolution, obtained from well characterized pure materials (1). It is also desirable to minimize the number of adjustable parameters.

Spectra were obtained from oxygen 1s electrons and fluorine 1s electrons when these atoms were present. Only carbon 1s spectra, however, were used to quantify the surface acetylation chemistry. Fortunately for these studies, there is sufficient resolution to determine the needed information. One advantage is that we need not be concerned with relative response of the electron multiplier to electrons of different energy (electrons from fluorine or oxygen, etc.). Another advantage is that we need not be concerned that spectra of different elements sample different depths of material. Because of their different kinetic energies, electrons from other elements have different escape depths (related to the distance they travel before an inelastic encounter) (8).

An example of the fitting process is illustrated for the simple case of unmodified cellulose. The high-resolution carbon 1s XPS spectrum of cellulose is shown in **Figure 1**, where the number of electrons measured versus binding energy is plotted. This spectrum is resolved into five Gaussian-Lorentzian components. The most intense component centered about 290 eV is attributed to electrons from carbon atoms that are bonded to oxygen atoms. Another component located 1.33 eV higher binding energy is attributed to carbon atoms bonded to two oxy-

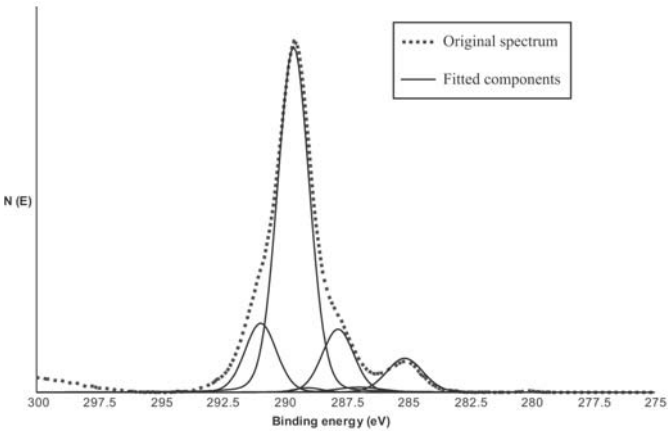

Figure 1. ~ *Carbon 1s spectrum of cellulose; the number of electrons measured (N (E)) plotted vs. binding energy (eV).*

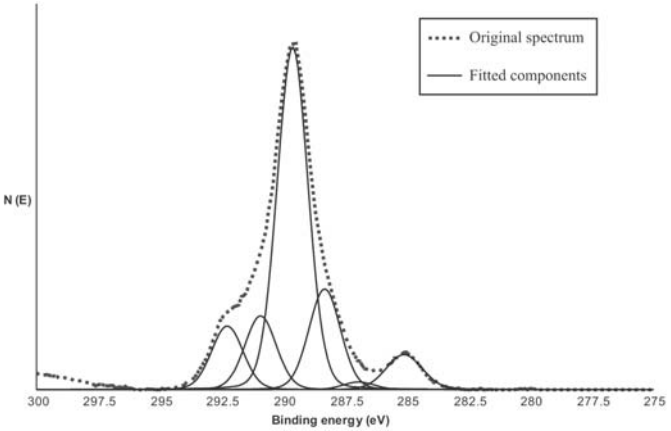

Figure 2. ~ *Carbon 1s spectrum of acetylated cellulose; the number of electrons measured (N (E)) plotted vs. binding energy (eV).*

gen atoms. In this analysis, the intensity of the higher binding energy component is fixed at 20 percent of the more intense component because this is the stoichiometry appropriate for these species in cellulose.

The small component near 288 eV is attributed to electrons emitted from hydrocarbon-like carbon atoms. Cellulose has none of these, but cellulose is a relatively high-surface energy material and thus attracts hydrocarbon contamination.

Another small component appears near 285 eV; this is attributed to hydrocarbon-like carbon on the stainless steel specimen stub. Electrons from this hydrocarbon source are distinguished from those contaminating the cellulose by conductivity. The cellulose (and contamination attached to cellulose) becomes positively charged because of the emission of electrons. This charge cannot be quickly neutralized because of the poor conductivity of cellulose. Electrons emitted from this material have a different reference state (ground potential) than electrons from hydrocarbon-like material on the conductive metal stub.

A separate spectrum was obtained from the contamination on the metal stub. This spectrum has components caused by other carbons species in addition to the hydrocarbon-like component. A scaled portion of this spectrum was fitted to each specimen spectrum to account for these contributions.

Degradation of Fluorocarbons by Electrons

Most molecular bonds are robust under exposure to low energy x-rays and electrons; however, halocarbons are known to degrade under exposure to electrons (17). Many electrons are generated by x-rays passing through the thin aluminum window which separates the x-ray source from the analytical chamber. These electrons flood the specimen and degrade the fluorocarbon bonds.

The kinetics of degradation were established by measurements of TFA-derivitized cellulose as a function of exposure time. The quantities reported here have all been adjusted to zero exposure time (17).

Results and Discussion

Acetylation of Cellulose

A high-resolution C1s spectrum for acetylated cellulose is shown in **Figure 2**. An additional component is near 292 eV in this spectrum because of the addition of carboxyl carbon – a carbon double-bonded to an oxygen and single-bonded to another oxygen. The addition of an acetate group also adds an equal contribution to the hydro-carbon-like component near 288 eV; this arises from the terminal methyl carbon.

We can determine the extent of acetylation by the contribution of the acetate components relative to the cellulose components. This process will be illustrated using the data shown in **Figure 2**. The component centered around 292 eV is caused by the carbonyl carbon in the acetate group; the methyl group of the acetate included in the component near 288 eV contributes equally. The acetate component areas are normalized by the contributions of the cellulose monomer near 289.6 eV and 291 eV. The contribution of the methyl group of the acetate is mixed with contributions from hydrocarbon contamination. Since this cannot be cleanly resolved, the contribution from the carbonyl carbon near 292 eV is doubled to represent the electron emission from all acetate carbons. This is shown in **Table 1** as 0.30 ± 0.01. If every available cellulose hydroxyl group were derivitized, the ratio would be 1.0. Thus, these data suggest that on average, about one out of three hydroxyl groups are modified by acetylation. Only the intensity (amplitude) is varied to fit the experimental spectrum; no other adjustable parameters

Table 1. ~ *Acetylation of cellulose.*

Treatment	Quantity	Value
Acetic anhydride	[Ac]/[Cellulose]	0.30 ± 0.01
Trifluroacetic anhydride	[TFA]/[Cellulose]	0.29 ± 0.01
TFA vapor > Acetylation	[TFA]/[Cellulose]	0.06 ± 0.01
TFA liquid > Acetylation	[TFA]/[Cellulose]	0.18 ± 0.03

are used in this fitting. All of the fitting is based upon an understanding of chemistry and physics using parameters obtained from very high-resolution spectra of well characterized materials.

Additional components are added to the XPS spectrum of trifluroacetylated cellulose (**Fig. 3**). When electrons are emitted from CF3, carbons signals are shifted to near 296 eV, and the electrons from the carboxyl carbon attached to CF3 are shifted to 293 eV. The extent of derivitization by TFA appears to be the same as that using acetic anhydride as shown in **Table 1**. This may or may not be surprising depending upon the reader's expectation. The situations are different. With TFA, the reactant is transported in the vapor phase and the reaction is at room temperature. The reaction with acetic anhydride is at high temperature in liquid phase. In both cases only the outermost portion of the specimen is probed by XPS. The depth sampled for a low atomic number material such as cellulose is about 5 nm (8).

If acetylated cellulose samples are subsequently treated with TFA vapor, there is some further derivitization. This is shown as the third treatment in **Table 1**. Somewhat greater reaction (0.18) is found if acetylated cellulose is treated with TFA in liquid state as shown by the fourth treatment in **Table 1**.

The TFA addition may occur by two mechanisms in acetylated cellulose: addition at unreacted OH sites and displacement of acetate groups. Fitting of high-resolution XPS spectra does not suggest any change in acetate composition after TFA reaction. This implies that new reactive sites have been modified. In the vapor phase, this could be a slow process transport depends on the mobility of macromolecules to expose unreacted hydroxyl groups. It does suggest that there is molecular mobility in the solid state and some possibility of rearrangement at least near the surface. This is somewhat analogous to the "fibrous acetylation process" described by Sassi and Chanzy, where diluents were added to the reaction medium (12).

In the cases where the TFA reaction occurred in the liquid state, the acetylated cellulose macromolecules at the surface likely are dissolved partly or swollen by the liquid. This would reveal unreacted OH sites. This is similar to the "homogeneous process" reported by Sassi and Chanzy (12). The studies suggested that acetylated cellulose polymers migrate away from cellulose fibrils to expose unreacted hydroxyl groups.

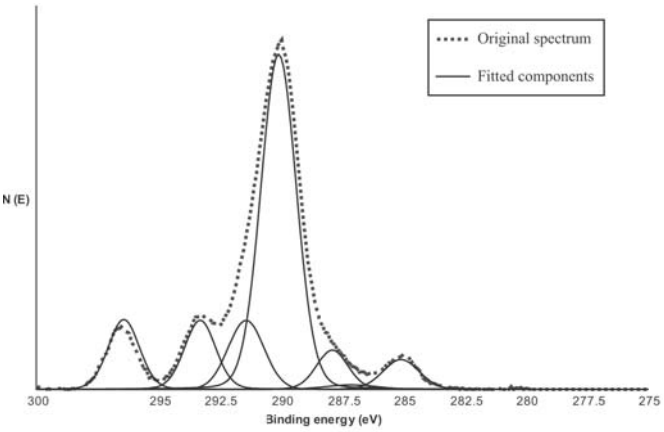

Figure 3. ~ *Carbon 1s spectrum of trifluoroacetylated cellulose; the number of electrons measured (N (E)) plotted vs. binding energy (eV).*

The crystalline portion of cellulose fibrils that is reinforced by hydrogen bonding resists the transport of even small water molecules. The accessibility to water molecules measured by nuclear magnetic resonance (NMR) is similar to our observations of extent of acetylation (2). Child and Jones measured the exchange of hydrogen for deuterium by NMR spectroscopy using deuterium oxide. For similar material (Whatman filter paper) at a similar time period (3 h), they observed that 32 percent of the sites were accessible to exchange.

Bulk methods (infrared and NMR spectroscopy) and XPS, which is very surface sensitive, agree well. The agreement is understandable when we realize that the 5-nm depth sampled by XPS is comparable to the width of an elementary cellulose fibril (4).

The results of this study and others (12) are in conflict with the observation that cellulose is resistant to acetylation (9). The reaction conditions used by Larsson et al. (acetic anhydride: xylene (1:1), 120°C, 1 to 16 h) were more vigorous than those employed by Sassi and Chanzy (acetic acid, acetic anhydride (~1:1), 60°C, trace of sulfuric acid, but somewhat lower temperature than we used, acetic anhydride, 140°C, 4 h). Previous studies offered no explanation for the absence of reaction (9), and we uncovered no further insight.

Acetylation of Wood

The vapor phase reaction of TFA with yellow-poplar sapwood was used to estimate the number of hydroxyl groups available near the surface. One of the advantages of this procedure is that no solvent or washing is involved. Three different specimens were examined: poplar, acetylated poplar, and acetylated poplar after planing. These specimens were from the same preparation used for adhe-

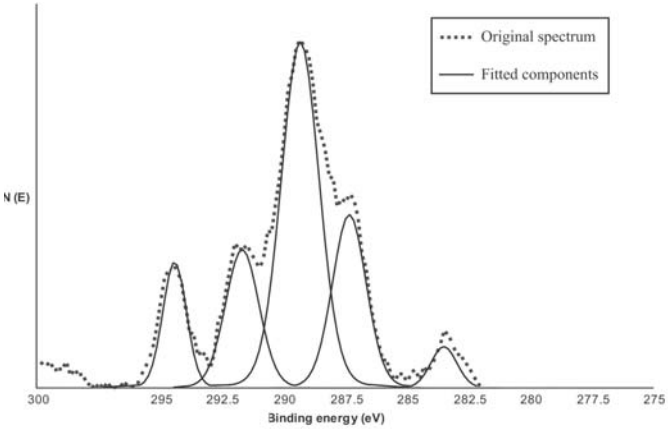

Figure 4. ~ *Carbon 1s spectrum of yellow-poplar treated with TFA; the number of electrons measured (N (E)) plotted vs. binding energy (eV).*

sion studies (7). A representative spectrum is shown in **Figure 4**.

We used a treatment of XPS data similar to that described above for cellulose Because of hydrocarbons on the specimen holder, however, the CF_3 component is normalized by the entire C1s emission excluding the small component. In this case, lignin contributes to the C1s spectrum, and the contribution from hemicellulose is very similar to that from cellulose. The major lignin contribution appears at the hydrocarbon-like component. Fortunately the CF_3 component is well isolated from any other contributions. The results are given in **Table 2**.

For the poplar specimens, about 13 percent of the carbon atoms near the outer surface (~ 5 nm) can be labeled by TFA. After acetylation, this is reduced to about 1 percent. After planing the acetylated poplar, a large number of reactive sites were uncovered – about 5 percent of the carbon atoms. This finding is consistent with the observations from the bond durability study. In bonding to acetylated wood, we found that the unplaned modified wood provided more durable bonds after water soaking than did the planed material (7). One hypothesis is that water absorption causes swelling of the surface and sufficient stress in the interphase region to fracture the bond. The additional hydroxyl groups on the surface of the planed acetylated wood could increase the swelling at the interface over that occurring with the unplaned acetylated wood.

Conclusions

The study of cellulose acetylation shows that cellulose is able to be acetylated by either reaction with acetic anhydride in liquid at elevated temperatures or trifluoroacetic anhydride as a vapor at room temperature. Only about a third of the hydroxyl groups, however, are available for modification. This is similar to previous observations concerning reaction or even accessibility to small molecules such as water.

Table 2. ~ *Trifluroacetylation of yellow-poplar.*

Specimen	$[CF_3]/[\Sigma$ carbon$]$
Yellow-poplar	0.13
Acetylated poplar	0.011
Acetylated poplar > planing	0.054

The acetylation of yellow-poplar sapwood in liquid acetic anhydride at elevated temperatures does not result in modification of all hydroxyl groups. Therefore, if the wood is planed after acetylation, additional unreacted hydroxyl sites are exposed at the surface. These previously unavailable sites can be labeled by trifluoroacetic anhydride.

The additional hydroxyl groups on the surface after planing of acetylated wood could explain why the unplaned acetylated wood gave more durable bonds to the epoxy than did the planed acetylated wood.

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