DIMENSIONAL STABILIZATION OF RUBBERWOOD (*HEVEA BRASILIENSIS*) WITH ACETIC OR HEXANOIC ANHYDRIDE

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SITI RAFIDAH, K., HILL, C. A. S. & ORMONDROYD, G. A. 2006. **Dimensional stabilization of rubberwood (*Hevea brasiliensis*) with acetic or hexanoic anhydride.** The dimensional stability of rubberwood (*Hevea brasiliensis*) modified with acetic or hexanoic anhydride was determined by the water-soak/oven-drying method. Acetylation was more effective at providing dimensional stability compared with hexanoylation below a weight percentage gain (WPG) of 10%. This was attributed to the different modification methods used with the two anhydrides. Volumetric swelling (oven-dry) due to modification was lower for acetylation compared with hexanoylation at comparable WPGs. Data were plotted in terms of anti-shrink efficiency (ASE) against a parameter ASE'. ASE' measures the ASE in terms of the sum of volume change due to modification and water soaking. A negative ASE' was found for hexanoylated rubberwood. This was attributed to over-swelling of the cell wall at WPGs in excess of 25%. The swelling was due to reaction of the wood with pyridine, which is known to be very effective at swelling the cell wall. Positive values of ASE' were found for acetylated rubberwood, but there was no clear relationship between ASE' and WPG. Although positive values of ASE' can be attributed to cross-linking occurring within the cell wall, this cannot be assumed, since changes in wood volume, as determined from external dimensions did not correlate with changes in cell wall volume.

Keywords: Modification, volumetric swelling/shrinkage, anti-shrink efficiency, hexanoylation, acetylation

SITI RAFIDAH, K., HILL, C. A. S. & ORMONDROYD, G. A. 2006. **Penstabilan dimensi kayu getah (*Hevea brasiliensis*) dengan anhidrida asetik atau anhidrida heksanoik.** Kestabilan kayu getah (*Hevea brasiliensis*) yang dirawat dengan anhidrida asetik atau anhidrida heksanoik dikaji dengan kaedah rendaman air atau kaedah kering ketuhar. Pengasetilan memberi kestabilan dimensi yang lebih baik berbanding dengan pengheksanoksilan pada nilai tambahan berat kering (WPG) di bawah 10%. Ini disebabkan oleh kaedah modifikasi yang menggunakan dua anhidrida berlainan. Pembengkakan isi padu berasaskan berat kering ketuhar adalah rendah bagi rawatan dengan anhidrida asetik berbanding dengan rawatan dengan anhidrida heksanoik pada WPG yang setara. Graf kecekapan antikecutan (ASE) lawan parameter ASE' dilukis. ASE' menyukat ASE dari segi perubahan isi padu kayu akibat modifikasi dan akibat rendaman air. Kayu yang dirawat dengan anhidrida heksanoik menunjukkan ASE' negatif disebabkan berlakunya pembengkakan berlebihan dinding sel pada WPG melebihi 25%. Ini berlaku akibat reaksi bahan pengembangan iaitu piridina. Nilai ASE' positif didapati pada kayu yang dirawat dengan anhidrida asetik. Hubung kait tidak begitu jelas antara ASE' dengan WPG. Walaupun nilai ASE' positif boleh dikaitkan dengan rangkaian silang di dalam dinding sel kayu, hasil kajian menunjukkan rangkaian silang tidak boleh dianggap sebagai penyebab ASE' yang positif kerana perubahan isi padu kayu yang terhasil daripada ukuran dimensi luaran tidak menunjukkan sebarang korelasi dengan perubahan isi padu dinding sel.

INTRODUCTION

Although primarily used for the production of latex, there has been increasing use of rubberwood (*Hevea brasiliensis*) for production of timber goods. Rubberwood is classified as non-durable and exhibits poor dimensional stability when exposed to moisture. Thus if it is to be used for exterior applications such as windows, doors and cladding, treatments need to be developed to overcome these disadvantages. One possible technology is chemical modification using acetic anhydride, which is a well-known method to improve the dimensional stability and

decay resistance of wood (Hill 2006).

There has been a resurgence of interest in this technology in Europe, with Titanwood presently nearing completion of a 30000 m^3 $year^{-1}$ production facility in the Netherlands. Although an expensive modification, the economics of this technology is now much more favourable due to the impact of environmental legislation on the use of more conventional biocidal treatments such as copper chromium arsenic.

Despite the potential of anhydride modification in upgrading rubberwood, there is little evidence in the literature of any substantive studies. In studies of the improvement of dimensional stability of rubberwood, two investigations have been reported on acetylated solid rubberwood (Ibrahim & Razak 1991, Liu *et al.* 1993) and one, on the dimensional stability of flakeboards made from acetylated rubberwood (Okino *et al.* 2001). Due to the paucity of information on this subject, a comprehensive study of the effects of anhydride modification on the swelling and dimensional stability of rubberwood was conducted.

Background theories

Acetic anhydride and hexanoic anhydride (Figure 1) were used to determine whether the dimensional stability was imparted solely as a result of cell wall bulking, as has been established for anhydride modified spruce (Stamm & Tarkow 1947) and Corsican pine (Hill & Jones 1996) or due to a more complex mechanism. The acetyl group has a molecular weight of 43 and the hexanoyl group, 99. Thus at comparable WPGs, approximately twice as many cell wall OH groups are substituted when wood is reacted with acetic anhydride, compared with hexanoic anhydride. Reaction of wood with these two anhydrides can therefore be used to determine the relative effects of cell wall bulking against hydroxyl substitution in property modification.
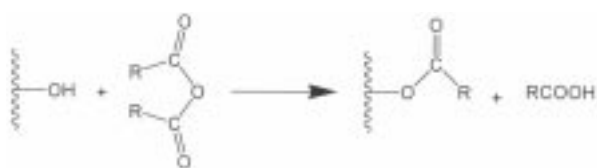
Further information regarding the mechanism by which modification enhances dimensional stability of wood can also be obtained by using a parameter discussed by Ohmae *et al.* (2002), which is termed ASE'. While as the conventional anti-shrink efficiency (ASE) is determined from the oven-dry and water-saturated volumes of the modified wood (see Eqns. 2, 3 and 4), ASE' is determined from the water-saturated volume of the modified wood, but the oven-dry volume of the same sample before modification (Eqns. 5 and 6).

The information that is obtained from such an analysis is illustrated with the aid of Figure 2. In Figure 2a, the difference in volume between an oven-dried and water-saturated sample is shown (V_1). In Figure 2b, the volume expansion due to modification with an anhydride reagent is given as V_3. When the same sample is then saturated in water, the further volume expansion that results is labelled V_2. If the sum of V_2 and V_3 is equal to V_1, then this can be interpreted as the dimensional stability being due solely to bulking and ASE' is zero.

However, if the modification of the wood results in a reduction in water-saturated volume (for example, due to cross-linking within the cell wall), then the situation is as illustrated in Figure 2c. Here the sum of the volume increase due to modification (oven-dry) (V_3) and the water-saturated volume of the modified wood

$$V_1 = V_2 + V_3 \quad ASE' = 0$$
$$V_1 > V_3 + V_4 \quad ASE' = +ve$$
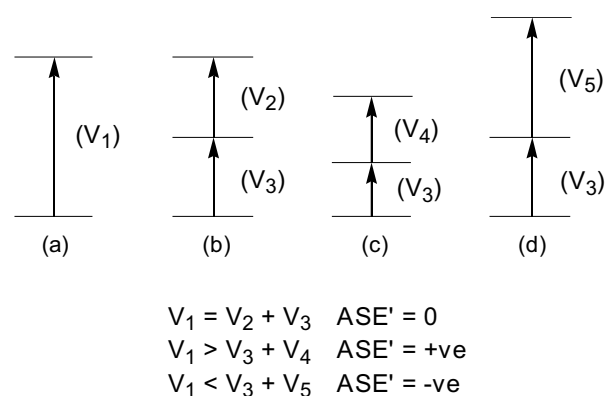$$V_1 < V_3 + V_5 \quad ASE' = -ve$$

Figure 2 Illustration showing the principles behind the determination of ASE' (a) an unmodified wood sample, (b) dimensional stability due to bulking, (c) dimensional stability due to cross-linking in the cell wall and (d) reaction plus water-saturation results in over-swelling of the cell wall. Source Ohmae *et al.* 2002

Figure 1 Wood anhydride reaction scheme acetylation (R=CH_3), hexanoylation (R=C_5H_{11})

(V_4) is less than that of the water-saturated volume of the same unmodified sample, resulting in a positive ASE' value.

Finally, in Figure 2d, the situation where the sum of the modified oven-dried volume (V_3) and the subsequent water-saturated volume increase (V_5) is greater than the water-saturated volume of the same unmodified sample gives a negative ASE'.

Such an analysis can, in principle, be used to determine the mechanism by which dimensional stability is rendered to the modified wood. However, an important caveat is that changes in the external dimensions of wood samples must be directly related to changes in cell wall volume. In other words, there must be no change in macro-void volume (wood volume attributed to pore, lumens, etc.) as the wood swells. This can be determined by measuring the volume occupied by the cell wall material and comparing it with the volume of the wood determined using exterior dimensions. The cell wall volume can be determined by pycnometric methods, and helium pycnometry is a simple commercially available method, which provides highly accurate determinations of the volume occupied by a dry material.

MATERIALS AND METHODS

Wood samples

For dimensional stability determinations, rubberwood was processed into samples of dimensions $20 \times 20 \times 5$ mm (radial $\times$ tangential $\times$ longitudinal), labelled and solvent extracted using a mixture of toluene/acetone/methanol (4:1:1 by volume) for 24 hours in a Soxhlet apparatus. Samples were air dried overnight, then placed in an oven at 105 °C for 24 hours. After oven drying, the blocks were placed in a desiccator to cool to ambient temperature before weight and dimensions (micrometer) were determined. For the helium pycnometry measurements, rubberwood discs were prepared with a diameter of 13 mm and a longitudinal dimension of 5 mm. Solvent extraction and oven drying were employed as detailed above. The weight percentage gain (WPG) of the wood samples was calculated according to:

$$\text{WPG } (\%) = [(W_{mod} - W_{unmod})/W_{unmod}] \times 100 \quad (1)$$

where W_{mod} is the oven-dry weight of the modified wood and W_{unmod} is the oven-dry weight of the same unmodified wood sample.

Chemical modification

Wood modification reactions were performed at 110 °C in acetic anhydride or in a mixture of hexanoic anhydride (1 M) in pyridine. Samples for chemical modification (batches of seven discs for helium pycnometry or six blocks for dimensional stability tests) were pre-impregnated with the appropriate reaction solution at ambient temperature for four hours, then added to the reaction flask containing the hot reaction solution at various time intervals to ensure a good range of WPG. The reaction was run for a total of 27 hours and samples added periodically to give reaction times of 15, 30, 45, 60 and 90 min, and 2, 3, 5, 7 and 27 hours. At the end of the reaction period, the hot reagent was decanted off from the blocks and the wood samples added to ice-cold acetone to quench the reaction. After two hours in the acetone, the samples were transferred to the Soxhlet apparatus for solvent extraction, followed by oven drying as detailed above. With acetylation of rubberwood, a 27-hour reaction period resulted in an average WPG of only 13% and two further reactions were performed at 94 and 144 hours to give average WPGs of 15 and 16.6% respectively.

Dimensional stability determinations

Wood samples were subjected to a five-day water soak, two-day oven-drying cycle, over a total of six cycles. Volume changes were determined by using a micrometer. Results for dimensional stability determinations were taken from the last five cycles only, with the results from the first cycle being discarded as unrepresentative (Rowell & Ellis 1978, Hill & Jones 1996). This is generally regarded as good practice for various reasons. When samples are first measured after solvent extraction and oven drying they often exhibit volumes slightly larger than when the wood is dried from a water-saturated state. This is because the forces collapsing the cell wall are greater with water compared with non-polar (or low polarity) solvents often used in the extraction procedures because of strong hydrogen-bonding interactions occurring with the former (Hill *et al.* 2004). There is also the possibility that there might be

non-bonded but cell wall bulking components remaining after the reaction, although this is not likely with anhydride reactions provided a rigorous solvent extraction procedure is adopted. The percentage swelling coefficient of the modified wood was calculated according to:

$$S_{mod} = [(Vws_{mod} - Vod_{mod})/Vod_{mod}] \times 100 \qquad (2)$$

where Vws_{mod} is the water-saturated volume of the modified wood sample and Vod_{mod} is the oven-dry volume of the modified wood sample. The percentage swelling coefficient of the unmodified wood was calculated according to:

$$S_{unmod} = [(Vws_{unmod} - Vod_{unmod})/Vod_{unmod}] \times 100 \quad (3)$$

where Vws_{unmod} is the water-saturated volume of the unmodified wood sample and Vod_{unmod} is the oven-dry volume of the unmodified wood sample.

The anti-shrink efficiency was calculated according to:

$$ASE = [(S_{unmod} - S_{mod})/S_{unmod}] \times 100 \qquad (4)$$

where S_{unmod} is the swelling coefficient of the unmodified wood samples and S_{mod} is the swelling coefficient of the modified wood samples at a given WPG.

An alternative measure of dimensional stability, as defined by Ohmae *et al.* (2002), was determined as follows:

$$ASE' = [1 - (S'_{mod}/S_{unmod})] \times 100 \qquad (5)$$

where S'_{mod} is the volumetric swelling coefficient of the modified wood sample calculated as follows:

$$S'_{mod} = [(Vws_{mod} - Vod_{unmod})/Vod_{unmod}] \times 100 \quad (6)$$

where Vws_{mod} is the water-saturated volume of the modified wood sample and Vod_{unmod} is the oven-dry volume of the same wood sample.

Helium pycnometry

Sets of samples were prepared in order to provide a good fit into the chamber of the helium pycnometer. The sample batch consisted of seven discs, which were measured individually using a micrometer and as a complete batch in the helium pycnometer. Oven-dried, pre-weighed samples were used for the measurements, with identical samples being measured before and after chemical modification. Full experimental details are given in Hill and Ormondroyd (2004).

Percentage volume change as a result of modification was calculated based on the original volume of the same sample before modification, as determined by helium pycnometry or by measurement of external dimensions:

$$VC (\%) = [(V_{mod} - V_{unmod})/V_{unmod}] \times 100 \qquad (7)$$

where V_{unmod} is the volume of the sample prior to modification and V_{mod} is the volume of the sample after modification. The macro-void volume (MV) of wood samples was determined by subtracting the sample volume as measured by helium pycnometry (V_{pyc}) from the sample volume as determined from measurement of external dimensions (V_{ext}):

$$MV = V_{ext} - V_{pyc} \qquad (8)$$

The percentage change in macro-void volume (MVC %) due to modification at different WPGs was then determined from:

$$MVC (\%) = [(MV_{mod} - MV_{unmod})/MV_{unmod}) \times 100 \quad (9)$$

where MV_{mod} is the macro-void volume of the modified wood and MV_{unmod} is the macro-void volume of the same samples prior to modification.

Thus an increase in MVC is a result of an increase in the macro-void volume of the modified wood, which can be interpreted as an increase in pore and lumen diameter of the wood. If MVC decreases, then this can be interpreted as an expansion of the cell wall into the macro void space of the wood.

RESULTS AND DISCUSSION

Volume change due to modification

The use of a helium pycnometer allows for extremely accurate determinations to be made of the cell wall volume in the wood samples. The percentage volume change (VC %) in samples due to modification as determined using the helium pycnometer is shown in Figure 3, and as

determined using the measurement of external dimensions, shown in Figure 4. With both sets of data, it is apparent that there is a linear relationship between VC % and WPG due to modifications in both the acetylated and hexanoylated samples. This contradicted a previous study of acetylated Corsican pine, in which the helium pycnometry data showed a linear relationship between VC % and WPG but not the data obtained by the measurement of external dimensions (Hill & Ormondroyd 2004).

There is a difference in the behaviour of acetylated samples compared with hexanoylated

Figure 3   Relationship between percentage volume change and weight percentage gain of rubberwood due to modification with acetic (□) or hexanoic (○) anhydride, as determined by helium pycnometer

Figure 4   Relationship between percentage volume change and weight percentage gain of rubberwood due to modification with acetic (□) or hexanoic (○) anhydride, as determined by external dimensions. Error bars show standard deviations

samples, with the VC % being slightly lower with acetylated samples as WPG increased. The larger volume change observed with hexanoylated samples is attributed to the use of pyridine as a solvent for these reactions. Pyridine was used because hexanoic anhydride will not react with wood. Pyridine not only serves as a reaction catalyst but also serves to swell the cell wall to permit penetration by this larger reagent. This also explains why it is possible to react to much higher WPG's with hexanoic anhydride.

Much higher VC % values were found using the helium pycnometry data compared with data obtained by measuring external dimensions. These data are not exactly comparable because each data set is based on a different initial volume (obviously the external dimensional volume is much greater than that recorded by helium pycnometry). At 20% WPG, the increase in the cell wall volume was about 20% but was remarkably high (over 50%) at WPG of about 35% with the hexanoylated samples (Figure 3). This could be interpreted as evidence of cell wall damage, although this would require confirmation using microscopy. What is clear from these data is that with hexanoylation there was a large increase in cell wall volume and that this was associated with an increase in external volume in excess of the 'green volume' of the wood (about 10% VC) above 20% WPG.
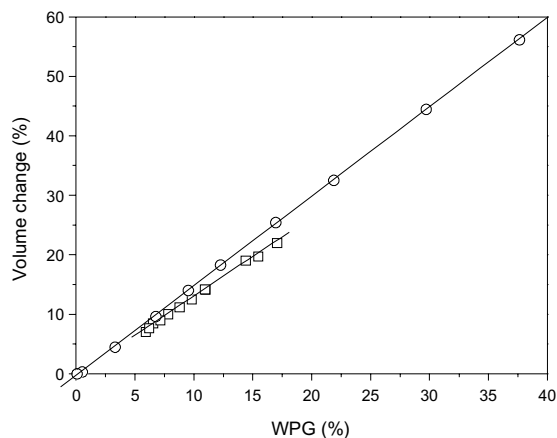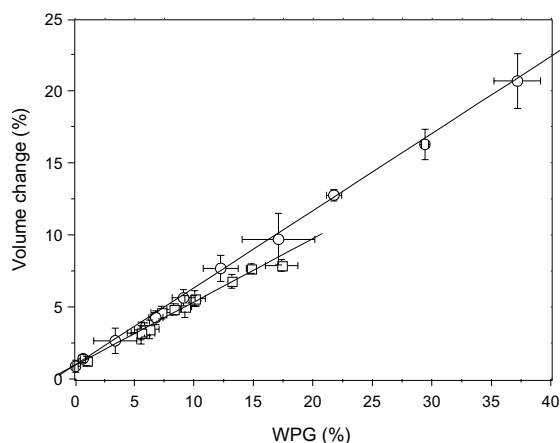
Dimensional stability

The relationship between ASE % and WPG for each individual sample is shown in Figure 5, which shows that acetylation was more effective at providing dimensional stability, particularly below 10% WPG. This contrasted the results obtained for Corsican pine by Hill and Jones (1996), where modification with a range of anhydrides resulted in comparable ASE values being obtained at a given WPG. The most likely explanation for the differences found in the present study is the use of pyridine for the hexanoylation reaction. It has been well established that pyridine is very efficient at swelling the wood cell wall. In the data presented by Hill and Jones (1996), pyridine was used for reaction with all the anhydrides studied.

Figure 6 shows the relationship between ASE' and ASE for each individual sample of acetylated and hexanoylated rubberwood. The large negative ASE' values found for hexanoylated

rubberwood at high ASE values could be interpreted as indicative of cell wall damage, but this was not confirmed independently. These data showed that the wood is swollen above the 'green volume' of the unmodified wood, as a result of reaction and water saturation. Rowell and Gutzmer (1975) suggested that cell wall damage occurred with alkylene oxide modified pine above 30% WPG. The data for acetic anhydride modified rubberwood generally exhibit positive values for ASE' but there is no relationship between ASE' and ASE. Although there is considerable scatter, the data indicate that the volume of water-saturated acetylated rubberwood with most of the samples is less than that of water-saturated unmodified rubberwood, which according to the interpretation given by Ohmae *et al.* (2002) suggests that a component of the dimensional stability can be attributed to cross-linking between macromolecular constituents within the cell wall.

This would appear to support a dimensional stability mechanism first proposed by Pizzi *et al.* (1994) where it was suggested that the acetic anhydride undergoes an α-set attack on the lignin aromatic nuclei, analogous to reactions found to occur with phenol formaldehyde resins. This acylation reaction then allows for the possibility for cross-linking between lignin aromatic units. However, a prerequisite for such reactions to take place is the use of strongly alkaline reaction media. Furthermore, such a reaction is not possible for

longer chain anhydride molecules, and the observation (until now) that the degree of dimensional stability imparted is a function of WPG only, irrespective of anhydride would argue against acetylation being a special case. Pizzi *et al.* (1994) also suggested that acylation of the aromatic components of lignin might occur by conventional acetylation of the phenolic hydroxyl, followed by a Fries rearrangement. However, this reaction requires the presence of Fridel-Crafts catalysts or UV radiation (March 1992), conditions not found in conventional acetylation reactions.

Although the possibility that acetylation of rubberwood may involve a degree of cross-linking cannot be completely discounted, an alternative explanation for this phenomenon can be advanced. The determination of ASE and ASE' rely on measurements of the external dimensions of samples only and therefore it is not possible to determine if there are any changes in the macro-void internal volume associated with pores, lumens, etc. The assumption which is used for this method is that changes in external dimensions can be correlated with changes taking place in the cell wall, which has been shown to be invalid at least in the case of Corsican pine sapwood (Hill & Ormondroyd 2004). The assumption that lumen volume is unchanged as wood swells cannot be made without supportive evidence. For example, if there is any intrusion of the cell wall into the macro-void space by the
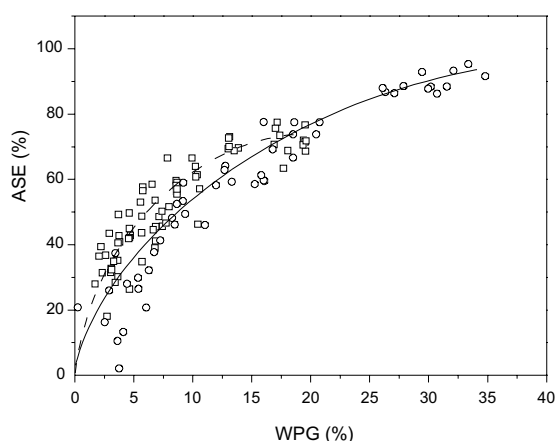

Figure 5   Relationship between percentage anti-shrink efficiency (ASE) and weight percentage gain (WPG) for acetylated (□) and hexanoylated (○) rubberwood. Dotted line shows best fit second-order polynomial curve through data for acetylated rubberwood. Solid line shows the same for hexanoylated rubberwood
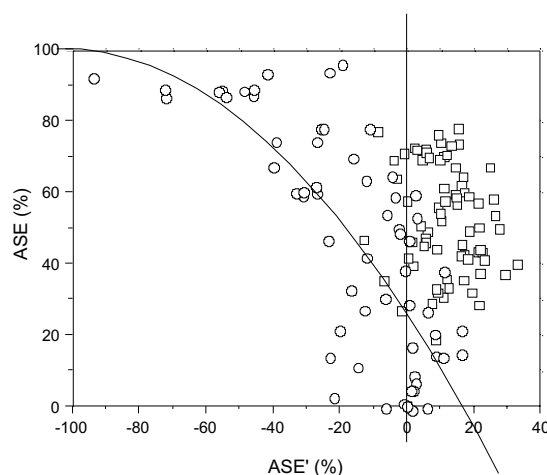

Figure 6   Relationship between ASE and ASE' for acetylated (□) and hexanoylated (○) rubberwood. Line shows best fit second-order polynomial through data points for hexanoic anhydride modified rubberwood. No relationship between ASE and ASE' for acetylated rubberwood was found

cell wall, this will not be detected in determinations of external dimensions. Therefore, a slight contraction in the external dimensions of the water-saturated modified sample when compared with the same unmodified sample could be falsely attributed to cross-linking within the cell wall. Although it was not possible to check for expansion of the cell wall into the lumen under conditions of water saturation, this can be performed on dry samples at different WPG's using the external dimensions and helium pycnometry data. External volume measurement with callipers gives (crudely) the total volume occupied by the sample (wood volume plus any voids), whereas helium pycnometry measures the volume occupied by cell wall material only. Subtracting the helium pycnometry volume from the volume obtained by determining external dimensions will therefore allow for an estimate to be made of the macro-void volume for a specific wood sample.

The percentage change in the void volume can then be calculated from the difference in the macro-void volume between modified and unmodified samples (Eqn. 9). The relationship between percentage change in macro-void volume and WPG is shown in Figure 7. Before any discussion of the analysis of this data, it should be noted that there is considerable scatter in the data, and that as a consequence interpretation is speculative. Although helium pycnometry is very accurate, the use of callipers to determine external dimensions is subject to considerable error. The plot shows that at WPGs below about 8%, the macro-void volume change is positive. This would then indicate that the cell wall adjacent to the lumen expands outwards at lower levels of modification but as the level of modification is increased, the cell wall then begins to encroach upon the pores, lumens and other macro-voids in the wood structure.

It should, however, be noted that the macro-void volume change at zero WPG is positive, whereas it would be expected that the value should be zero at this point. It is not clear why this behaviour is observed, but this may be related to loss of cell wall material associated with the oven drying and extraction procedures adopted for the modification protocol. It is known that rubberwood contains high levels of starch and it is possible that this component is susceptible to degradation. In any event, this plot shows that
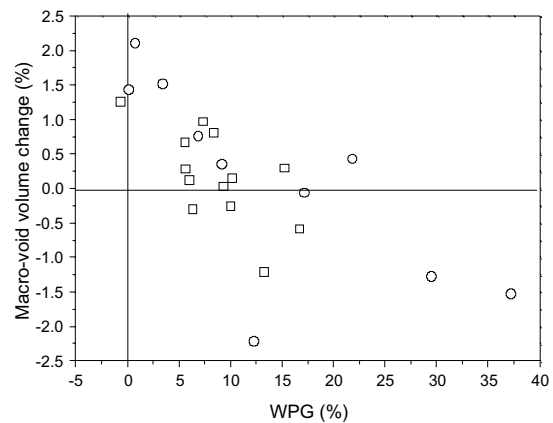

Figure 7 Relationship between percentage macro-void volume change and weight percentage gain for acetylated (□) and hexanoylated (○) rubberwood

at higher levels of modification the cell wall does intrude into the macro-void volume of the wood. If this also occurs during the water-soaking phase in dimensional stability determinations, then measurement of the volume of the water-saturated wood by external dimensions will not give a true representation of behaviour at the cell wall level. Any movement of the cell wall into or contraction away from the lumen will not be accounted for in measurements of the external dimensions of samples. As a consequence, ASE' cannot be used as evidence that cross-linking has occurred in the cell wall in this case.

CONCLUSIONS

Rubberwood was modified using two different anhydride reagents under significantly different reaction conditions. Differences were observed in the volumetric swelling of samples due to reaction and dimensional stability imparted by reaction. When the dimensional stability data was analysed in terms of ASE' vs. ASE, hexanoylated wood exhibited high negative ASE' values as WPG increased, related to over-swelling of the cell wall. However, acetylated wood generally exhibited marginally positive ASE' values, which is thought to be indicative of a cell wall cross-linking dimensional stabilisation mechanism. Interpretation of ASE' data relies on the assumption that the change in wood volume as determined by measurement of the external

dimensions of the sample is directly related to changes in cell wall volume. Changes in internal macro-void volume with WPG can result in possible misinterpretation of ASE' data.

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