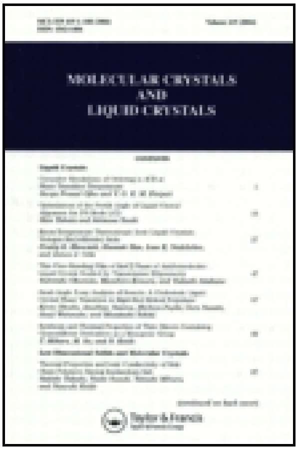

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# Acetylation of natural fibers to improve performance

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# ACETYLATION OF NATURAL FIBERS TO IMPROVE PERFORMANCE

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*One of the advantages of using natural fibers in composites is that they are biodegradable and renewable. Many applications for composites, however, require high performance and durability. While our attention is being drawn to renewable, biodegradable and sustainable resources for materials, we are faced with the concern about durability. For the most part, we have designed and used natural fiber-based composites accepting performance limitations such as dimensional instability, ultraviolet and biological degradation, and thermal instability. However, with a better understanding the relationship between chemistry, properties, and performance of natural fibers, we can produce a new generation of value added composites that will be performance driven and durable.*

*Properties such as dimensional instability, flammability, biodegradability, and degradation caused by acids, bases, and ultraviolet radiation are all a result of chemical degradation reactions (hydrolysis, oxidation, dehydration, and reduction) which can be prevented or, at least slowed down if the cell wall chemistry is altered. This approach is based on the premise that the properties of any resource are a result of the chemistry of the components of that resource. In the case of natural fibers, cell wall polymers, extractives, and inorganics are the components that, if modified, would change the properties of the resource. Based on performance requirements, chemical modifications can be carried out to change the chemistry of the resource that will result in a change in performance.*

*Many chemicals have been used to chemically modify natural fibers to improve performance including anhydrides, acid chlorides, ketene carboxylic acids, isocyanates, aldehydes, β-propiolactone, acrylonitrile, and epoxides [16–19,28]. After reaction, the natural fibers are made into a composite and the composite has greatly improved dimensional stability and biological resistance. Chemistries can also be selected to improve thermal resistance and resistance to UV radiation.*

*Keywords*: acetylation; biological resistance; chemical modification; dimensional stability; fungal resistance; moisture sorption; natural fiber

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## INTRODUCTION

Natural lignocellulosic fiber, like wood, were designed, after millions of years of evolution, to perform, in nature, in a wet environment and nature is programmed to recycle these resources, in a timely way, back to basic building blocks of carbon dioxide and water through biological, thermal, aqueous, photochemical, chemical, and mechanical degradations.

Wood composites, such as plywood, particleboard, chipboard, and fiberboard are generally sold as high volume, low performance products. We have used these composites for so long, as a building material, that we tend to accept there limitations in use. By accepting these failures and limitation, we also limit our expectations of performance, which, ultimately, limits our ability to accept new concepts for improved performance.

The properties of any resource are, in general, a result of the chemistry of the components of that resource. In the case of lignocellulosic fibers, the cell wall polymers (cellulose, hemicelluloses, and lignin) are the components that, if modified, would change the properties of the resource. If the properties of the lignocellulosics are modified, the performance of composites made using the modified fiber would be changed. This is the basis of chemical modification to change properties and improve performance of composites made from them.

## CHEMICAL MODIFICATION SYSTEMS

The term "chemical modification" has been used to mean different things by different authors over the years. For this discussion, chemical modification will be defined as a chemical reaction between some reactive part of a lignocellulosic and a simple single chemical reagent, with or without catalyst, to form a covalent bond between the two. This excludes all simple chemical impregnation treatments, which do not form covalent bonds, monomer impregnation that polymerize *in situ* but do not bond with the cell wall, polymer inclusions, coatings, hear treatments, etc.

In general, the chemicals to be laboratory tested must be capable of reacting with lignocellulosic hydroxyls under neutral, mildly alkaline or acid conditions at temperatures below 170°C. The chemical system should be simple and capable of swelling the structure to facilitate penetration. The complete molecule should react quickly with lignocellulosic components yielding stable chemical bonds, and the treated lignocellulosic must still possess the desirable properties of untreated lignocellulosics.

## CHEMICAL MODIFICATION FOR PROPERTY ENHANCEMENT

As was stated before, because the properties of lignocellulosics result from the chemistry of the cell wall components the basic properties of a lignocellulosic can be changed by modifying the basic chemistry of the cell wall polymers. Many chemical reaction systems have been published for the modification of agro-fiber and these systems have been reviewed in the literature several times in the past [4,10,16,17,19,23,28].

While there has been a lot of research on many different chemical reaction systems, the most research and interest, both in the past and the present, has been done on the reaction of acetic anhydride with cell wall polymer hydroxyl groups to give an acetylated lignocellulosic. Many different laboratories in the world have or are working in this area. For this reason, the acetylation of lignocellulosics will be the focus of this paper. To keep the paper short, only improvements in moisture sorption, dimensional stability and biological resistance will be covered and only one type of lignocellulosic fiber will be used as an example. Other performance improvements resulting from acetylation and other types of lignocellulliscs are covered in other literature [20,28].

## PROPERTIES OF ACETYLATED LIGNOCELLULOSICS

The reaction of acetic anhydride with a lignocellulosic results in esterification of the accessible hydroxyl groups in the cell wall with the formation of byproduct acetic acid.

$$\begin{gathered}\text{Cell Wall} - \mathrm{OH} + \mathrm{CH_3C({=}O) - O - C({=}O) - CH_3} \rightarrow \\ \text{Cell Wall} - \mathrm{O - C({=}O) - CH_3 + CH_3C({=}O) - OH}\end{gathered}$$

The preferred method of acetylating lignocellulosics today is to use a limited amount of liquid acetic anhydride without a catalyst or cosolvent and at reaction temperatures between 120 and 160°C [27]. The fact that only a limited quantity of acetic anhydride is used means that less chemical has to be heated during the reaction and less chemical has to be cleaned up after the reaction. A small amount of acetic acid seems to be needed in the reaction mixture to swell the cell wall initiate the reaction.

### Moisture Sorption

By replacing some of the hydroxyl groups on the cell wall polymers with bonded acetyl groups, the hygroscopicity of the lignocellulosic material is reduced. Table 1 shows the fiber saturation point for acetylated pine [19]. As the level of acetylation increases the fiber saturation point decreases.

Table 2 shows the equilibrium moisture content (EMC) of control and acetylated several types of lignocellulosic fibers at three levels of relative humidities. In all cases, as the level of acetyl weight gain increases, the EMC of the resulting wood goes down.

Figure 1 shows the adsorption-desorption isotherms for acetylated spruce fibers [29]. The 10 minute acetylation curve represents a WPG of 13.2 and the 4 hour curve represents a WPG of 19.2. The untreated spruce reaches an adsorption/desorption maximum at about 35% moisture content, the 13.2 WPG a maximum of about 30%, and the 19.2. WPG a maximum of about 10%. There is a very large difference between the adsorption and desorption curves for both the control and the 13.2 WPG fibers but much less difference in the 19.2 WPG fiber.

## Dimensional Stability

Changes in dimensions in tangential and radial direction in solid wood and in thickness and in linear expansion for composites are a great problem in lignocellulosic composites. In composites, they not only undergo normal

**TABLE 1** Fiber Saturation Point (FSP) for Acetylated Pine

| WPG | FSP (%) |
|---|---|
| 0 | 45 |
| 6 | 24 |
| 10.4 | 16 |
| 18.4 | 14 |
| 21.1 | 10 |

**TABLE 2** Equilibrium Moisture Content of Control and Acetylated Fibers

| Specimen | WPG | Equilibrium 30%RH | Moisture 65%RH | Content at 27°C 90%RH |
|---|---|---|---|---|
| Pine | 0 | 5.8 | 12.0 | 21.7 |
| | 6.0 | 4.1 | 9.2 | 17.5 |
| | 10.4 | 3.3 | 7.5 | 14.4 |
| | 14.8 | 2.8 | 6.0 | 11.6 |
| | 18.4 | 2.3 | 5.0 | 9.2 |
| | 20.4 | 2.4 | 4.3 | 8.4 |

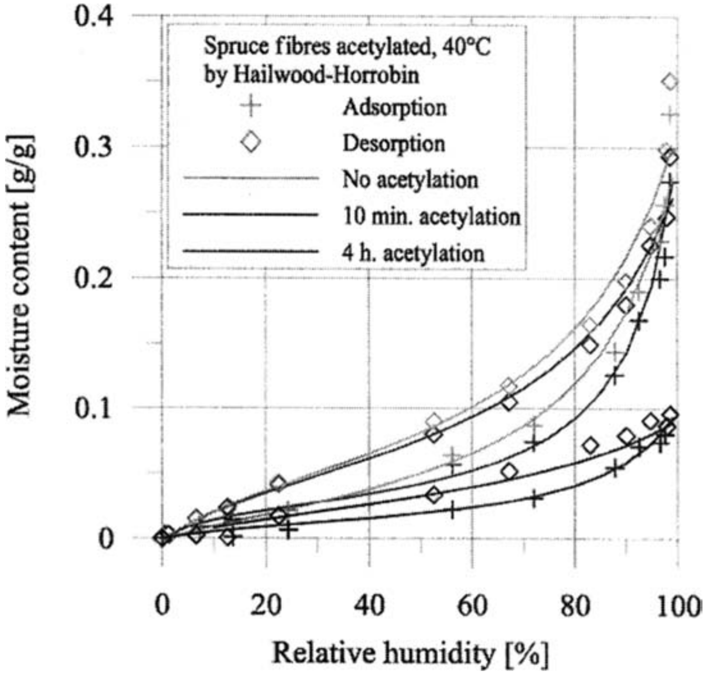

**FIGURE 1** Sorption/desorption isotherms for control and acetylated spruce fiber.

swelling (reversible swelling) but also swelling caused by the release of residual compressive stresses imparted to the board during the composite pressing process (irreversible swelling). Water sorption causes both reversible and irreversible swelling with some of the reversible shrinkage occurring when the board dries.

**TABLE 3** Thickness Swelling (TS) of Aspen Fiberboards made from Control and Acetylated Fiber

| WPG | Phenolic resin content (%) | 30% RH (%) | 65% RH (%) | 90% RH (%) |
|---|---|---|---|---|
| 0 | 5 | 0.7 | 3.0 | 12.6 |
| | 8 | 1.0 | 3.1 | 11.2 |
| | 12 | 0.8 | 2.5 | 9.7 |
| Acetylated (17.9 WPG) | 5 | 0.2 | 1.8 | 3.2 |
| | 8 | 0.2 | 1.7 | 3.1 |
| | 12 | 0.1 | 1.7 | 2.9 |

**TABLE 4** Rate and Extent of Thickness Swelling in Liquid Water of Pine Fiberboards made from Control and Acetylated Fiber (8% Phenolic Resin)

| | Percent thickness swelling at | | | | | | |
|---|---|---|---|---|---|---|---|
| | Minutes | | | Hours | | | Days |
| Fiberboard | 15 | 30 | 60 | 3 | 6 | 24 | 5 |
| Control | 25.7 | 29.8 | 33.5 | 33.8 | 34.0 | 34.0 | 36.2 |
| Acetylated (21.6 WPG) | 0.6 | 0.9 | 1.2 | 1.9 | 2.5 | 3.7 | 4.5 |

Thickness swelling at three levels of relative humidity is greatly reduced as a result of acetylation (Table 3). The rate and extent of thickness swelling in liquid water of fiberboards made from control and acetylated fiber is shown in Table 4. Both the rate and extent of swelling are greatly reduced as a result of acetylation. At the end of 5 days of water soaking, control boards swelled 36% whereas boards made from acetylated fiber swelled less than 5%. After drying the boards at the end of the test showed that control boards show a greater degree of irreversible swelling as compared to boards made from acetylated fiber.

Table 5 shows the volumetric swelling and antishrink efficiencies for control and acetylated pine solid wood. $S_1$ and $ASE_1$ are calculated from the wood going from oven dry to water soaked [22]. $S_2$ and $ASE_2$ are then calculated from the wood going from water soaked back to the oven dry state. It can be seen that the control specimens change volumes about the same from dry to wet and back to dry. The acetylated cycle have an antishrink efficiency of about 61% for the 16.3 WPG specimens and about 70% for the 22.5 WPG specimens. This data shows that the acetyl groups are very stable to swelling and shrinking cycles.

The results of both water vapor and liquid water tests show that acetylation of lignocellulosics greatly decreases moisture sorption and improves dimensional stability of both solid wood and composites.

**TABLE 5** Volumetric Swelling (S) and Antishrink Efficiency (ASE) of Acetylated Pine

| WPG | $S_1$ | $ASE_1$ | $S_2$ | $ASE_2$ | $S_3$ | $ASE_3$ | $S_4$ | $ASE_4$ |
|---|---|---|---|---|---|---|---|---|
| 0 | 13.8 | – | 13.3 | – | 13.6 | – | 13.3 | — |
| 16.3 | 5.1 | 63.0 | 5.1 | 61.7 | 5.3 | 61.0 | 5.3 | 60.2 |
| 22.5 | 4.1 | 70.3 | 3.8 | 71.4 | 4.0 | 70.6 | 4.1 | 69.2 |

## Biological Resistance

Particleboards and flakeboards made from acetylated pine flakes have been tested for resistance to several different types of organisms [1,7,12,21,24–26]. In a 2-week termite test using *Reticulitermes flavipes* (subterranean termites), boards acetylated at 16 to 17 WPG were very resistant to attack, but not completely so (Table 6). This may be attributed to the severity of the test. However, since termites can live on acetic acid and decompose cellulose to mainly acetic acid, perhaps it is not surprising that acetylated wood is not completely resistant to termite attack. The data shows that termite survival is quite high at the end of the test showing that the acetylated wood is not toxic to them.

Acetylated composites have been tested with decay fungi in several ways. Control and acetylated pine particleboards were exposed to a 12 week soil block test using the brown rot fungus *Gloeophyllum trabeum* the white rot fungus *Trametes versicolor* (Table 7). All the boards were

**TABLE 6** Weight Loss and Termite Survival in Acetylated Pine Flakeboards after 2 Weeks Exposure to *Reticulitermes flavipes* (5% Phenolic Resin)

| WPG | Wood weight Loss (g) | Final termite biomass[1] (g) |
|---|---|---|
| 0 | 0.13 | 0.61 |
| 8.7 | 0.10 | 0.62 |
| 11.5 | 0.07 | 0.63 |
| 13.6 | 0.08 | 0.60 |
| 16.3 | 0.08 | 0.63 |
| 17.6 | 0.06 | 0.54 |

[1] Starting weight, 1 g.

**TABLE 7** Resistance of Acetylated Pine Particleboard against Brown- and White-rot Fungi

| | Weight loss after 12 weeks | |
|---|---|---|
| WPG | Brown-rot fungus (%) | White-rot fungus (%) |
| 0 | 61.3 | 7.8 |
| 6.0 | 34.6 | 4.2 |
| 10.4 | 6.7 | 2.6 |
| 14.8 | 3.4 | <2 |
| 17.8 | <2 | <2 |

made using a phenolic resin [13,25]. All of the acetylated boards at a WPG over about 14 shows good resistance to brown- and white-rot.

Weight loss resulting from fungal attack is the method most used to determine the effectiveness if a preservative treatment to protect wood composites from decaying. In some cases, especially for brown-rot fungal attack, strength loss may be a more important measure of attack since large strength losses are known to occur in solid wood at every low wood weight loss [2]. A dynamic bending-creep test has been developed to determine strength losses when wood composites are exposed to a brown- or white-rot fungus [6,14,15].

Using this bending-creep test on aspen flakeboards, control boards made with phenon-formaldehyde adhesive failed in an average of 71 days using the brown-rot fungus *Tyromyces palustris* and 212 days using the white-rot fungus *Traetes versicolor* [8,25]. At failure, weight losses averaged 7.8% for *T. palustris* and 31.6% for *T. Versicolor*. Isocyanate-bonded control flakeboards failed in an average of 20 days with *T. palustris* and 118 days with *T. versicolor*, with an average weight loss at failure of 5.5% and 34.4%, respectively. Very little or no weight loss occurred with both fungi in flakeboards made using either phenon-formaldehyde or isocyanate adhesive with acetylated flakes. None of these specimens failed during the 300 days test period.

Mycelium fully covered the surfaces of isocyanate-bonded control flakeboards within 1 week, but mycelial development was significantly slower in phenol-formaldehyde-bonded control flakeboards. Both isocyanate- and phenol-formaldehyde-bonded acetylated flakeboards showed surface mycelium colonization during the test time, but the fungus did not attack the acetylated flakes so little strength was lost.

In similar bending-creep tests, both control and acetylated pine particleboards made using melamine-urea-formaldehyde adhesive failed because *T. palustris* attacked the adhesive in the glueline [8]. Mycelium invaded the inner part of all boards, colonizing in both wood and glueline in control boards but only in the glueline in acetylated boards. These results show that the glue line is also important in protecting composites from biological attack.

After a 16-week exposure to *T. palustris*, the internal bond strength of control aspen flakeboards made with phenon-formaldehyde adhesive was reduced over 90% and that of flakeboards made with isocyanate adhesive was reduced 85% [7]. After 6 months of exposure in moist unsterile soil, the same control flakeboards made with phenol-formaldehyde adhesive lost 65% of their internal bond strength and those made with isocyanate adhesive lost 64% internal bond strength. Failure was due mainly to great strength reductions in the wood caused by fungal attack. Acetylated aspen flakeboards lost much less internal bond strength during the 16-week exposure to *T. palustris* or 6-month soil burial. The isocyanate adhesive was somewhat more resistant to fungal attack than the phenol-formaldehyde adhesive was somewhat more resistant to fungal attack than the phenon-formaldehyde adhesive. In the case of acetylated composites, loss in internal bond strength was mainly due to fungal attack in the adhesive and moisture, which caused a small amount of swelling in the boards.

Another test for biological resistance that has been done on acetylated composites is with brown-, white-, and soft-rot fungi and tunneling bacteria in a fiungal cellar (Table 8). Control blocks were destroyed in less than 6 months while flakeboards made from acetylated furnish above 16 WPG showed no attack after 1 year. [13,25]. This data shows that no attack occurs until swelling of the wood occurs [23,25]. This fungal cellar test was continued for an additional 5 years with no attack at 17.9 WPG.

This is more evident that the moisture content of the cell wall is critical before attack can take place [5].

In ground tests have also been done on acetylated solid wood and flakeboards [3,11,21]. Specimens have been tested in the United States, Sweden, New Zealand, and Indonesia. The specimens in the United States, Sweden and New Zealand are showing little or no attack after 10 years while the specimens in Indonesia failed in less than three years [3]. The failure in Indonesia was mainly due to termite attack.

Table 9 shows the data for chemically modified pine flakeboards in a marine environment [9]. As with the termite test, all types of chemical modifications of wood help resist attack by marine organisms. Control flakeboards were destroyed in 6 months to 1 year, mainly because of attack of

**TABLE 8** Fungal Cellar Tests of Aspen Flakeboards Made from Control and Acetylated Flakes[1,2]

| | Rating at intervals (Months)[3] | | | | | | | |
|---|---|---|---|---|---|---|---|---|
| WPG | 2 | 3 | 4 | 5 | 6 | 12 | 24 | 36 |
| 0 | S/2 | S/3 | S/3 | S/3 | S/4 | – | – | – |
| 7.3 | S/0 | S/1 | S/1 | S/2 | S/3 | S/4 | – | – |
| 11.5 | 0 | 0 | S/0 | S/1 | S/2 | S/3 | S/4 | – |
| 13.6 | 0 | 0 | 0 | 0 | S/0 | S/1 | S/2 | S/3 |
| 16.3 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 |
| 17.9 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 |

[1] Nonsterile soil containing brown-, white-, and soft-rot fungi and tunneling bacteria.

[2] Flakeboards bonded with 5% phenol-formaldehyde adhesive.

[3] Rating system: 0 = no attack; 1 = slight attack; 2 = moderate attack; 3 = heavy attack; 4 = destroyed; S = swollen.

**TABLE 9** Ratings of Chemically Modified Southern Pine Exposed to a Marine Environment[1]

| WPG | Exposure | Years of *Limnoriid and Teredinid Borers* | Mean rating due to attack by *Shaeroma terebrans* |
|---|---|---|---|
| 0 | 1 | 2–4 | 3.4 |
| 22 | 3 | 8 | 8.8 |

[1] Rating system – 10 = no attack; 9 = slight attack; 7 = some attack; 4 = heavy attack; 0 = destroyed.

*Limnoria tripunctata*, while acetylated flakeboards show some attack after 3 years. Similar tests were run in Sweden and acetylated wood failed in marine tests after 2 years [11]. The failure was due to attack by crustaceans and moluscs.

All laboratory and field tests for biological resistance conducted to this point show that acetylation is an effective means of reducing or eliminating attack by soft-, white-, and brown-rot fungi, and tunneling bacteria. Acetylated wood is moderately effective in preventing attack by subterranean termites but not effective in a marine organisms.

## CONCLUSIONS

Acetylation is on method of chemically modifying natural lignocellulosic fibers to improve properties of dimensional stability, resistance to biological attack, and degradation due to ultraviolet energy. The technology has been studied by several laboratories around the world and is presently under consideration for commercialization in Sweden, United Kingdom, Holland, and the United States. It is already in commercial use in Japan.

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