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Chemically modified wood – a review with consideration of the opportunities for application to Irish timber

C. Birkinshaw

Department of Materials Science and Technology, University of Limerick,
Plassey Technological Park, Limerick.

Abstract

Chemical modification offers the opportunity to improve the dimensional stability and biological durability of wood. In the modification process, reagents are introduced which react permanently with the structural polymers cellulose and lignin, reducing their hygroscopicity and attractiveness as a fungal food source. Acetylation, in which the wood is reacted with acetic anhydride, was one of the earliest approaches used, and remains the most promising technique, with at least one European project now at the pilot plant stage. Although the reactive materials and the process require careful handling, the treated material presents none of the toxicity hazards associated with wood preserved by conventional means. Acetylation may give sufficient property improvement to allow fast grown softwoods to be used in higher value joinery applications, thus increasing the scope for utilisation of native materials.

Keywords: Acetylation, chemical modification, durability, anti-shrink efficiency

Introduction

Chemical modification has long been seen as a way of improving the stability and durability of wood, but despite extensive investigation since the key publications on reaction with formaldehyde (Tarkow and Stamm, 1953) and on acetylation of wood in lumber thickness (Goldstein *et al.*, 1961), relatively little practical use has been made of the idea. Attempts at commercialisation in the USA in 1961 (Anon., 1961) and in the USSR in 1974 (Otle snow and Nikitini, 1977) were discontinued, presumably for commercial rather than technical reasons. This situation may soon change, as a number of European centres have projects underway which are approaching industrial scale, and awareness of the potential advantages of chemical modification has increased. The purpose of this review is to consider the general principles involved, to examine some of the recent developments, and to comment on opportunities for application to Irish timber.

The improvement in properties associated with chemical modification is largely obtained through the control of the moisture uptake capacity of the wood. Dimensional instability in wood subject to varying environmental conditions is largely caused by the absorption or loss of moisture by the hydroxyl-rich structural polymers. The associated swelling or shrinkage is highly anisotropic, and the resulting mechanical stresses lead to checking and other forms of gross damage. A high concentration of available hydroxyl groups is also important for maintaining the minimum 20% moisture content needed for

decay-causing fungi to thrive. If wood is treated in such a way as to reduce the hydroxyl concentration, or to permanently prevent the hydroxyls from binding with water, then a considerable improvement in physical and biological properties can be expected, and this is the basis of chemical modification. Figure 1 compares wetting isotherms for modified wood with a control, illustrating the point about moisture absorption.

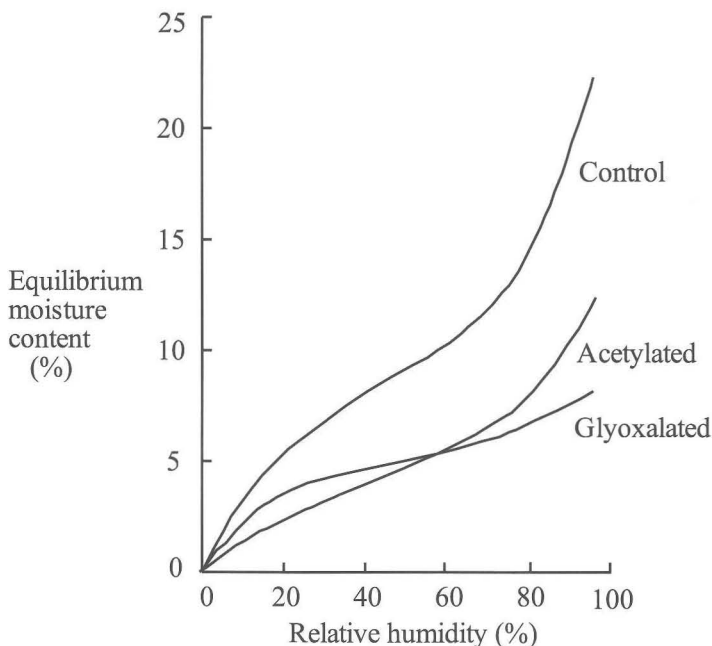


Figure 1. Absorption isotherms for Sitka spruce (*Picea sitchensis* (Bong.) Carr.) modified with acetic anhydride (21.3 WPG) and with glyoxal (17.2 WPG). From Yasuda et al. (1995).

Chemical modification also brings extra benefit in that the chemistry of decay depends upon the ability of fungally produced enzymes to 'recognise' the wood polymer repeat units as a food source, and then release them for metabolisation. Modification of the wood substrate can prevent the enzyme binding reactions (Peterson and Thomas, 1978) and the subsequent depolymerisation. Thus there are two rather different methods of protection at work in modified materials, one physical and the other chemical. The physical contribution is through improvement in hydrophobicity, while the chemical contribution is through substrate modification. Hydrophobicity can be altered by wax impregnation (Rowell and Banks, 1985; Banks and Voulgaridis, 1980), which tends to change the rate of approach to the equilibrium moisture content, but not the ultimate value. Chemical modification changes the kinetics of moisture absorption and wood swelling, and reduces the equilibrium moisture content at any particular humidity level. Figure 2 illustrates the difference between chemically modified wood and wood treated with a hydrophobic substance such as wax. The stabilisation of the moisture response explains why acetylated wood has been proposed (Yano *et al.*, 1993) for the manufacture of musical instruments.

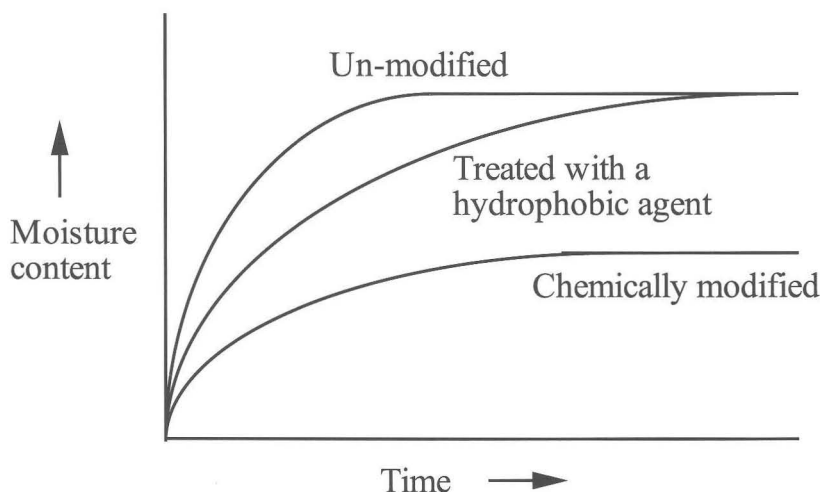


Figure 2. Relative moisture uptake of chemically modified wood and wood treated with a hydrophobic agent such as wax.

Most effective wood preservation methods present considerable environmental hazard, but this should not be the case with chemical modification. Although the reagents used are themselves toxic, if the process is carried out properly, reactions go to completion and the final products contain no harmful residues. Treated wood can be handled with complete safety, and waste wood disposal problems are greatly reduced.

Much experimental evidence is now available from many laboratories to support these general assumptions about durability improvement and environmental benefit, forcing the question about why the process has not been widely adopted. Problems are both technical and commercial. The technical problems of chemical modification are associated with the practicalities of conducting chemical reactions in a semi-solid cellular matrix, where physical factors of penetration and diffusion may dominate the chemistry. Although many of the reactions are simple in principle, the complex nature of wood makes it very difficult to work out important details about which molecular species are reacting and how it is happening. Penetration of reactants was always seen as a difficulty. The majority of the earlier publications dealt with modification of feedstocks for wood composites, such as flakeboards, with the feasibility of treatment of timber in joinery sizes hardly ever considered, despite Goldstein *et al.*'s (1961) landmark paper. Recently, reports of extensive practical trials (Beckers and Militz, 1994; Beckers *et al.*, 1994 & 1995) have shown that timber of practically useful dimensions can be treated by acetylation at the laboratory scale. The greatest challenge now lies in translating the process from the laboratory to the mill or factory. Scale-up is currently being attempted in the Netherlands, where approximately 40 companies have come together to form the acetylation company Acetyleer Kennis B.V. This initiative has been led by the independent Foundation for Timber Research at Wageningen.

Questions of economics are more complicated, as they are intertwined with environmental factors. Costs are going to be considerable, but if modified native timber can com-

pete on the grounds of stability and durability with the highest grade Canadian or Scandinavian imports, then clearly the process has merit. Indications from Wageningen are that acetylated spruce (*Picea* spp.) claddings will work out at approximately 1,150 Guilders (approximately IR£520)/m³, and pine (*Pinus* spp.) for joinery at 1,700-1,750 Guilders (approximately IR£770-800)/m³. These can be compared with meranti (*Shorea* spp.), currently widely used in the Netherlands, at 1,800 Guilders (approximately IR£820)/m³. When scale-up is achieved there, it will be easier to assess the likely cost structures which would apply in Ireland.

Measuring the benefits of modification

If wood is going to be subjected to the expense of modification, and if different modification techniques are to be compared, then it is important to be able to quantify the benefits obtained. Weight percent gain (WPG) is the most common term used to specify the degree of modification, and has obvious meaning. It is important, however, to remember that it does not distinguish between chemically and physically bound materials which may respond in different ways to continued extraction with water. Acetylated wood is typically treated to give approximately 16 WPG.

The mechanical and physical benefits of modification can be conveniently expressed in terms of two parameters. The anti-shrink efficiency (ASE) of the modification can be obtained (Rowell and Ellis, 1978) by comparing the swelling of the modified material with that of a control, as the materials are cycled between oven dry and equilibration at 98% RH:

$$ASE = ((S_c - S_m)/S_c) \times 100$$

where S is the volumetric swelling in percent, and is given by:

$$S = ((V_w - V_d)/V_d) \times 100$$

V_w and V_d are the wet and dry volumes, and S_c and S_m are the shrinkages.

Anti-shrink behaviour is often described by plotting dimensional change through a series of wetting and drying cycles. Figure 3 shows such a plot and compares modified wood with a control. A high ASE confers the obvious benefit of overall dimensional stability, and greatly reduces the problems posed by knots and grain defects. Paint adhesion is also consequently improved.

Anti-creep efficiency (ACE) can be measured by 3-point bending tests. It has been proposed (Norimoto *et al.*, 1992) that it should be expressed in the same way as ASE:

$$ACE = ((dJ_c - dJ_m)/dJ_c) \times 100$$

where dJ_c and dJ_m are the creep compliances of control and modified materials, and are given by:

$$dJ = (4wt^3 / F I^3) \times (f_0 - f_1)$$

F is the applied load, f_0 is the deflection before unloading, f_1 is the deflection after loading, I is the span, w is the width and t is the thickness.

ACE is of interest because of the central role of water and hydrogen bonding in mechano-sorptive creep. If modification interferes with hydrogen bond concentration and water interaction, then it can be expected to have an impact on creep properties. Norimoto *et al.* (1992) plotted ASE versus ACE to map property change for wood subject to a series of treatments. A simplified version of their plot is shown in Figure 4. The treatments which have a beneficial effect on both ASE and ACE can be clearly identified and compared.

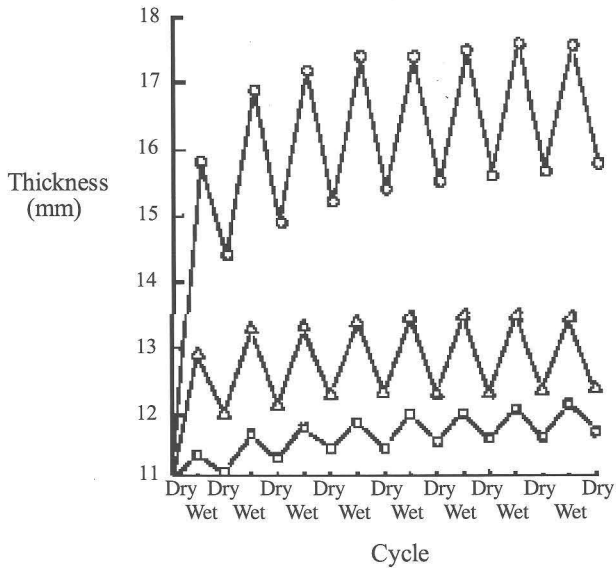


Figure 3. Thickness of chemically modified flakeboard. Symbols: \circ control; Δ butylene oxide at 18.2 WPG; \square acetylated at 24.4 WPG. From Rowell et al. (1986a).

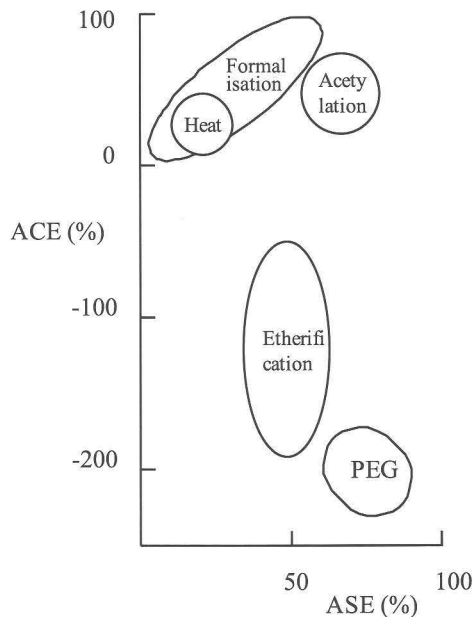


Figure 4. Plot of anti-shrink efficiency versus anti-creep efficiency. Only materials with an ACE above zero are likely to be of value for structural applications. After Norimoto et al. (1992).

Fungal durability improvement is more difficult to quantify, due to inherent biological variability and the range of test methods. There are, however, numerous publications detailing the fungal resistance of chemically modified wood under all kinds of test conditions. These include field trials (Beckers *et al.*, 1995), laboratory soil block tests (Chen, 1994) and fungal cellar observations (Nilsson *et al.*, 1988), in which parallel exposures of modified and unmodified materials have been followed by visual assessment, weight loss or strength loss measurement. All show impressive improvement in durability. In tests according to EN113 and EN 807, non-durable hardwood and softwood acetylated to a WPG of 15 reach durability class 1. This means that acetylated beech (*Fagus* spp.) will have a durability at least equal to iroko (*Chlorophora excelsa*), a timber which is still very popular for door and window joinery in Ireland. This opens up the possibility that a native species with high amenity value could be used to replace tropical hardwood.

Modification and bulking

Before looking at the chemical detail of modification processes, it is useful to clarify the relationship between modification and bulking. Bulking of wood with materials such as polyethylene glycol (PEG) is well known and is used in the preservation of cellulose based historical artefacts. It relies upon the hydroxyls of the glycol creating strong hydrogen bonds with the wood polymers and replacing water lost from the cell wall during drying (Stamm, 1964). In this way, shrinkage, distortion and cracking are minimised. Bulking agents such as PEG are very effective as dimensional stabilisers, but greatly increase the creep propensity of the wood (Norimoto *et al.*, 1992). This is probably because applied mechanical stresses exceed the activation energy barrier for hydrogen bond hopping, and the bulking agent effectively functions as a molecular level lubricant, facilitating chain rearrangement. Leaching is also a problem. Therefore, although important in the specialised field of archaeological conservation, hydrophilic bulking agents have little to offer to industrial timber technology.

Chemical modification with reactive materials such as anhydrides or epoxides, relies on the formation of primary chemical bonds between the reactant and the lignocellulosic materials, usually through reaction with the hydroxyls. The wood polymers are therefore chemically changed, and dimensional stability, creep resistance and biological durability are all improved, as previously described. All chemical modifiers will have an associated bulking effect, the magnitude of which will depend upon the size and type of chemical group which becomes attached. This means that a given molar addition of, for example, propionic anhydride, will bulk the cell wall more than the equivalent amount of acetic anhydride. Hill and Jones (1996) reacted wood with a homologous series of anhydrides, to give uniform percentage weight gain but different percent hydroxyl substitution. They found that dimensional stabilisation depended simply upon the WPG and not on the hydroxyl substitution, as might have been expected. From this, the authors concluded that dimensional stabilisation can be accounted for purely as a bulking phenomenon. This must mean that in wood modified with a higher molecular weight anhydride, free hydroxyls are present in the cell wall but are screened by the hydrocarbon tail of the modifier.

Figure 5 schematically illustrates the actions of various types of agent. The key point with the chemical modifier is that it is tied to the wood by primary chemical bonds, eliminates some hydroxyls and limits access to others. The modifier is not free to facilitate creep, which is why wood treated with anhydrides has better creep resistance than unmodified wood.

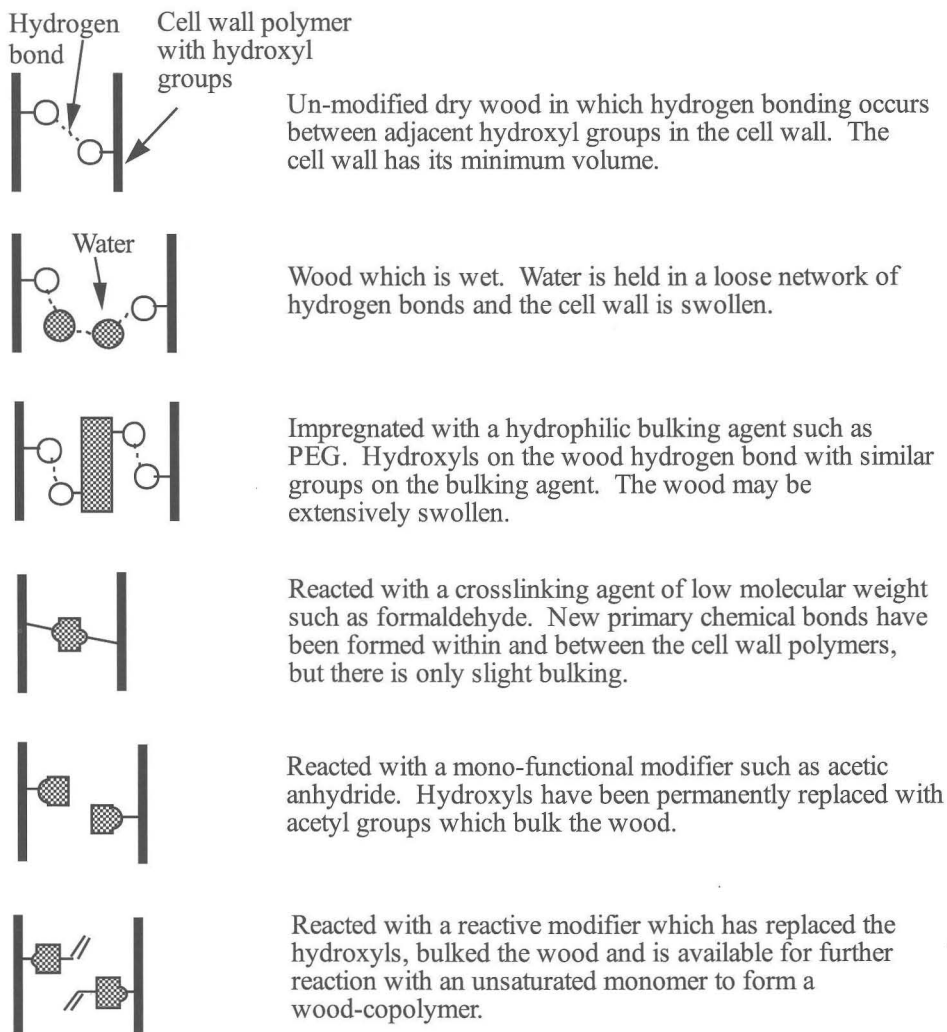


Figure 5. Schematic illustration of the effects of different types of chemical agent on the cell wall.

Both simple bulking and chemical modification result in a volume change which is broadly predictable (Rowell and Ellis, 1978) from consideration of the molar volumes of the species involved. The bulking effect is important, as it means that shrinkage on drying will be followed by expansion during modification. The wood is therefore stressed twice, with opportunity for manifestation of faults. Due to this, one of the goals of those trying to develop the process is direct modification of wet wood, so that the drying shrinkage is counterbalanced by the modifier bulking effect. If this can be achieved, then the problems of knots and grain irregularity will be minimised. With Irish softwoods, this clearly has to be a major consideration.

The chemistry of modification

Figure 6 details some of the important reactions used in chemical modification. The scheme is based on reaction with cellulose hydroxyls. Reactions with lignin are not given because, although in some cases they are important, it is still not clear what they are. As shown, much, but not all, modification chemistry depends upon the reaction of electrophiles with the cell wall hydroxyl groups. Anhydrides, which react fairly rapidly with hydroxyls to give an ester link, have received the greatest attention. Three are of particular importance; acetic, succinic and maleic. There are important differences in the way they react.

Acetic anhydride reacts with cellulose, replacing a hydroxyl and liberating acetic acid, which must then be removed as it damages the wood and is corrosive to fixings. The acetylation reaction is acid or base catalyzed. Many catalysts are mentioned in the literature, but most pose insuperable practical problems for commercial processes. Strong acids degrade the wood, while pyridine and dimethyl formamide, which have been used extensively in some of the small scale trials, present unacceptable problems of residual contamination. The general trend has therefore been to move towards un-catalyzed reactions, driven forward by heat alone. As already indicated, questions remain about the details of acetylation chemistry. Pizzi *et al.* (1994) have suggested that acylation of the lignin is an important process. It is not clear if this occurs directly or through the rearrangement of acetylated lignin groups, but the implication is that crosslinking of the matrix material may occur and that some linking of the cellulose to the lignin may be possible. Such reactions would explain the improved creep properties of acetylated wood.

Maximum practical acetyl substitution is achieved at about 25 WPG, and acetylation to about 20 WPG in wood such as poplar (*Populus* spp.) gives an ASE value of about 70%. Wood acetylated to 15 WPG or above is effectively resistant to fungal decay (Takahashi *et al.*, 1989; Peterson and Thomas, 1978), but is not fungi-toxic in the way that materials treated with copper-chromium-arsenic (CCA) are. In agar plate tests, the hyphal tips will grow up to the modified wood specimen, but are not able to penetrate the cell wall.

Of the three commonly used anhydrides, succinic is the most reactive, followed by acetic and then maleic. Maleic and succinic react with wood to replace hydroxyls with new and potentially reactive groups. They do not release a by-product such as acetic acid, which is obviously an advantage. The attached reactive groups may be exploitable as a site for copolymerisation of the cell wall polymers with an unsaturated monomer, and it has been suggested that if the process is used as part of fibreboard manufacture, then the wood-polymer composite may have sufficient thermoplasticity at the intermediate stage to allow the production of profiles by extrusion. Experiments with maleic anhydride and methyl methacrylate monomer (Banks *et al.*, 1995) have shown that some copolymerisation does occur.

Of the anhydride processes, acetylation, using either liquid or gas phase reaction, stands out as that closest to commercialisation (Beckers and Militz, 1994; Beckers *et al.*, 1994 & 1995), even though some details of the chemistry still remain to be resolved. The reaction is carried out by exposing the wood, using vacuum impregnation in a stainless steel reactor, to acetic anhydride, followed by heating to promote reaction. A typical cycle (Beckers *et al.*, 1995) comprises 1.5 hours at a vacuum of 0.04 MPa, followed by exposure to anhydride at a pressure of 8.0 MPa, after which the excess anhydride is drained off and the wood heated under vacuum at 120°C for 3 hours. Time of reaction appears to be more significant than temperature. It is important that acetic acid released during the

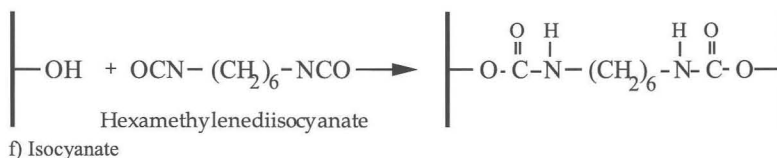
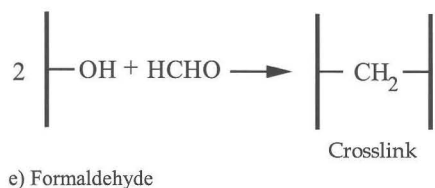
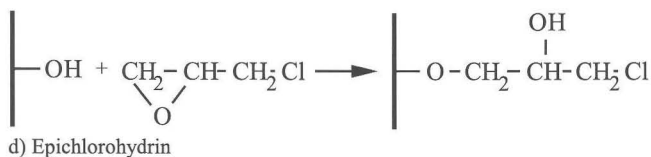
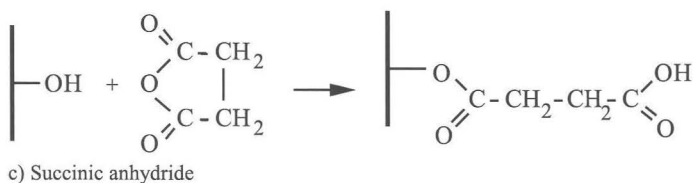
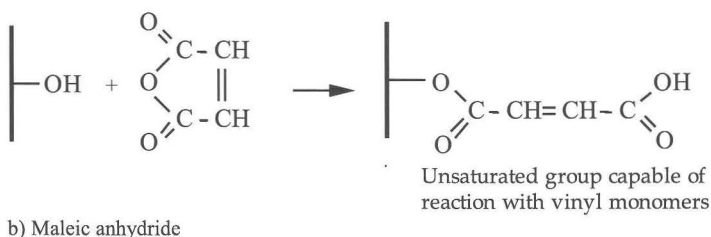
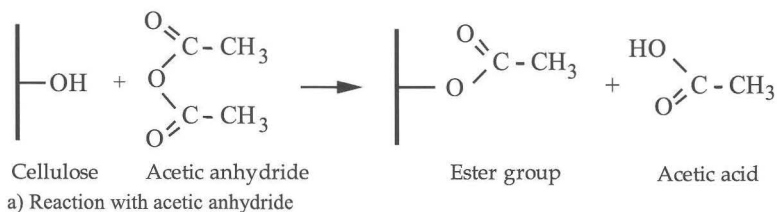


Figure 6. Some of the chemical reactions used in modification.

reaction is removed from the final product. The wood must also be relatively dry before treatment, as residual moisture competes for anhydride, wastefully reacting with it to liberate more acetic acid.

Wood morphology has a large effect on the efficiency of the acetylation process, but this is not simply a question of penetrability, as beech and poplar are both readily penetrated but show different rates of acetylation (Beckers *et al.*, 1995). Spruce is particularly difficult to acetylate, although acetic anhydride has been shown to achieve better penetration than normal aqueous preservative. It has been proposed (Militz and Homan, 1992) that this is because of degradation of the pectin in aspirated bordered pits by acetic acid. During acetylation, the wood swells almost to green state dimensions, and as reaction may preferentially occur closer to the surface in wood such as spruce, subsequent profiling may lose much of the benefit. This suggests that the process sequence should match the timber. In current work in the Netherlands, spruce cladding is profiled before treatment, while pine for structural applications is profiled after treatment. Profiling waste is completely harmless and can be disposed of or used along with other process waste.

Acetylation of the feedstocks for wood-based composites has also been extensively described (e.g. Rowell *et al.*, 1986a & 1986b; Clemons *et al.*, 1992), but again processes have not reached commercial realisation. The technical advantage of easy access of reactant to fibres or flakes makes the idea attractive, and Rowell's work shows that significant improvement in ASE can be obtained. This would lead to composites of improved mechanical integrity under wet conditions (Rowell *et al.*, 1988). Fitting the modification process into existing composite production lines would, however, not be easy, probably explaining why development emphasis has gone on binder research.

Beside the anhydrides, many other reagents have been investigated as possible wood modifiers. The more important of these are aldehydes, such as formaldehyde, isocyanates and epoxides. Formalisation, the reaction with formaldehyde, results in cross-linking within the cell wall, probably involving the lignin matrix, and because of this, there is a beneficial reduction in plasticity (Akitsu *et al.*, 1993) and creep (Norimoto *et al.*, 1992). Low levels of treatment provide a very high level of dimensional stability, with 7 WPG giving an ASE of 90% (Tarkow and Stamm, 1953). Unfortunately, the reaction is catalyzed by strong acids which cause wood degradation (Stevens *et al.*, 1979; Stevens and Parameswaran, 1981), with impact strength being particularly badly affected. There is also the additional health problem of free formaldehyde. The dialdehyde, glyoxal, presumably undergoes similar condensation reactions to formaldehyde, but has also been used in conjunction with glycol to form more complex condensation linkages.

Isocyanates have a high reactivity towards hydroxyls and react with the cell wall cellulose, without the use of catalysts, to form carbamate groups. Binders based on isocyanates are already in use in Ireland for the production of medium density fibreboard, and the reactions here are probably the same as those used in the modification of solid wood. Treatment with mono-isocyanates such as the n-butyl isocyanate leads to cell wall bulking and significant reduction in moisture absorption (Martin and Banks, 1991). Reaction of wood with a diisocyanates such as hexane diisocyanate or toluene diisocyanate may crosslink the cell wall material, as shown in Figure 6. Direct modification with isocyanates can be difficult because of premature reaction with moisture, and so acyl hydrazides have been proposed (Gerardin *et al.*, 1995) as the starting materials. Wood is impregnated with the pre-cursors and the *in-situ* release of the isocyanates, provoked by heating, limits the opportunity for premature reaction. The technique has, however, only been used with spruce meal and small 3 cm x 2 cm x 1.5 cm blocks, with pyridine as sol-

vent. Weight gains were around 3.5% and 70% with block and meal respectively, when reacted with $C_6H_5CON_3$. Chen (1994) has reported the use of chlorosulphonyl isocyanates reacted in pyridine, which acts as a solvent and catalyst, with blocks (1.9 cm) subject to rigorous drying. The wood was soaked in the reactant mixture for 24 hours and then reacted at 60°C for up to 24 hours to give a WPG of up to 14%. Some results are given in Table 1.

Table 1. *Effect of isocyanate and epichlorohydrin modification of loblolly pine (Pinus taeda L.) on resistance to decay by Gloeophyllum trabeum in a 12-week soil block test. From Chen (1994).*

<i>Treatment</i>	<i>WPG</i>	<i>Weight loss by decay (%)</i>
Chlorosulphonyl isocyanate	2.56	14.9
	6.06	8.4
	14.69	0.6
Epichlorohydrin	2.15	37.4
	5.45	12.7
	10.86	2.3
Control		49.7

Epoxides will react with wood hydroxyls to give ether links, but there is good evidence (Norimoto *et al.*, 1992) that creep properties are poor. Some interesting observations (Chen, 1994) have been made with the epoxide epichlorohydrin, which, in the presence of triethylamine catalyst, reacts with wood to give a combination of alkoxy bonded side chains and wood-wood crosslinks. Table 1 compares the performance of isocyanate and epichlorohydrin modified wood, and illustrates what can be achieved in relation to fungal resistance. Creep resistance data does not seem to be available. Combinations of epichlorohydrin and glutaric anhydride have been reported (Goethals and Stevens, 1994) to give good ASE results. Sequential esterification and epoxidation, by elimination of the carboxyl group, should give a substituent with reduced hydrophilicity.

Another approach which has been considered (Rowell and Chen, 1994) is to use epichlorohydrin as a coupling agent to bind a bioactive compound such as pentachlorophenol to the wood. If the bioactive group contains a hydroxyl, it will condense with epichlorohydrin to give a glycidyl ether. This will still have an epoxy group and be able to further condense with the hydroxyls of the cell wall, binding on the bioactive group. In the same way, glycidylmethacrylate can be directly reacted with wood (Goethals and Stevens, 1994), and offers an unsaturated reaction site for subsequent copolymerisation.

Wood-polymer composites are a specialised area of modification. The target is usually copolymerisation of the lignocellulosic materials with a vinyl monomer such as methyl methacrylate (MMA). If this can be achieved, then substrate modification and hydrophobicity will be maximised, but the problems of diffusion of reactants are large and the process economics do not look promising. Maleic anhydride-methyl methacrylate combinations (Banks *et al.*, 1995) have already been mentioned. Lumen fill with MMA can be achieved and slightly reduces the rate of water swelling but does not reduce the extent

of swelling (Feist *et al.*, 1991), implying that little cell wall penetration occurs. The MMA polymer acts as an additional matrix adhesive material and holds the cellulose fibres together during weathering, and as weathering is a surface phenomenon, it may be possible to simply surface treat the finished wood product. Many other wood polymer composites have been examined, including wood-phenol formaldehyde resins (Akitsu *et al.*, 1993) and wood-styrene systems (Stevens and Schalck, 1978).

Beside the few materials considered here, there are many other potential modifiers, and research into new systems continues. For example, the EU-supported Chemowood project, involving groups in Belgium, the UK, the Netherlands and Finland, is currently investigating a range of substances, including N-methylolacrylamide, furfuryl alcohol and various modified anhydride systems. These, and the other non-anhydride reactants previously mentioned, may in the long run prove useful, but their practical realisation does not appear to be imminent. Many of the reagents are expensive and somewhat difficult to handle, and scale-up work has not been attempted.

Conclusion

There is no doubt that a number of different chemical modification procedures are effective at improving the stability and durability of wood, but acetylation is the only process currently close to commercialisation. Within the next few years, significant amounts of acetylated timber may be in use in the Netherlands, and the technical and economic position of the process will become clearer. Timber utilisation patterns in Ireland differ from those in other European countries, but many of the same environmental and economic pressures apply. Before getting involved in modification, Irish mills and joinery manufacturers will require detailed cost information. Costs are going to be considerable, as the nature of the processes, involving corrosion-resistant pressure vessels, ovens and chemical handling, recovery and storage facilities, indicates a large investment compared with a CCA plant. It is therefore important to realise that acetylated timber is not simply a new method of preservation which will compete with use of CCA or borates, but is a way of upgrading the material to allow it to be used for higher value applications.

A significant cost benefit may arise through savings in waste wood disposal. European and national regulations are making handling, and in particular, disposal, of CCA treated materials problematic and potentially expensive. Acetylated wood can be dealt with and disposed of without any special precautions, and no environmental hazards are presented.

The investment required is within the capability of the large wood composite companies, but would be more of a challenge for the smaller units involved in solid wood processing. Economies of scale make it very difficult to envisage more than a very small number of modification plants in Ireland, but decisions about how these will be owned and operated rest with the industry and State bodies. The Netherlands example of the establishment of a joint company is one approach.

From a technical perspective, the way ahead is more straightforward. As the capability to acetylate timber becomes available on the Continent, sample quantities of Irish materials can be supplied for treatment. COFORD are currently funding a project at the University of Limerick to do this. In this project, mechanical and chemical properties of acetylated wood will be evaluated using standard tests, and biological durability investigated using accelerated procedures. Species being examined include Sitka spruce (*P. sitchensis* (Bong.) Carr.), lodgepole pine (*P. contorta* Dougl.) and Japanese larch (*Larix kaempferi* (Lamb.) Carr.). In the longer term, larger quantities of modified fast grown

wood can be converted into products for field trials. Any problems of treatment penetration with fast grown spruce or pine would become clearer and could be addressed. Such a series of trials would be relatively cheap, and the information generated would allow a more informed decision about the adoption of the process and the economics involved.

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