

# **PRESERVATIVE TREATMENT AND CHEMICAL MODIFICATION OF RUBBER WOOD**

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BY

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## **DECLARATION**

*I hereby declare that the thesis entitled **Preservative Treatment and Chemical Modification of Rubber wood**, has not previously formed the basis for the award of any degree, diploma, associateship, fellowship or other similar titles or recognition.*

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## ABSTRACT

Rubber wood is an important low cost and plentifully available timber in Kerala. Because of the acute timber scarcity, the timber from rubber trees now finds many applications. The highly perishable nature of this timber due to its susceptibility to insect and fungal attack limits its wider utilization. The present study was aimed at developing appropriate techniques for converting this highly perishable timber into durable, value-added products.

A highly toxic but conventional preservative, the copper-chrome- arsenate (CCA) composition as well as more environmentally safer chemicals of low mammalian toxicity, viz., boric acid and borax, were investigated. In addition, the feasibility of developing a more environmentally friendly and non-conventional method of wood preservation through chemically modifying the wood by acetylation technique was also studied. In order to ensure the availability of raw material throughout the year and to prevent the possibility of fungal and borer attack during the storage period, the potential of under-water storage of the timber was also investigated. The effect of under-water storage on the physical and mechanical properties as well as on treatability was investigated in order to see whether the utilization value was affected or not and also to arrive at an optimum under-water storage period.

It is found that rubber wood can easily be diffusion treated with 10% boric acid equivalent (BAE) solution to improve its durability. The study determined the maximum thickness of wood that could be diffusion treated and the distribution of chemicals in the treated wood. Wood of thickness up to 50 mm can be easily diffusion treated with 10% BAE solution to adequate loading of chemicals as

prescribed in the New Zealand and British Wood Preserving Association's (BWPA) specifications (0.2% BAE retention in the core of sample and 0.4% BAE retention as a whole respectively). Wood of 75 mm and 100 mm thickness can be diffusion treated with 10% BAE solution, but only the New Zealand specification can be achieved. Reducing the duration of dipping time from 40 minutes to 30 minutes for the 25 mm thick material and from 160 minutes to 80 minutes for the 50 mm thick material does not affect the achievement of desired dry salt retention (DSR). The optimum storage period is two, three, nine and twelve weeks for 25, 50, 75 and 100 mm thick wood respectively, in order to get uniform distribution of chemicals.

Rubber wood is easy to treat by the vacuum-pressure impregnation method using boron chemicals as well as with CCA preservatives. An economical treatment schedule consisting of 15 minutes initial vacuum of 85 kPa followed by a pressure of 1000 kPa for 15 minutes and a final vacuum of 85 kPa for 5 minutes (denoted by 15'/15'/5') was arrived at for treating rubber wood in air-dried, partially-dried and green conditions with boron as well as CCA compounds. Further, it was also found that small variations in the extent of vacuum or pressure did not significantly affect the achievement of desired DSR.

Treating with 3% BAE solution by employing the economical schedule gave nearly double the loading of chemicals suggested by the Indian Standard ( $6.5 \text{ kg/m}^3$  for use in humid condition but not in ground contact) in air-dried wood and just adequate retention for partially dried wood. For rubber wood in green condition, the use of 4 to 5% BAE solution gave the desired DSR.

It is found that in rubber wood the penetration of chemicals through lateral sides of the timber is equally efficient as that in the longitudinal direction. The thickness and length of wood have more influence than the surface area per unit volume for the achievement of the desired DSR. Due to active lateral penetration of preservative, the various lengths and thicknesses of sizes required for common

end-uses are not a problem for the achievement of desired DSR. Also it is found that altering the strength of boron compounds in the treatment solution does not in any way adversely affect the solution pick-up. As the variation in solution pick-up with the concentration of boron solution is little, the concentration of solution required to achieve any desired DSR range can be predicted at an agreeable level of accuracy.

Commercial scale testing of the economical schedule with full load wood in the treatment cylinder showed that the schedule was capable of achieving the desired DSR in the case of air-dried, partially dried and green wood, both for boron and CCA treatments, provided solutions of optimum concentrations are employed depending upon the thickness and moisture content of the material. Further, it is seen that the newly developed economical schedule is more productive, much time- and energy-saving than the traditional schedules being followed in some commercial units.

Commercial scale testing of the CCA impregnation of green rubber wood showed that the use of 3 to 4% solution provides desired DSR in treated wood while employing the economical schedule. Except for very high thicknesses in the range of 100 mm, the use of 3% CCA solution is effective for green rubber wood (about 65% moisture content). When the moisture content is higher than this level it is desirable to use 4% CCA solution.

The penetration of both boron and CCA preservatives in the wood treated at air-dried, partially dried and in green condition using the economical schedule is found to be uniform and through and through.

Chemical analysis of the treated wood indicates that a net average measured DSR of  $3.0 \text{ kg/m}^3$  is sufficient to fulfil the requirements of New Zealand and BWPA specifications in the diffusion as well as impregnation treatment. Chemical analysis of CCA impregnated wood shows that the absorption of the

constituent elements of CCA in the timber treated using the economical schedule is in the desired ratio, provided the treatment conditions are properly monitored (i.e., the pH and concentration are not altered and sludge formation due to successive treatments is prevented). Hence, care has to be taken to ensure proper treatment conditions during CCA impregnation treatment for achieving the desired absorption of the different constituents of the preservative.

As far as storage method is concerned, it was found that rubber wood can be stored under water without any threat of sapstain development. Even though under-water storage for a period of 3 to 4 months reduced the physical and mechanical properties of wood, except for maximum compressive stress (MCS) the reduction of the properties (density, fibre stress at limit of proportionality (FSLP), modulus of rupture (MOR) and modulus of elasticity (MOE)) was found insignificant as to affect the utilization value of the timber. As the MCS was found significantly lowered even with a storage period of one month, it is advisable to avoid the use of under-water stored wood for end-uses where MCS is critical. The treatability was found to improve due to under-water storage. The economical schedule was found to be capable of achieving the desired DSR during impregnation treatment of under-water stored wood at all moisture content levels. For the treatment of under-water stored wood at air-dried condition the use of 3% BAE solution is sufficient, whereas for the treatment of under-water stored wood at very high moisture content levels (95% MC), the use of 6% BAE solution is essential to achieve the desired DSR levels.

The feasibility of chemically modifying rubber wood by acetylation technique for improved dimensional stability and biological resistance was investigated on solid wood blocks. It was found that an acetyl weight percent gain (WPG) of 18-22 can be achieved, which has been reported to be adequate to offer protection from biodegradation. Anti-shrink efficiency (ASE) in the range of 85-88% was achieved, indicating very good dimensional stability for the

acetylated rubber wood. Effect of chemical modification on physical and mechanical properties was also studied. MOR decreased slightly as the WPG increased. MOR of acetylated wood is slightly less than that of the untreated wood, but the difference is not large enough to affect the utilization value. MCS was found unaffected by acetylation. Density is improved to an average extent of 9%. In general, acetylation technique was found promising as a non-conventional and non-toxic, environmentally friendly way of protecting rubber wood and to impart improved dimensional stability required for specialty products.

Medium density fibre boards (MDF) made from acetylated fibres at a WPG of 14 showed that their dimensional stability was superior than that of the control boards. An average ASE of 94.8% was achieved for the thickness swelling of acetylated boards. Acetylation was found to improve the MOR of the MDF boards. The study has shown that rubber wood can be utilized as a potential raw material for the manufacture of MDF boards particularly for specialty products by applying the acetylation technique.

In brief, appropriate technology for the storage and preservative treatment of rubber wood to prevent biodegradation has been developed through the present study. This will help in the optimum utilization of this highly perishable but plentifully available non-conventional timber resource. This will indirectly help in conserving our natural forests since the pressure on forests for timber could be partially relieved by utilizing this resource.

## 1. INTRODUCTION

Wood is the most important and major forest product. It is one of the oldest known raw materials and from the pre-historic period man used it for fuel, tools, boat making and house construction. It is biological in origin, renewable and biodegradable.

Wood is also a modern raw material as well. Huge timber vaultings and precious furniture exemplify its glory. Besides solid wood applications, wood has become a valuable material in reconstituted forms such as plywood, particle board, fibre board, etc. It forms the basic substance for pulp and paper, films, fibres, additives, chemicals and many other products.

About one third of the world's land surface is covered by forests which contain a total growing stock of about 300 billion m<sup>3</sup> of wood (Steinlin 1979). Hence it is one of the most important products of nature. The global consumption of wood increased drastically since the early part of this century and further rapid increase in consumption is predicted for the future (FAO 1981). The world's total annual requirement of round wood by 2000 A.D. is estimated to vary between 3800 and 6200 million m<sup>3</sup>. The demand for industrial round wood and pulp wood is increasing day by day. The consumption of the most important product of chemical conversion of wood, pulp, is also increasing day by day. The decrease in the internal sources of supply and a growing concern for environmental protection in the developed countries resulted in an increased exploitation of tropical forests in the developing countries culminating in the fast depletion of tropical hardwoods and the resultant increase in price. FAO (1992) reported a substantial raise in the prices of various forms of wood.



One of the primary and historic reasons for the continuous decrease in the forest wealth of India is the increasing demand for fuelwood with the growing population. A further burden was from the excess demand for timber during the two world war periods for ship building and railways. The post world war period witnessed major agricultural expansion activities which resulted in a significant loss of forest cover, causing a further decrease in timber production. The demand for timber for construction, joinery, furniture and for the manufacture of modern construction materials like plywood, fibre board, etc. increased over the years. All these developments exerted pressure on the limited timber resources resulting in a rapid increase in timber prices. In India, the projected demand for wood is 63 million m<sup>3</sup> by 2000 A.D. (Govt. of India 1976).

In Kerala State, the imbalance between demand and supply of wood has been on the increase due to factors like growth of population and increased demand for timber with the increase in standard of living. Also, the negligence of sustained yield management practices of the forests led to loss of forest cover. The rapid rate of declining forest wealth created more environmental awareness which resulted in the stoppage of clearfelling of natural forests, and consequent shortage of timber supply. This has led to a further increase in timber prices in the State.

Krishnankutty (1990) reported that the effective demand for industrial timber in Kerala during 1987-'88 was 2.5 million m<sup>3</sup> round wood equivalent. The long term trend in timber prices, as reported by Krishnankutty (1989 a), gives a clear indication of scarcity of timber resources in the State. Projections for the future indicate that the likely supply-demand disparity can be neutralized to some extent by economizing the timber use.

## **1.1 WHY RUBBER WOOD?**

It is clear that timber plays a significant role in the economy of Kerala. Development of wood-based industries is therefore of great importance to the

State. But with the current rate of dwindling timber resources, there is uncertainty over the availability of raw material for timber industries. This critical situation calls for drastic changes in the pattern of raw material supply and utilization. One approach to partially solve the problem is to evolve appropriate technologies for the utilization of non-conventional timbers.

Rubber plantations are an important non-conventional source of timber. Rubber plantations ensure a sustained supply of rubber wood as the plantations are managed on a rotation of 25-30 years. Utilization of this resource can indirectly contribute to the conservation of natural forests. The major rubber growing ASEAN countries have the potential of producing 19 million m<sup>3</sup> of rubber wood logs per annum based on an annual replanting rate of 3% and an estimated yield of 100 m<sup>3</sup> per hectare (Ser 1990).

India is one among the leading rubber growing countries in the world. According to the estimates of the Rubber Board of India (RRII 1992), the total annual wood production is about 1.27 million m<sup>3</sup>, out of which 60% is stem wood and the remaining 40% branch wood. Kerala State accounts for about 86% of the total area under rubber cultivation in India (Mannothra 1993, George and Joseph 1993).

A study conducted by Krishnankutty (1989 b) showed that rubber wood accounted for around 65% of the total consumption of industrial wood, excluding sawmilling in Kerala, indicating the significant contribution of rubber wood in the industrial economy of the State.

The consumption pattern of rubber wood in India, as reported by the Rubber Board of India is given below (RRII 1992):

Consuming sector	Quantity consumed (million m <sup>3</sup> )	Relative share (%)
Packing case	0.49	64.5
Safety matches	0.13	17.1
Plywood	0.10	13.1
Processed wood	0.03	3.9
Others	0.01	1.4
Total	0.76	100.0

This clearly shows the need for more value addition and more efficient utilization, as is evident from the insignificant share of processed rubber wood in the total consumption.

It is estimated that about 50% of the demand-supply imbalance of wood can be resolved by rubber wood (Mannothra 1993). Thus rubber wood can play a very important role in the timber scenario of the State. Conversion of this perishable wood into value added products through chemical treatments warrants attention since it is available at comparatively low cost and the plantations are likely to become one of the most promising sources of wood in the state. Because of the acute timber scarcity, the stem wood of rubber tree now finds many applications. Effective utilization of this wood will relieve pressure on our forests to a significant extent.

## **1.2 NATURE OF THE PROBLEM**

Perishable nature of rubber wood - its susceptibility to fungal and insect attack - limits its wider utilization. Rubber wood will continue to be under-utilized if it is not treated with preservative chemicals for protection against fungi and

insects. It is because of this reason that rubber wood has been traditionally used for firewood, packingcases and match veneers and splints. Only through developing appropriate techniques for the protection, wood from rubber tree can be effectively and efficiently utilized. The protection can be approached in two different angles, one with the use of conventional toxic preservatives and the other by non-conventional method using environmentally friendly non-toxic chemicals, like chemical modification.

### **1.2.1 Biodegradation**

Rubber wood is classed under the category of perishable timbers. It is highly susceptible to the attack of biological organisms such as fungi, insect borers, termites and marine borers. Through the cut ends of the logs, soon after felling, fungal infection starts. The sapstaining fungi causes a bluish black stain and the aesthetic value of the timber gets affected. Along with this, the infestation of decay fungi causes deterioration of the wood which affects its physical and mechanical properties. Also rubber wood is highly susceptible to the attack of insect borers during the air-drying period. Heavy infestation of insect borers can lower its mechanical strength. Under marine conditions rubber wood is affected by the intense attack of marine borers.

### **1.2.2 Natural durability**

The high susceptibility of rubber wood to the attack of biological organisms makes it to be grouped under the 'durability class 3 type' of timbers, whose average life in ground contact is less than 60 months (BIS 1982). Balasundaran and Gnanaharan (1990 a) reported rubber wood as 'non-resistant' to decay fungi. Varma and Gnanaharan (1989) recorded it as highly susceptible to termite attack in ground contact. Santhakumaran and Srinivasan (1989) and Rao *et al.* (1993) found that rubber wood was easily degradable in marine conditions. All these studies clearly indicate that rubber wood is not naturally durable.

### **1.3 OBJECTIVES OF THE STUDY**

Preservative treatment is the most important aspect to be taken care of for the effective utilization of this perishable timber. Appropriate preservation techniques suitable for small scale industries as well as medium to large scale industries need to be developed/standardised. Diffusion treatment, suitable for both sectors, and vacuum-pressure impregnation (VPI) treatment, suitable for the latter, have to be standardized. Even though conventional wood preservative chemical composition such as copper-chrome-arsenate (CCA) is more effective, due to the increased environmental concerns emphasis has been given in the present study for the use of environmentally safer chemicals like boron compounds.

To ensure the availability of raw material throughout the year, rubber wood will have to be stored for few months. Because of its acute susceptibility to biodegradation, the method of storage is very important. Under-water storage is an effective way to protect timber from the attack of biological organisms. This method of storage needs to be evaluated in the case of rubber wood. It is also important to assess the effect of under-water storage on the physical and mechanical properties as well as on the treatability.

As environmental concerns are becoming more and more important these days, the possibility of using non-toxic compounds for wood preservation needs to be explored. Chemical modification of wood structure is a novel and environmentally friendly method of wood preservation. This can be achieved by blocking the confirmational sites required for the highly specific enzyme-substrate reactions, thereby modifying the wood so that the attacking microorganisms do not recognize it as a food source. Further, replacing the polar hydroxyl groups with less polar groups results in reduction in hygroscopicity which means increased dimensional stability.

It was decided to develop and standardize the optimum treatment conditions for the production of 'modified' or 'improved' rubber wood through chemical modification by acetylation technique. Another aspect of the study was to evaluate the effect of acetylation on the physical and mechanical properties of rubber wood for different end-use applications. The utilization potential of modified rubber wood fibres for the preparation of medium density fibre board (MDF) and the effect of acetylation on the dimensional stability and strength properties of MDF were also included as part of the study.

Thus the broad objective of the study was to develop appropriate techniques for converting the highly perishable rubber wood into durable, value-added products; thus to effect saving of our valuable natural forest from the pressure for timber.

## 2. REVIEW OF LITERATURE

Rubber wood becomes available at the time of harvesting the trees after their economic life. In earlier days, majority of this wood was used only for fuelwood purposes and later for the manufacture of short-life packing cases. Of late, due to the acute scarcity of timber, the utilization potential of rubber wood is realized on a worldwide level and several workers have evaluated its suitability for various end-uses. The timber has an attractive pale straw colour, straight grain and moderately coarse texture. Its characteristic smooth finish and colour make it a favourable choice for making attractive items. Rubber wood has a density range of 500-600 kg/m<sup>3</sup> and is classified as a light hardwood. The wood possesses medium strength properties comparable with many conventional structural timbers (Gnanaharan and Dhamodaran 1993). It has good machining properties (Hong 1985, Khoo 1993, Shukla *et al.* 1984). It has good drying characteristics, and kiln-drying schedules have been reported (Asano and Choo 1990, Choo and Grewal 1983, FRIM 1980, Grewal 1979 a & b, Hong 1985, Sharma and Kukreti 1981, Sharma 1989).

Several authors have reported its anatomical (Ashaari Haji Amin 1986, Panikkar 1971, Pendlebury and Petty 1993, Rao *et al.* 1983, Rao and Hemavathi 1989, Reghu *et al.* 1989 a & b, Tisseverasinghe 1970); chemical (Azizol and Sudin 1989, Chinchole and Apte 1969, D'Auzac and Pujarniscle 1967, Guha and Lee 1972, Khoo 1993, Lim 1985, Patrick 1956, Sandermann *et al.* 1965, Simatupang *et al.* 1993, Sudin *et al.* 1990, Tan 1989, Tomimura *et al.* 1989 a, Tomimura 1993); physical (Bhat *et al.* 1984, Sharma *et al.* 1982) and mechanical (Gnanaharan and Dhamodaran 1993, Kamala and Krishna Rao 1993, MTIB

1982, Sanyal and Dangwal 1983, Shukla and Lal 1985 and Shukla 1989) properties.

The timber is widely suggested for various end-uses such as structural timber for construction purposes (Tan and Hong 1989, Shukla 1989), furniture (Cheow 1982, Tan 1975, Todd 1970) parquet flooring (Ali Zafar 1990, Jagadeesh and Damodaran 1992, Mohd. Shukri 1983, Tan and Mohd. Ali 1980), plywood (Birje *et al.* 1989, Wong 1979), particle boards (Lew and Sim 1982, Wong and Ong 1979), fibre boards (Jain 1965, Khoo *et al.* 1990), cement and gypsum bonded particle boards (Gnanaharan and Dhamodaran 1985, Simatupang *et al.* 1993, Sudin *et al.* 1990), solid bent works (Ser and Lim 1980, Sharma *et al.* 1982), moulding works (Sim and Mohd. Arshad 1980), handicrafts (Krishna Rao *et al.* 1993), wood-polymer composites (Kaderkutty 1989), etc. From the strength point of view, rubber wood was evaluated for its suitability for door and window shutters, furniture, dunnage pallets, tool handles and as a structural timber for construction purposes, besides its wide use for match veneers and splints as well as for compressed shuttle blocks (Kamala and Krishna Rao 1993, Shukla 1989).

Even though the timber is having many merits, the most serious problem for its efficient utilization is its perishable nature. It is highly susceptible to the attack of sapstaining and decay fungi. Also it is susceptible to insect borers and termites. Hence, for any utilization purpose, the timber needs to be protected with preservatives. It is this aspect that is reviewed in depth in this thesis.

## **2.1 BIODEGRADATION**

The fungal problem in rubber wood has been studied by Ali (1977), Ali *et al.* (1980), Florence (1989), Florence and Sharma (1990) and Tisseverasinghe (1969, 1970). The common blue stain fungus *Botryodiplodia theobromae* together with surface moulds like *Aspergillus*, *Penicillium* and *Trichoderma* and



basidiomycetous decay fungi like *Ganoderma applanatum*, *Lenzites palisotti*, *Poria* spp. and *Trametes corrugata* are reported to attack rubber wood. Blue stain fungi cause deep penetrating discolouration in the untreated rubber wood. The moulds cause only superficial infection which can easily be removed during product manufacture while the rotting fungi are more serious as they cause wood decay under humid conditions.

Hong (1982) reported that the decay fungus *Ganoderma applanatum* caused the greatest weight loss in wood samples and showed a preference for lignin degradation. *Schizophyllum commune* decayed the samples at a slower rate, but preferred lignin to cellulose whereas *Poria* spp. caused the least weight loss and showed a slight preference for cellulose. Out of the 10 commercially important timber species studied by Florence (1989, 1991) and Florence and Sharma (1990) rubber wood was the most affected by *Botryodiplodia theobromae* and this species has been consistently isolated from blue stain infection of rubber wood by various workers (Hong 1980, 1981, Hong *et al.* 1982, 1985).

Balasundaran and Gnanaharan (1990 a) reported the decay resistance of rubber wood against *Gloeophyllum trabeum* (brown-rot fungus) and *Polyporus versicolor* (white-rot fungus) and classified the timber as 'non-resistant' towards decay fungi. Ananthapadmanabha *et al.* (1989) tested the differential decay resistance of rubber wood against *Ceratocystis ulmi*, *Chaetomium globosum*, *Fomes annosus*, *Gloeophyllum trabeum*, *Polyporus hirsutus*, *P. meliae* and *P. versicolor*. Both the above studies concluded that rubber wood had the least natural resistance to some of the common tropical decay fungi, even though the resistance to different fungi appeared to be relatively different.

Norhara (1981), Tan *et al.* (1979) and Tisseverasinghe (1970) documented the insect borer attack in rubber wood. Bostrychid borers *Heterobostrychus unicornis* and *Sinoxylon conigerum* were found to be the main insect species attacking rubber wood in Sri Lanka. *Heterobostrychus aequalis*, *Minthea*

*rugicollis*, *Sinoxylon anale* and *Xylothrips flavipes* caused serious damage to rubber wood in Malaysia. Abood *et al.* (1992) reported that *Minthea rugicollis* was the most destructive and commonly found insect borer attacking dried rubber wood in Malaysia. Gnanaharan and Mathew (1982) listed 10 species of insect borers attacking rubber wood in Kerala, India and reported that *Sinoxylon anale* caused the most serious damage.

Rao *et al.* (1993) and Santhakumaran and Srinivasan (1989) stated that rubber wood was easily degradable in marine conditions. Main species of marine borers encountered were *Bankia comapanellata*, *B. rochi*, *Lyrodus pedicellatus*, *Martesia striata* and *Nausitora hedleyi*.

## 2.2 PROPHYLACTIC TREATMENTS

Application of anti-transpirant endcoats was found effective as prophylactic treatment against sapstain of tropical hardwoods (Olofinboba 1973). Hong *et al.* (1980) found incorporation of 2% Captafol and 1% Fennotox S2 in Shellkote, a bituminous compound, prevented the development of sapstain for a period of 4 weeks. In a similar study, Tan *et al.* (1980) reported about the control of fungal attack during the storage of rubber wood. End treatment with 2% Captafol or 3% sodium pentachlorophenoxide (NaPCP) incorporated in Shellkote gave a fairly good control of fungal attack for a period of 4 months.

Lewis and Spence (1985) stated that effective prophylactic treatments have a significant role in increasing the viability of rubber wood as a raw material. They also stressed the need for the treatment integrating from the log to the sawn timber in conjunction with rapid extraction and conversion to obtain full, practical benefit. They reported that end-grain sealing with 'Shellkote' containing fungicides such as 4% NaPCP or 1.5% 'Antiblu 3739' (active ingredient, methylene-bis-thiocyanate (MBT) and 2-thio cyano methyl thio benzo thiazole (TCMTB)) or 4% Captafol could reduce the rate of sapstain penetration, though

not prevent it. Significant stain and mould on sawn timber can be prevented by a 1 or 2 minute dip treatment in 1.5% 'Antiblu 3739' or 2% NaPCP. Shi and Tan (1986) suggested the treatment with a BBP mixture (3.5% borax, 3% boric acid and 3.5% NaPCP) for the protection of rubber wood in China.

Jose *et al.* (1989) reported that NaPCP was the best preservative and in combination with boric acid and borax controlled fungal and insect borer attack. They also reported that oxycarboxin, next to NaPCP, showed promise in controlling fungi and incorporation of insecticide, viz., monocartophos and phosphamidon was effective against insect borer attack for two months. According to them the use of 10% copper sulphate solution was effective against insect borer attack for two months.

### **2.3 SAPSTAIN CONTROL**

Sodium salt of pentachlorophenol (NaPCP) was the most widely used preservative for the control of sapstain. A concentration of 0.4-0.5% was suggested (BCL 1972) to control sapstain development. The compound is highly toxic to fungi. PCP is known to be toxic to humans and other animals also. NaPCP has been known to produce irritant skin problems after long periods of handling. Now it has been banned in a number of countries, including India. Because of the concern for the toxicity of PCP there is a constant search for finding alternatives which could be readily available for the control of sapstain.

Hong (1981) screened 18 preservatives for their efficacy against sapstain and mould fungi (*Botryodiplodia* spp., *Sphaeronaema* spp. and *Trichoderma* spp.) isolates from rubber wood. NaPCP was used as the standard. Only one, Basiment 9230, was found to match NaPCP in terms of overall antifungal activity. Six other commercial products, Busan 30, Benomyl, Fennotox S2, Mitrol PQIC and Mitrol PQIL were found promising but warranted further tests. Hong *et al.* (1982) reviewed the biodeterioration problems of rubber wood and the control measures

against sapstain, mould, fungi and insect borers. The common method of control suggested is the dip treatment using a preservative mixture of 1-2% NaPCP and 1.5% borax. Synthetic pyrethroids and organic solvent-based preservatives are also used, though in much smaller quantities. Gnanaharan and Mathew (1982) reported that 0.3% Busan in 10% boric acid equivalent (BAE) solution could not control fungal growth in rubber wood. Higher doses of Busan were not tried as they would not be cost effective. In field tests conducted at Brazil, only a higher dose of Busan (1.5%) showed effectiveness against sapstain and mould fungi (Milano 1981). Plackett (1982) found that treatments with 1% and 2% Busan were 89% and 97% effective respectively against sapstain and mould over a 5-month field test period.

Hong *et al.* (1982) reviewed the work on the protection of rubber wood against stains caused by sapstaining fungus (*Botryodiplodia theobromae*) and mould fungi (*Aspergillus niger*, *Curvularia* spp., *Fusarium* spp., *Penicillium* spp., *Sphaeronaema* spp. and *Trichoderma* spp.) as well as against decay fungi (*Chaetomium* spp., *Ganoderma* spp., *Polyporus* spp., *Pycnoporous sanguineus*, *Schizophyllum commune* and *Stereum* spp.). The control measures suggested include application of endcoats incorporated with preservative chemicals, keeping logs under water in log ponds (for long-term storage), etc. for unconverted billets. For sawn timber, immediate dip treatment in anti-stain chemicals, followed by preservative treatment and proper drying practices can protect the timber from biodegradation. Hong *et al.* (1983) compared the standard treatment (1.5% NaPCP + 2% borax) with four less toxic preservatives in a simulated timber-yard trial with boards of rubber wood. Sapstain attack was not significantly different between treatments.

Tan *et al.* (1983) suggested the incorporation of 2% NaPCP in the treatment solution for effective control of sapstain infection during seasoning. As an alternative to NaPCP, incorporation of 2% 'Basillit SAB' in the treating solution

appeared to be moderately effective against sapstaining infection, if the wood was treated on the same day of conversion.

Hong *et al.* (1985) reported that the alkyl ammonium compound (AAC), benzylkonium chloride, at a concentration of 0.12% completely inhibited the growth of the sapstain fungus, *Botryodiplodia theobromae*; mould (*Fusarium* spp.) as well as two decay fungi (*Pycnoporus sanguineus* and *Schizophyllum commune*). Gnanaharan (1986) reported that two formulated products of alkyl ammonium compounds, viz., 'Sinesto B' (at 2.0% active ingredient concentration) and 'Akzo EN 494' (at 0.7% a.i. concentration) had a potential to replace the NaPCP, the environmentally unfriendly compound used for the purpose.

Florence (1989) and Florence and Sharma (1993) reported that the application of sodium azide could inhibit the fungi. These studies report the efficacy of the compound at various concentration levels but the compound is hazardous in nature. Florence and Sharma (1990) reported that the bacterium *Bacillus subtilis* had the potential as a bio-control agent against the sapstain fungus, *Botryodiplodia theobromae*. Florence *et al.* (1993) studied the influence of moisture content of rubber wood on the growth of sapstain fungus *Botryodiplodia theobromae* and suggested that if the moisture content of the wood was reduced to less than 24%, the wood can be protected from fungal sapstain.

## **2.4 BORON DIFFUSION TREATMENT**

Diffusion treatment with boron compounds is one of the simplest, cheapest and effective way of protecting wood from biodeterioration. Because of the advantages of borates such as low toxicity, colourless, odourless nature and ease to handle, boron treatment has been a popular method of protecting timber in New Zealand and Australia for the last 40 years (Carr 1957, Harrow 1952, Tamblyn *et al.*

1968, Tamblyn 1975). Research on preserving wood with borates has been reviewed by Cockcroft and Levy (1973) and Bunn (1974). Because of the environmental concerns on toxic chemicals like arsenic and chromium in the copper-chrome-arsenic (CCA) type of preservatives, recently renewed worldwide interest is focused on the application of boron compounds for timber preservation. Although the method of simple diffusion treatment with boron compounds is cheaper, one disadvantage is that a long diffusion storage period is required to meet the core retention specifications. The Timber Preservation Authority of New Zealand suggests 4-6 weeks diffusion storage period for 25 mm thickness and 8-12 weeks for 50 mm thickness (NZTPA 1986, cf. Page *et al.* 1987). Thus a good portion of the production will always be immobilised in the diffusion storage stage. Further, it has been reported that certain sapstain and mould fungi grow prolifically on boron-treated timbers during diffusion storage period (McQuire 1959). This problem was observed in the case of rubber wood also (Tisseverasinghe 1969, 1970). This necessitates the incorporation of a suitable anti-sapstain chemical in the preservative solution. The most effective and cheap chemical, though banned now-a-days, is still NaPCP. Substitutes for NaPCP as anti-sapstain chemical are under search. McQuire and Goudie (1972) tried to develop methods for an accelerated boron diffusion treatment of timber. They tried the hot and cold process with subsequent diffusion storage at a temperature of 40°C. By this method the diffusion storage time was brought down to half a week from the normal 4 weeks for the 25 mm thick sizes. Though the method drastically cuts down the diffusion storage period, it requires sophisticated machinery. This method will not be suitable for small size sawmills in India.

Tisseverasinghe (1975) developed a method to introduce preservatives into wood by employing the principle of differential release of capillary tension in the vessels of wood. This method involves spraying a boron solution of lower concentration on green wood with a spray cycle of 15 minutes on and 15 minutes

off. It was claimed that full penetration was obtained in 25 mm square within 12-15 hours. A modification of this method will be to have repeated immersions in the preservative solution for shorter times instead of one immersion for longer time. But this will also be a cumbersome process.

Vinden and McQuire (1980) reviewed the developments in wood preservation processing techniques in New Zealand. Vinden *et al.* (1985) reviewed the practical alternatives and options for the accelerated boron diffusion treatment. The status of research on borates as wood preserving compounds in the United States was reviewed by Barnes *et al.* (1989). An international conference was held in 1990 to take stock of the worldwide status of boron compounds as wood preservatives (Hamel 1990). Trials on protection of timber with boron compounds, mainly with fused borate rods and sodium octaborate tetrahydrate, were reported by Lebow and Morrell (1989), Morrell and Lebow (1991) and Morrell *et al.* (1990, 1992). The latest development in this field is the vapour phase boron treatment, developed by Burton *et al.* (1990), Turner *et al.* (1990) and Bergervoet *et al.* (1992). In developing countries interest still exists in the boron diffusion treatment mainly because of the fact that the treatment method is inexpensive and simple.

With regard to rubber wood, Gnanaharan and Mathew (1982) reported that it can be treated by diffusion process with 10% boric acid equivalent (BAE) solution containing 0.5% NaPCP. Reports of Gnanaharan (1982), Gnanaharan *et al.* (1983) and Dhamodaran and Gnanaharan (1989) showed the achievement of 0.4% BAE dry salt retention (DSR) in samples of thickness 25 mm with a dipping time of 40 minutes in 10% BAE solution. Treatment by this method provides protection against decay fungi (Balasundaran and Gnanaharan 1990 b) and insect borers (Gnanaharan *et al.* 1983) and termites (Varma and Gnanaharan 1989). The effect of immersion time on loading and distribution of boric acid in rubber wood was reported by Dhamodaran and Gnanaharan (1984). This study

showed that DSR increased with increase in immersion time, although the rate of increase was low. For higher thicknesses prolonged immersion time reduced the DSR. Hong *et al.* (1985) suggested a diffusion storage period of 10 weeks for rubber wood with 35% moisture content (MC) to ensure 100% penetration of test pieces of cross-section 50 x 50 mm. Fresh sawn wood (75% MC) and wood with 45% MC required only a shorter diffusion period of 3 weeks and 6 weeks respectively for 100% penetration. A minimum diffusion period of 21 days was suggested for sizes of cross-section 50 x 50 mm to achieve 0.2% BAE retention in the core of treated rubber wood. They also indicated the possibility of using multi-salt formulations such as boron, fluorine, chromium and arsenic (BFCA) compounds for the dip-diffusion treatment of rubber wood.

## **2.5 VACUUM-PRESSURE IMPREGNATION (VPI) TREATMENT**

### **2.5.1 Boron impregnation treatment**

While using a 2% BAE solution, Tan *et al.* (1979) obtained a mean solution absorption of around 200 kg/m<sup>3</sup> in the freshly converted wood and 350 kg/m<sup>3</sup> in one-week-old pieces. Green rubber wood is considered relatively easy to impregnate with preservatives when compared with other Malaysian light hardwoods (Tan *et al.* 1983). Tan (Unpublished, cf. Hong *et al.* 1982) suggested a treatment schedule of 90 kPa initial vacuum for 45 minutes followed by a pressure of 1050 kPa for 75 minutes and a final vacuum of 90 kPa for 15 minutes while using a 3% borax pentahydrate solution for the treatment of fresh sawn rubber wood boards of thickness 50 to 70 mm. Hong *et al.* (1982) also suggested to air-dry the pressure-treated timber for at least a week to achieve sufficient distribution of the preservatives before kiln drying. They reported that air drying periods of less than one week did not achieve the toxic threshold value of 0.2% BAE at depths of 12 mm or more in the treated timber. To overcome this problem the freshly sawn timber could be air-dried for a week prior to treatment. Such



timbers were found to contain more than 0.2% BAE up to a depth of 28 mm, even though they were kiln-dried immediately after treatment.

Tan *et al.* (1983) compared the effects of two different treatment schedules on 57 mm thick green rubber wood with a moisture content of about 60% and 35% while using a 3% BAE solution. They employed an initial vacuum of 90 kPa for a period of 45 minutes for both the schedules. The effect of varying the pressure period on solution absorption was studied while exerting a pressure of 1050 kPa for two different lengths of time, viz., 150 minutes and 75 minutes. A final vacuum of 90 kPa was maintained for a period of 15 minutes in both the schedules. They found that reducing the pressure treatment time from 2.5 hours to 1.25 hours did not result in an appreciable change in the absorption by freshly sawn and partially dried (MC around 35%) wood. They achieved a DSR of around 7.5 kg BAE/m<sup>3</sup> for the green wood with average MC around 60%. Hong *et al.* (1985) reported that rubber wood could be treated under the Bethel full-cell process with boron compounds achieving a DSR of 16 kg/m<sup>3</sup> for air-dried wood. They also found that rubber wood could be treated in green condition and the preservative retentions were almost directly proportional to the moisture content of the boards.

Hong and Liew (1989) reported the effect of three different treatment schedules, viz., full-cell, full-cell to refusal and full-cell with 12 cycles of vacuum/pressure while using a 3% BAE solution. In their study, an increase of pressure time from 45 minutes to 90 minutes allowed slightly higher retention values. They also concluded that it is possible to treat freshly sawn rubber wood with boron preservatives using the full-cell VPI process provided adequate pressure time was given to achieve the desired loading of chemicals. According to them the application of repeated rapid cycles of vacuum and pressure was a possible modification of the full-cell process for shortening treatment time to achieve adequate levels of preservative loadings.

Salamah *et al.* (1989) reported on the double vacuum process for the treatment of rubber wood and they achieved a core loading of 0.2% BAE. But the treatment schedule employed by them was too lengthy, an initial vacuum period of 225 minutes and pressure period of 150 minutes and a final vacuum for 10 minutes. However, for green wood, they reported that a combination of diffusion-vacuum pressure process was necessary to achieve the required chemical loading. This chemical loading is achieved within a period of less than a week only, after pressure treatment.

### **2.5.2 CCA impregnation treatment**

Tan *et al.* (1979) and Hong *et al.* (1982) reported that rubber wood was easily treatable by the Bethel vacuum-pressure process using copper-chrome-arsenic (CCA) preservatives. They reported that complete penetration of CCA was achieved in 50 mm thick boards at an average DSR of about 16 kg/m<sup>3</sup>. Tan *et al.* (1979) suggested a treatment schedule of 90 kPa initial vacuum for 45 minutes, followed by a pressure of 1500 kPa for 150 minutes and a final vacuum of 90 kPa for 20 minutes while using a 3% Tanalith CT 106 solution and achieved a solution absorption of 500 kg/m<sup>3</sup> CCA (cf. Hong *et al.* 1982).

Kuppusamy and Srinivasan (1989) reported a case study of penetration and absorption of the components of CCA in a treated rubber wood door shutter received from a commercial source. Although the penetration of the preservative as well as DSR were found to be good, data on absorption of individual components of CCA showed significantly low arsenic and high copper retention. This study throws light on the need for taking proper care during the CCA treatment of rubber wood.

Sonti *et al.* (1989) reported that rubber wood can be pressure impregnated at a moisture content of about 55-66% using CCA compound.

### 2.5.3 Impregnation treatment with other chemicals

Martawidjaja and Martawidjaja (1971) reported that pressure impregnation of rubber wood posts with creosote resulted in a mean durability greater than 22 years.

Hong *et al.* (1985) reported that rubber wood could be effectively pressure-treated with alkylammonium compounds (AAC) to desired retention levels. Tests using 1.5% benzylkonium chloride achieved a retention of around  $7.5 \text{ kg/m}^3$ . This value is very much higher than the toxic thresholds, ranging from  $0.2 \text{ kg/m}^3$ , as reported by Butcher *et al.* (1977). AACs have both insecticidal and fungicidal activity, but have comparatively low mammalian toxicity. They have good fixative action when impregnated into the wood. Rubber wood is found to fix between  $7.5$  and  $9.0 \text{ kg/m}^3$  of benzylkonium chloride even after subjecting the treated samples to leaching.

Synthetic pyrethroids, which are available in the form of emulsifiable concentrates as deltamethrin, permethrin and cypermethrin, and other aqueous preservative formulations such as TCMTB (thio cyano methyl thio benzo thiazole), MBT (methylene-bis-thiocyanate) and TBP-M (tribromophenol-metaborate) are some other compounds which have potential for pressure treatment. But, according to Hong *et al.* (1985), all this needs more rigorous evaluation for their performance. Sonti *et al.* (1989) reported that the formulations 'Antiblu' (MBT) and 'Antiborer' (cypermethrin) are effective for protecting rubber wood. 'Antiblu' at a concentration of 1.5% and 'Antiborer' at concentrations 0.1-0.25% were suggested. Ananathapadmanabha and Srinivasan (1993) tested a proprietary chemical possessing insecticidal and fungicidal properties with active ingredient 'deltamethrin', a photo-stable synthetic pyrethroid, and sold under the trade name 'K-Otek-Combi', which was effective in protecting rubber wood against biodeterioration and maintaining the natural colour.

The oscillating pressure method (OPM) has been practised in Malaysia for the treatment of rubber wood with synthetic pyrethroids. However, the efficacy in terms of penetration and the retention of the compounds has not been assessed. Salamah *et al.* (1992) treated rubber wood samples at green, partially dried and dried conditions by employing the OPM method using cypermethrin at 0.2% concentration. The amount of cypermethrin detected in treated wood by High Performance Liquid Chromotography (HPLC) was much lower than that by calculation. Rubber wood samples of low moisture content gave better penetration of cypermethrin. Also it was found that the amount of cypermethrin detected decreased as the drying time increased. A formulation that can compensate for the amount lost during drying may be able to provide a better retention in rubber wood.

## **2.6 METHOD OF STORAGE**

Storage of raw material for a few months is most likely required for running a medium to large-scale rubber wood processing unit, as the availability of this timber may not be always regular throughout the year. Because of its acute susceptibility to biodegradation, the method of storage of rubber wood is very important. Tan *et al.* (1980) showed that under-water storage is an effective way to protect wood from immediate fungal attack. They tried under-water storage up to a period of one month only. They found that this method of storage of raw material was superior to the application of endcoats to prevent sapstain development during the storage period.

In brief, a perusal of available literature on rubber wood indicates that it is a timber with great potential. It is apparent that if proper chemical treatment is given rubber wood can substitute any commercial grade timber used for various purposes.

## **2.7 CHEMICAL MODIFICATION**

Chemical modification of wood structure is one among the many methods of non-conventional wood preservation techniques using non-toxic compounds to improve the biological resistance and dimensional stability required for specialty products. It essentially consists of a chemical reaction between some reactive part of a wood component and a chemical reagent, with or without catalysts, to form a covalent bond between the two. Rowell (1975, 1977, 1983, 1988, 1991 a & b) reviewed the diverse aspects of chemical modification of wood. Recently, Kumar (1994) also reviewed the chemical modification of wood and stated that acetylation holds a great potential out of the various wood modification methods.

### **2.7.1 Acetylation**

Acetylation, the esterification reaction, is a single site reaction with the most abundant reactive sites, viz., the hydroxyl groups in lignin, hemicelluloses and cellulose in wood. Bulking the hydroxyl groups with acetyl groups results in blocking the conformational sites required by the highly stereospecific enzyme-substrate reactions to undergo. Thus the attacking organisms cannot recognize the modified wood as a food source. Acetylation results in a reduction of hygroscopicity due to the non-availability of hydroxyl groups for hydrogen bonding with water from the atmosphere and thereby imparting increased dimensional stability. The external volumetric shrinkage will also decrease as a result of the bulking of the fibres and resulting increase in the volume of dry cell-wall.

Acetylation has a number of advantages: the reagent, acetic anhydride, is non-toxic to humans; decay resistance can be achieved; catalysts and solvents are not essential; mechanical properties of treated wood are not significantly reduced; there is little colour change, good penetration and good dimensional stability, etc. (Rowell 1975).

### 2.7.2 Acetylation procedures

The most common reagent used for acetylation is acetic anhydride, which reacts with wood hydroxyls with or without catalysts forming wood ester and releasing acetic acid in the wood structure.

Many of the properties of acetylated wood depend on the method of acetylation. The temperature of treatment, duration of reaction and types and amount of catalysts all play a significant role in the extent of fibre degradation during treatment. The amount of moisture present in the wood is also important. Some moisture (2- 5%) seems to be needed for best reaction, but above this level the water hydrolyses the acetic anhydride to acetic acid.

Several acetylation procedures using acetic anhydride vapour have also been published (Arora *et al.* 1979, 1981, Ozolina and Svalbe 1966). Singh and Kumar (1981) evaluated various catalysts for vapour-phase acetylation with thio acetic acid. Rowell *et al.* (1986 b) found that the rate of acetylation was much lower for the vapour than for the liquid phase reaction, even though weight percent gains (WPG) above 20 was achieved by both methods. Liquid phase acetylation was more effective in reducing swelling in both liquid and water vapour tests than was the vapour phase acetylated wood (Rowell *et al.* 1986 a & b).

Rowell (1986) and Rowell *et al.* (1986 c) suggested a new procedure to acetylate flakes that greatly reduced the reaction time and simplified the chemical recovery. This procedure eliminated both catalyst and organic co-solvent by a simple dip procedure followed by heat curing, recovering the excess or unreacted chemical and finally oven-drying the reacted flakes. An acetyl WPG of 15-20% could be achieved in 1-3 hours with southern pine and 2-4 hours with aspen flakes. Flake boards made from flakes acetylated by the new dip procedure performed nearly identically in dimensional stabilization tests with boards made from flakes acetylated by refluxing acetic anhydride/xylene and was superior to

boards made from flakes acetylated with vapour phase acetylation with acetic anhydride alone.

### 2.7.3 Biological resistance

At weight percent gain (WPG) above 17, acetylated wood has been found to be resistant to attack by different fungi such as *Contophora cerebella* (Bekere *et al.* 1978, Ozolina and Svalbe 1966, Rugevitsa 1977, Svalbe *et al.* 1978); *Contiphora puteana* (Goldstein *et al.* 1961); *Gloeophyllum trabeum* (Goldstein *et al.* 1961, Koppers 1961, Peterson and Thomas 1978); *Lentinus lepideus* (Goldstein *et al.* 1961); *Polyporous versicolor* [*Coriolus versicolor*] (Goldstein *et al.* 1961, Koppers 1961, Tarkov *et al.* 1950); *Poria incrassata* (Goldstein *et al.* 1961, Tarkov *et al.* 1950); *Poria microsporia* (Tarkov *et al.* 1950) and *Poria monticola* (Goldstein *et al.* 1961).

Acetylated wood exhibits good resistance to most decay fungi (Miltz 1991, Takahashi *et al.* 1989), but has been found ineffective in controlling the attack of lower fungi. Even a WPG of 20 conferred no protection against mould and stain fungi (Wakeling *et al.* 1992) and acetylated samples failed in cooling tower trials within one year (Kumar 1994).

Wood flakes acetylated by the dip procedure suggested by Rowell (1986) and flake boards prepared from such material (Rowell *et al.* 1987) also showed greatly improved resistance to attack by brown-, white- and soft-rot fungi and tunnelling bacteria at acetyl WPG above 15. Veneer-faced low density particle boards made from totally acetylated material were shown to have resistance to attack by fungi, *Trametes versicolor* and *Tyromyces palustris* in a 8-week soil block test.

The average service life of acetylated yellow birch stakes in ground contact was reported to be 17.5 years compared with 2.7 years for the untreated control (Gjovik and Davidson 1973). Acetylation improved resistance against decay

at 6-20 WPG and against subterranean termites at 13-18 WPG (Kumar and Agarwal 1982, Kumar and Kohli 1986, Videlov 1986). Agarwal *et al.* (1986) reported that mango wood acetylated with thio acetic acid to a WPG of 12.8 became resistant to termites by making it unpalatable.

Acetylated wood resisted marine borer attack, but the efficiency does not match with CCA or creosote (Johnson and Rowell 1988).

#### **2.7.4 Dimensional stabilization**

An antishrink efficiency (ASE) of over 60% is achieved by acetylation to 20-25 WPG (Koppers 1961, Kumar and Agarwal 1982, Tarkov *et al.* 1950). Cell-wall modification results in lowering of cell-wall moisture and the fibre saturation point (Codd *et al.* 1992, Kumar *et al.* 1991, Miltz 1991).

Singh and Kumar (1981) obtained a maximum ASE of 66.6 at an acetyl content of 15.3%. Rowell (1982) observed that acetylation took place on the hydroxyl groups of cell-wall polymers with the formation of stable acetate groups. At WPG of approximately 20, modified wood will have an ASE of 60-70 in repeated water soaking tests and at this level of WPG, the oven-dry acetylated specimen was of the same size as under green condition.

Boards made with aspen flakes, acetylated to about 15% weight gain exhibited one-sixth to one-seventh the thickness swelling in comparison to unacetylated controls when samples were subjected to 24-hour water-soaking tests. Water sorption and thickness swelling is greatly reduced at a WPG of around 20 (Youngquist *et al.* 1986 a).

#### **2.7.5 Physical and mechanical properties**

Acetylated wood is more dense than untreated wood and has fewer fibres of lignocellulose per unit volume (Dreher *et al.* 1964). There is a slight wood colour change (usually darkening) upon acetylation.



Generally, most reaction systems, even those done under relatively mild conditions, cause a slight reduction in strength properties. Depending upon the nature of wood species and the reaction conditions this effect can be significant sometimes. Narayanamurty and Handa (1953) reported a slight decrease in modulus of elasticity (MOE) due to acetylation. Similarly, shear strength parallel to grain is found decreased (Dreher *et al.* 1964). Acetylated bamboo showed reduced strength properties (Zhou *et al.* 1985).

Reactions under very mild conditions yielded products without much significant loss in mechanical properties. Goldstein *et al.* (1961), Millitz (1991) and Tarkov *et al.* (1950) produced acetylated wood without adversely affecting the mechanical strength to a significant extent. Impact strength is found unaffected (Koppers' 1961). Rowell and Banks (1987) found no significant loss of tensile strength due to acetylation of pine and lime (*Pinus sylvestris* and *Tilia vulgaris*). Further, they also reported that any massive loss in toughness is unlikely due to acetylation.

Increase in certain mechanical properties are also reported in some cases. Compressive strength (MCS), fibre stress at limit of proportionality (FSLP) and work to proportional limit are reported to be increased. Modulus of rupture (MOR) of softwoods is increased (Narayanamurty and Handa 1953). Hardness is also reported to be increased due to acetylation (Goldstein *et al.* 1961). These increases in certain strength properties may be due to the influence of higher density of the acetylated wood.

Reconstituted board products made from acetylated wood can behave in various ways as far as their mechanical properties are concerned, depending upon the manufacturing conditions and resin ingredients. Acetylation process adversely affected the strength properties of aspen flake board at an acetyl weight gain of 20 (Youngquist *et al.* 1986 b). Creep, an important strength parameter for

long-term loading of wood members, is largely reduced in acetylated wood (Norimoto *et al.* 1987, 1992).

Dynamic mechanical properties of acetylated wood are reported to improve, probably due to the lowering of cell-wall moisture (Akitsu *et al.* 1993). Resonance frequency, an important factor in instrument tones, gets stabilized in the case of acetylated wood (Sasaki *et al.* 1988, Yano *et al.* 1988). Acetylation also reduces the effect of changing humidity on acoustic properties due to stabilization of the cell-wall (Yano *et al.* 1986).

### **2.7.6 Weathering properties**

Acetylated wood exhibited colour stability and reduced incidence of surface checking (Dunningham *et al.* 1992, Plackett *et al.* 1992). Resistance of acetylated wood to degradation by natural weathering was investigated at different levels of acetylation by Imamura (1993). Even after exposure to sunlight for more than one year, acetylated wood of above 20% weight gain retained nearly its original surface colour. The erosion rate of early wood and latewood, and wood substance loss during weathering were significantly reduced in wood which had been chemically modified by acetylation. Acetylated wood behaved differently than untreated controls when exposed to sunlight or sunlight and rain, as revealed by electron microscopic studies. Acetylation also reduced the rate of surface degradation due to UV radiation, but the protection was lost after exposure for 8-weeks due to loss of acetyl content (Plackett *et al.* 1992). Weathering degrade and rate and extent of swelling can be reduced by combining acetylation and lumen-filling treatment with methyl methacrylate (MMA) (Feist *et al.* 1991).

### **2.7.7 Acetylation of rubber wood**

Not much work has been done on the chemical modification of rubber wood. Tomimura *et al.* (1989 b) found that medium density fibre (MDF) boards made with acetylated rubber wood showed excellent water resistance, but their

mechanical properties decreased considerably. In another study, Ibrahim and Mohd. Ali (1991) reported that in the case of rubber wood the weight gain during acetylation with acetic anhydride increased with longer curing time. The dimensional stability of modified rubber wood samples was considerably improved.

Even though the high processing costs do not justify the modification treatments at the current level of development, as traditional commercial wood preservation techniques are relatively much cheaper and equally effective, rising environmental concerns have, however, threatened to phase out some of the conventional wood preservatives. In this context, chemical modification seems to be a good alternative. Due to this reason cell-wall modification studies have been receiving greater attention during the recent years, and many reactions like acetylation, yielding enhanced wood properties, have shown technical feasibility. As of today, the main property advantage in cell-wall modification is its resistance to biodegradation and improved dimensional stability.

### 3. MATERIALS AND METHODS

#### 3.1 BORON DIFFUSION TREATMENT

Boric acid ( $H_3BO_3$ ) and borax (sodium tetraborate decahydrate,  $Na_2B_4O_7 \cdot 10H_2O$ ), both technical grade, were used as preservative chemicals. A 10% boric acid equivalent (BAE) solution prepared by dissolving 5 kg of boric acid and 7.5 kg of borax in 100 l water was used as the treatment solution. Sodium pentachlorophenoxide (NaPCP) at a concentration of 0.5% was incorporated in the boron treatment solution for preventing the development of sapstain and mould fungi during the diffusion storage period.

Fresh green rubber wood billets from slaughter tapped trees of age 30-35 years, collected from felling areas in central Kerala were used in the study. The billets were sawn into cross- sections 25 x 25, 50 x 50, 75 x 75 and 100 x 100 mm with a length of 300 mm. Fifteen samples of each cross-section were treated by simple diffusion method with a 10% BAE solution.

The weight of each sample before and after the treatment was recorded in order to calculate the dry salt retention (DSR).

$$\text{Solution pick-up (kg/m}^3\text{)} = \frac{\text{difference in weight (kg)}}{\text{volume of wood (m}^3\text{)}}$$

$$\text{DSR (kg/m}^3\text{)} = \text{solution pick-up (kg/m}^3\text{)} \times \text{concentration (\%)}$$

The DSR, in terms of BAE % was obtained as,

$$DSR (\%BAE) = \frac{DSR (kg/m^3)}{\text{density of wood } (kg/m^3)} \times 100$$

The density and moisture content of samples were determined by taking sub-samples from the samples undergoing treatment.

The dipping time for the different thicknesses of samples was determined by the following formula suggested by Harrow (1952).

$$\frac{\text{Solution concentration } (\%) \times \sqrt{\text{Time } (hr)}}{\text{Loading } (\%) \times \text{thickness of wood } (cm)} = K (\text{constant})$$

Harrow arrived at a K value of 7 for radiata pine and Winters (n.d) suggested a K value of 8 for different species. Dhamodaran and Gnanaharan (1984) also found that a K value of 8 holds good for thicker samples of rubber wood. A net average DSR of 0.4% BAE was taken as the minimum threshold level of DSR that should be achieved by diffusion treatment. Hence in the above formula, loading % can be substituted as 0.4 and K as 8. Therefore the above formula can be rearranged as,

$$T = \left\{ \frac{3.2 t}{C} \right\}^2$$

where T = dipping time (hr); t = thickness of wood (cm) and

C = solution concentration (%BAE).

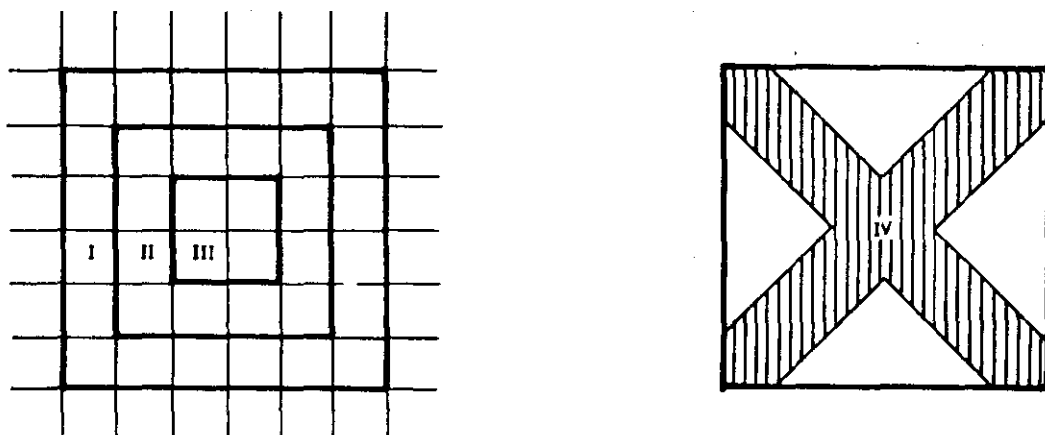
Using this formula, dipping time for various thicknesses of wood was calculated and it was rounded to 40, 160, 360 and 640 minutes respectively for 25, 50, 75 and 100 mm thick samples. To determine the effect of thickness on the diffusion treatability of rubber wood, the above dipping times were employed.

### 3.1.1 Effect of diffusion storage period

After immersing the samples of varying thicknesses for the appropriate dipping time, the solution was drained and the samples were kept for diffusion storage (close packing in polythene bags). In order to determine the optimum storage period, out of the 15 samples in each size category kept for diffusion storage, a set of five samples was withdrawn at three different intervals as given below:

Sample size (mm x mm x mm)	Diffusion storage withdrawal period (No. of weeks)		
25 x 25 x 300	2	3	4
50 x 50 x 300	3	5	8
75 x 75 x 300	6	9	12
100 x 100 x 300	9	12	16

From each sample, sub-samples were taken from the surface, middle and core portions (Fig. 1 a) for determining the distribution of chemicals. Also, two diagonal samples were taken as 'over-all' (Fig. 1 b) in order to determine the net



**Fig. 1** Sub-sample for chemical analysis. (A). I. 'Surface' (area of cross-section - 5/9); II. 'Middle' (area of cross-section - 3/9); III. 'Core' (area of cross-section - 1/9). (B). IV. 'Overall' (two diagonals)

average DSR in each sample. The samples were sliced into very thin sections and subjected to chemical analysis, as per the procedure of Wilson (1959) (section 3.2.4.1).

### **3.1.2 Effect of surface area per unit volume**

In order to know whether it is the surface area or the thickness of the wood which plays the major role in solution pick-up, diffusion treatment with 10% BAE solution was repeated with samples of length 300 mm and cross-sections 25 x 25; 25 x 50; 25 x 75; 25 x 100; 50 x 50; 50 x 75 and 50 x 100 mm with 10 samples in each set. The mean DSR was determined. The DSR per surface area per unit volume was calculated.

### **3.1.3 Effect of dipping duration**

In order to determine the effect of treatment duration on the DSR, samples of 300 mm length and of cross-section 25 x 25 and 50 x 50 mm were kept dipped in 10% BAE solution for various length of time as given below:

Sample size (mm x mm x mm)	Diffusion treatment period (minutes)
25 x 25 x 300	10, 20, 30 and 40
50 x 50 x 300	20, 40, 60, 80 and 100

There were 10 samples in each set. The DSR was determined.

### **3.1.4 Variation of solution pick-up with concentration**

The basal portion of one rubber tree of age 30-35 years, collected from a felling area in central Kerala was selected for preparing the study material. A clear billet of 300 mm length, having no apparent defect was peeled in a rotary peeling lathe.

The thickness of the veneer was 1.5 mm. The outermost layers and the layers close to the inner core were rejected. The veneer sheet was then cut into samples of 15 mm width. These samples were then carefully air-dried and 15 defect free clear specimens were selected for the dip treatment with solutions of varied concentrations. The treatment solution concentration varied from 1 to 10% BAE and dipping time was 10 minutes for every batch. The volume of each samples was recorded and the weight of material before and after treatment was noted. The moisture content, density and the solution pick-up were determined.

The viscosity of treatment solution was determined by capillary flow and surface tension by capillary rise method in order to correlate with solution pick-up. The variation of pH with concentration of treatment solution was also recorded. The density of treatment solutions was also measured with hydrometer for the purpose of correlating it with actual concentrations.

## **3.2 VACUUM-PRESSURE IMPREGNATION (VPI) TREATMENT**

### **3.2.1 Treatment with boron chemicals**

Air-dried (MC 12-18%) samples of size 63 x 63 x 1000 mm were impregnated with 3% BAE solution. The study was conducted in a pilot-type preservation cylinder (0.3 m diameter and 2.0 m long). The Bethel full-cell process was employed for the treatment.

After an initial vacuum period the preservative solution was introduced into the treatment cylinder containing the timber charge, with the vacuum pump working. After filling the cylinder the vacuum pump was stopped. The charge was subjected to a pressure for a particular period after which the preservative solution was withdrawn from the cylinder to the storage tank and a vacuum was once again applied to free the timber from excess preservatives.



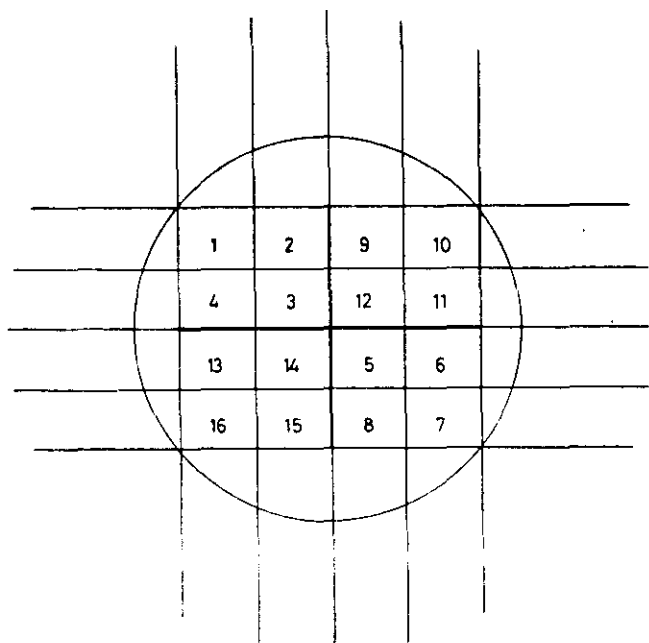
The maximum obtainable vacuum of 85 kPa and pressure 1000 kPa were used in the study. To arrive at the optimum treatment schedule, the effect of varying the initial vacuum time and pressure treatment time on the solution pick-up was studied. Final vacuum was maintained at 85 kPa for 5 minutes in all the trials.

To study the effect of different moisture levels on the uptake of chemical solution, three levels of moisture were tried: green wood (MC about 75%); partially dried wood (MC about 50%) and air- dried wood (12-18% MC). The dimension of the test samples remained the same as above. Trials were taken with 6% BAE solution for green wood. The dry salt retention was determined for every trial.

The penetration of chemicals in the treated wood was tested as per IS:2753 (BIS 1964) (section 3.2.3.1). The chemical analysis of boron impregnated samples was done as per the same procedure and sampling pattern mentioned earlier (section 3.1.1).

#### **3.2.1.1 Lateral penetration**

The butt log of two rubber trees of age 30-35 years was collected from a felling area in Trichur, Kerala. One log of 1 m length and another of 2 m length were prepared. The sampling pattern is as shown in Figure 2. This particular pattern was suggested in order to minimize the variation in solution pick-up due to the position of samples in the wood. Eight replicate samples of length 1 m and 2 m, having cross-sections 25 x 25 and 50 x 50 mm with and without end-sealing were treated with 3% BAE solution and DSR was determined. The schedule employed consisted of an initial vacuum of 85 kPa for 15 minutes followed by a pressure of 1000 kPa for 15 minutes and a final vacuum of 85 kPa for 5 minutes. The significance of penetration through lateral sides and effect of doubling the sample length on solution pick-up was analyzed by subjecting the DSR data to statistical analysis.



**Fig. 2 Pattern of sample selection for studying the effect of lateral penetration of preservatives**

### **3.2.2 Treatment with CCA preservative**

Freshly felled 1.5 m long rubber wood billets, converted into 25 x 25, 50 x 50, 75 x 75 and 100 x 100 mm cross-sections were used for the study. A 2% concentration of CCA ('ASCU' brand) preservative was used. Treatability was studied at two moisture levels: partially dried (MC about 50%) and air-dried (MC about 15%). The full-cell Bethel process was followed for the VPI treatment. An initial vacuum of 85 kPa followed by a pressure of 1000 kPa and a final vacuum of 85 kPa was applied in all the trials. The final vacuum period was maintained for 5 minutes in all the trials (same as in boron impregnation treatment). The effect of three different treatment schedules on the DSR was examined: (1) 15 minutes initial vacuum and 30 minutes pressure (referred as 15'/30'); (2) 30'/60' and (3) 30'/90'. DSR was determined as mentioned earlier.

### **3.2.3. Determination of penetration of preservatives**

Penetration of preservatives in treated wood was tested as per the Indian Standard IS:2753 (Part I)-1964 (BIS 1964).

The extent of penetration was tested by cutting the middle portion of treated test sample and spraying chemicals on the cut surface.

#### **3.2.3.1 Penetration of boron**

The following two reagents were prepared.

Reagent 1: Alcoholic extract of turmeric powder, prepared by refluxing 2 g turmeric powder with 100 ml 95% alcohol for 1 hour followed by cooling and filtering.

Reagent 2: Extract of salicylic acid and hydrochloric acid, prepared by saturating with salicylic acid a mixture of 80 ml distilled water and 20 ml of 30% hydrochloric acid.

The alcoholic extract of turmeric powder was applied on a reasonably dry surface of the material to be tested. The surface was allowed to dry for a few minutes and the salicylic acid - HCl extract was applied. The treated surface will develop red colour, while the untreated surface will remain yellow.

#### **3.2.3.2 Penetration of CCA**

Reagent was prepared by dissolving 0.5 g chrome azurol S and 5.0 g sodium acetate in distilled water and diluted to 100 ml.

The reagent was applied in the form of a mist, evenly over the surface to be tested. Immediately after spraying, the parts of the surface treated with CCA preservative change to blue colour, while the untreated parts will remain red.

### **3.2.4 Chemical analysis of treated wood**

#### **3.2.4.1 Analysis for boron**

Chemical analysis was carried out for the determination of boron in diffusion treated as well as VPI treated timber, as per the procedure of Wilson (1959), in order to determine the actual DSR. The method essentially consists of leaching the chemicals by refluxing with a very dilute solution of hydrochloric acid, followed by titration with alkali in the presence of neutral mannitol using pyrocatechol violet as indicator.

#### **3.2.4.2 Analysis for CCA**

Integrated Coupled Plasma Spectrophotometry (ICP) and Atomic Absorption Spectrophotometry (AAS) were followed in order to determine the amount of copper, chromium and arsenic present in the CCA treated rubber wood samples.

### **3.2.5 Full scale testing in commercial plants**

#### **3.2.5.1 Treatment with boron compounds**

Full scale trials were taken in six commercial plants. The concentration of boron treatment solution was measured with a hydrometer and was further confirmed by chemical analysis. Observations for the determination of DSR were taken on marked samples. DSR, density and moisture content of test samples were determined.

#### **3.2.5.2 Treatment with CCA preservative**

CCA treatment was tested at two commercial scale plants. The sample sizes, concentrations of solution and other observations required for DSR determination were recorded with respect to marked samples.

### **3.3 DETERMINATION OF PHYSICAL PROPERTIES**

#### **3.3.1 Moisture content**

Moisture content (MC) of wood samples was determined by the procedure prescribed by Indian Standard IS: 1708-1986 (BIS 1986). After determining initial weight, the sample was kept in an oven at 105°C for about 48 hours and weighed after cooling in a desiccator. The heating, cooling and weighing were repeated until constant weights were obtained. The moisture content was calculated as follows:

$$\text{Moisture content (MC) \%} = \frac{\text{Initial weight} - \text{Oven dry weight}}{\text{Oven dry weight}} \times 100$$

#### **3.3.2 Density**

Basic density was determined as per the Indian Standard IS:1708-1986 (BIS 1986).

It was determined by measuring the volume of wood sample by water displacement method and then placing the sample in an oven at 105°C for 48 hours and again determining its oven-dry weight after cooling in a desiccator.

$$\text{Basic density (kg/m}^3\text{)} = \frac{\text{Oven-dry weight (kg)}}{\text{Green volume (m}^3\text{)}}$$

### **3.4 DETERMINATION OF MECHANICAL PROPERTIES**

The main mechanical properties investigated in the study are the bending and compressive strength. The mechanical properties were determined according to Indian Standard IS:1708-1986 (BIS 1986). The specimens were defect free and 20 x 20 x 300 mm in size for static bending test and 20 x 20 x 80 mm for compression parallel to grain test. The span for static bending test was 280 mm. Load was applied continuously during the test at a constant rate. Fibre stress

at limit of proportionality (FSLP), modulus of rupture (MOR), modulus of elasticity (MOE) and maximum compressive stress (MCS) were determined using the formulae given below:

#### 3.4.1 Fibre stress at limit of proportionality (FSLP)

$$\text{Fibre stress at limit of proportionality, FSLP (N/mm}^2\text{)} = \frac{3Pl}{2bh^2}$$

where,  $P$  = load, in N, at the limit of proportionality which shall be taken as the point in load-deflection curve above which the curve deviates from straight line.

$l$  = span of the test specimen, in mm (280 mm)

$b$  = breadth of test specimen, in mm (20 mm)

$h$  = depth of the test specimen, in mm (20 mm)

#### 3.4.2 Modulus of rupture (MOR)

$$\text{Modulus of rupture (MOR) (N/mm}^2\text{)} = \frac{3P'l}{2bh^2}$$

where,  $P'$  = maximum load, in N. Other parameters are the same as mentioned in the earlier section, 3.4.1.

#### 3.4.3 Modulus of elasticity (MOE)

$$\text{Modulus of elasticity (MOE) (kN/mm}^2\text{)} = \frac{Pl^3}{4000 \Delta b h^3}$$

where,  $\Delta$  = deflection, in mm, at the limit of proportionality (from graph)

#### 3.4.4 Maximum compressive stress (MCS)

$$\text{Maximum compressive stress (MCS) (N/mm}^2\text{)} = \frac{P'}{\Delta}$$

where  $P'$  = the maximum crushing load (in N) and

$A$  = the cross-sectional area (in  $\text{mm}^2$ ) of sample

### **3.5 UNDER-WATER STORAGE**

Fresh rubber wood billets of 500-750 mm girth and 1 m length were used for the study. Eighteen billets were kept in round form with bark intact and 18 billets were squared. Some more billets were converted to planks of 150 mm width and 25, 50 and 75 mm thickness. There were 30 planks for each thickness. Squared billets and sawn planks were kept under water (tap water) in one tank and the round billets with bark were kept in another. Both the tanks were in an open (outside) place. Water was replaced every 10 days.

At the end of 45, 60, 90, 120 and 180 days, 5 planks each of 25, 50 and 75 mm thickness, 3 squared billets and 3 round billets were removed. The billets and sizes were sawn as shown in Fig.3 to look for the extent of sapstain.

#### **3.5.1 Evaluation of physical and mechanical properties of under-water stored rubber wood**

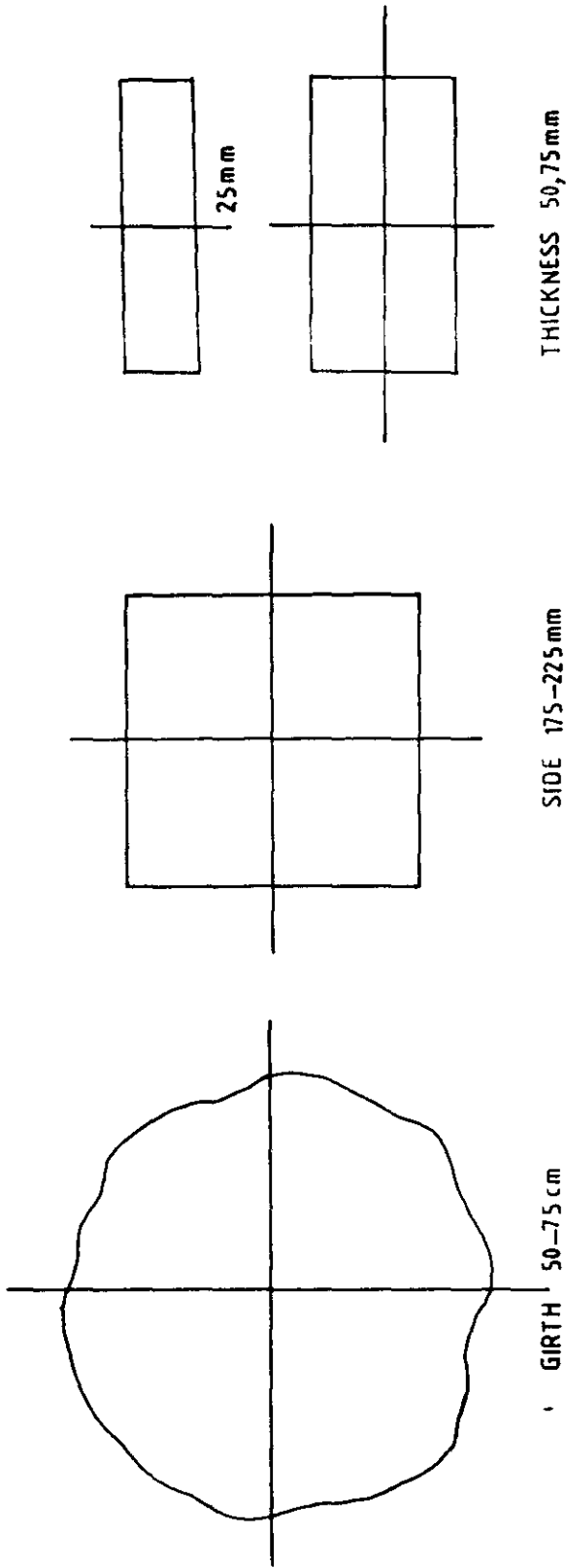
Rubber wood stored under water for 3, 4, 5 and 6 months were subjected to testing for their physical and mechanical properties as described earlier.

All strength data were corrected to 12% moisture content for comparison purposes, using the formula suggested by Sekhar and Rajput (1968). According to them,

$$\text{strength property at 12\% MC, } S_{12} = \frac{M_d S_d}{12}$$

where,  $S_d$  is the strength property at a moisture content  $M_d$ .

All the tests were repeated on samples prepared from non-ponded, air-dried control material for comparison purpose.



**Fig. 3** Sawing pattern of under-water stored billets and sizes, to examine the extent of sapstain penetration



### **3.5.2 Treatability of under-water stored rubber wood**

Samples after storage in water were grouped for sizes for the boron impregnation treatment trials. A 3% BAE solution was employed for the treatment. Wood at air-dried condition as well as at high moisture content level was treated. For the treatment of material at high moisture content, trials were conducted with 6% BAE solution also, to study the effect of concentration on DSR.

To study the effect of under-water storage on treatability, material stored under water for a period of 45 days and sawn into sizes 63 x 63 x 1000 mm and air-dried was used. This material was treated with 3% BAE solution using three different schedules, viz., 15 minutes initial vacuum of 85 kPa followed by a pressure of 1000 kPa for another 15 minutes and a final vacuum of 85 kPa for 5 minutes (denoted by 15'/15'/5'), 15'/30'/5' and 30'/30'/5' (vacuum was 85 kPa and pressure 1000 kPa in all the cases). The effect of duration of under-water storage on treatability was studied on material stored under water for 2, 4 and 6 months and sized into 50 x 50 x 1800 mm. In order to observe the effect of moisture content, this batch was treated at high moisture content level (95% mc), with 3% BAE solution. In order to determine the effect of concentration, 3 and 6 months ponded materials at high moisture content level (MC 95%) was subjected to treatment with 6% BAE solution also. The schedule, 15'/15'/5', was employed.

The effect of different treatment schedules on the DSR of under-water stored rubber wood at high moisture content (MC 95%) was studied on various sawn sizes with 3% BAE solution. Since the study material was of different sizes, in order to observe how the surface area available for penetration affected the achievement of desired DSR, the recorded DSR was divided with surface area of each samples.

Penetration of boron in the treated timber was also tested.

## 3.6 CHEMICAL MODIFICATION OF RUBBER WOOD

### 3.6.1 Acetylation technique

Sawn sizes obtained from freshly felled rubber trees were first air-dried and then oven-dried (105°C for 48 hours). They were re-sized into samples of cross-sections 10 x 10 mm and 20 x 20 mm. Oven-dried and weighed samples of size 20 x 20 x 5 mm were used for acetylation, which was carried out with 1 molar acetic anhydride solution in pyridine for various lengths of time at reflux condition (temperature - about 120°C). After the reaction, the treated samples were washed with acetone and again refluxed in acetone for three hours in order to remove the unreacted reagents and byproducts. The samples were oven-dried and weighed again for the determination of weight percent gain (WPG). The treatment duration yielding the best or optimum WPG was repeated with two different concentrations of acetic anhydride, viz., 1.5 and 3.0 molar solutions. In this trial, in order to achieve an easy and more loading of chemicals, vacuum impregnation of acetic anhydride followed by refluxing in the same reagent was carried out.

### 3.6.2 Determination of weight percent gain (WPG) of chemicals

In order to determine the WPG, the initial weight of individual samples (OD) was recorded. The oven-dry weight of samples after completing the acetylation procedure was noted.

$$\text{Then, WPG} = \frac{\text{OD weight sample after acetylation} - \text{OD weight of sample before acetylation}}{\text{OD weight of sample before acetylation}} \times 100$$

### 3.6.3 Determination of antishrink efficiency (ASE) of acetylated rubber wood

For dimensional stability studies, samples of size 20 x 20 x 5 mm were used. Dimensional stability was calculated by determining the volumetric swelling

coefficients of modified as well as control samples. The volumetric swelling coefficients are determined from the volume measurements made on samples before and after water soaking. Antishrink efficiency (ASE) percent values were calculated as per the formula suggested by Rowell (1983).

$$\text{Swelling coefficient, } S = \frac{V_2 - V_1}{V_1} \times 100$$

where,  $V_1$  = wood volume of OD sample before humidity conditioning or wetting

$V_2$  = wood volume of the same sample after humidity conditioning or wetting with water

$$\text{Then, ASE\%} = \frac{S_1 - S_2}{S_1} \times 100$$

where,  $S_1$  = un-treated volumetric swelling coefficient

$S_2$  = treated volumetric swelling coefficient

ASE = Antishrink efficiency or reduction in swelling (%)

The untreated volumetric swelling coefficient  $S_1$  was determined on control un-treated, oven-dried rubber wood samples of size 20 x 20 x 5 mm. The samples were prepared in such a way that the dimensions were accurate to the extent possible. The volume of samples before and after wetting was determined by measuring the dimensions of individual samples by using a digital vernier. For this purpose, points were marked on all the six faces of the samples and on all the occasions measurements were taken between these marked points. After measuring the volume, samples were kept immersed in water for 10 days and the volume was again recorded. In this way,  $S_1$  was calculated.  $S_2$ , the treated volumetric swelling coefficient, was also determined by the same procedure, by taking observations on treated samples.

Rowell and Ellis (1978) reported that values of percent dimensional stabilization were more realistic after the first soaking cycle for those treatments where the bulking chemicals have reacted with the cell-wall components. Hence, the effect of repeated water leaching of modified samples on dimensional stability was determined. For this, volumetric swelling coefficient,  $S_{2.1}$ , was determined from initial OD volume and the first water swollen volume, and  $ASE_1$  was calculated based on  $S_{2.1}$ . Similarly,  $S_{2.2}$  was determined from first water swollen volume and re-oven dried volume, and  $ASE_2$  was calculated based on  $S_{2.2}$ .  $S_{2.3}$  was determined from re-oven dry volume and second water swollen volume, and  $ASE_3$  was calculated based on  $S_{2.3}$ .  $S_{2.4}$  was determined from second water swollen volume and second re- oven dried volume, and  $ASE_4$  was calculated from  $S_{2.4}$ .

The first leaching duration was 10 days and the second, 20 days. The weight loss of samples due to the total 30 days leaching was also noted by recording the weight before and after leaching of the samples.

#### **3.6.4 Evaluation of physical and mechanical properties of modified rubber wood**

Static bending test was carried out on samples of size 10 x 10 x 150 mm, acetylated by the following three different conditions: (1) Treatment in 1 molar acetic anhydride solution in pyridine under reflux conditions for 12 hours, (2) vacuum impregnation treatment with 1.5 molar solution followed by refluxing for 3 hours and (3) vacuum impregnation with 3 molar solution followed by refluxing for 3 hours. The WPG and density were also determined from the weight and volume data recorded before and after the reaction.

Compression test parallel to grain was conducted on samples of size 10 x 10 x 30 mm, acetylated by vacuum impregnation with 1.5 molar solution. Here also the density and WPG were calculated.

Due to the technical restrictions and difficulties in acetylating thicker and longer samples in the particular acetylating system employed for this work, test samples selected were smaller in size. All the mechanical tests were carried out on oven-dried material, both on treated and non-treated control samples for comparison purpose.

### **3.7 PREPARATION OF MEDIUM DENSITY FIBRE BOARDS (MDF) FROM ACETYLATED RUBBER WOOD FIBRES**

A 0.5 m long billet from the basal portion of a rubber tree of age 30-35 years, was collected from a felling area in Trichur, Kerala, debarked, sawn and chipped, defibrated and oven dried. The material was acetylated using an excess of acetic anhydride without any solvents or catalysts for 2 hours at 120°C in a glass reactor. The acetylated fibre was washed with acetone to remove the unreacted or excess reagents and byproducts at reflux conditions and filtered under vacuum. All the modified fibre material were oven dried and weighed again and the WPG was calculated.

MDF boards were made with a phenol-formaldehyde (PF) resin (J1047+). Fibres were mixed with resin (8% by weight), made into a mat form and hot pressed at 160°C in a hydraulic press for 10 minutes. About 6mm thick boards were thus prepared. The boards were conditioned at 65% RH at 23°C for 48 hours. Control boards were also made in a similar way with non-acetylated fibres.

#### **3.7.1 Evaluation of physical and mechanical properties and dimensional stability of MDF prepared from acetylated rubber wood fibres**

The MDF thus made were cut into 150 x 15 mm size and subjected to static bending test. Boards made with control (non-acetylated) fibres were also subjected to the same test.

Dimensional stability tests were carried out on samples of size 40 x 40 mm. The initial thickness and volume of the samples were recorded using a digital vernier and subjected to swelling by keeping in water for 24 hours. The volumetric and thickness swelling was determined by measuring the thickness and volume of swelled samples. The swelling coefficient and antishrink efficiency (ASE) percentage values were calculated as per the formula suggested by Rowell (1983), as described in section 3.6.3. Density of sample was calculated from the OD weight and volume data.

## 4. RESULTS AND DISCUSSION

### 4.1 SPECIFICATIONS FOR DRY SALT RETENTION (DSR) OF PRESERVATIVES IN TREATED WOOD FOR PERISHABLE TIMBERS

As the very purpose of preservative treatment is to protect timber from biodeterioration, the preservative retention in the treated timber should be checked against the standard threshold value of the chemical for ensuring effective protection. In this regard, various countries have specified minimum threshold levels of different wood preserving chemicals in the treated wood. These values may vary depending upon the severity of the prevailing conditions existing in these countries.

The New Zealand specification for hardwoods susceptible to the insect borer, *Lyctus*, is 0.2% boric acid equivalent (BAE) in the core of treated wood (McQuire 1962). In 1980, even though New Zealand reduced the core retention requirement in softwoods to 0.1%, it retained the retention level of 0.2% for hardwoods (NZTPA 1986). The core is defined as one-ninth of the area of cross-section taken at the geometrical centre of the timber (BCL 1972). To ensure specified core loading of 0.2% BAE, McQuire (1962) found that the overall DSR measured by weight differences should be about 1% BAE. However, Gnanaharan and Mathew (1982) found that McQuire's (1962) findings in the case of radiata pine were not applicable in the case of rubber wood. They found that the chemicals penetrated throughout the cross-section of rubber wood suggesting that it was very permeable and did not pose any problem for the chemical to diffuse into the core.

The British Wood Preserving Association's (BWPA) specification is in terms of net dry salt retention in the whole sample. It recommends an average total DSR of 0.4% BAE (BCL 1972). This means when the whole sample is chemically analysed, it should contain an average 0.4% BAE retention.

According to the Australian specifications also, for effective protection against infestation by *Lyctid* beetle and *Anobiid* borers (house long-horn beetle), a minimum retention of 0.2% in the susceptible wood is required (Taylor 1967, Anonymous 1980). Even though none of these specifications are recommended specifically for rubber wood, adoption of the above specified threshold levels can effectively protect the timber. Malaysia is following a core loading of 0.2% BAE in the treated rubber wood (see Hong *et al.* 1982, Tan *et al.* 1983, Hong and Liew 1989, Salamah *et al.* 1989). A number of wood preservation companies in South East Asia have specified treating rubber wood to an average retention of 0.2% BAE.

According to Williams and Amburgey (1987), the minimal loading that prevents survival of *Lyctid* beetle larvae in wood is much below 0.2% BAE. According to them a threshold value range of 0.5% BAE retention will effectively offer total protection from decay fungi.

As is evident from the above discussions, there are two types of specifications: one specifying a core loading of 0.2% BAE; the other specifying a net average DSR of 0.4% BAE in the total sample. For ensuring a very effective protection, the present study was concerned to achieve a core loading of 0.2% BAE and over-all retention of 0.4% BAE. This will ensure protection of a perishable timber like rubber wood, in a tropical country like India.

An overall retention of 0.4% BAE equates to an average DSR of  $2.4 \text{ kg/m}^3$  boron chemicals in the treated rubber wood with a density range of  $500\text{-}600 \text{ kg/m}^3$ .



Even though boron compounds have been used for over 30 years in Australia and Europe for protecting timber, a standard for boron- treated timber for above ground, protected uses has been issued only recently by the American Wood Preservers' Association (AWPA). AWPA has promulgated a pressure treatment standard calling for a retention of 0.17 pound per cubic feet (pcf)  $B_2O_3$ , which is equal to  $2.72 \text{ kg/m}^3$  in the outer one inch (25 mm) for material greater than 50 mm thick (AWPA 1991). Further, it is also required to attain penetration to a depth of 2.5 inches (62 mm), or 85% of the sapwood.

The Indian Standard IS:401-1982 (BIS 1982) recommends a minimum DSR of  $6.5 \text{ kg/m}^3$  boron compounds, for building timbers for internal use but in ground contact or in humid conditions;  $5 \text{ kg/m}^3$  for internal use and not in contact with ground, and in dry conditions. It further envisages penetration of preservatives in the treated timber to a depth of 19 mm in the former and 12 mm in the latter. The recommended retention, in the case of CCA preservative, can vary in the range of  $5.0\text{-}6.5 \text{ kg/m}^3$ . For use as shooks for packing cases, ammunition boxes, cable drums and pallets (above ground, not under cover), the minimum recommended DSR of CCA is  $4 \text{ kg/m}^3$  with a minimum penetration of 6 mm. All these values are recommended not specifically for rubber wood, but the recommended levels of retention will hold good for rubber wood too.

## **4.2 BORON DIFFUSION TREATMENT**

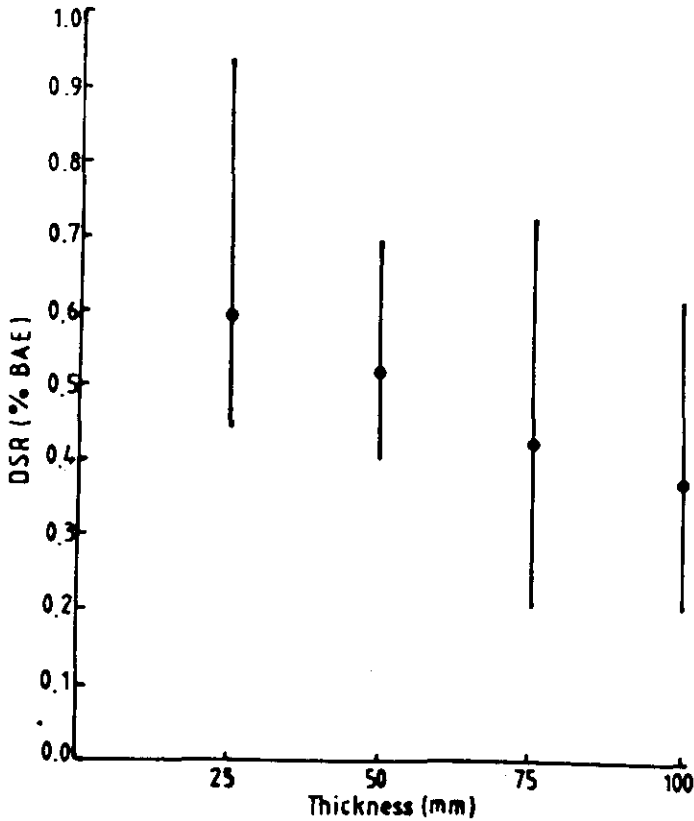
### **4.2.1 Developing simple diffusion treatment with boron chemicals**

The average moisture content (MC) of rubber wood samples was 60% and density  $550 \text{ kg/m}^3$ . The mean dry salt retention (DSR) values achieved by the treatment with 10% BAE solution with samples of varying thicknesses are given in Table 1 and Figure 4. Mean DSR, in terms of  $\text{kg BAE/m}^3$  varied from 3.4 to 2.1, and in terms of %BAE, from 0.6 to 0.38 in the case of 25 to 100 mm thick sections. The values of %BAE retentions reported in Table 1 are the calculated values.

**Table 1. Dry salt retention (DSR) obtained in boron diffusion treatment (CV values (in %) are given within brackets)**

Sample size (mm x mm x mm)	(DSR) (kg/m <sup>3</sup> )		% BAE	
	Mean	Range	Mean	Range
25 x 25 x 300	3.4 (27.5)	2.5-5.2	0.60	0.45-0.94
50 x 50 x 300	2.9 (13.8)	2.2-3.8	0.53	0.41-0.70
75 x 75 x 300	2.3 (29.1)	1.1-3.9	0.43	0.21-0.73
100 x 100 x 300	2.1 (28.3)	1.2-3.4	0.38	0.21-0.62

n = 15



**Fig. 4 Variation of dry salt retention (DSR) with thickness in boron diffusion treatment**

Results of an earlier trial reported by Gnanaharan and Mathew (1982) for the diffusion treatment of 25 mm thick cross-section with 10% BAE solution for the identical dipping time of 40 minutes are also in comparable range with the present results. They obtained a DSR of  $3.06 \text{ kg/m}^3$  which is equivalent to a calculated retention of 0.57% BAE. By chemical analysis they got an average value of 0.43% BAE retention for the same material kept for a diffusion storage period of 1 month. Further, Gnanaharan *et al.* (1983) proved that the material thus treated to a DSR of about 0.4% BAE was protected from the attack of insect borer, *Sinoxylon anale*. The mean DSR value obtained in the present trial is  $3.4 \text{ kg/m}^3$  and 0.6% in terms of %BAE. Thus the earlier finding (Gnanaharan 1982) that immersing 25 mm thick rubber wood material in 10% BAE solution at room temperature for 40 minutes gave adequate loading of chemicals, is further confirmed by the present study.

#### **4.2.2 Optimum storage period**

The effect of different diffusion storage periods on the distribution of chemicals in the treated wood is presented in Table 2 for cross-sections varying from 25 to 100 mm. It can be seen that the BWPA specification requirement of a net average DSR of 0.4% BAE (BCL 1972) can be achieved within a diffusion storage period of two weeks for 25 mm thick section and within three weeks for 50 mm thick cross-section. In the case of 75 and 100 mm thick sections, the 'overall' DSR achieved was around 0.3% BAE only. But, it is evident that in all these cases, the DSR in the core is greater than or equal to 0.2% BAE, which is sufficient to offer protection, as per the New Zealand specification for hardwoods susceptible to *Lyctus* beetle attack (McQuire 1962). This clearly points out that there is no need for keeping rubber wood of up to 50 mm thickness for longer diffusion storage period as normally suggested for other hardwood species: 4 weeks per every 25 mm thickness (see Vinden *et al.* 1990). In the case of 75 and 100 mm thick sizes, the minimum diffusion storage period tested, viz., 6 weeks and 9 weeks respectively, are just adequate to achieve the DSR required as per the

New Zealand specification of 0.2% BAE in the core. To ensure better penetration, it would be desirable to go for a diffusion storage period of 9 weeks and 12 weeks for the 75 and 100 mm thick cross-sections respectively. As seen in Table 2, this results in a core loading of 0.24 and 0.22% BAE retention for the 75 and 100 mm thick sizes respectively.

**Table 2. Effect of varying diffusion storage period on dry salt retention (DSR) (%BAE) (DSR determined by chemical analysis)**

Position	Size (mm x mm)	2 weeks	3 weeks	4 weeks
25 x 25				
Overall		0.45	0.47	0.54
Surface		0.59	0.60	0.61
Middle		0.48	0.51	0.56
Core		0.41	0.41	0.42
	Size (mm x mm)	3 weeks	5 weeks	8 weeks
50 x 50				
Overall		0.40	0.43	0.48
Surface		0.42	0.46	0.50
Middle		0.35	0.39	0.40
Core		0.30	0.34	0.36
	Size (mm x mm)	6 weeks	9 weeks	12 weeks
75 x 75				
Overall		0.32	0.33	0.37
Surface		0.33	0.35	0.38
Middle		0.26	0.29	0.32
Core		0.21	0.24	0.25
	Size (mm x mm)	9 weeks	12 weeks	16 weeks
100 x 100				
Overall		0.30	0.31	0.34
Surface		0.31	0.34	0.35
Middle		0.24	0.28	0.32
Core		0.20	0.22	0.25

n = 5

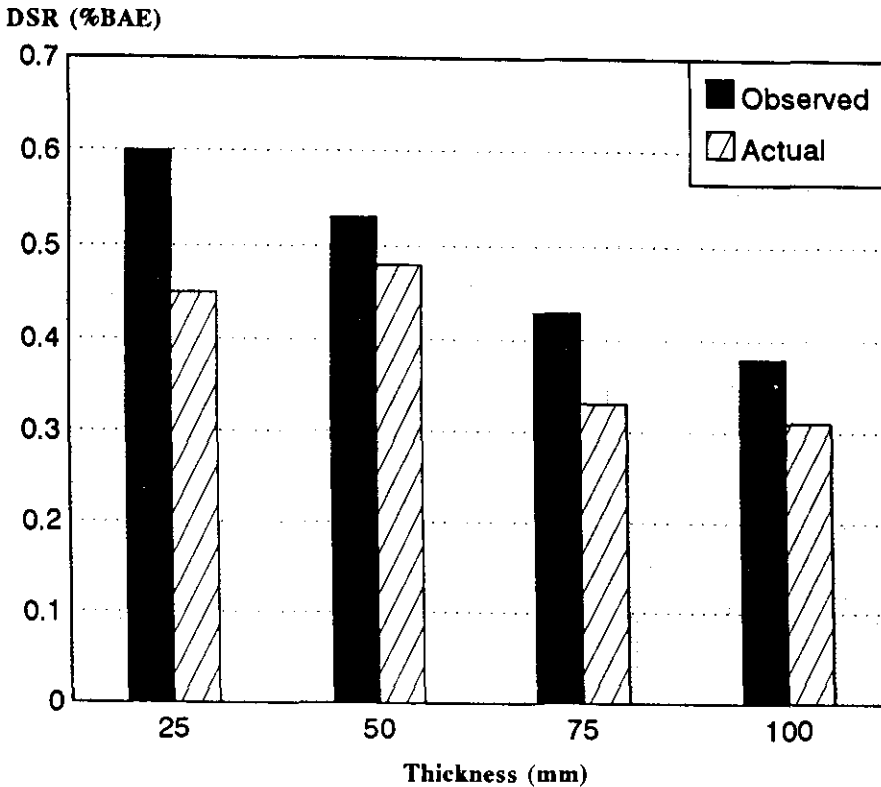
Further increase in the diffusion storage period did not result in appreciable increase in the DSR. Also it can be seen that the chemicals are distributed more or less uniformly within the samples of various thicknesses for the suggested diffusion storage periods.

Earlier studies by Balasundaran and Gnanaharan (1990 b), Gnanaharan *et al.* (1983) and Varma and Gnanaharan (1989) reported that a net average DSR of 0.4% BAE offered protection from fungi, termites and insect borers.

Table 3 and Figure 5 shows a comparison between the %BAE values obtained from the volume pick-up and density data and the actual values obtained by chemical analysis. The results summarized in Table 3 are those extracted from Table 2. The treated wood samples of 25, 50, 75 and 100 mm thick cross-sections subjected to chemical analysis mentioned in this Table were kept for diffusion storage for a period of 2, 3, 9 and 12 weeks respectively. The calculated values of total net average DSR in terms of %BAE retention and the mean of actual values obtained by chemical analysis differ by about 20% within a range of 9 to 25 per cent. This indicates that a total net average DSR of about 0.48% BAE (determined by gross absorption) gives an actual net average DSR of 0.4% (determined by chemical analysis) in the wood. This is equivalent to a measured DSR of about  $3.0 \text{ kg/m}^3$ , for a density range of  $500\text{-}600 \text{ kg/m}^3$  for rubber wood. So, if the BWPA specification of a total net average DSR of 0.4% BAE has to be achieved, then a total DSR of  $3.0 \text{ kg/m}^3$ , as measured by gross absorption has to be achieved. This is also in agreement with the retention value specified by the American Wood Preservers' Association (AWPA 1991).

**Table 3. Comparison between the calculated values (mean of 15) of %BAE retention and the actual values (mean of 5) obtained by chemical analysis in diffusion treated material**

Size (mm x mm x mm)	Calculated (A)	Actual (B)	Difference % $[(A-B) \times 100]/A$
25 x 25 x 300	0.60	0.45	25.0
50 x 50 x 300	0.53	0.48	9.4
75 x 75 x 300	0.43	0.33	23.3
100 x 100 x 300	0.38	0.31	18.4



**Fig. 5 Variation of dry salt retention (DSR) with thickness in boron diffusion treatment - Results of chemical analysis**

#### **4.2.3 Effect of surface area per unit volume**

Table 4 shows the results of the effect of surface area per unit volume of the samples on the DSR. No clear trend is observed. Rather than the surface area per unit volume of wood, it may be the thickness of sizes that is playing a significant role in the achievement of desired DSR.

**Table 4. Effect of surface area on dry salt retention (DSR) achieved by boron diffusion treatment**

Sample size (mm x mm x mm)	DSR (kg/m <sup>3</sup> )		CV (%)	Surface area (m <sup>2</sup> )	DSR
	Mean	Range		Volume (m <sup>3</sup> )	Surface area per unit volume
25 x 25 x 300	3.4	2.5-5.2	27.5	167	0.020
25 x 50 x 300	2.4	1.7-3.2	19.5	127	0.019
25 x 75 x 300	2.8	2.1-3.9	21.6	113	0.024
25 x 100 x 300	3.1	2.4-4.0	15.5	107	0.029
50 x 50 x 300	4.1	3.6-4.7	8.5	87	0.048
50 x 75 x 300	4.1	3.2-4.7	10.7	73	0.056
50 x 100 x 300	4.0	3.1-5.6	17.4	67	0.060
75 x 75 x 300*	2.3	1.1-3.9	29.1	60	0.039
100 x 100 x 300*	2.1	1.2-3.4	28.3	47	0.044

\*Extracted from Table 1; n = 15. In all other cases n = 10.

#### 4.2.4 Effect of treatment duration

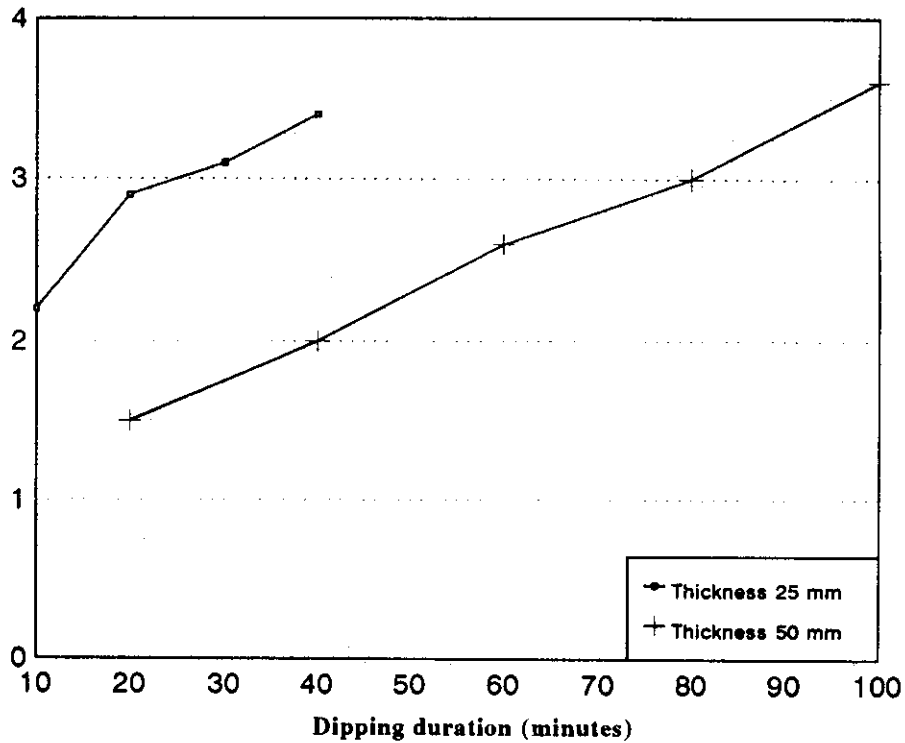
Table 5 and Figure 6 shows the effect of treatment duration (dipping time) on the DSR in the treatment of 25 x 25 and 50 x 50 mm thick sizes. It can be seen that a dipping time of 30 minutes is sufficient to achieve the minimum requirement of DSR for 25 mm thick sizes, and for 50 mm thick sizes, the minimum requirement of DSR can be achieved by a treatment time of 80 minutes. So it is possible to reduce the duration of diffusion treatment for 25 mm thick material from 40 minutes to 30 minutes and from 160 minutes to 80 minutes in the case of 50 mm thick sizes. This again confirms that rubber wood is easily treatable and the dipping time (determined by the formula suggested by Gnanaharan 1982, Dhamodaran and Gnanaharan 1984) can be further decreased for material of up to 50 mm thickness. As it was found that diffusion treatment with 10% BAE solution cannot achieve a total net average DSR of 0.4% BAE in sizes beyond 50 mm cross-sections (Tables 1 and 2), there was no point in trying to reduce the dipping period for these sizes.

**Table 5. Effect of duration of dipping on the dry salt retention (DSR)**

Dipping period (No. of minutes)	25 x 25 x 300 mm				
	DSR (kg/m <sup>3</sup> )		CV (%)	Av. BAE%	Range
	Mean	Range			
10	2.2	1.6-2.7	18.7	0.40	0.29-0.49
20	2.9	1.9-3.9	30.6	0.53	0.35-0.71
30	3.1	2.2-3.6	16.3	0.55	0.40-0.65
40	3.4	2.5-5.2	27.5	0.60	0.45-0.94
50 x 50 x 300 mm					
20	1.5	1.1-2.1	18.0	0.26	0.19-0.36
40	2.0	1.3-2.5	18.3	0.35	0.23-0.43
60	2.6	1.9-3.2	14.5	0.44	0.33-0.55
80	3.0	2.7-3.5	10.2	0.52	0.45-0.60
100	3.6	3.2-4.3	11.2	0.62	0.54-0.73

n = 10.

DSR (% BAE)



**Fig. 6 Effect of dipping time on dry salt retention (DSR)**



#### 4.2.5 Variation of solution pick-up with concentration

Table 6 shows the variation of solution pick-up with concentration of treatment solution while dip treating air-dried rubber wood veneers. It can be seen that the variation is very little except for solutions of 1-2% concentration where the chemical pick-up is only slightly higher than that with other higher concentrations. Hence the concentration required to achieve any desired DSR can be worked out to an acceptable level of accuracy.

**Table 6. Variation of solution pick-up with concentration of treatment solution**

Concentration of treatment solution (%)	Average solution pick-up* (kg/m <sup>3</sup> )
1	220
2	220
3	200
4	205
5	200
6	200
7	200
8	200
9	205
10	200

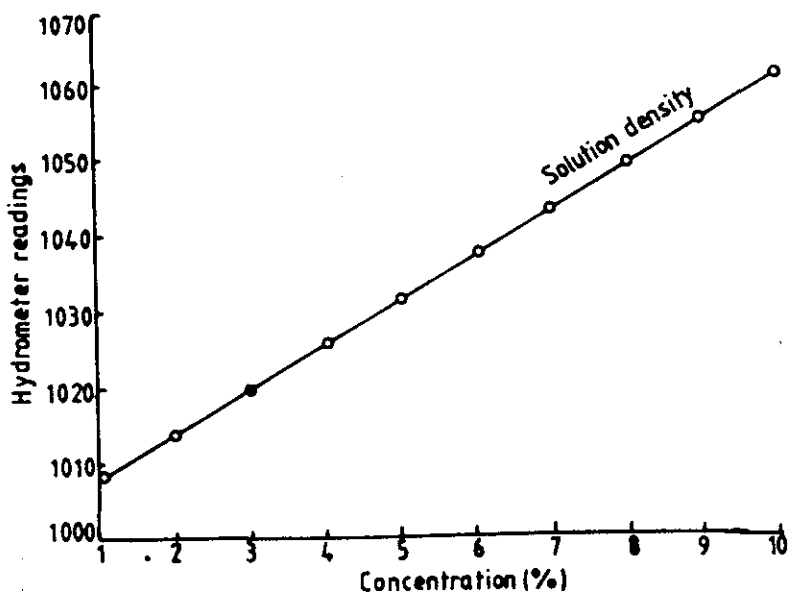
\*CV range 1-10%

Variation in surface tension and viscosity with solution concentration is also not large enough to influence the chemical pick-up significantly (Table 7). The variation of pH of treatment solution with concentration is also found negligible. Hence increasing the concentration of boron compounds in the treatment solution will not in any way adversely affect the solution pick-up. The density of treatment solution is found linearly related to concentration (Figure 7). Hence the variation of concentration of treatment solution during different batches of treatment can be monitored by checking the solution density by using a hydrometer.

**Table 7. Variation of solution properties with concentration of treatment solution**

Solution concentration (% BAE)	Mean viscosity of treatment solution (centipoise)	Mean surface tension of treatment solution ( $\times 10^{-3}$ N/m)	Hydrometer readings*	pH of treatment solution
1.0	1.0	3.7	1008	7.95
2.0	1.1	3.7	1014	7.93
3.0	1.1	3.8	1020	7.92
4.0	1.1	3.9	1026	7.94
5.0	1.1	3.9	1032	7.95
6.0	1.1	3.9	1038	7.97
7.0	1.2	3.9	1044	7.99
8.0	1.2	4.0	1050	7.93
9.0	1.3	4.2	1056	7.95
10.0	1.3	4.2	1062	7.97

\*A hydrometer reading of, say 1008, means solution density of 1.008 g/cc



**Fig. 7 Plot of solution density (measured as hydrometer readings) versus concentration of treatment solution**

#### **4.2.6 Summary of diffusion treatment**

If we were to meet the New Zealand specification of 0.2% BAE in the treated hardwood core, up to 100 mm thick sizes can be diffusion treated. Instead, if the BWPA specification of a net average DSR of 0.4% BAE has to be achieved, then only up to 50 mm thick sizes can be diffusion treated with 10% BAE solution. This means, to satisfy both these specifications, rubber wood up to 50 mm thickness can be diffusion treated effectively with 10% BAE solution. In tropical countries where biodegradation is more intense, it is desirable to adopt the BWPA specification of a net average DSR of 0.4% BAE in the treated rubber wood.

For end-uses requiring thicker sections and/or high chemical retentions, it is advisable to treat the timber by employing the vacuum-pressure impregnation process in order to attain the specified or recommended DSR.

The optimum storage period for diffusion treated materials were standardized as 2 weeks for 25 mm thick section and 3 weeks for 50 mm thick cross-section. The optimum dipping duration is standardized as 30 minutes for 25 mm thick material and 80 minutes for the 50 mm thick sizes.

### **4.3 VACUUM-PRESSURE IMPREGNATION (VPI) TREATMENT — PILOT PLANT INVESTIGATIONS**

The objective of VPI treatment was to offer effective protection to thicker sections where boron diffusion treatment is not adequate and to achieve higher loading of chemicals required for specific end-uses. This includes a DSR range of around 3 to 6.5 kg/m<sup>3</sup> BAE. In the case of CCA treatment the objective was to achieve a DSR in the range of 5-6.5 kg/m<sup>3</sup>.

### 4.3.1 Boron impregnation treatment

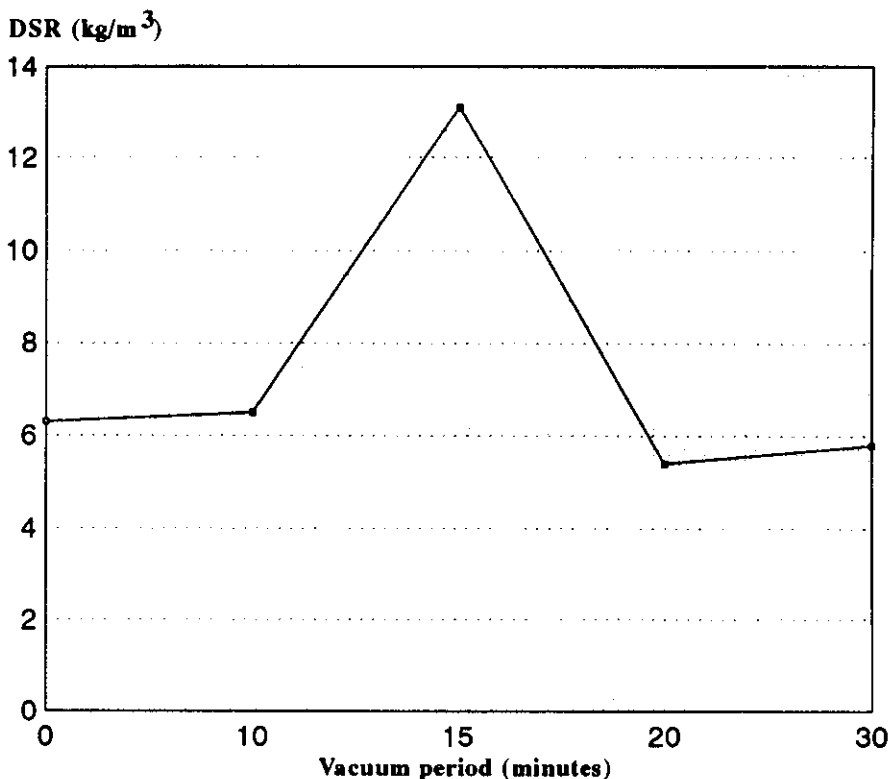
#### 4.3.1.1 Effect of varying the initial vacuum period

In the full-cell process, initial vacuum is applied to ensure that the air-pockets in the wood are removed. However, there are no reports in the literature on the effect of varying the duration of initial vacuum period on DSR. While varying the vacuum period from 0 minutes to 30 minutes at an interval of 15 minutes, an interesting phenomenon was noticed (Table 8). The DSR was maximum when the initial vacuum was retained for 15 minutes. One might expect that the chemical pickup will increase with increasing duration of vacuum period. However, maintaining the vacuum for 30 minutes resulted in low DSR (Figure 8). The DSR obtained with 15 minutes vacuum was more than double the value obtained with no vacuum (0) or vacuum for 30 minutes. This is rather difficult to explain.

**Table 8. Effect of varying the initial vacuum period on the dry salt retention (DSR) of air-dried rubber wood by pressure treatment**

Treatment Schedule vac/pr/vac (minutes)	(DSR) (kg/m <sup>3</sup> )		CV(%)
	Mean	Range	
<b>Trial I</b>			
0/15/5	6.3	5.2-7.1	11.8
15/15/5	13.1	12.0-14.3	7.8
30/15/5	5.8	4.5-8.2	24.9
<b>Trial II</b>			
10/15/5	6.5	4.9-7.1	30.2
15/15/5	13.9	9.3-18.8	24.6
20/15/5	5.4	4.0-6.3	17.2

n = 5



**Fig. 8 Effect of varying vacuum period (keeping pressure period constant) on dry salt retention (DSR) in boron impregnation treatment of air-dried wood**

It was decided to confirm this phenomenon. This time, trials were taken with initial vacuum period of 10, 15 and 20 minutes to see whether there was any inversion effect on DSR. Here also it was noticed that DSR was maximum with the vacuum period of 15 minutes. Decreasing or increasing the duration of vacuum even by 5 minutes resulted in low DSR. While these trials confirmed the trend observed in the first set of trials, it failed to explain the peculiar phenomenon observed. It was decided to verify this while studying the effect of varying the pressure treatment duration.

#### **4.3.1.2 Effect of varying the pressure period**

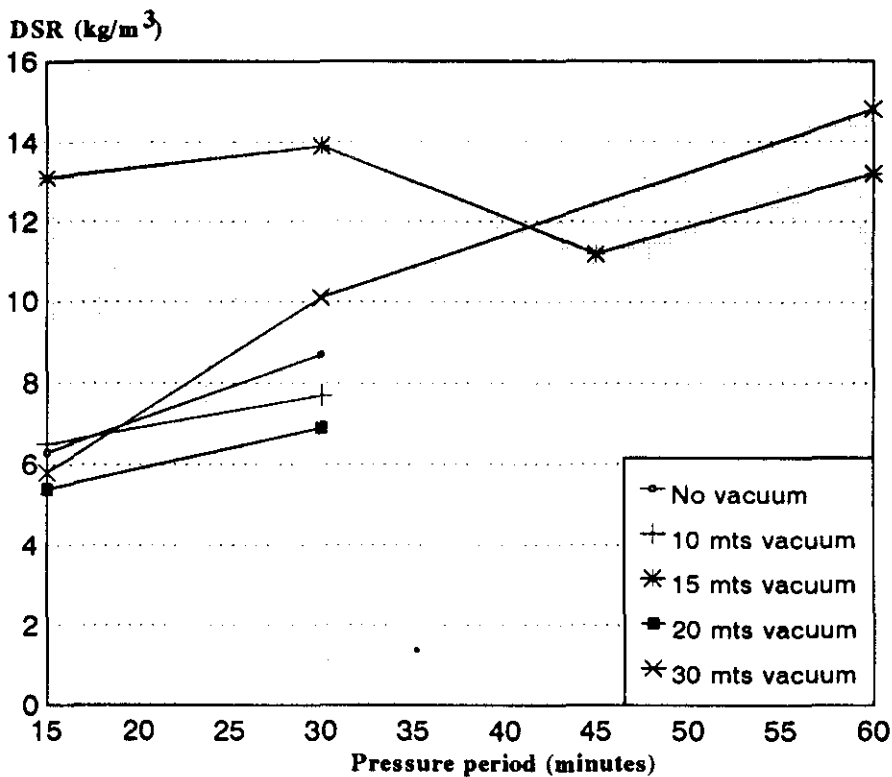
The following treatment schedules, both to study the effect of varying pressure treatment period and also to see the peculiarity of 15 minutes initial vacuum

period, were tried: initial vacuum for 0 minutes and pressure for 30 minutes (referred as 0/30); 10/30; 15/30; 15/45; 15/60; 20/30; 30/30 and 30/60. Results obtained with these schedules and results obtained in the previous trials are combined and presented in Table 9 for easy comparison. It can be seen from Table 9 and Figure 9 that increasing the pressure treatment time (for the initial vacuum period of 0, 10, 20 and 30) resulted in increased DSR. This is to be normally expected. However, in the case of initial vacuum period of 15 minutes, there was no advantage in increasing the pressure treatment period. This clearly shows that the optimum initial vacuum period is 15 minutes. Further studies are needed to understand why the chemical pickup reaches maximum with initial vacuum period of 15 minutes and then drops.

**Table 9. Effect of varying initial vacuum and pressure period the dry salt retention (DSR) of air-dried rubber wood under pressure treatment**

Treatment Schedule vac/pr/vac (minutes)	DSR (kg/m <sup>3</sup> )		CV(%)
	Mean	Range	
0/15/5*	6.3	5.2-7.1	11.8
0/30/5	8.7	7.1-10.2	3.4
10/15/5*	6.5	4.9-7.1	30.2
10/30/5	7.7	5.9-10.0	19.4
15/15/5*	13.1	12.0-14.3	7.8
15/15/5*	13.9	9.3-18.8	24.6
15/30/5	13.9	12.6-15.4	7.4
15/45/5	11.2	9.3-15.7	23.0
15/60/5	13.2	10.9-15.9	16.5
20/15/5*	5.4	4.0-6.3	17.2
20/30/5	6.9	6.3-7.5	7.4
30/15/5*	5.8	4.5-8.2	24.9
30/30/5	10.1	8.9-11.1	9.4
30/60/5	14.8	13.6-16.5	9.3

\*Taken from Table 8.  
n = 5



**Fig. 9 Effect of varying initial vacuum and pressure period on dry salt retention (DSR) in boron impregnation treatment**

Indian Standard (BIS 1982) recommends a boric acid retention of  $6.5 \text{ kg/m}^3$  for building timbers for internal use. Out of the 13 different schedules tried, 10 schedules met this requirement. Out of these 10 schedules, the most economical schedule is one with 15 minutes initial vacuum, 15 minutes pressure and 5 minutes final vacuum period (15'/15'5').

The DSR obtained in air-dried rubber wood using the economical schedule arrived in this study is more than double the amount suggested by the Indian Standards. This clearly shows that rubber wood can be treated even at higher moisture levels.

#### 4.3.1.3 Effect of varying moisture levels and concentration of treatment solution

The economical schedule was chosen and 3% BAE solution was used for treating partially dried (50% MC) and green (75% MC) rubber wood. The results are given in Table 10. Whereas partially dried rubber wood barely met the DSR value recommended in the Indian Standard, it was very low ( $3.8 \text{ kg/m}^3$ ) in the case of green rubber wood. Tan *et al.* (1983), using a 3% BAE solution, obtained a DSR of  $7.5 \text{ kg/m}^3$  in 57 mm thick green rubber wood. However, this was possible with a pressure treatment time of 75 minutes at 1050 kPa pressure. As boron chemicals are diffusible, it was decided to increase the concentration of the treatment solution rather than increasing the pressure treatment time. One more trial of green wood was taken with 6% BAE solution and the average DSR obtained was  $13.5 \text{ kg/m}^3$  (Table 10). This shows that rubber wood in green condition can be treated quite easily. By choosing the right concentration level of the treatment solution, the required chemical retention values can be met even when the moisture level is high.

**Table 10. Effect of moisture levels on the dry salt retention (DSR) of rubber wood under boron impregnation treatment**

Moisture level and concentration solution	DSR ( $\text{kg/m}^3$ )		CV (%)
	Mean	Range	
<b>3% BAE solution</b>			
Air-dried (12-18% MC)	13.9	9.3-18.8	24.6
Partially dried (50% MC)	6.5	5.2-7.1	11.5
Green (75% MC)	3.8	3.3-4.6	12.9
<b>6% BAE solution</b>			
Green (75% MC)	13.5	12.3-14.5	6.6

n = 5



#### 4.3.1.4 Effect of length of samples

Table 11 shows a decrease in DSR while doubling the sample length, from 1 m to 2 m while treating under the economical schedule. But this decrease does not affect adversely the achievement of desired DSR. The achievement of a DSR of 12.5 kg/m<sup>3</sup> for 2 m long samples shows that sample length does not cause problems in the achievement of desired DSR.

**Table 11. Effect of sample length on dry salt retention (DSR) of green rubber wood under boron impregnation treatment with 6% BAE solution**

Size of wood (mm x mm x mm)	DSR (kg/m <sup>3</sup> )		CV (%)
	Mean	Range	
63 x 63 x 1000*	13.5	12.3-14.5	6.6
63 x 63 x 2000	12.3	9.1-14.6	17.3

\*Extracted from Table 10.  
n = 5

#### 4.3.1.5 Effect of end-sealing

The average moisture content at the time of treatment was 40% for samples of cross-section 25 x 25 mm and 13% for samples of cross-section 50 x 50 mm and the density was 550 kg/m<sup>3</sup>.

The extent of penetration of preservatives through the lateral side of the timber were tested on end-sealed samples. Table 12 shows the effect of end-sealing as well as length on the DSR in sizes of cross-section 25 x 25 mm and 50 x 50 mm. The results are subjected to test of significance by the 3-way-ANOVA (Table 13). There is no significant difference between the DSR values achieved in samples with and without end-sealing. The F-value for the interaction of thickness and length on DSR is found highly significant. This explains the situation observed

in here as well as in many earlier occasions that as the thickness and length of sample increases DSR decreases correspondingly. Thus thickness has a major role in influencing the DSR and as the thickness increases the length of samples will also affect DSR.

**Table 12. Effect of end-sealing and sample length on dry salt retention (DSR)**

Sample size (mm x mm x mm)	Without end-sealing			With end-sealing		
	DSR (kg/m <sup>3</sup> )		CV (%)	DSR (kg/m <sup>3</sup> )		CV (%)
	Mean	Range		Mean	Range	
25 x 25 x 1000 <sup>*</sup>	11.19	10.0-12.0	6.0	11.16	10.3-11.9	4.6
25 x 25 x 2000 <sup>*</sup>	11.49	11.2-11.9	2.1	11.37	10.7-11.9	5.2
50 x 50 x 1000 <sup>**</sup>	13.0	12.0-14.1	5.5	12.73	11.2-14.6	9.5
50 x 50 x 2000 <sup>**</sup>	9.58	8.5-11.1	9.7	9.11	7.3-10.0	9.3

<sup>\*</sup> Average MC 40%  
<sup>\*\*</sup> Average MC 13%  
n = 8

**Table 13. Test of significance (3 way ANOVA) on the effect of end-sealing and length**

Source of variation	DF	MSS	F-value
With and without end-sealing (S)	1	876.160	1.352 <sup>ns</sup>
Thickness (T)	1	686.440	1.059 <sup>ns</sup>
Length (L)	1	47295.376	72.963 <sup>**</sup>
S x T	1	377.331	0.582 <sup>ns</sup>
S x L	1	92.160	0.142 <sup>ns</sup>
T x L	1	63504.000	97.968 <sup>**</sup>
S x T x L	1	13.876	0.021 <sup>ns</sup>
Error		56	648.212

<sup>\*\*</sup> = significant at 1% P level  
ns = not significant

This study shows that penetration of chemicals through the lateral surfaces is very active in rubber wood. Due to this reason, for the boron impregnation treatment of rubber wood, as far as common end-uses are concerned, sample length up to 2 m does not cause any significant problem in achieving the desired DSR. The results thus indicate that the thickness and length of wood influences DSR inversely.

#### 4.3.2 Chemical analysis of boron impregnated rubber wood

Table 14 shows the distribution of chemicals in 'surface' 'middle' and 'core' portions as well as the net average %BAE retention as 'overall' value, as determined by chemical analysis. All the values are very much higher than the requirement.

**Table 14. Chemical analysis of boron impregnated rubber wood - dry salt retention (DSR) values in terms of %BAE (CV (%) values in brackets)**

	63 x 63 x 1000 mm			63 x 63 x 2000 mm
	Air-dried wood treated with 3% BAE solution	Partially dried (MC 50%) wood treated with 3% BAE solution	Green wood (MC 75%) treated with 6% BAE solution	Green wood (MC 75%) treated with 6% BAE solution
'Overall'	2.31 (20.3)	0.70 (26.2)	1.70 (11.1)	1.57 (13.7)
'Surface'	2.43 (20.7)	1.05 (17.4)	2.55 (9.2)	1.99 (24.8)
'Middle'	2.05 (30.3)	0.78 (11.6)	0.97 (16.5)	1.42 (17.9)
'Core'	1.16 (14.0)	0.47 (9.4)	0.97 (12.6)	0.96 (9.5)

n = 5

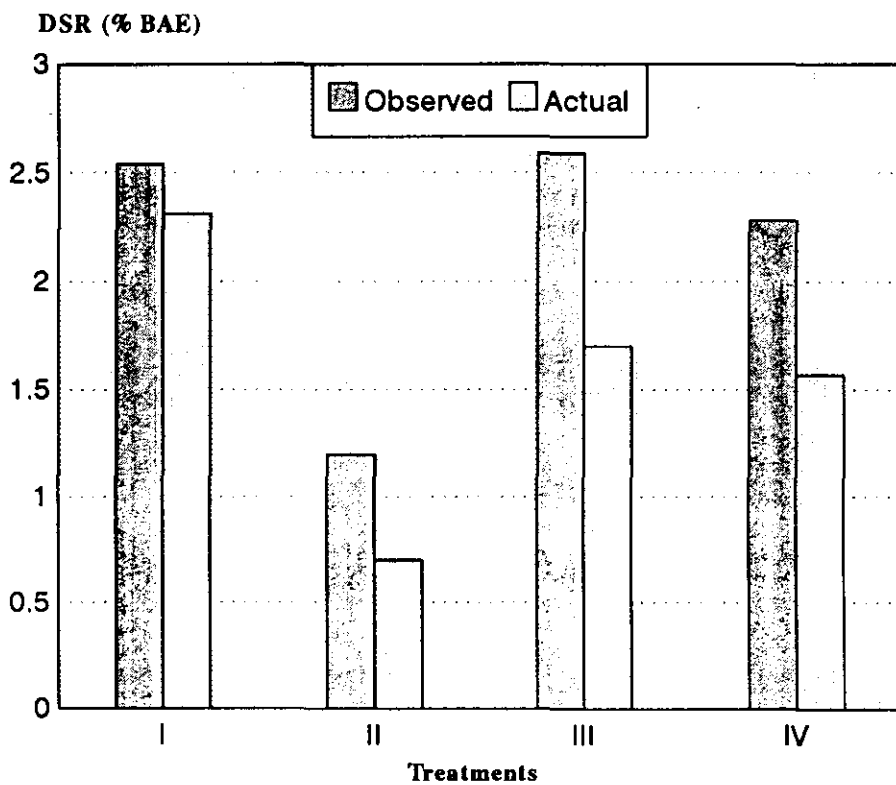
Table 15 and Figure 10 shows the difference between the 'calculated' and 'actual' dry salt retention in terms of %BAE. The measured DSR values in  $\text{kg}/\text{m}^3$  and the corresponding retention in terms of %BAE are also given in this Table. The calculated values of total net average DSR in terms of %BAE retention and the mean of actual values obtained by chemical analysis differ by about 30% within a range of 9 to 42 percent.

**Table 15. Comparison between the calculated values of %BAE and the actual values obtained by chemical analysis in pressure treated material**

Condition of wood and concentration of treatment solution	Measured DSR ( $\text{kg}/\text{m}^3$ )	Calculated mean value of % BAE (A)	Actual value of %BAE (overall sample) by chemical analysis (B)	Difference % [(A-B) x 100]/A
<b>63 x 63 x 1000 mm</b>				
Air-dried wood treated with 3% BAE solution	14.0	2.54	2.31	9.1
Partially-dried wood (MC 50%) treated with 3% BAE solution	6.5	1.20	0.70	41.7
Green wood (MC 75%) treated with 6% BAE solution	13.5	2.59	1.70	34.4
<b>63 x 63 x 2000 mm</b>				
Green wood (MC 75%) treated with 6% BAE solution	12.3	2.28	1.57	31.1

n = 5

Comparison of chemical analysis of boron impregnated wood (Table 14 & 15) with that of diffusion treated wood (Table 2 & 3) proved the earlier conclusion that for the boron treatment of cross- sections greater than 50 mm thickness vacuum-pressure impregnation method needs to be employed for achieving the



**Fig. 10 Results of chemical analysis of boron impregnated rubber wood**

desired 'core' and 'overall' retentions. Table 14 clearly shows that even for partially dried wood (MC 50%), treatment with 3% BAE solution achieves an actual core retention of 0.47% and 'overall' retention of 0.70% BAE. Also, results are similar in respect of the impregnation treatment of green wood with 6% BAE solution. All the DSR values in terms of %BAE obtained from the chemical analysis of boron impregnated wood are much higher than the requirements. Comparing with the data on chemical analysis of diffusion treated wood (Table 3), it can be seen from Table 14 that the DSR achieved for sizes greater than 50 mm thickness in the boron impregnation treatment with 3% BAE solution also satisfies the New Zealand and BWPA specifications. As mentioned in section 4.2.2, the measured DSR required to satisfy the New Zealand and BWPA specifications, viz.,  $3 \text{ kg/m}^3$ , can be achieved by adopting the economical schedule in the VPI method in sizes greater than 50 mm thickness. Hence it can be concluded that for samples of thicker cross-sections (greater than 50 mm thickness) where diffusion treatment is not able to achieve the desired DSR

required for ensuring effective protection, impregnation treatment is the best alternative. Also in cases where more quantity of timber needs to be treated within a short time, whether in smaller or bigger cross-sections, impregnation method can be employed.

### 4.3.3 Reconfirmation trial in pilot plant

#### 4.3.3.1 Air-dried wood

Air-dried rubber wood samples of different sizes obtained as balance material while sawing for various purposes were utilized for a further reconfirmation of the economical schedule, 15'/15'/5'. The treatment was carried out in the pilot plant used for the earlier experiments.

The average moisture content of the material was about 17% and density 500 kg/m<sup>3</sup>. Results of the trial are given in Table 16. A DSR of 11.2 kg/m<sup>3</sup> was achieved for material of length 2 m and thickness 13 mm by using a 3% BAE solution. While the length was reduced to half, the DSR obtained was 14.2 kg/m<sup>3</sup> for the 50 mm thick sections. The DSR value was slightly low while the thickness increased to 63 mm. The greater length of the samples may be the reason for a low DSR in smaller cross-sections.

**Table 16. Boron impregnation treatment of air-dried rubber wood with 3% BAE solution. Reconfirmation trial in pilot plant**

Size of wood (mm x mm x mm)	DSR (kg/m <sup>3</sup> )		CV (%)	n
	Mean	Range		
23 x 13 x 2000	11.2	9.6-15.4	13.0	15
50 x 50 x 1000	14.2	12.6-15.6	8.9	5
63 x 63 x 1000	12.7	9.3-14.7	16.3	5

All the values are much higher than the IS recommendation of 6.5 kg/m<sup>3</sup> boron chemicals in the treated wood for indoor purposes in humid conditions (BIS 1982). As all the results from this trial satisfy this requirement, it is proved

beyond doubt that the schedule, 15'/15'/5', can be utilized economically for the pressure impregnation treatment of air-dried rubber wood with boron compounds.

#### 4.3.3.2 Partially dried wood

In connection with a training programme meant for popularizing the utilization of treated rubber wood among rural carpenters, rubber wood of various sizes was impregnated with 3% BAE solution. The sizes were treated by employing the schedule 15'/15'/5' in the pilot plant. The sizes were partially dried and had an average moisture content of 50% and possessed an average density of 556 kg/m<sup>3</sup>. Sizes of different thicknesses, varying from 25 to 63 mm (sample size of 10 to 40 pieces) were treated.

Table 17 gives details on the various sizes that were treated, the DSR achieved, and number of samples in each thickness. The average DSR for various sizes ranged from 5.1 to 9.7 kg/m<sup>3</sup>. For rubber wood with density of 500 to 600 kg/m<sup>3</sup>, the BWPA specification of 0.4% BAE in the treated wood corresponds to 3.0 kg/m<sup>3</sup> BAE in terms of gross absorption (section 4.2.2 and 4.3.2). The values obtained in this study are much higher, and especially for indoor furniture a DSR of 3 kg/m<sup>3</sup> BAE will be sufficient.

**Table 17. Boron impregnation treatment of partially dried rubber wood (50% MC) of assorted sizes with 3% BAE solution employing a treatment schedule of 15'/15'/5'**

Size (mm x mm x mm)	DSR (kg/m <sup>3</sup> )		CV (%)	No. of bsamples (n)
	Mean	Range		
1050 x 25 x 25	9.7	7.9-11.4	15.7	40
600 x 50 x 30	9.1	7.1-10.9	11.7	20
450 x 63 x 38	7.0	6.6-7.8	5.5	10
458 x 50 x 50	6.9	5.2-8.8	16.9	10
825 x 56 x 56	5.8	5.2-6.3	5.7	10
450 x 100 x 63	5.1	4.3-5.5	7.2	10

The penetration of chemicals in the treated wood was tested as per IS: 2753-1964 (Part I) (BIS 1964) and found to have uniform through and through distribution.

The DSR decreased with increase in thickness. This is to be expected as all the sizes were treated together using the same schedule. It would be ideal to sort out sizes by thickness and carry out treatment separately to have control on chemical pick-up. Otherwise, as was observed here, lower thickness material will tend to absorb more chemical at the cost of thicker material. This can be seen in the higher retention of chemicals ( $6.5 \text{ kg/m}^3$  BAE) when the samples of  $63 \times 63$  mm cross-section were treated in the previous trial (Table 10), compared to only  $5.8 \text{ kg/m}^3$  BAE obtained even for cross-section of  $56 \times 56$  mm (in this trial), when treated with samples of other thicknesses. If a mixture of different thicknesses cannot be avoided, it should be ensured that the thickest material gets the minimum required DSR.

If we are aiming for a DSR of  $3.0 \text{ kg/m}^3$  BAE for indoor furniture, this study indicates that there is scope to reduce the concentration of the chemical solution. This trial has confirmed the validity of the treatment schedule for partially dried rubber wood, even for treating sizes of different thicknesses together.

#### **4.4 FULL SCALE TESTING OF THE ECONOMICAL SCHEDULE IN COMMERCIAL SCALE PLANTS**

##### **4.4.1 Air-dried wood**

A full scale testing of the economical schedule for the boron impregnation treatment was conducted for air-dried wood with an average moisture content of 18% and density  $550 \text{ kg/m}^3$  in a commercial scale treatment cylinder of length 3 m and diameter 1.25 m, available with M/s. Wood Tech Industries, Irinjalakuda, Kerala. There was a slight modification with regard to the extent of vacuum employed. The treatment plant was set for the application of a vacuum of 115 kPa and for this particular study the same extent of vacuum was



employed. A treatment solution of 4% BAE concentration was used. The trial was carried out with full load of wood in the cylinder.

Table 18 shows the mean DSR achieved in the treatment. There is not much variation between the DSR values of the two different sizes treated. A pooled mean DSR of 12.35 kg/m<sup>3</sup> was achieved. This falls in the same range of DSR found for the boron impregnation treatment of air-dried rubber wood in the earlier trials using a pilot plant. The DSR achieved is much higher than the IS recommendation. The initial and final vacuum applied in the earlier trials was only 85 kPa. The findings of the present study indicate that as far as air-dried rubber wood is concerned, small increase in the amount of vacuum does not significantly increase the DSR. Further, this trial established the utility of the economical schedule for commercial scale treatment of air-dried rubber wood.

**Table 18. Boron impregnation treatment of air-dried rubber wood in a commercial scale treatment plant using the economical schedule**

Size of wood (mm x mm x mm)	DSR (kg/m <sup>3</sup> )		CV (%)
	Mean	Range	
80 x 56 x 2500	12.2	11.1-12.7	4.3
25 x 38 x 2000	12.5	9.7-17.9	22.6

n = 7

#### 4.4.2 Partially dried wood - Effect of length of sizes

The boron impregnation treatment facilities available with M/s. P. Subbaramayyar & Brother, a rubber wood processing industry in Chalakudy, Kerala, were utilized for the commercial scale testing of the economical schedule on partially dried rubber wood. The treatment cylinder had a diameter of 0.9 m and length 6 m. In order to observe the effect of length of samples, it was decided to go for samples of length 0.45, 0.90 and 1.8 m with cross-section 63 x 63 mm.

The concentration of treatment solution used was 1.6% BAE. The factory has been regularly treating the above cross-section with a solution of the above mentioned concentration for the purpose of manufacturing shuttle blocks. The effect of length of samples on DSR was investigated by employing the economical treatment schedule. Due to some technical reason, it was possible to apply a pressure of 500 kPa only. This also helped in another way to see the effect of a reduced pressure on DSR.

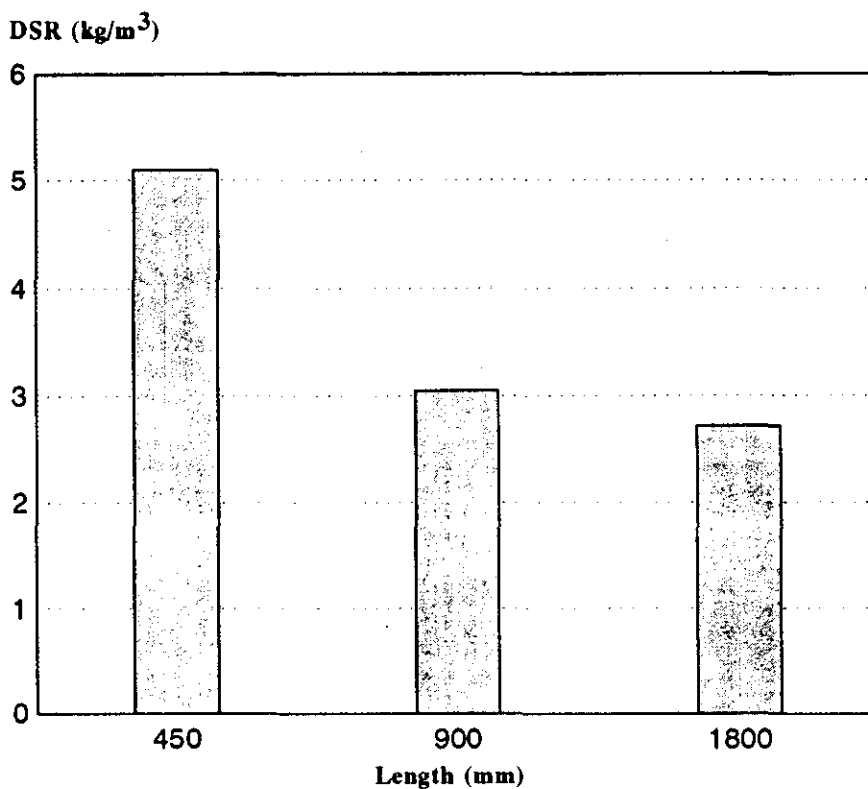
The average moisture content of the samples was around 50% and density about 550 kg/m<sup>3</sup>.

The effect of the variation of sample length on DSR by employing the schedule 15'/15'/5' is detailed in Table 19. The results showed that the DSR decreased as the sample length increased (Figure 11). The DSR varied from 5.1 to 2.7 kg/m<sup>3</sup>, as the sample length varied from 0.45 to 1.8 m. The corresponding DSR in terms of %BAE varies from 0.46 to 0.88. This data showed that for partially dried rubber wood the concentration of solution used, viz., 1.6% BAE, is adequate to achieve only low DSR, as desired in the case of shuttle blocks. For end-uses where more loading of preservatives is desirable, a solution of higher concentration should be employed. Hypothesis tests for means (t-test) shows significantly different values of DSR in all the three length categories studied (Table 20). Length affects the DSR in thicker sections as already shown (Table 13). But even then, in all the length classes studied, sufficient DSR was achieved as per the BWPA specifications. This shows that as far as the end-uses are considered, even though the length of samples is significant in determining the DSR, up to 1.8 m length does not pose problems in achieving the desired DSR, provided solutions of appropriate concentrations are used. The same is the case for the results shown in Table 11 also. For the end-uses such as for shuttle blocks of size 63 x 63 x 450 mm, where only low boron retention is sufficient, the use of 1.6% BAE solution is suggested in the schedule 15'/15'/5' for partially dried rubber wood.

**Table 19. Effect of length of samples on dry salt retention (DSR) in partially dried wood while employing the treatment schedule 15'/15'/5', using 1.6% BAE solution**

Size of wood (mm x mm x m)	DSR (Kg/m <sup>3</sup> )		CV (%)	Av. BAE%	Range
	Mean	Range			
63 x 63 x 450	5.10	3.6-6.5	14.9	0.88	0.58-1.24
63 x 63 x 900	3.05	3.1-2.5	10.9	0.54	0.41-0.67
63 x 63 x 1800	2.72	1.7-3.5	15.4	0.46	0.29-0.65

n = 15



**Fig. 11 Effect of length of samples on dry salt retention (DSR) in partially dried wood**

**Table 20. Test of significance of means for the effect of length of samples on dry salt retention (DSR) of partially dried wood while employing the economical schedule**

Source of variation	DF	t-value
<b>Between lengths</b>		
450 mm vs 900 mm	28	10.6307**
450 mm vs 1800 mm	28	9.5575**
900 mm vs 1800 mm	28	-2.4108**

\*\* Significant at 1% P level.

#### 4.4.3 Green wood

M/s. Sakthi Wood Treats, Trivandrum, Kerala, extended facility for impregnating green wood (MC around 70%) with 6% BAE solution by employing the economical schedule, 15'/15'/5'; with the exception that the maximum pressure exercised was only 800 kPa, instead of 1000 kPa. The treatment cylinder had a length of 6 m and a diameter of 0.9 m. The cylinder was fully loaded with rubber wood of length 2.25 m and of two different cross-sections, 75 x 75 and 88 x 88 mm.

The results of the trial run are given in Table 21. A mean DSR of 12.9 and 11.8 kg BAE/m<sup>3</sup> was achieved for samples of cross-sections 75 x 75 mm and 88 x 88 mm respectively. As in earlier studies, here also it was observed that as the thickness increased DSR decreased. The DSR achieved for both sizes is much higher than the requirement of various specifications. Hence this study confirms that the reported schedule, 15'/15'/5', could be adopted for commercial scale boron impregnation treatment of rubber wood in green condition.

**Table 21. Commercial scale testing of boron impregnation treatment of green rubber wood (MC 70%) with 6% BAE solution employing a treatment schedule of 15'/15'/5' - dry salt retention (DSR) data**

Size of wood (mm x mm x m)	DSR (kg/m <sup>3</sup> )		CV (%)
	Mean	Range	
75 x 75 x 2.25	12.9	9.5-15.9	16.5
88 x 88 x 2.25	11.8	10.1-13.0	8.0

n = 10

Tan *et al.* (1983) treated 57 mm thick green rubber wood with a 3% BAE solution by employing an initial vacuum of about 90 kPa for 45 minutes followed by a pressure of 1050 kPa for 75 minutes and a final vacuum for 15 minutes. They achieved a DSR of 7.5 kg BAE/m<sup>3</sup>. However, this study, using 6% BAE solution and employing the economical schedule of 15'/15'/5', achieved a pooled mean DSR of 12.4 kg/m<sup>3</sup> in green rubber wood. This trial carried out in a commercial plant with full load of wood confirms the validity of the economical schedule developed in a pilot plant. As the pressure used in this study was only 800 kPa compared to 1000 kPa in the pilot plant study it can be seen that lowering the pressure slightly did not affect the achievement of the required DSR. The present study also suggests that the concentration of the treatment solution can be reduced according to the DSR requirements.

M/s. Plantation Corporation of Kerala Ltd (PCKL) extended their facilities for the commercial scale boron impregnation treatment of green rubber wood. The company was treating rubber wood with a solution of concentration 6.67% BAE employing a vacuum of 85 kPa and a pressure of 1000 kPa. The economical schedule was tested in this plant. Table 22 shows the results of a trial on three different sizes of rubber wood in green condition. The mean DSR varies from 13.7 to 12.2 kg/m<sup>3</sup> for sizes of thickness 20 to 40 mm. This shows that high levels of retention can be achieved. As the DSR achieved is much higher than the requirement, it is clear that the concentration of treatment solution can be lowered.

**Table 22. Commercial scale boron impregnation treatment of rubber wood in green condition with 6.67% BAE solution by employing a vacuum of 85 kPa and a pressure of 1000 kPa employing the economical schedule**

Size of wood (mm x mm x mm)	DSR (kg/m <sup>3</sup> )		CV (%)	n
	Mean	Range		
250 x 20 x 2150	13.7	13.0-14.2	4.8	3
115 x 40 x 1700	12.2	11.0-12.9	9.0	3
115 x 40 x 2150	12.2	10.9-14.7	11.3	6

In order to observe the effect of a lower concentration, another trial was conducted with a solution of 5% BAE concentration on assorted sizes in almost green condition. As the company was interested in carrying out the treatment with a reduced pressure for technical reasons, this trial was taken with pressure at 800 kPa. Table 23 shows the results of the second trial. The DSR achieved varies from 11.3 to 5.6 kg/m<sup>3</sup> for sizes of thickness 15 to 75 mm. A pooled mean DSR of 8.9 kg/m<sup>3</sup> achieved shows that the concentration of treatment solution can be lowered for the economy of the treatment system. Also it showed that lowering the pressure slightly not adversely affect the achievement of desired DSR. The relationship between moisture content and DSR is evident from this study. For materials of almost similar thickness, higher DSR is achieved in wood with lower moisture content. Partially dried (MC 45-50%) wood picked up more chemical than green wood.

**Table 23. Commercial scale boron impregnation treatment of rubber wood in green condition with 5% BAE solution by employing a vacuum of 85 kPa and a pressure of 800 kPa in the economical schedule**

Size of samples (mm x mm x mm)	DSR (kg/m <sup>3</sup> )	Av. BAE%	MC%	Density (kg/m <sup>3</sup> )
140 x 15 x 775	11.3	2.24	51.3	501.7
50 x 25 x 875	9.1	1.69	83.9	542.3
150 x 40 x 1125	11.1	2.22	50.3	498.5
150 x 40 x 1350	9.2	1.93	74.7	477.2
50 x 50 x 875	9.1	1.89	46.6	484.3
140 x 50 x 1275	6.8	1.35	74.1	508.3
140 x 75 x 1300	5.6	1.04	71.2	537.9

It was possible to conduct another commercial scale treatment trial of rubber wood in green condition by utilizing the boron impregnation facilities available with M/s. Emson Treat Wood, Aluva, Kerala. This Company was making use of a vacuum of 100 kPa followed by a pressure of 800 kPa. The Company was following their traditional schedule of 30 minutes initial vacuum followed by a pressure period of 60 minutes and a final vacuum of 30 minutes (30'/60'/30').

They were treating wood with 2.4% BAE solution. Table 24 shows the results of the treatment trial. In general, it can be seen that the solution strength is not sufficient to achieve the desired DSR. Application of the newly developed schedule also did not achieve the desired DSR. So it was clear that the solution concentration should be above 2.4% BAE for treating rubber wood in green condition. Comparison of the present results (Table 24) with the results of the earlier trial in the PCKL Plant (Tables 22 and 23) showed that the newly developed economical schedule is more productive, much time-and energy-saving than the traditional schedule being followed, provided treatment solution of appropriate concentration is employed.

**Table 24. Commercial scale boron impregnation treatment of rubber wood in green condition with 2.4% BAE solution by employing a vacuum of 100 kPa and a pressure of 800 kPa**

Size of sample (mm x mm x mm)	Schedule	DSR (kg/m <sup>3</sup> )		CV (%)	n
		Mean	Range		
80 x 30 x 700	30'/60'/30'	6.6	5.2-7.4	13.1	5
80 x 40 x 1500	30'/60'/30'	5.7	3.9-6.9	17.7	10
110 x 45 x 1300	15'/15'/30'	4.6	3.5-6.3	20.5	10

Average moisture content = 70%  
 Mean density = 560 kg/m<sup>3</sup>

#### 4.4.4 Effect of a more intensive initial vacuum on DSR

The full scale testing reported under section 4.4.1 was conducted employing the economical schedule, but by exerting a more intensive vacuum, 115 kPa instead of 85 kPa. The extent of pressure was the same as what was regularly used, 1000 kPa. The results of the treatment detailed in Table 18 show that a pooled mean DSR of 12.35 kg/m<sup>3</sup> can be achieved. As this value is in comparable range with the results obtained from the earlier trials (see Tables 10 & 16), the present trial indicated that small increase in the extent of vacuum does not significantly affect the achievement of desired DSR.

#### 4.4.5 Effect of sample size on DSR

The commercial scale boron impregnation facilities available with M/s. Supreme Tech Wood Exporters, Ollur, Kerala were utilized for the present study. The economical schedule was employed for the treatment, but the extent of vacuum employed was 100 kPa instead of 85 kPa and the pressure exerted was 1200 kPa, instead of 1000 kPa. A 4% BAE solution was employed for the treatment. The effect of thickness on DSR was studied on samples of length 900 mm and with cross-sections 25 x 25, 50 x 50, 75 x 75 and 100 x 100 mm.

The samples were treated in green condition (MC 70%). The average density of samples was around 525 kg/m<sup>3</sup>.

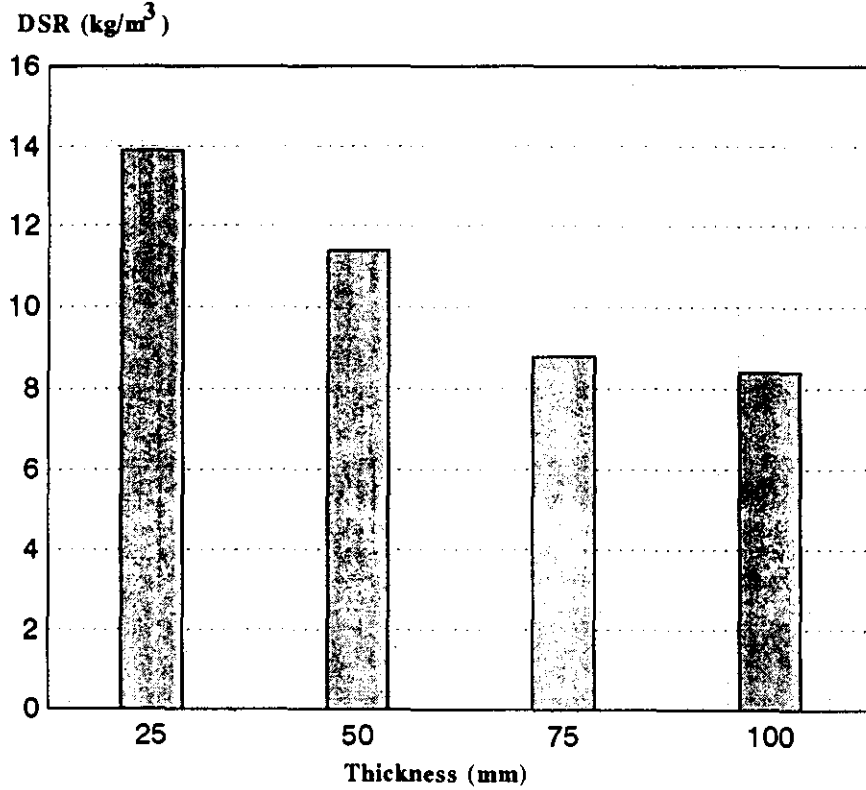
The effect of size on DSR as revealed from the experiment is detailed in Table 25. It can be seen from Figure 12 that the DSR varies from 8.4 to 14.0 kg/m<sup>3</sup> in the case of sizes of cross- sections 100 x 100 mm to 25 x 25 mm. It was found that the smaller sizes achieved higher and the bigger sizes lower DSR. So, as far as possible, treatment of similar sizes in one charge is always desirable. In treatments with different sizes in the same charge, it should be ensured that the thickest sizes acquired the desired or specified DSR.

**Table 25. Effect of size on DSR of green rubber wood while employing economical schedule for the commercial scale boron impregnation treatment**

Size of wood (mm x mm x mm)	DSR (kg/m <sup>3</sup> )		CV (%)
	Mean	Range	
25 x 25 x 900	13.9	11.9-15.6	7.8
50 x 50 x 900	11.4	9.4-13.0	9.1
75 x 75 x 900	8.8	7.2-9.4	8.2
100 x 100 x 900	8.4	8.0-8.8	3.7

n = 10





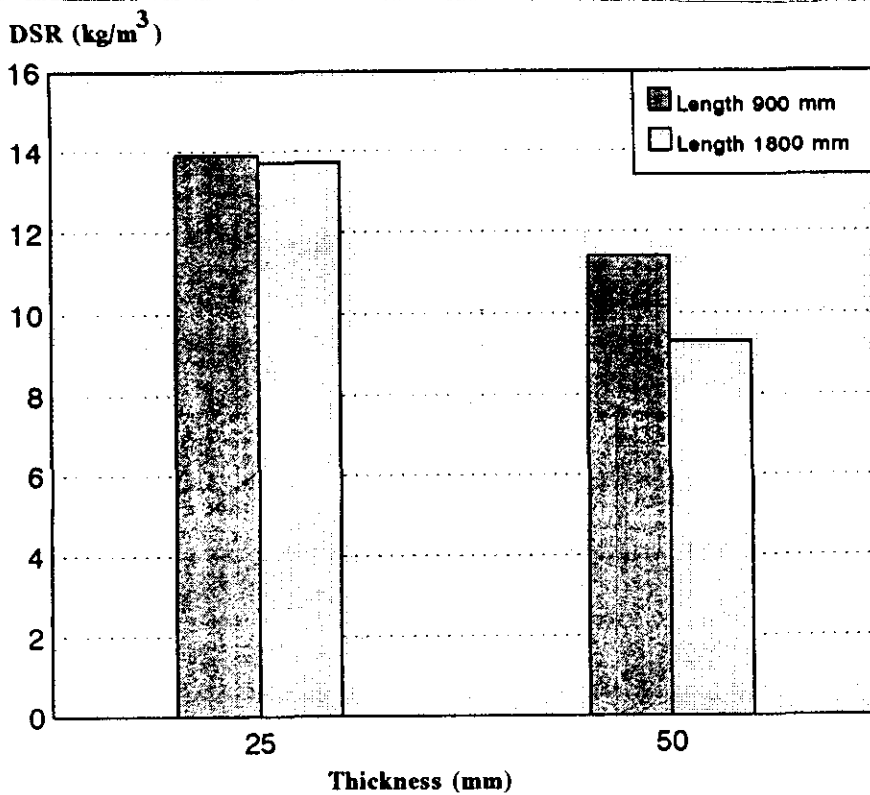
**Fig. 12 Effect of size of samples on dry salt retention (DSR) in green wood**

Table 26 and Figure 13 shows the effect of sample length on DSR. Test of significance of means (t-test) indicated that doubling the sample length from 900 mm, in cross-sections 25 x 25 mm does not affect the DSR significantly. The effect is significant for cross-section 50 x 50 mm (Table 27). In earlier trials also a similar trend was noticed.

**Table 26. Effect of length of samples on DSR while boron impregnation treatment of green wood**

Size of wood (mm x mm x mm)	DSR (kg/m <sup>3</sup> )		CV (%)
	Mean	Range	
25 x 25 x 900*	13.9	11.9-15.6	7.8
25 x 25 x 1800	13.7	10.5-15.1	9.2
50 x 50 x 900*	11.4	9.4-13.0	9.1
50 x 50 x 1800	9.3	8.2-10.3	7.1

\* n = 10  
 \* Extracted from Table 25.



**Fig. 13 Effect of length of samples on dry salt retention (DSR) in green wood**

**Table 27. Test of significance for means (t-test) on the effect of sample length on DSR**

Source of variation	DF	t-value
25 x 25 x 900 mm vs 25 x 25 x 1800 mm	18	0.2848 <sup>ns</sup>
50 x 50 x 900 mm vs 50 x 50 x 1800 mm	18	5.3208 <sup>**</sup>

ns = not significant  
 \*\* = significant at 1% P level

As all the DSR values in Table 25 and 26 are much higher than that in the IS recommendations, the applicability of newly developed schedule, 15'/15'/5', is once more reiterated for commercial scale applications.

As the earlier trials were with 3 and 6% BAE solutions, the present study with 4% BAE solution indicated that the concentration of treatment solution can be brought down to 4% BAE from 6% BAE, for the treatment of green rubber wood. But it should be remembered that the vacuum and pressure applied were slightly higher than in the earlier trials.

Table 28 shows the DSR achieved while treating wide planks of larger lengths (150 x 20 x 3000 mm) in green condition with a 4% BAE solution. The high DSR (14 kg/m<sup>3</sup>) achieved may be due to the low thickness of the planks.

**Table 28. Boron impregnation treatment of green rubber wood planks with 4% BAE solution by employing the economical schedule with 100 kPa vacuum and 1200 kPa pressure**

Size (mm x mm x mm)	DSR (kg/m <sup>3</sup> )		CV (%)	n
	Mean	Range		
150 x 20 x 3000	14.0	10.2-20.7	23.8	10

## 4.5 CCA IMPREGNATION TREATMENT

### 4.5.1 Pilot plant trials

#### 4.5.1.1 Air-dried wood

Air-dried rubber wood (MC around 14%) was subjected to vacuum- pressure treatment with 2% CCA solution in the pilot-plant. Table 29 shows the effect of variation of thickness on DSR while treating with a schedule 15'/30'/5'. The vacuum applied was 85 kPa and pressure 1000 kPa. The DSR varied from 9.9 to 8.0 kg/m<sup>3</sup> for sizes of cross-sections 25 x 25 to 100 x 100 mm (Figure 14). It can be seen that for air-dried wood, sufficient DSR as per the recommendations of Indian Standard (6.5 kg/m<sup>3</sup>) for exposed non- ground contact applications

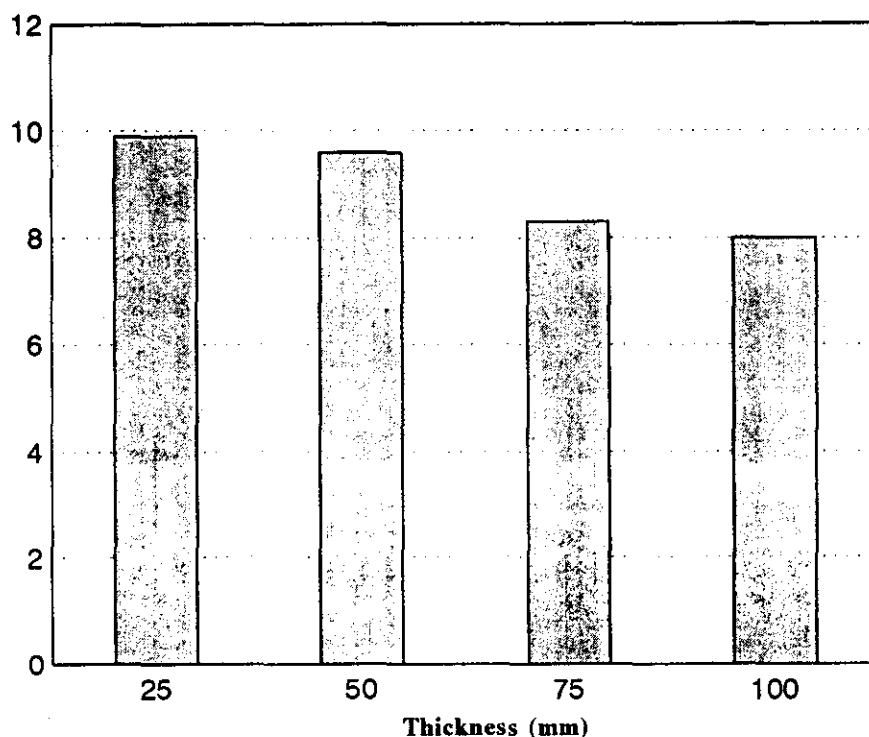
can be achieved by using 2% CCA solution under this particular schedule, even for samples of higher thickness (up to 100 mm). This means that as far as sizes required for common end-uses are concerned, CCA impregnation of rubber wood is not at all difficult and required loading can be achieved.

**Table 29. Effect of thickness variation on the dry sal retention (DSR) of CCA while impregnating air dried rubber wood using 2% solution under the schedule 15'/30'/5'**

Sample size (mm x mm x mm)	DSR (kg/m <sup>3</sup> )		CV (%)
	Mean	Range	
25 x 25 x 1500	9.9	8.5-12.8	17.7
50 x 50 x 1500	9.6	9.3-9.9	2.3
75 x 75 x 1500	8.3	7.5-9.4	8.5
100 x 100 x 1500	8.0	7.8-8.2	1.9

n = 5

DSR (kg/m<sup>3</sup>)



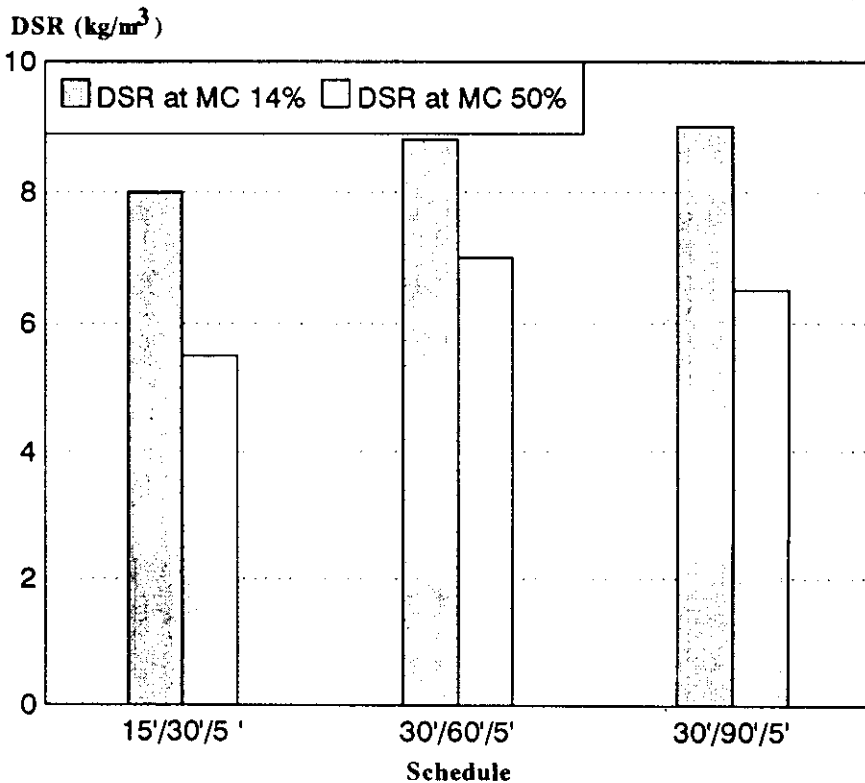
**Fig. 14 Effect of thickness variation on the dry salt retention (DSR) of CCA**

Table 30 and Figure 15 shows the effect of three different schedules on the DSR while using a 2% solution for the treatment of air-dried wood. All the three schedules, viz., 15'/30'/5', 30'/60'/5' and 30'/90'/5' are found capable of achieving the desired DSR. For saving time and energy, the shortest schedule, 15'/30'/5', could be utilized for achieving effective protection.

**Table 30. Effect of different treatment schedules on the dry salt retention (DSR) of CCA while treating air-dried (MC around 14%) and partially dried (MC around 50%) wood with 2% solution on samples of size 100 x 100 x 1500 mm**

Treatment schedule	MC 14%			MC 50%		
	DSR (kg/m <sup>3</sup> )		CB (%)	DSR (kg/m <sup>3</sup> )		CV (%)
	Mean	Range		Mean	Range	
15'/30'/5'	8.0	7.8-8.2	1.9	5.5	5.0-6.2	7.9
30'/60'/5'	8.8	8.6-8.9	1.5	7.0	6.3-8.0	9.0
30'/90'/5'	9.0	8.7-9.7	4.7	6.5	5.8-7.7	11.1

n = 5



**Fig. 15 Effect of treatment schedule on dry salt retention (DSR) of CCA in air-dried and partially dried wood**

#### **4.5.1.2 Partially dried wood**

Table 30 also shows the effect of the above mentioned three different treatment schedules on the DSR of CCA while treating partially dried (MC around 50%) rubber wood of the thickest cross-sections (100 x 100 mm) with a 2% solution. It can be seen that the schedule, 30'/60'/5', satisfies the DSR requirements even for the thickest sizes. This implies that for sizes of lower thickness, even shorter schedule may satisfy the DSR requirements.

#### **4.5.2 Full-scale testing of CCA impregnation of green rubber wood in commercial scale plants**

The commercial scale vacuum pressure impregnation treatment plant available with M/s. Wood Tech Industries, Irinjalakuda, Kerala, was utilized for the CCA impregnation of rubber wood in green condition (MC around 75%) with a 4% solution. A more concentrated solution than the earlier trials was selected due to the higher moisture content of the test material. The cylinder had a length of 3 m and 1.2 m diameter. This plant was set for applying a vacuum of 115 kPa and a pressure of 1000 kPa. The same conditions were followed for the present trial and the economical schedule developed for boron impregnation treatment, 15'/15'/5', was tested for the CCA impregnation. The treatment was done with full load wood in the cylinder.

Table 31 shows the result of the treatment. Achievement of a mean DSR of 10.4 kg/m<sup>3</sup> shows the applicability of the economical schedule arrived at for the boron treatment of green rubber wood for CCA treatment too. The value of DSR achieved is higher than the IS recommendation for building timbers (BIS 1982). The high DSR value achieved indicates that the treatment solution concentration can still be lowered in order to economise the treatment.

**Table 31. CCA impregnation of green rubber wood (MC 75%) with 4% solution while employing the economical schedule with 115 kPa vacuum and 1000 kPa pressure**

Size of wood (mm x mm x mm)	DSR (kg/m <sup>3</sup> )		CV (%)	n
	Mean	Range		
105 x 38 x 2000	10.4	7.2-13.8	15.6	20

It was possible to carry out another commercial trial with a CCA solution of 3% concentration at M/s. Sitaramson Wood Chem Process Pvt. Ltd., Ollur, Kerala. In this particular plant, the economical schedule, 15'/15'/5', was employed as in the above trial with the exception that the vacuum applied was only 85 kPa. Here also the treatment was conducted with a full load of wood in the cylinder.

Table 32 shows the results of the above trial on green rubber wood (average MC around 65%) employing the economical schedule with 85 k a vacuum and 1000 kPa pressure. It can be seen that except in the case of very thick sections (150 x 100 mm), the desired DSR is achieved by treating with 3% CCA solution. For thicker sizes it is desirable to go for solutions of higher concentration in order to achieve the required loading of preservatives.

**Table 32. CCA impregnation treatment of green rubber wood (MC 65%) with 3% solution while employing the economical schedule with 85 kPa vacuum and 1000 kPa pressure**

Size of wood (mm x mm x mm)	DSR (kg/m <sup>3</sup> )		CV (%)
	Mean	Range	
70 x 25 x 2475	8.7	7.5-10.3	8.8
150 x 30 x 2475	6.5	6.1-7.1	5.0
90 x 40 x 1500	6.9	5.7-8.3	11.1
150 x 100 x 2200	4.7	3.6-6.2	14.7

n = 10

In all the cases the penetration of CCA in the treated wood was tested as per the Indian Standard (BIS 1964) and found uniform and through and through penetration and distribution of preservative.

These two commercial scale trials confirmed the applicability of the economical schedule, 15'/15'/5', for the CCA impregnation too.

#### **4.5.3 Chemical analysis of CCA treated wood**

Table 33 shows the result of ICP and AAS analyses for the quantity of individual constituents (copper, chromium and arsenic) of CCA in the treated timber. Results of ICP and AAS analyses are in agreeable range. The range of coefficients of variation is 12-41 percent. Table 34 is the summarized form of Table 33. This compares the mean values of Cu, Cr, and As retention in treated wood, between the calculated values from gross solution absorption measurements and the mean values of the AAS and ICP analyses. It was found that there is no significant difference between the DSR as measured by gross absorption and as determined by the chemical analysis. The total oxide content also shows the same trend between the calculated and determined values. For a DSR of 7.5 kg/m<sup>3</sup> CCA as determined by chemical analysis, an equivalent oxide content of 3.0 kg/m<sup>3</sup> is detected in the chemical analysis. The ratio of As : Cu : Cr in the treated wood is found to be 1.0 : 2.8 : 3.2, conforming to the ratio of 1:3:4 in the treatment solution. This implies that treating rubber wood in green condition with CCA is not a problem as far as the absorption of the individual components in the treated wood is concerned. But this may be an exceptional situation. In a recent study, Kuppusamy and Sharma (1987) reported that the pH of the solution increased from about 2 when freshly prepared to more than 2.5 or 3.0 when the same CCA solution was repeatedly used for treatment, resulting in the formation of sludge. They observed that more arsenic than copper and chromium got precipitated and there was a progressive reduction in the strength of the preservative solution after each treatment charge. As a consequence, they found that the treated



**Table 33. Results of chemical analysis of rubber wood treated in green condition with 3% CCA under the economical schedule, 15'/15'/5'**

Sl. No.	Data from gross solution absorption measurements (kg/m <sup>3</sup> )										Data from AAS measurements (kg/m <sup>3</sup> )										Data from ICP measurements (kg/m <sup>3</sup> )									
	DSR	CrO <sub>3</sub>	CuO	As <sub>2</sub> O <sub>5</sub>	Total oxides	Cr	Cu	As	CrO <sub>3</sub>	CuO	As <sub>2</sub> O <sub>5</sub>	Total oxides	Cr	Cu	As	CrO <sub>3</sub>	CuO	As <sub>2</sub> O <sub>5</sub>	Total oxides	Cr	Cu	As								
1.	8.4	1.41	1.00	0.91	3.32	0.73	0.80	0.59	2.10	0.90	0.49	2.68	1.09	0.72	0.32	1.92	0.98	0.44	3.34	1.00	0.78	0.29								
2.	5.2	0.87	0.62	0.56	2.06	0.45	0.50	0.37	1.33	0.83	0.38	2.53	0.69	0.66	0.25	1.29	0.90	0.31	2.49	0.67	0.72	0.20								
3.	9.3	1.56	1.11	1.01	3.68	0.81	0.89	0.66	2.01	1.25	0.49	3.75	1.05	1.00	0.32	1.79	1.45	0.43	3.67	0.93	1.16	0.28								
4.	6.1	1.02	0.73	0.66	2.41	0.53	0.58	0.43	1.76	0.70	0.48	2.94	0.91	0.56	0.31	1.50	0.66	0.40	2.55	0.78	0.53	0.26								
Mean	7.3	1.22	0.87	0.79	2.87	0.63	0.69	0.51	0.51	1.80	0.46	2.98	0.94	0.74	0.30	1.00	1.00	0.39	3.01	0.69	0.80	0.26								
CV (%)	26.3	26.5	26.3	26.6	26.4	26.6	26.4	26.7	26.7	19.3	12.0	18.3	19.3	25.4	11.9	33.2	33.1	15.2	19.3	40.8	33.2	15.1								

timber contained less arsenic than expected. The proportions of the components of CCA in the treated timber increased from 1: 2.9 : 3.5 (As:Cu:Cr) in their first charge to 1: 4.4 : 5 in their fifth charge, the retention of arsenic being much lower compared to the copper and chromium in the material treated in the fifth charge. Kuppusamy and Srinivasan (1989) also reported the case of a selective absorption of the constituents of CCA in a door shutter made out of rubber wood, received from a commercial source. In their study, although the penetration of the preservative was found to be good, data on the absorption of individual components showed a significantly low arsenic and high copper retention. As against the normal ratio of 1: 3: 4 of As, Cu and Cr in the treating solution, they found that the material received by them showed an average ratio of 1: 7.1: 5.3, indicating a very low arsenic retention compared to copper and chromium.

**Table 34. Comparison of mean values of the results of chemical analysis with the results calculated from gross solution absorption measurements (CV (%) values are given in brackets)**

Parameters	By gross absorption measurements (kg/m <sup>3</sup> )	By chemical analysis (kg/m <sup>3</sup> )
DSR	7.30 (26.3)	7.50 (17.3)
Total oxides	2.87 (26.4)	3.00 (17.3)
CrO <sub>3</sub>	1.22 (26.5)	1.71 (18.3)
CuO	0.87 (26.3)	1.05 (24.8)
As <sub>2</sub> O <sub>5</sub>	0.79 (26.6)	0.43 (13.3)
Cr	0.63 (26.6)	0.89 (18.3)
Cu	0.69 (26.4)	0.77 (29.5)
As	0.51 (26.7)	0.28 (13.3)

The disproportionate or selective absorption of the constituents of the CCA solution may be attributed to the treatment conditions, especially the pH of solution, concentration and the extent of sludge formation after every charge in

the solution. This kind of loss of preservative in the form of sludge can be avoided by monitoring the pH of the treating solution at every possible stage of treatment and correcting it with chromic acid whenever it exceeds 2.5, which incidentally may bring back into solution the portion of the preservatives sedimented out (Kuppusamy and Srinivasan 1989). Another way to tackle this problem, even though cumbersome, is to chemically analyse the treatment solution after every treatment charge and make good the loss of the components observed to keep the treating solution in the proper proportion. Hartford (1986) and Pizzi (1983) also gave a comprehensive account on the importance of pH, sludge, impurities, plant keeping, etc. while the treatment of timber with CCA. Unless proper care is taken during CCA treatment, it is possible that the strength of the solution may get reduced considerably after few charges which may not only result in poor retention than expected, but also inadequate distribution of the toxic components in the treated timber. The present study clearly showed that treating rubber wood in green condition with CCA is not a problem as far as the proportionate absorption of the individual constituents of the preservative formulation is concerned, provided the treatment conditions, especially the treatment solution concentration and pH, are carefully monitored. Treating green timber with 3% CCA solution achieves the desired chemical loading by employing the economical schedule, 15'/15'/5'.

#### **4.5.4 Summary of vacuum-pressure impregnation (VPI) treatment trials**

It is found that the new economical schedule arrived at is suitable for employing in the treatment of rubber wood at air-dried, partially dried and in green conditions. Desired DSR requirements can be achieved by using treatment solution of appropriate concentration.

Commerical scale trials proved the utility of the economical scheudle not only for boron impregnation, but also for the impregnation of CCA preservative, and even for the treatment of green wood (MC around 75%). Thickness, length and moisture content of the samples affect the DSR.

It is found that for the treatment of green timber the use of 4% BAE solution is sufficient to achieve the desired DSR levels while employing the economical schedule. For wood in air-dried and partially dried conditions, the concentration of treatment solution can be lowered to 3% BAE. For the treatment of green material using CCA preservative, the use of 3% solution is found sufficient to achieve desired DSR. For wood in air-dried and partially dried condition, the concentration of CCA solution can be lowered to 2 percent. For wood samples with high thicknesses (around 100 mm) and with very high moisture content the concentration of treatment solution has to be increased in the order of 5-6% for boron and 4% for CCA, in order to get desired DSR levels.

Even though thickness and length are found to affect DSR, the general conclusion is that as far as sizes required for various end-uses are concerned, the effect of variation of these parameters was found not causing any problem in the achievement of desired DSR levels (due to the active lateral penetration of preservative), provided treatment solution of appropriate concentration is employed.

## **4.6 UNDER-WATER STORAGE**

### **4.6.1 Optimum storage conditions**

No sapstain development was observed in billets either with intact bark or without bark kept under water for a period up to 6 months. Beyond 3 months algal growth was observed on the samples causing a greenish colour on the surface of the samples. However, this was found limited only to the surface and the wood inside was clean as found from sawn samples. More bacterial activity was noticed after 4 months causing foul smell. This was in spite of the fact that water was changed frequently (every ten days) to reduce algal and bacterial growth.

To avoid extractives from the bark polluting the water, it is advisable to store wood under water in squared billet form. This will further help to accommodate more volume. Sawn sizes can also be stored under water.

#### 4.6.2 Effect of under-water storage on physical and mechanical properties

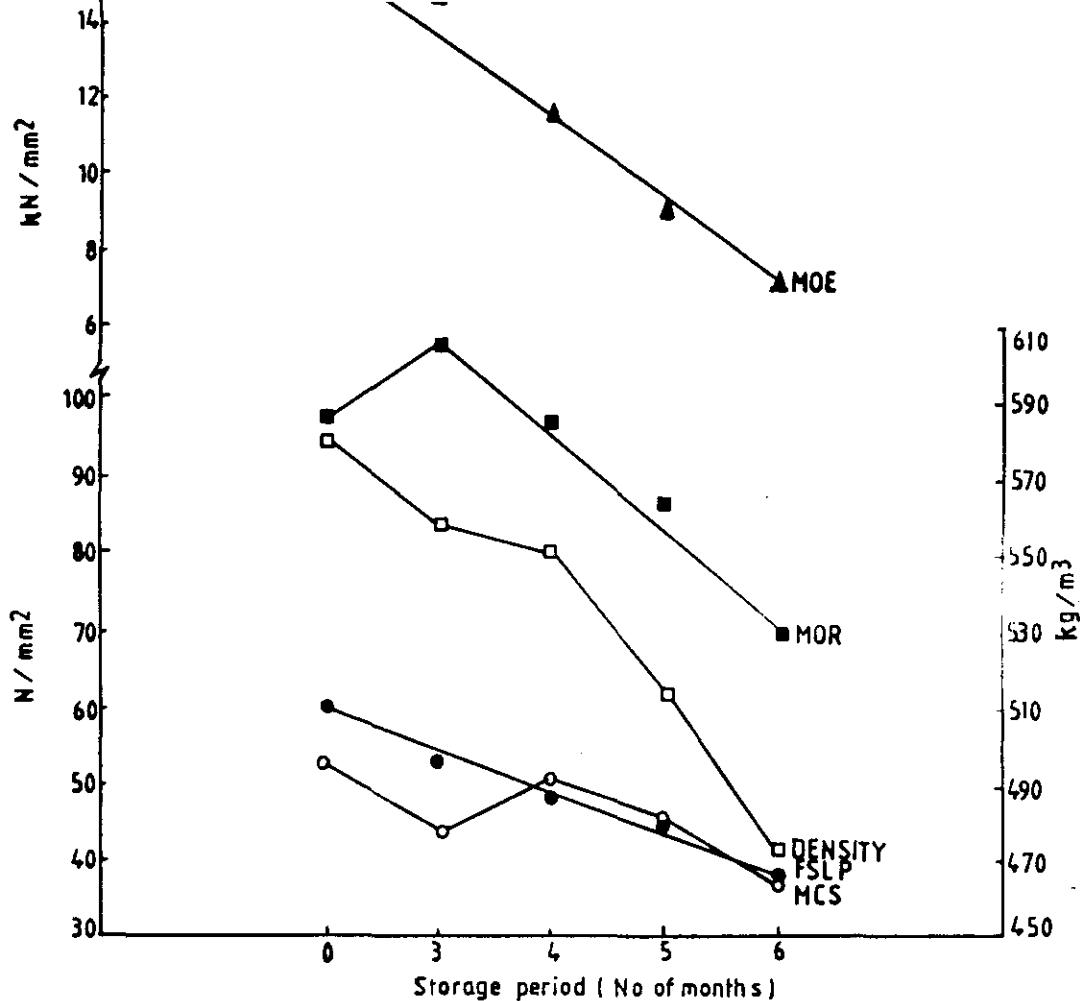
Table 35 shows the effect of under-water storage on the physical and mechanical properties of rubber wood in air-dried conditions (MC 12%). For comparison purpose, the physical and mechanical properties data of non-ponded, air-dried rubber wood at 12% MC were extracted from earlier report of Gnanaharan and Dhamodaran (1993). It can be seen that from an under-water storage period of 3 months onwards up to 6 months, values of physical and mechanical properties show a decreasing trend (Figure 16). The slightly high value of MOR for wood stored under water for 3 months may be due to the sampling errors. Except this, the values of all other mechanical properties of under-water stored wood are lower than the non-ponded, air-dried wood.

**Table 35. Effect of under-water storage on the mechanical properties of rubber wood (CV (%) values are given in brackets)**

Property	Under-water storage period (No. of months)				
	0**	3	4	5	6
FSLP (N/mm <sup>2</sup> )	60.45 (24.4)	53.47 (19.0)	48.70 (19.3)	45.83 (5.6)	38.49 (24.0)
MOR (N/mm <sup>2</sup> )	98.35 (4.2)	107.90 (13.2)	96.83 (9.4)	87.55 (12.4)	70.16 (22.3)
MOE (kN/mm <sup>2</sup> )	15.67 (37.8)	14.77 (34.9)	11.69 (22.6)	9.06 (27.1)	7.23 (32.4)
MCS (N/mm <sup>2</sup> )	52.73 (9.5)	44.01 (7.1)	50.12 (11.8)	45.01 (14.1)	36.75 (15.7)
Density* (kg/m <sup>3</sup> )	580.0 (5.5)	557.9 (5.9)	551.3 (3.4)	513.9 (6.7)	473.3 (8.3)

\* Mean of 12 samples, all other parameters, mean of 6 samples.

\*\* Values of the mechanical properties of non-ponded air-dried rubber wood at 12% MC, extracted from Gnanaharan and Dhamodaran (1993).



**Fig. 16 Effect of under-water storage period on the physical and mechanical properties of rubber wood**

Liese (1984), in his review on the wet storage of wind-blown conifers in Germany, observed a reduction of up to 10-15% in impact bending and compression strength after 3 years of wet sprinkler storage of pine wood. He did not observe further reduction after 5 years wet storage. MOR and MOE after 4 years sprinkling were sufficiently high to satisfy the standard requirements of marine (boat) timber for which the level is higher than the construction timber. He found no significant strength reduction affecting later utilization due to prolonged wet storage. The findings of this study are contrary to the above findings, may be due to the difference in nature of the timber species.

The percentage reduction in properties of wood stored under water for various duration does not show any uniform trend. The data were subjected to test of

significance by the ANOVA technique and Table 36 gives the results of the test. Pair-wise comparison was made between non-ponded, air-dried wood and 3, 4 and 5 months ponded wood. It can be seen that the reduction in values of FSLP, MOR and MOE due to under-water storage is not significant up to a storage period of 3-4 months. The reduction in wood density is also not significant for 3 months, but significant at 5% level for wood stored for 4 months and highly significant (1% level) for wood stored for a period of 5 months. Naturally, storage beyond 5 months will adversely affect all the mechanical properties.

**Table 36. Analysis of variance (ANOVA) of the data on the effect of under-water storage on mechanical properties of rubber wood - Test of significance**

Property	Between non-ponded and 3 months ponded			Between non-ponded and 4 months ponded			Between non-ponded and 5 months ponded		
	D.F	M.S	F. ratio	D.F	M.S	F. ratio	D.F.	M.S	F. ratio
FSLP	1	146.091	0.913 <sup>NS</sup>	1	320.333	2.543 <sup>NS</sup>	1	641.526	5.722 <sup>*</sup>
MOR	1	245.708	2.318 <sup>NS</sup>	1	6.871	0.137 <sup>NS</sup>	1	352.242	7.379 <sup>*</sup>
MOE	1	2.493	0.081 <sup>NS</sup>	1	46.138	2.216 <sup>NS</sup>	1	131.209	6.389 <sup>*</sup>
MCS	1	228.202	13.045 <sup>**</sup>	1	20.384	0.681 <sup>NS</sup>	1	178.641	5.471 <sup>*</sup>
Density	1	3037.500	2.854 <sup>NS</sup>	1	5075.042	7.388 <sup>*</sup>	1	26467.042	24.038 <sup>**</sup>

NS = Not significant  
<sup>\*</sup> = Significant at 5% P level  
<sup>\*\*</sup> = Significant at 1% P level

In the case of MCS, 3 months under-water stored wood is significantly different (at 1% level) from the non-ponded air-dried material. But for 4 months under-water stored wood it was found non-significant. For 5 months under-water stored wood, it is again found significant. To clarify the situation, wood stored under water for a period of 1 and 2 months were also subjected to compression parallel to grain test at air-dried condition. The mean values of MCS were 38.39 and 41.66 N/mm<sup>2</sup> respectively (mean of 6 samples each). The mean MCS for non-ponded air-dried rubber wood is 52.73 N/mm<sup>2</sup>. This clearly shows that the higher values obtained for the MCS of 4 months under-water stored air-dried

wood material may be due to sampling problems. The general conclusion drawn from this data is that, as far as MCS is concerned, under-water storage significantly affects this property. Hence for end-uses where MCS is critical, it is advisable to avoid under-water storage of rubber wood.

#### 4.6.3 Effect of under-water storage on treatability

The treatability was tested for under water stored wood at air- dried condition (MC 16%) and at a high moisture content (95%) level.

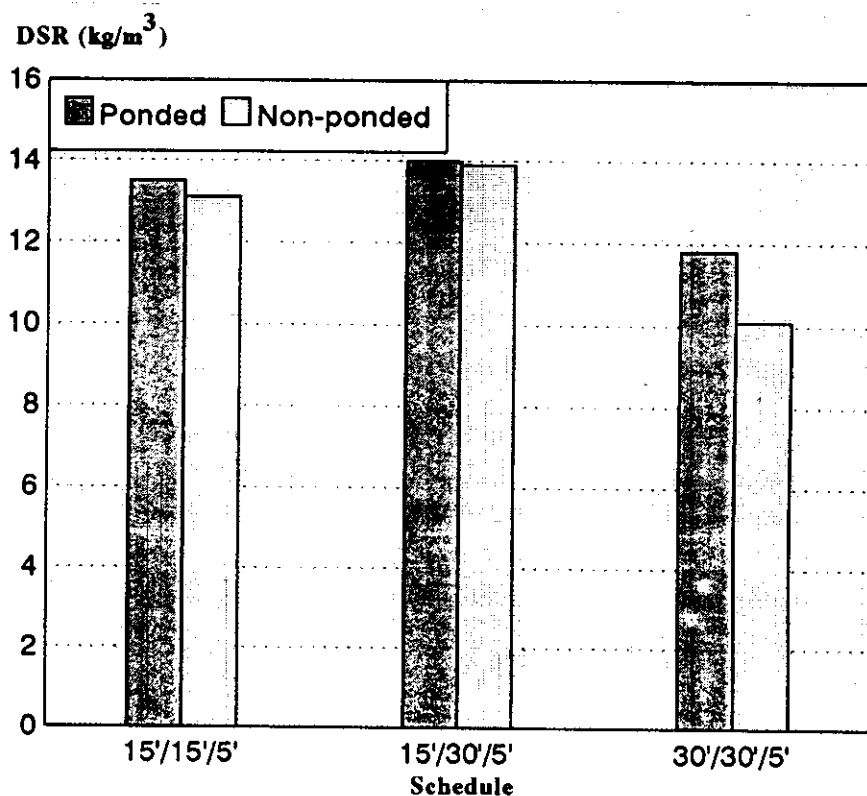
Table 37 shows the effect of under-water storage on the DSR of air-dried material treated with 3% BAE solution, under three different treatment schedules. It can be seen that for the treatment of air-dried wood, under-water storage for a period of 1.5 months does not make an appreciable difference in the achievement of DSR when compared with the non-ponded, air-dried wood (Figure 17). As revealed in earlier studies, here also it has been shown that the application of the schedule 15'/15'/5' is adequate to achieve the desired DSR.

**Table 37. Effect of under-water storage and treatment schedule on the dry salt retention (DSR) of rubber wood, while boron impregnation treatment with 3% BAE solution, in air-dried condition**

Treatment schedule	1.5 months under-water stored and air-dried wood			Non-ponded air-dried wood		
	DSR (kg/m <sup>3</sup> )		CV (%)	DSR (kg/m <sup>3</sup> )		CV (%)
	Mean	Range		Mean	Range	
15'/15'/5'	13.5	12.5-14.6	5.7	13.1	11.9-14.4	7.8
15'/30'/5'	14.0	13.3-15.2	5.5	13.9	12.7-15.4	7.3
30'/30'/5'	11.8	9.3-13.8	15.8	10.1	8.9-11.1	9.4

n = 5





**Fig. 17 Effect of treatment schedule on the dry salt retention (DSR) of under-water stored wood treated in air-dried condition with 3% BAE solution**

As immediate impregnation after conversion can reduce the possibility of biodegradation during the air-drying stage, possibility of treating under-water stored wood at high moisture content (MC 95%) was investigated. Two, four and six months under-water stored wood was impregnated with 3% BAE solution employing the treatment schedule 15'/15'/5'. Data in Table 38 show that only low DSR values were achieved (The average DSR in terms of %BAE varies from 0.58 to 1.08). But there is significant difference in DSR as the under-water storage period increased. This clearly indicates that as the duration of under-water storage increases the treatability at high moisture level also improves. This may be attributed to some bacterial attack as well as due to the removal of some starch and other water leachable extractives from wood due to ponding thereby improving the permeability. The DSR recommendation as per

the Indian Standard (BIS 1982) was not achieved with the use of 3% BAE solution under the particular treatment schedule employed for the material at very high moisture content. However, if we are aiming for DSR of around  $3 \text{ kg/m}^3$  (on an average, 0.48 %BAE retention) (see section 4.2.2 and 4.3.2), the concentration of the solution used (3% BAE) seems to be sufficient. Otherwise, the concentration should be increased accordingly.

**Table 38. Effect of duration of under-water storage on DSR, while boron impregnation treatment with 3% BAE solution at high moisture content (95% MC), employing the treatment schedule 15'/15'/5'**

Under- water storage period (No. of months)	DSR ( $\text{kg/m}^3$ )		CV (%)	Av. BAE%	Range	Mean density
	Mean	Range				
2	2.8	2.3-3.3	14.0	0.58	0.53-0.67	481.9
4	4.6	4.0-4.8	6.9	0.95	0.75-1.11	491.1
6	4.9	4.2-5.9	2.8	1.08	0.94-1.27	449.5

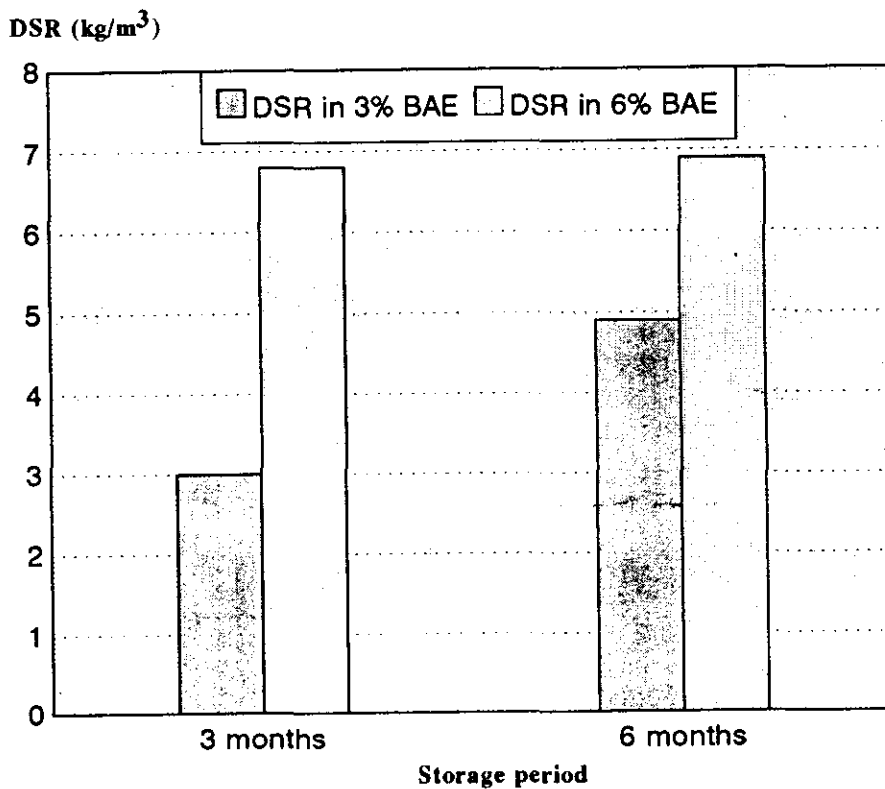
n = 5

By doubling the concentration of treatment solution from 3%, higher DSR values were achieved (Table 39) for the ponded material with high moisture content (Figure 18). With the use of a solution of higher concentration, the improvement in treatability is not found significant in the case of material at very high moisture content (95% MC). The earlier experiments on the treatability of non-ponded wood at green condition (75% MC) with a 6% BAE solution resulted in very high loading of chemicals. The lower DSR values achieved in the treatment of under-water stored wood may be due to much higher moisture content level (an average moisture content of 95%). However, this study proves conclusively that for the treatment of under-water stored rubber wood at comparatively higher moisture levels, the use of 6% BAE solution is a must to achieve the DSR requirements.

**Table 39. Effect of doubling the concentration of treatment solution from 3% BAE on the DSR of 3 and 6 months under-water stored rubber wood with high moisture content (95%) level under boron impregnation treatment by employing the treatment schedule 15'/15'/5'**

Under-water storage period (No. of months)	3% BAE			6% BAE			
	DSR (kg/m <sup>3</sup> ) *		CV (%)	DSR (kg/m <sup>3</sup> )		CV (%)	Mean density (kg/m <sup>3</sup> )
	Mean	Range		Mean	Range		
3	3.0	2.7-3.5	11.0	6.8	6.0-8.5	14.5	447.9
6	4.9	4.2-5.9	12.8	6.9	5.6-8.2	15.0	473.7

n = 5



**Fig. 18 Effect of concentration of treatment solution on the dry salt retention (DSR) of under-water stored wood treated at high moisture content (MC 95%) level**

Table 40 shows the effect of different treatment schedules on the DSR of 6 months under-water stored wood at high moisture content level (MC 95%) with the use of a 3% BAE solution. As the sizes used for this treatment under various schedules were not uniform, the DSR values were divided by the surface area of samples in order to see whether the DSR per unit surface area exhibits any uniform trend. No specific trend in DSR or DSR per unit surface area is noticed for the various treatment schedules attempted. A DSR of above 6.5 kg/m<sup>3</sup> BAE was achieved only by the treatment schedule 60'/60'/5'.

Penetration of chemicals in the treated wood was tested in all the cases and found uniform and through and through.

**Table 40. Effect of treatment schedule on the dry salt retention (DSR) of 6 months under-water stored rubber at high moisture content, while boron impregnation treatment with 3% BAE solution**

Treatment schedule	DSR (kg/m <sup>3</sup> )		CV (%)	Mean value of DSR/ Surface area (kg/m <sup>3</sup> /m <sup>2</sup> )	Size of samples
	Mean	Range			
0'/15'/5'	3.0	2.5-3.7	16.7	17.8	S <sub>2</sub>
0'/30'/5'	2.4	1.8-2.8	16.0	14.2	S <sub>2</sub>
0'/60'/5'	2.9	2.6-3.4	11.8	14.7	S <sub>3</sub>
15'/15'/5'	3.5	2.3-5.7	39.9	20.7	S <sub>2</sub>
15'/30'/5'	4.5	3.9-5.9	18.1	26.7	S <sub>2</sub>
15'/60'/5'	4.4	3.2-5.5	25.3	20.6	S <sub>1</sub>
30'/15'/5'	4.1	3.7-4.5	8.2	19.2	S <sub>1</sub>
30'/30'/5'	2.9	2.5-3.3	10.4	17.2	S <sub>2</sub>
30'/60'/5'	5.3	4.6-5.7	13.7	31.4	S <sub>2</sub>
45'/15'/5'	2.2	2.0-2.5	9.8	10.3	S <sub>1</sub>
60'/15'/5'	5.9	4.5-8.9	33.7	27.7	S <sub>1</sub>
60'/30'/5'	2.9	2.3-3.5	14.6	13.6	S <sub>1</sub>
60'/60'/5'	7.4	5.6-8.9	16.0	34.7	S <sub>1</sub>

\* Size of sample S<sub>1</sub> = 93.8 x 43.8 x 1050 mm  
 S<sub>2</sub> = 75.0 x 37.5 x 1050 mm  
 S<sub>3</sub> = 81.3 x 81.3 x 1050 mm

n = 5

#### **4.6.4 Summary of the effect of under-water storage**

It was found that for long term storage of rubber wood, under- water storage can be employed effectively without any problem of sapstain development. Even though due to under-water storage all physical and mechanical properties are found lowered, the reduction in property, except MCS, is not significant for a storage period of 4 months. The reduction in MCS due to under- water storage is significant for a storage period of even one month. Hence in end-uses where MCS is critical, it is advisable to avoid under-water storage practice. For all other purposes, an under-water storage of 3-4 months does not adversely affect the utilization value of the timber.

The under-water stored wood is found treatable in any moisture content level by employing the economical schedule. Generally, under-water storage improves the treatability. Any desired DSR levels can be achieved by using proper concentration of treatment solution.

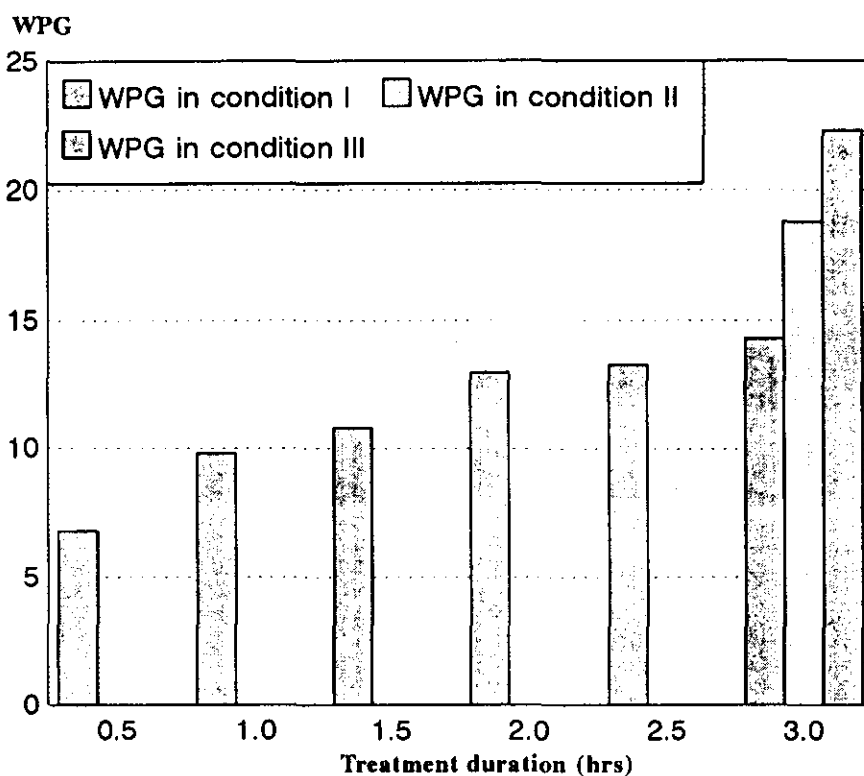
### **4.7 CHEMICAL MODIFICATION OF RUBBER WOOD BY ACETYLATION**

#### **4.7.1 Effect of treatment conditions on acetyl weight percent gain**

Table 41 shows the effect of treatment duration as well as concentration of reagent on weight percent gain (WPG). A WPG of 14.3 achieved by reacting with 1 molar acetic anhydride solution for 3 hours on samples of 20 x 20 x 5 mm cross-sections improved to 18.8 by vacuum impregnation with 1.5 molar solution followed by refluxing for 3 hours. The WPG value is improved to 22.3 by vacuum impregnation with 3 molar solution followed by refluxing for 3 hours. This clearly indicates that the reaction conditions have got a significant influence on the WPG that can be achieved by acetylation (Figure 19).

**Table 41. Effect of acetylation conditions on Weight Percent gain (WPG)**

	Refluxing in 1 molar solution						Vacuum impregnation with 1.5 molar solution and refluxing	Vacuum impregnation with 3.0 molar solution and refluxing
	Treatment Duration (hr)							
	0.5	1.0	1.5	2.0	2.5	3.0	3.0	3.0
Mean WPG	6.8	9.8	10.8	13.0	13.3	14.3	18.8	22.3
CV (%)	6.8	6.9	5.1	3.9	3.7	3.2	2.9	7.0
n	5	5	5	5	5	5	20	10



**Fig. 19 Effect of acetylation conditions on the weight percent gain (WPG)**

At WPG above 17, acetylated wood has been found by soil block tests to be resistant to attack by the fungi *Coniphora puteana*, (Bekere *et al.* 1978, Goldstein *et al.* 1961, Ozolina and Svalbe 1966, Rugevitsa 1977 and Svalbe *et al.* 1978), *Gloeophyllum trabeum* (Goldstein *et al.* 1961, Koppers' 1961 and Peterson and Thomas 1978), *Lentinus lepideus* (Goldstein *et al.* 1961), *Polyporus versicolor* [*Coriolus versicolor*] (Goldstein *et al.* 1961, Koppers' 1961 and Tarkov *et al.* 1950), *Poria incrassata* (Goldstein *et al.* 1961 and Tarkov *et al.* 1950), *Poria microsporia* (Tarkov *et al.* 1950) and *Poria monticola* (Goldstein *et al.* 1961). The present study on the effect of treatment condition on the chemical modification shows that adequate levels of acetyl weight percent gains required to achieve effective biological resistance, as per the above mentioned reports, can be achieved in the case of rubber wood also.

#### **4.7.2 Dimensional stability of acetylated rubber wood**

Rowell (1975) reported good dimensional stability at 15-20 acetyl WPG. Hence, samples with WPG above 15 were subjected to dimensional stability tests and the results are summarized in Table 42. Sufficient leaching periods were provided by keeping the samples submerged in water for 10 days for the first soaking and 20 days for the second soaking. As such the modified samples were subjected to prolonged leaching, the antishrink efficiency (ASE) values will really reflect the extent of dimensional stabilization achieved through the treatment. Data from Table 42 reveals the fact that fairly good dimensional stability can be achieved by acetylating rubber wood above 18% weight gain. Rowell and Ellis (1978) reported that acetylation of southern pine with acetic anhydride gave the most consistent ASE values after repeated water wetting tests. The values reported by them vary from 69.6 to 65.4 for ASE<sub>1</sub> to ASE<sub>4</sub> at 21.1 WPG. For rubber wood, the present study achieved ASE values ranging from 94.0 to 77.6% for ASE<sub>1</sub> to ASE<sub>4</sub> at 18.8 WPG and 96.4 to 81.4% for ASE<sub>1</sub> to ASE<sub>4</sub> at 22.3 WPG. This indicates a strong bonding and very little loss of chemical during the leaching cycle. Actually, it was observed that only 5.2% weight loss occurred

after 1 month soaking of modified wood with 18.8 WPG. Hence, the data on dimensional stability tests by repeated water leaching on modified rubber wood indicates the possibility of achieving good dimensional stability by acetylation. It is found that a pooled mean ASE of 85.5 to 87.8% can be achieved with an acetyl weight gain of 18.8 to 22.3 percent.

**Table 42. Dimensional stability of acetylated rubber wood - Effect of repeated water leaching of modified samples on antishrink efficiency (ASE). (CV (%) values are given in brackets)**

WPG	ASE <sub>1</sub> %	ASE <sub>2</sub> %	ASE <sub>3</sub> %	ASE <sub>4</sub> %	Pooled mean
18.8* (2.9)	94.0 (6.6)	83.5 (9.7)	87.0 (11.1)	77.6 (11.8)	85.5 (7.9)
22.3** (7.0)	96.4 (3.1)	89.0 (4.0)	84.2 (2.5)	81.4 (1.8)	87.8 (7.5)

\* n = 20  
\*\* n = 10

#### **4.7.3 Effect of acetylation on physical and mechanical properties**

The results of the effect of acetylation on mechanical properties of rubber wood are detailed in Tables 43 and 44. There is a slight reduction in modulus of rupture (MOR) as the WPG increases. But the extent of reduction in MOR due to acetylation is not big enough to affect the utilization value, as seen from the MOR value of untreated wood (Figure 20). Oven dried untreated control samples showed a mean MOR value of 101.7 N/mm<sup>2</sup> and maximum compressive stress (MCS) of 50.9 N/mm<sup>2</sup>. Gnanaharan and Dhamodaran (1993) reported MOR value of 98.4 N/mm<sup>2</sup> and MCS value of 52.7 N/mm<sup>2</sup> at 12% MC. The slightly low MCS value (50.9 N/mm<sup>2</sup>) obtained for the oven dried control material may be due to the small sample size. The present study material came from the bottom portion of one single tree only. For air-dried wood a modulus of elasticity (MOE) of 15.7 kN/mm<sup>2</sup> is reported in the earlier study. The present study showed a MOE of 8.3 kN/mm<sup>2</sup> for oven-dried untreated wood. This lower value may be



due to the very low moisture content of the present material. The MOE of treated wood does not differ from that of untreated material to such an extent that the utilization value is lowered. The MCS is not found different for treated and untreated samples.

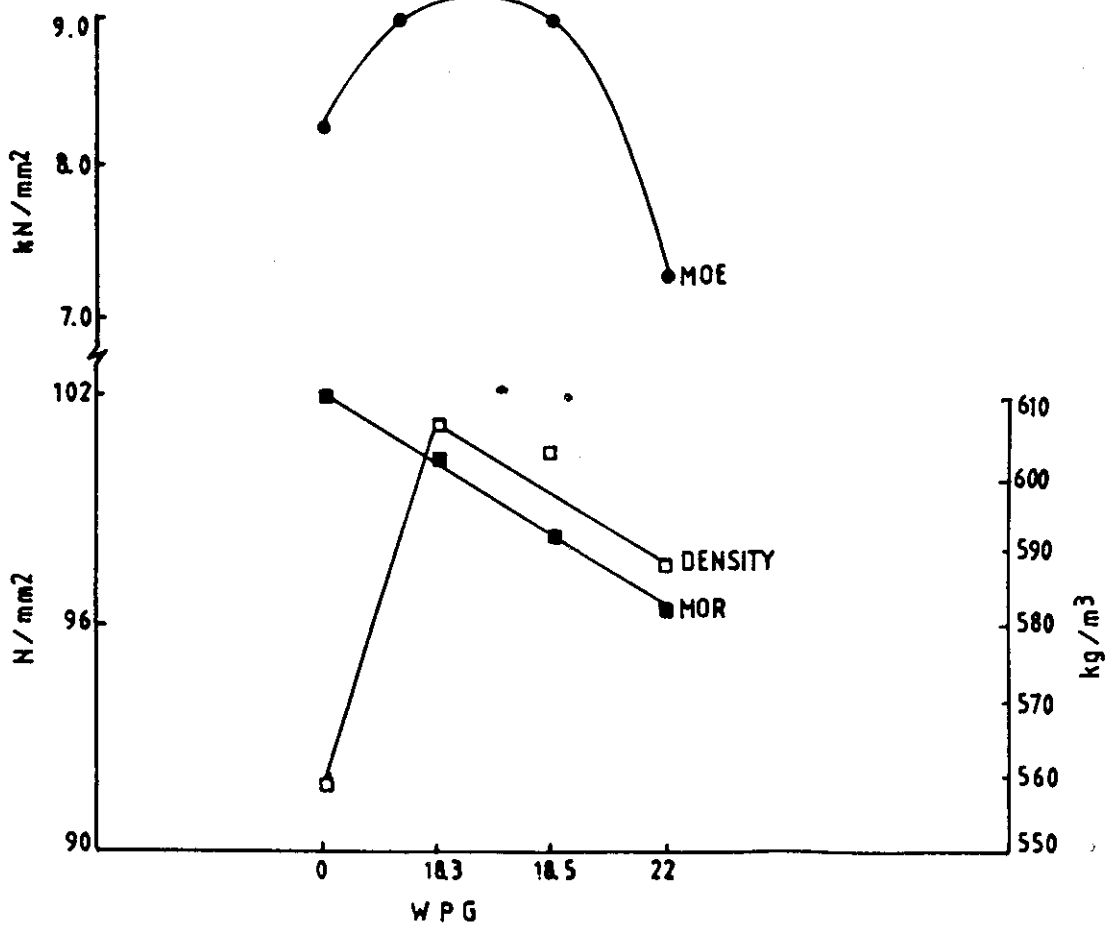
**Table 43. Effect of acetylation on modulus of rupture (MOR) and modulus of elasticity (MOE) (CV (%) values are given in brackets)**

Details of treatment process	Mean WPG	Mean density (kg/m <sup>3</sup> )	Mean MOR (N/mm <sup>2</sup> )	Mean MOE (kN/mm <sup>2</sup> )	n
12 hr refluxing in 1 molar solution	18.3 (7.9)	605.7 (4.8)	100.4 (15.6)	9.0 (10.0)	15
Vacuum impregnation with 1.5 molar solution and 3 hr refluxing	18.5 (6.7)	602.9 (4.0)	98.4 (13.7)	9.0 (16.4)	15
Vacuum impregnation with 3.0 molar solution and 3 hr refluxing	22.0 (5.0)	588.1 (4.9)	96.4 (17.7)	7.3 (7.8)	5
Oven-dried untreated control samples	0.0	557.9 (4.5)	101.7 (9.8)	8.3 (11.9)	15

**Table 44. Effect of acetylation on maximum compressive stress (MCS) at 18.5 WPG. (CV (%) values are given in brackets)**

	Modified wood	Control (untreated, OD)
Mean MCS(N/mm <sup>2</sup> )	50.9 (9.6)	50.9 (6.2)
Density	596.7 (4.3)	547.4 (5.0)

n = 15



**Fig. 20 Effect of acetylation on the physical and mechanical properties of rubber wood**

Density of oven-dried control samples was found to be  $545.8 \text{ kg/m}^3$ . Due to acetylation this increased to  $603\text{-}606 \text{ kg/m}^3$  for a WPG range of  $18.3 - 18.5$ . The lower value obtained for the density of acetylated material at WPG  $22.0$  may be due to the limited number of samples ( $n=5$  only). On an average, density is found improved to the extent of 9 percent.

Acetylated wood remains in a permanent swollen state and there are less fibres per unit area compared with untreated wood (Agarwal *et al.* 1985). The less number of fibres per unit area has a tendency to lower the strength value. This may be the reason for lower MOR values for treated wood, even at higher density. In general, it is found that acetylation of rubber wood does not affect the bending and compressive strength significantly. Narayanamurthy and Handa (1953)

reported a slight decrease in MOE due to acetylation. Here also MOE is found to be lower at 22 percent weight gain than the untreated material. Koppers' (1961), and Dreher *et al.* (1964) reported that acetylation increased the compressive strength. MOR is increased for softwoods, but decreased for hardwoods. All these reports indicate the possibility of a species effect also. Agarwal *et al.* (1985) reported that the MOR and MCS improved with increase in chemical add on. They reported that at optimum treatment level, the mechanical properties are higher than those of untreated control. But the fact remains that their acetyl WPG were very much lower than in the present study. For fir, mango and gurjan wood they tested, the WPG range was from 4.1 to 8.6 only. Further, they used thioacetic acid as the reagent.

The general conclusion is that rubber wood could be acetylated effectively with good weight percent gain needed to prevent biological degradation and to achieve good dimensional stability. The mechanical properties are also not lowered due to the modification reaction to such an extent that the utilization value is lowered.

Even though chemical modification may not be cost-effective when compared to the conventional wood preservation methods, due to the increasing environmental concerns, in future, it may attract further attention for specified end-uses. The present study clearly indicates the potential of utilizing rubber wood for such purposes.

## **4.8. PREPARATION OF MEDIUM DENSITY FIBRE (MDF) BOARD FROM ACETYLATED RUBBER WOOD FIBRES**

### **4.8.1 Method for acetylating rubber wood fibres and preparing MDF board**

Sawn wood from the basal portion of a rubber tree was utilized for this study. The wood was chipped, then defibrated and oven-dried. The oven dried, weighed fibre material was reacted with excess acetic anhydride without any solvent or

catalyst for 2 hours at 120°C in a glass reactor. The acetylated fibre was washed with acetone to remove unreacted or excess reagents and byproducts and filtered under vacuum. All the materials were again oven-dried. The weight percent gain (WPG) achieved was found to be 14.0.

MDF boards were made as detailed in section 3.7. Mean density of the wood material used was 545 kg/m<sup>3</sup>. After acetylation, at an acetyl WPG of 14, the mean density of MDF boards made with acetylated fibres was around 835 kg/m<sup>3</sup>. The control boards made with non-acetylated fibres showed a density of around 780 kg/m<sup>3</sup>.

#### 4.8.2 Dimensional stability of acetylated MDF board from rubber wood

Table 45 shows the results of volumetric and thickness swelling tests on acetylated boards. At an acetyl WPG of 14, the antishrink efficiency (ASE) of boards made with acetylated fibres was 85.5% for volumetric and 94.8% for thickness swelling. The study made by Tomimura *et al.* (1989 b) reported about 75% decrease in thickness swelling at a WPG of 15.4 for MDF boards made with acetylated rubber wood. Youngquist *et al.* (1986 a) reported that the wet thickness swell is about 4.5 times more for the controls. Rowell *et al.* (1989) also reported excellent dimensional stability for acetylated wood, as determined from liquid water tests.

**Table 45. Effect of acetylation on dimensional stability of MDF boards made with acetylated rubber wood fibres at an acetyl WPG of 14 - Antishrink efficiency (ASE) values (CV (%) values are given in brackets)**

Antishrink efficiency percentage (ASE%)	
Volumetric swelling	Thickness swelling
85.5	94.8
(17.0)	(4.7)

n = 5

### 4.8.3 Effect of acetylation on the bending strength of MDF board from rubber wood

Boards made from acetylated fibres showed slight improvement in bending strength than the control boards (Table 46). This is contrary to the findings of Tomimura *et al.* (1989 a), where the mechanical properties of acetylated rubber wood MDF boards were reported to decrease considerably. The present findings on the improvement in bending strength is favoured by another report by Agarwal *et al.* (1985) on the acetylation of solid wood with thioacetic acid. They reported that MOR, MCS and toughness improved with increase in chemical add on and at optimum treatment level, properties were higher than that of the untreated controls. Rowell *et al.* (1989) reported that MOR and MOE were slightly reduced for totally acetylated veneer-faced low-density particle boards of 20 mm thickness made from acetylated seraya (*Shorea* spp.) wood and faced with 3 mm thick veneers from Douglas-fir (*Pseudotsuga menziesii*). Youngquist *et al.* (1986 b) found that acetylation process adversely affected the strength properties of aspen flake board at an acetyl WPG of 20. All these reports indicate that the wood species and board manufacturing conditions affect the mechanical properties of the modified wood.

**Table 46. Effect of acetylation on bending strength of MDF boards made with acetylated rubberwood fibres at an acetyl WPG of 14 (CV (%) values are given in brackets)**

Property	MDF boards made from acetylated fibres	MDF boards made from control (non-treated) fibres
MOR (N/mm <sup>2</sup> )	24.60 (30.7)	20.89 (36.0)
MOE (kN/mm <sup>2</sup> )	2.09 (26.3)	2.11 (34.5)
Density (kg/m <sup>3</sup> )	834.7 (13.1)	788.7 (15.0)

n = 10

#### **4.8.4 Summary of chemical modification of rubber wood**

It is found that rubber wood could be acetylated effectively achieving good weight percent gains (WPG) needed for imparting biological resistance and dimensional stability. The treatment conditions were found to have influence on the achievement of desired WPG levels. Antishrink efficiency (ASE) in the range of 85-88% are achieved with acetyl weight percent gains of 18.8-22.3 percent. The physical and mechanical properties are found not lowered due to the modification reactions to such an extent that the utilization value is lowered.

Also, it was found that MDF board made from acetylated rubber wood possess superior properties as far as its dimensional stabilization is concerned. At an acetyl WPG of 14, the ASE of MDF boards made with acetylated fibres was found 85.4% for volumetric and 94.8% for thickness swelling, indicating the achievement of excellent dimensional stability. The mechanical property, bending strength, of the MDF board is also found not adversely affected due to acetylation.

The higher values of bending strength and dimensional stability show that chemical modification through acetylation has great potential to improve the properties of rubber wood for its applications in re-constituted wood products. This opens up the possibility of utilizing rubber wood for specialty products through chemical modification.

## 5. CONCLUSIONS

The conclusions drawn from the various experiments conducted during the study are listed below.

- Rubber wood is easily treatable by diffusion method using boron preservatives. Desired dry salt retention (DSR) can be achieved by the use of 10% boric acid equivalent (BAE) solution.
- Up to 50 mm thick sizes of rubber wood can be effectively diffusion treated with 10% BAE solution, fulfilling the New Zealand and BWPA specifications (0.2% BAE retention in the core and 0.4% net average retention as a whole respectively). Both 75 and 100 mm thick sizes can be diffusion treated with 10% BAE solution, but only the New Zealand specification of 0.2% BAE retention in the core can be achieved. For imparting effective protection to thicker sections and in cases where DSR greater than  $3 \text{ kg/m}^3$  is desired, it is advisable to opt vacuum-pressure impregnation treatment.
- The study has optimized the diffusion storage period for rubber wood. A storage period of 2, 3, 9 and 12 weeks is required for 25, 50, 75 and 100 mm thick material respectively, to attain uniform distribution of chemicals in the treated wood.
- Rather than the surface area per unit volume of wood, it is the thickness of sizes that is playing a significant role in the achievement of desired DSR in the boron diffusion treatment.

- Reducing the duration of dipping time from 40 minutes to 30 minutes for the 25 mm thick material and from 160 minutes to 80 minutes for the 50 mm thick material does not affect the achievement of desired DSR as recommended by the BWPA specification while employing the boron diffusion treatment.
- Rubber wood is easy to treat by the vacuum-pressure impregnation method using boron chemicals. The initial vacuum period was found to be critical for rubber wood. A vacuum of 85 kPa for a duration of 15 minutes was found to be optimum. Increasing the vacuum period beyond 15 minutes was found to reduce the chemical pick-up considerably.
- Although the chemical pick-up increased with increase in pressure treatment time, even a pressure of 1000 kPa for 15 minutes was found to give more than adequate loading of chemicals in air-dried, partially dried and even in green wood.
- The optimum treatment schedule of 15 minutes initial vacuum of 85 kPa followed by 15 minutes pressure of 1000 kPa and a final vacuum of 85 kPa for 5 minutes (denoted by 15'/15'/5') is the most economical treatment schedule. Employing this schedule, when air-dried wood was treated with 3% BAE solution nearly double the level of loading of chemicals recommended by the Indian Standard was achieved.
- If the concentration of treatment solution is increased to 5-6% BAE, even green rubber wood can be pressure treated to the required loading of chemicals prescribed in the Indian Standard.
- As rubber wood can be pressure treated even in green condition using a higher concentration of treatment solution, attack by



sapstaining fungi before treatment can be avoided. However, as there are certain fungi which can attack boron-treated wood, a suitable fungicide should be incorporated with the boron solution to take care of the staining problem that might take place after treatment and before kiln drying.

- It is estimated from the chemical analysis of boron impregnated as well as diffusion treated timber that, a measured DSR of around  $3 \text{ kg/m}^3$  equivalent to a calculated retention of 0.5% BAE will satisfy the New Zealand and BWPA specifications.
- The economic schedule arrived at is capable of achieving the IS prescription of  $6.5 \text{ kg/m}^3$  DSR (as measured from gross solution absorption), provided proper level of concentration of treatment solution is selected depending upon the moisture content of the material and the thickness of sizes.
- In rubber wood, the penetration of chemicals through lateral sides of the timber is equally efficient as with that of longitudinal side. The thickness and length of wood are the most influencing factors than the surface area per unit volume of the material for the achievement of desired DSR. The penetration of preservatives through the lateral side of the samples is found very active.
- The variation in solution pick-up due to change in concentration of treatment solution is found to be negligible in the case of rubber wood. Hence, the concentration required for achieving any desired DSR range can be worked out to an acceptable level of accuracy. Similarly, as the variation in surface tension, viscosity and pH due to concentration change is not very large, altering the strength of boron compounds in the treatment solution will not in any way adversely affect the solution pick- up.

- The density of the boron solution is found linearly related to concentration. Hence, the variation of concentration of treatment solution during different batches of treatment can be monitored by checking the solution density using a hydrometer.
  
- Full scale testing of the economical schedule arrived at for the boron impregnation treatment in commercial scale plants showed that the schedule is capable of achieving the desired DSR in the case of air-dried, partially dried and green wood.
  
- Comparison of the economical schedule with the schedules followed in some commercial treatment units showed that the newly developed economical schedule is more productive, much time-and energy-saving.
  
- As far as the effect of size of wood on DSR is concerned, it was found that as the size increased DSR decreased correspondingly. As thickness and length of samples play a significant role in determining the extent of solution pick-up, in treatment charges where materials of different thickness and length are unavoidable, care must be taken to ensure that the thickest and longest material achieves the desired DSR. But in general, it was found that as far as common end-uses are concerned, neither the variation in thickness nor the length of the material pose problems for the achievement of the desired DSR due to the active lateral penetration of preservative, provided treatment solution of adequate concentration is used.
  
- From the results of full scale testing of the economical schedule using solutions of various concentrations in different commercial plants, the following points are brought out:

- (a) The use of 3% BAE solution provided more than sufficient DSR in the case of air-dried wood, and just adequate DSR for partially dried rubber wood.
  - (b) For the treatment of rubber wood in green condition, use of 4% BAE solution will achieve the desired DSR.
  - (c) A concentration of 1.6% BAE is sufficient to offer protection of sizes used in the manufacture of compressed shuttle blocks (63 x 63 x 450 mm) if treated in partially dry condition (MC around 50%). This will achieve a boron retention level of around 3.0 kg/m<sup>3</sup> BAE.
- Small variations in the extent of vacuum (ranging from 85-115 kPa) and pressure (ranging from 800-1200 kPa) do not affect the achievement of desired DSR. Even the application of 500 kPa pressure is found sufficient to give the desired DSR in small dimension sizes required for the production of compressed shuttle blocks.
  - Rubber wood is easy to treat with CCA also. Pilot plant trials showed that for sizes of cross-sections varying from 25 x 25 to 100 x 100 mm, in air-dried and partially dried conditions, sufficient DSR as prescribed in the Indian Standard for non-ground contact applications can be achieved by using 2% CCA solution.
  - Full scale testing of the CCA impregnation of green rubber wood in commercial scale plants showed that 3-4% CCA solution can be employed effectively for achieving the desired DSR while employing the economical schedule, 15'/15'/5'. It has also been shown that except for high thicknesses (100 x 100 mm) the use

of 3% CCA solution is adequate to achieve desired DSR in green rubber wood with around 65% moisture content. When the moisture content is higher than this level, at around 75% MC, and for thicker sizes, it is desirable to go for a CCA solution of higher concentration, viz., 4 per cent.

- The penetration of CCA in the treated wood (treated in air-dried, partially dried and in green condition) was found to be uniform and through and through.
  
- Chemical analysis of CCA impregnated wood showed that the absorption of the constituents of CCA (copper, chromium and arsenic) in the treated timber is in the desired ratio, provided the treatment conditions are ideal (i.e., the pH and concentration of solution are not altered and sludge formation due to successive treatments is avoided).
  
- For commercial scale treatment of rubber wood with CCA solution, as the solution is reported to be extremely sensitive to pH and hence due to the possibilities of selective precipitation or sludge formation, the right concentration and pH of solution after every charge of operation have to be ensured for the proper absorption of the constituents of CCA (copper, chromium and arsenic) in the treated timber.
  
- Rubber wood can be stored under water without any problem of sapstain development. To accommodate more volume and to avoid the problem of polluting water with the extractives from bark, it is advisable to store wood under water in squared billet form. Sawn sizes can also be stored under water. If the storage is in an artificial pond, the water needs to be changed once in a week or ten days to avoid foul smell.

- Under-water storage reduces all the physical and mechanical properties of wood when compared with non-ponded wood. Up to three months under water storage does not significantly affect the wood density; up to four months the reduction is significant at 5% level. Whereas fibre stress at limit of proportionality (FSLP), modulus of rupture (MOR) and modulus of elasticity (MOE) are not significantly affected up to a storage period of four months, MCS is significantly lowered due to under-water storage, even for a period of one month. Hence for all other end-uses except in cases where MCS is important, a storage period of 3-4 months does not significantly affect the utilization value of the timber. For end-uses where MCS is critical, it is advisable to avoid the use of under-water stored wood.
  
- It has been found that the economical schedule is adequate to achieve the desired DSR in the case of under-water stored wood at air-dried condition and at comparatively high moisture content level (95% MC). In general, the treatability improves as the under-water storage period increases. The use of 3% BAE solution is sufficient to achieve only the BWPA specification requirement of DSR for under-water stored wood at high moisture content level (95%), while treating the wood employing the economical schedule. By increasing the concentration of solution to 6% BAE, adequate DSR can be achieved for the under-water stored wood at high moisture content level.
  
- The effect of different treatment schedules on the DSR of 6 months under-water stored wood at high moisture content level (95%) indicates that using 3% BAE solution with the economical schedule, 15'/15'/5', does not achieve the DSR as desired by the Indian Standard. In this case the treatment schedule has to be modified to 60'/60'/5'. The DSR per unit surface area of wood

does not exhibit any uniform trend. Hence, rather than the surface area, it may be the thickness of wood and length that may be influencing significantly the achievement of desired DSR while treating in a particular schedule. If the economical schedule has to be employed, the concentration of treatment solution has to be increased to 6% BAE in the case of material with very high moisture content.

- Whenever the under-water stored wood treated at high moisture content level passes the DSR requirement, the penetration of boron in the treated wood is found uniform and through and through.
- In the chemical modification of rubber wood, the reaction conditions have significant influence in the weight percent gains (WPG) that can be achieved by acetylation. WPG up to 22.3 was achieved in the present study, and WPG above 17 is reported to be sufficient to achieve good biological resistance. Hence, rubber wood could be chemically modified by acetylation so as to achieve adequate weight percent gains needed for improving its biological resistance.
- A pooled mean antishrink efficiency (ASE) of 85.5 to 87.8% achieved for the acetylated rubber wood with an acetyl WPG of 18.8-22.3 showed the achievement of very good dimensional stability of acetylated rubber wood.
- Density is found to increase to an average extent of 9% due to acetylation.
- The mechanical properties are not lowered due to the modification reaction to such an extent that the utilization value

is lowered. In general, chemical modification of rubber wood by acetylation technique holds a good potential for the environmentally friendly way of non-conventional wood preservation.

- At an average WPG of 14, the mean ASE of medium density fibre boards (MDF) made from acetylated rubber wood fibres is found to be 85.5% for volumetric and 94.8% for thickness swelling, indicating excellent dimensional stability for the acetylated boards.
  
- Boards made from acetylated fibres showed slight improvement in bending strength. Hence, acetylation of rubber wood is not adversely affecting the utilization value of the re-constituted board product made out of it. Hence, acetylation has great potential to improve the properties of rubber wood for its applications in re-constituted board products such as MDF.

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\* Original not seen.

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# **PUBLICATIONS**

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## Plant investigation of boron treatment of rubber wood: g at an economical treatment schedule\*

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al treatment schedule (15 minutes initial vacuum of  
minutes pressure of 1000 kPa; 5 minutes final vacuum  
as arrived at for treating air-dried rubber wood in a  
cylinder. Treating with 3% BAE (boric acid equivalent)  
led to a dry salt retention of 13.1 kg/m<sup>3</sup>. Also, the  
that green rubber wood can be treated to required  
retention level by employing the above treatment schedule  
by increasing the concentration of the treatment solution to

### Wirtschaftlich vertretbare Borsäure-Behandlung von Pilot-Maßstab

wirtschaftlich vertretbare Borsäure-Behandlung von Hevea  
wurde unter folgenden Bedingungen erreicht: 15 min  
um bei 85 kPa, 15 min Druckbehandlung bei  
min Endvakuum bei 85 kPa. Mit einer Konzentration  
säure-Äquivalent führte diese Behandlung bei luft-  
trockenem Holz zu einem Salzeintrag von 13,1 kg/m<sup>3</sup>. Auch in  
grünem Holz kann die geforderte Salzmenge eingebracht werden,  
wenn die Konzentration der Lösung auf 6% erhöht wird.

tion

among the major rubber (*Hevea brasiliensis*  
Swartz, syn. *Hevea*) growing countries of the world.  
Brazil, with an equable climate, accounts for more  
than 50% of India's rubber plantations. Area under  
cultivation increases each year and as of 1986–87, it was  
1.2 million ha (Anonymous 1988). Now nearly 1.2 million  
ha of rubber wood is available in the market every year.  
It is estimated that by 2000 AD, about 2.0 million  
ha of rubber wood per year will become available. As  
rubber wood is available in plenty at comparatively low  
prices, rubber plantations have become a major source of  
timber supply in Kerala. Rubber wood, despite the  
fact that it is highly perishable, accounted for 65% of the  
industrial wood consumption in Kerala during  
1980–85 due to the acute shortage of industrial timbers  
and the increasing price (Krishnankutty 1989). This

clearly indicates that rubber wood plays a significant role  
in the industrial economy of Kerala. Considering the  
industrial scope of this wood from a non-conventional  
source, the Kerala Forest Research Institute started  
working on the preservative treatment of rubber wood.  
Adding value to this perishable timber not only improves  
the economy of the state but also helps in reducing  
pressure on forests for industrial timber.

Gnanaharan (1982) standardised a simple diffusion  
treatment with boron chemicals. The treatment was found  
to be effective in giving protection to rubber wood against  
borers (Gnanaharan et al. 1983), decay fungi (Balasun-  
daran, Gnanaharan 1990) and termites (Varma, Gnana-  
haran 1989). Dhamodaran and Gnanaharan (1984) stu-  
died the effect of immersion time on the retention of  
boron chemicals in rubber wood. It was found that  
chemical retention increased with immersion time even  
though the rate of increase was low, but with increase in  
thickness of wood, prolonged immersion proved to be  
detrimental. Further, Gnanaharan and Dhamodaran  
(1990) studied methods of storage and the effect of  
under-water storage on the treatability of rubber wood.

The above studies showed that rubber wood was easy  
to treat and the boron chemicals were able to diffuse  
across the whole cross-section even up to 100 mm.  
However, during the diffusion storage, *Botryodiplodia*  
*theobromae*, *Fusarium decemcellulare*, *Aspergillus sydowii*  
and *Penicillium citrinum* were found to grow and stain  
the wood (Balasundaran, Gnanaharan 1990). Gnanaha-  
ran (1983; 1984; 1986) evaluated different anti-stain  
chemicals. Even though a number of chemicals were  
found to have potential to replace sodium penta-  
chlorophenoxide (NaPCP) they were not cost effective.  
As NaPCP has been banned in most of the countries, it  
has become imperative to find an alternative way to  
overcome the problem. As boron chemicals are highly  
diffusible, and as they do not get precipitated, treating  
green rubber wood by pressure method will be advanta-  
geous.

Tan et al. (1983) examined the feasibility of treating  
green rubber wood by vacuum-pressure impregnation.  
They found that reducing the pressure treatment time  
from 150 minutes to 75 minutes did not lower the  
absorption of chemicals in green wood. However, they  
did not examine reducing the pressure treatment time  
further. From the experience gained in conducting the  
treatability study on rubber wood, we felt that there was  
scope for reducing the pressure time considerably. There-

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July 1993

This study was undertaken to optimise the treatment schedule for treating rubber wood, and also to see the effect of different moisture levels on the chemical uptake.

## Materials and methods

Sh sawn green rubber wood pieces of size  $63 \times 63 \times 1000$  mm were brought to the laboratory from the sawmill. The test samples, carefully air-dried to 18% moisture content (MC) were used in this study. The study was conducted in a pilot-type preservative treatment cylinder (0.3 m diameter and 2.0 m long). The maximum obtainable pressure of 1000 kPa and vacuum of 15 kPa were used in the study. Final vacuum was maintained at 85 kPa for 5 minutes in all the trials. To arrive at the optimum treatment schedule, the effect of varying the initial vacuum time and the pressure treatment time on solution pickup was studied.

To study the effect of different moisture levels on the uptake of chemical solution, three levels of moisture were used: green wood (MC of about 75%); partially air-dried (MC of about 50%) and air-dried (12–18% MC). The dimensions of the test samples remained the same as above.

Trials were taken with 3% BAE (boric acid equivalent solution (w/w)). The solution uptake ( $\text{kg/m}^3$ ) of each test sample was determined from the weight difference before and after treatment and the volume of wood; the dry salt retention (DSR) was calculated by multiplying the uptake by solution concentration. BAE% retention was determined by dividing the DSR ( $\text{kg/m}^3$ ) by the density of wood ( $\text{kg/m}^3$ ).

## Results and discussion

### Effect of varying the initial vacuum period

In a full-cell process, initial vacuum is applied to ensure the cell lumina become empty and the whole lumen to be filled with the preservative chemical solution. However, we could not find any study reported in the literature on the effect of varying the duration of initial vacuum period. While varying the vacuum period from 0 minutes to 30 minutes at the interval of 15 minutes, an interesting phenomenon was noticed (Table 1). The DSR was maximum when the initial vacuum was retained for 15 minutes. One might expect that the chemical uptake to increase with increasing the duration of vacuum

period. However, maintaining the vacuum for 30 minutes resulted in low DSR. The DSR obtained with 15 minutes vacuum was more than double the value obtained with no vacuum (0') or vacuum for 30 minutes. This finding is difficult to explain.

It was decided to confirm this phenomenon. This time, trials were taken with initial vacuum period of 10, 15 and 20 minutes to see whether there was any inversion effect on DSR. Here also it was noticed that DSR was maximum with the vacuum period of 15 minutes. Decreasing or increasing the duration of vacuum even by 5 minutes resulted in low DSR. While these trials confirmed the trend observed in the first set of trials, it failed to explain the peculiar phenomenon observed. It was decided to verify this while studying the effect of varying the pressure treatment duration.

### 3.2 Effect of varying the pressure treatment period

The following treatment schedules, both to study the effect of varying pressure treatment period and also to see the peculiarity of 15 minutes initial vacuum period, were tried: initial vacuum for 0 minutes and pressure for 30 minutes (referred as 0'/15'); 10'/30'; 15'/30'; 15'/45'; 15'/60'; 20'/30'; 30'/30' and 30'/60'. Results obtained with these schedules and results obtained in the previous trials are combined and presented in Table 2 for easy comparison. It is clear from Table 2 that increasing the pressure treatment time (for the initial vacuum period of 0', 10', 20' and 30') resulted in increased DSR. This is to be normally expected. However, in the case of initial vacuum period of 15 minutes, as can be seen from the Table, there was no advantage in increasing the pressure treatment period. This clearly shows that the optimum initial vacuum period is 15 minutes. Further studies are needed to understand why the chemical pickup reaches maximum with an initial vacuum period of 15 minutes and then drops.

The Indian Standard Specification (ISI 1982) recommends a boric acid retention of  $6.5 \text{ kg/m}^3$  for building timbers for internal use. Out of the 13 different schedules tried, 10 schedules met this requirement. Out of these 10 schedules, the most economical schedule is one with 15 minutes initial vacuum, 15 minutes pressure and 5 minutes final vacuum period.

In New Zealand, the requirement of retention for hardwoods susceptible to *Lyctus* borers is 0.2% BAE in the core (McQuire 1962). To ensure the above requirement in radiata pine, McQuire (1962) found that the overall DSR should be about 1% BAE. As the average density

1. Effect of varying initial vacuum period on the dry salt retention (DSR) of treated rubber wood

Treatment Schedule vac/pr/vac (minutes)	DSR ( $\text{kg/m}^3$ )		CV(%)	BAE%	
	Average	Range		Average	Range
<i>Trial I</i>					
0/15/5	6.3	5.2– 7.1	11.8	1.1	0.94–1.27
15/15/5	13.1	12.0–14.3	7.8	2.3	2.14–2.56
30/15/5	5.8	4.5– 8.2	24.9	1.2	0.88–1.78
<i>Trial II</i>					
10/15/5	6.5	4.9– 7.1	30.2	1.3	0.90–1.88
15/15/5	13.9	9.3– 18.8	24.6	3.2	1.70–4.90
20/15/5	5.4	4.0– 6.3	17.2	1.1	0.79–1.26

Effect of varying initial vacuum period on the dry salt retention in rubber wood

Treatment schedule vac/pr/vac (minutes)	DSR (kg/m <sup>3</sup> )			CV(%)	BAE%	
	Average	Range			Average	Range
0/15/5*	6.3	5.2-7.1	11.8	1.1	0.94-1.27	
0/30/5	8.7	7.1-10.2	3.4	1.6	1.33-1.92	
10/15/5*	6.5	4.9-7.1	30.2	1.3	0.90-1.88	
10/30/5	7.7	5.9-10.0	19.4	1.6	1.19-2.24	
15/15/5*	13.1	12.0-14.3	7.8	2.3	2.14-2.56	
15/15/5*	13.9	9.3-18.8	24.6	3.2	1.70-4.90	
15/30/5	13.9	12.6-15.4	7.4	2.5	2.16-2.75	
15/45/5	11.2	9.3-15.7	23.0	2.2	1.87-2.74	
15/60/5	13.2	10.9-15.9	16.5	2.8	2.20-3.60	
20/15/5*	5.4	4.0-6.3	17.2	1.1	0.79-1.26	
20/30/5	6.9	6.3-7.5	7.4	1.4	1.19-1.59	
30/15/5*	5.8	4.5-8.2	24.9	1.2	0.88-1.78	
30/30/5	10.1	8.9-11.1	9.4	1.8	1.57-2.09	
30/60/5	14.8	13.6-16.5	9.3	2.9	2.60-3.29	

\* Taken from Table 1

wood is around 600 kg/m<sup>3</sup>, 1% BAE will be to DSR of 6 kg/m<sup>3</sup>. This is close to the Indian Specification recommendation of 6.5 kg/m<sup>3</sup>. The amount obtained in air-dried rubber wood using the most economical schedule arrived in the study is more than the required amount. This clearly shows that rubber wood can be treated even at higher moisture levels.

#### Effect of varying moisture levels

The most economical schedule was chosen and 3% BAE solution was used for treating partially dried (50% MC) and green (75% MC) rubber wood. The results are given in Table 1. Whereas partially dried rubber wood barely meets the DSR value recommended in the Indian Standard Specification, it was very low (3.8 kg/m<sup>3</sup>) in the case of green rubber wood. Tan et al. (1983), using a 3% BAE solution, obtained a DSR of 7.5 kg/m<sup>3</sup> in 57 mm thick partially dried rubber wood. However, this was possible with a treatment time of 75 minutes at 1050 kPa pressure. As boron chemicals are diffusible, it was decided to increase the concentration of the treatment solution and increase the pressure treatment time. One block of green wood was taken with 6% BAE solution and the average DSR obtained was 13.5 kg/m<sup>3</sup>. This shows that treating green rubber wood is not a problem. By choosing the right concentration of the treatment solution, the required chemical values can be met even when the moisture level

#### 4 Conclusion

The study has confirmed that rubber wood is easy to treat and the pressure treatment time can be considerably reduced. The initial vacuum period was found to be critical for rubber wood and a vacuum of 85 kPa for a duration of 15 minutes was found to be optimum. Increasing the initial vacuum period beyond 15 minutes was found to reduce the chemical pickup considerably.

Although chemical pickup increased with increase in pressure treatment time, even a pressure of 1000 kPa for 15 minutes was found to give more than adequate loading of chemicals.

The most economical treatment schedule (15 minutes initial vacuum of 85 kPa; 15 minutes pressure of 1000 kPa; 5 minutes final vacuum of 85 kPa) gave nearly double the required loading of chemicals in air-dried rubber wood, when treated with 3% BAE solution.

If the concentration of the treatment solution is increased to 6% BAE, even green rubber wood can be treated to achieve the required loading of chemicals.

As rubber wood can be treated even in green condition using a higher concentration of treatment solution, attack by the sapstaining fungi before treatment can be avoided. However, as there are certain fungi which can attack boron-treated wood, a suitable fungicide should be incorporated with the boron solution to take care of the staining problem that might take place after treatment.

The economical treatment schedule arrived at in this study is both time and energy saving. Also, the boron

Effect of moisture levels on the dry salt retention (DSR) of rubber wood

Condition of wood	DSR (kg/m <sup>3</sup> )		CV%	BAE%	
	Average	Range		Average	Range
<i>3% BAE solution</i>					
Air-dried* (12-18% MC)	13.1	12.0-14.3	7.8	2.3	2.14-2.56
Partially dried (50% MC)	6.5	5.2-7.1	11.5	1.1	0.86-1.18
Green (75% MC)	3.8	3.3-4.6	12.9	0.64	0.53-0.76
<i>6% BAE solution</i>					
Green (75% MC)	13.5	12.3-14.5	6.7	2.59	2.30-2.87

\* Taken from Table 1

ical penetration was found to be across the whole section of wood.

The treatment schedule arrived at by using a pilot-treatment cylinder should be verified in a commercial cylinder. Also, further study should be conducted to understand the role of the initial vacuum period on chemical pickup.

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# Commercial scale trial of an economical schedule for boron impregnation treatment of green rubber wood

T. K. Dhamodaran, R. Gnanaharan

Commercial treatment schedule, developed at the Kerala Forest Research Institute for boron impregnation of rubber wood, was tested in a commercial wood treatment plant. A mean dry salt retention (DSR) of 12.4 kg/m<sup>3</sup> was achieved for green rubber wood in green condition with average moisture content around 70%, using a 6% boric acid equivalent solution. This study confirms the success of the schedule for commercial application as the DSR achieved is much higher than the standard requirement of many countries.

## Wirtschaftliche Anwendung eines wirtschaftlichen Verfahrens zur Borimprägnierung von Gummibaumholz

Das Forest Research Institut in Kerala wurde ein wirtschaftliches Verfahren zur Borimprägnierung entwickelt. Dieses Verfahren wurde in einem Gewerbebetrieb an frischem Gummibaumholz mit mittleren Holzfeuchte um 70% getestet. Eine Tränklösung, die 6% Bor-säure-Äquivalente enthielt, erreichte die mittlere Salzretention von 12,4 kg/m<sup>3</sup>. Dies bestätigt die erfolgreiche Übertragung des Verfahrens für kommerzielle Zwecke; denn die Salzretention ist höher als es die Anforderungen vieler Länder erfordern.

## Introduction

The present investigation of an economic schedule (application of an initial vacuum of 85 kPa followed by a pressure of 800 kPa for 15 minutes and a final vacuum of 85 kPa for 5 minutes) is reported earlier (Gnanaharan and Dhamodaran 1993). The study on green rubber wood (average moisture content of about 70%) under this schedule with 6% boric acid equivalent (BAE) solution resulted in a dry salt retention (DSR) of 13.5 kg/m<sup>3</sup>. As this is more than twice the Indian Standard requirement of 6 kg/m<sup>3</sup> for construction wood (ISI 1982), this indicated the suitability of this schedule for boron impregnation treatment of green rubber wood even in commercial wood treatment plants.

## Materials and methods

The Forest Research Institute, Trivandrum, Kerala extended facility conducted the trial in their treatment cylinder of 0.9 m diameter and 6 m length. The same schedule, 15'/15'/15', as tried in the pilot plant investigation, was used for the treatment, with the condition that the maximum pressure exercised was only 800 kPa and 6% BAE solution, as in the earlier study for green wood,

the cylinder was loaded with rubber wood of length 2.25 m and two different cross sections, 75 × 75 mm and 88 × 88 mm. Samples in each size were marked and their initial weight

and volume recorded. The full charge was subjected to vacuum and it took about 15 minutes to reach the maximum achievable vacuum of 85 kPa and then the vacuum was retained in that condition for 15 minutes. After filling the cylinder with the treatment solution, pressure was applied and it took 30 minutes to reach the maximum pressure of 800 kPa and the pressure was maintained at that condition for 15 minutes. After the treatment, the solution was returned to the storage tank and a final vacuum of 85 kPa was applied for just 5 minutes. After the treatment, the marked samples were weighed again. From the weight difference, volume of wood, concentration of treatment solution and density of wood, the dry salt retention (DSR) in terms of kg BAE/m<sup>3</sup> and %BAE was calculated.

## 3

### Results and discussion

The average moisture content of the material was around 70% and density 550 kg/m<sup>3</sup>.

The results of the trial run are given in Table 1. An average DSR of 12.9 and 11.8 kg BAE/m<sup>3</sup> (2.36 and 2.14% BAE respectively) was achieved for samples of cross sections 75 × 75 mm and 88 × 88 mm respectively. As found in earlier studies, it was observed that as the thickness increased DSR decreased. The pooled mean DSR was 12.4 kg/m<sup>3</sup>. The DSR achieved for both sizes is much higher than the requirement of Indian Standard (ISI 1982), and the specifications of New Zealand (see McQuire 1962) and British Wood Preserving Association (see BCL 1972) for furniture wood. Hence this study confirms that the recently suggested schedule, 15'/15'/15', could be adopted for commercial scale boron impregnation treatment of rubber wood.

Tan et al. (1983) treated 57 mm thick green rubber wood with a 3% BAE solution by employing an initial vacuum of about 90 kPa for 45 minutes followed by a pressure of 1050 kPa for 75 minutes and a final vacuum for 15 minutes. They achieved a DSR of 7.5 kg BAE/m<sup>3</sup>. However, this study, using 6% BAE solution and employing the economic schedule of 15'/15'/15', achieved a pooled mean DSR of 12.4 kg/m<sup>3</sup> in green rubber wood. This trial carried out in a commercial plant with full load of wood confirms the validity of the economic schedule developed in a pilot plant. As the pressure used in this study was only 800 kPa compared to 1000 kPa in the pilot plant study it can be seen that lowering the pressure slightly did not affect the achievement of the required DSR. This also suggests that the concentration of the treatment solution can be reduced according to the DSR requirement.

Table 1. Vacuum-pressure impregnation treatment of green rubber wood (MC 70%) with 6% BAE solution with a treatment schedule of 15'/15'/15' - DSR data (n = 10)

Size of wood (mm × mm × m)	DSR (kg/m <sup>3</sup> )		CV (%)	BAE(%)	
	Mean	Range		Mean	Range
75 × 75 × 2.25	12.9	9.5–15.9	16.5	2.36	1.73–2.89
88 × 88 × 2.25	11.8	10.1–13.0	8.0	2.14	1.84–2.37

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## Conclusion

Study has confirmed that rubber wood in green condition can be pressure treated with boron compounds employing the optimum schedule developed at the Kerala Forest Research Institute (15 minutes initial vacuum of 85 kPa followed by application of a pressure of 800 to 1000 kPa for 15 minutes and a final vacuum for 5 minutes) in commercial scale plants also.

## 5

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## VERIFICATION OF AN ECONOMICAL SCHEDULE FOR BORON IMPREGNATION TREATMENT OF PARTIALLY DRIED RUBBER WOOD

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### Introduction

Gnanaharan and Dhamodaran (1993) presented an economical schedule for the boron impregnation treatment of rubber wood (*Hevea brasiliensis*). This schedule consists of an initial vacuum of 560 mm Hg for 15 minutes followed by a pressure of 10 kg/cm<sup>2</sup> for another 15 minutes and a final vacuum of 560 mm Hg for 5 minutes. This schedule was arrived at by treating air-dried rubber wood, and it was checked with partially dried and green rubber wood. It was found that, in the case of partially dried rubber wood, a dry salt retention (DSR) of 6.5 kg/m<sup>3</sup> could be achieved in samples of 63×63 mm cross-section, which just met the DSR value recommended in the Indian standard for building timbers in internal use (ISI, 1982).

In connection with a training programme for popularising the utilization of treated rubber wood among rural carpenters, rubber wood of various sizes were impregnated with 3% BAE solution. The sizes were treated by employing the above mentioned schedule (15'/15'/15') in a pilot plant of 300 mm diameter and 2 m long cylinder. The sizes were partially dried and had an average moisture content of

50%. Sizes of different thicknesses, varying from 25 to 63 mm (sample size of 10 to 40 pieces), were marked, and initial weight and dimensions were noted. Also samples were taken for testing the moisture content and density. The DSR was calculated from the weight difference and the concentration of treatment solution, and the percentage boric acid equivalent (%BAE) retention in the treated samples was calculated from the DSR and density data.

### Results and Discussion

The average moisture content of the material before treatment was found to be 50% and possessed an average density of 556 kg/m<sup>3</sup>.

Table 1 gives details on the various sizes that were treated, the DSR achieved, the average BAE% retention and number of samples in each thickness. The average DSR for various sizes ranged from 5.1 to 9.7 kg/m<sup>3</sup>. The pooled mean DSR is 7.3 kg/m<sup>3</sup>, which equals to a calculated net average retention of 1.3% BAE and is more than adequate as per the prescription of Indian Standards for building timbers in internal use (ISI, 1982). The British Wood Preserving Association's (BWPA) specification for retention of boron is 0.4% BAE (BCL,

Table 1

Vacuum-pressure impregnation treatment of partially dried rubber wood (50% MC) of assorted sizes with 3% BAE solution employing a treatment schedule of 15'/15'/15'

Size (mm × mm × mm)	DSR (kg/m <sup>3</sup> )	Range	CV%	Av. BAE%	Range	No. of samples (n)
1050 × 25 × 25	9.7	7.9–11.4	15.7	1.75	1.42 — 2.05	40
600 × 50 × 30	9.1	7.1–10.9	11.7	1.64	1.28 — 1.96	20
450 × 63 × 38	7.0	6.6 – 7.8	5.5	1.26	1.19 — 1.40	10
458 × 50 × 50	6.9	5.2— 8.8	16.9	1.24	0.94 — 1.58	10
825 × 56 × 56	5.8	5.2— 6.3	5.7	1.05	0.94 — 1.13	10
450 × 100 × 63	5.1	4.3— 5.5	7.2	0.92	0.77 — 0.99	10

1972). For rubber wood with density of 500 to 600 kg/m<sup>3</sup>, DSR will work out to 2.0 to 2.4 kg/m<sup>3</sup> BAE. The values obtained in this study are very much higher, and especially for indoor furniture a DSR of 2.5 kg/m<sup>3</sup> BAE will be sufficient. The penetration of chemicals in the treated wood was tested as per IS: 2753 part I (ISI, 1964) and found to have uniform through and through distribution.

The DSR decreased with increase in thickness. This is to be expected as all the sizes were treated together by the same schedule. It would be ideal to sort out sizes by thickness and carry out treatment separately to have control on chemical pick up. Otherwise, as was observed here, lower thickness material will tend to absorb more chemical at the

cost of thicker material. This can be seen in the higher retention of chemicals (6.5 kg/m<sup>3</sup> BAE) when the samples of 63 × 63 mm cross-section were treated in the previous trial, compared to only 5.8 kg/m<sup>3</sup> BAE obtained even for cross-section of 56 × 56 mm (in this study), when treated with samples of other thicknesses. If a mixture of different thickness can not be avoided, it should be ensured that the thickest material gets the minimum required DSR.

If we are aiming for a DSR of 2.5 kg/m<sup>3</sup> BAE for rubber wood, this study indicates that there is scope to reduce the concentration of the chemical solution. This trial has confirmed the validity of the treatment schedule for partially dried rubber wood, even for treating sizes of different thicknesses together.



### Summary

*An economical schedule developed at the Kerala Forest Research Institute for boron impregnation treatment of rubber wood was further verified on partially dried samples (about 50% moisture content). A pooled mean dry salt retention of 7.3 kg/m<sup>3</sup> was achieved by using a 3% boric acid equivalent solution of boric acid and borax. This confirms the utility of the newly developed schedule for the boron impregnation treatment of partially dried rubber wood.*

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## MECHANICAL PROPERTIES OF RUBBERWOOD FROM A 35-YEAR-OLD PLANTATION IN CENTRAL KERALA, INDIA\*

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**GNANAHARAN, R. & DHAMODARAN, T. K. 1993. Mechanical properties of rubberwood from a 35-year-old plantation in central Kerala, India.** The mechanical properties of air-dried rubberwood (*Hevea brasiliensis*) from a 35-year-old plantation in the central region of Kerala were determined. It was found that the mechanical properties tested, namely, modulus of rupture (MOR), modulus of elasticity (MOE) and maximum compressive stress (MCS) were higher for the 35-year-old material than for lower age material reported in the literature. These properties were comparable with those of other conventional timbers grown in Kerala. The study showed that rubberwood possesses medium strength properties.

Key words: Rubberwood - strength properties

**GNANAHARAN, R. & DHAMODARAN, T. K. 1993. Ciri-ciri mekanikal kayu getah dari ladang getah berumur 35 tahun di Kerala tengah, India.** Ciri-ciri mekanikal kayu getah (*Hevea brasiliensis*) yang telah di kering udara di laporkan. Kayu getah ini berasal dari ladang getah berumur 35 tahun di Kerala tengah, India. Hasil-hasil kajian yang lalu melaporkan, ciri-ciri mekanikal seperti modulus kerekanan (MOR), modulus kekenyalan (MOE) dan ketegangan mampatan maximum (MCS) adalah lebih tinggi bagi kayu getah yang berasal dari ladang getah berumur 35 tahun berbanding dengan kayu getah dari ladang getah yang lebih muda. Ciri-ciri ini menempatkan kayu getah dalam rejal yang setanding dengan kayu balak yang tumbuh di Kerala. Kajian ini menunjukkan kayu getah mempunyai kekuatan sederhana.

### Introduction

Kerala State accounts for a major share, nearly 90%, of rubber (*Hevea brasiliensis*) plantations in India. Rubber plantations have become a major source of industrial timber in Kerala. Knowledge of the physical and mechanical properties of rubberwood is essential for its proper utilization. Sanyal and Dangwal (1983) reported the results of physical and strength properties of rubberwood from 25- to 30-year-old trees from Kottayam, Kerala. Shukla and Mohan Lal (1985) tested wood from 22-year-old trees from Quilon, Kerala. It was seen from the above studies that rubberwood from 25- to 30-year-old trees had 22% lower MOR values and 26% lower MOE values than those of 22-year-old trees (Table 2). Generally, one would expect the strength values to taper off after attaining the maximum. It is not clear whether these lower values were due to locality differences, even though both Kottayam and Quilon are in the southern region of Kerala. The low strength values may be due to any one of the following reasons. Either the trees were of lower age, around 20 y or less, or they had such fast growth resulting in low strength values. Sanyal

\* KFR I Scientific Paper No. 234

and Dangwal (1983) tested the material which had been kiln-dried. If the kiln-drying had been carried out properly, there would be no reason for the kiln-drying to have reduced the strength values so drastically. It is probable that the kiln-drying was not done according to the right schedule. One more possibility is that the tested material had tension wood present in them. In this uncertainty, it was decided to confirm whether strength values of rubberwood, after reaching a maximum around the age of 20 years, declined afterwards. Also, in Kerala rubber trees are generally felled at the age of 35 y for replanting. So, this study was conducted to evaluate the strength properties of wood from 35-year-old trees from central Kerala.

### **Material and methods**

As the general practice in Kerala is to fell rubber trees at the age of around 35 y for replanting, the study material was obtained from a 35-year-old plantation which was being clearfelled. The plantation was located near Trichur in the central region of Kerala (76°16' E longitude and 10°31' latitude). The plantation, in general, was undulating with gentle slopes and good drainage. Stones and boulders were present on the surface but sheet rocks were absent. The soil of the area varied from reddish brown to yellowish red in colour, very gravelly loamy sand to gravelly sandy loam in texture, granular to massive in structure and with low organic matter.

Trees selected had girth varying from 80 to 100 cm. These trees were raised from bud material and not seeds. The logs were immediately sawn into sizes of 3 × 3 × 30 cm and air-dried. From the air-dried material six clear specimens, one from each tree, were selected randomly and re-sized to 2 × 2 × 30 cm for static bending test and 2 × 2 × 8 cm for compression test.

Static bending test and compression parallel to grain test were carried out in a 'Amsler' Universal Testing Machine. Tests were limited to these two as modulus of rupture (MOR), modulus of elasticity (MOE) and maximum crushing stress (MCS) will give a fairly good indication of the utilization potential. Density was determined by the water displacement method and moisture content by oven drying. The tests were done as per the Indian Standard IS: 1708 (Bureau of Indian Standards 1986). All the strength data were corrected to 12% moisture content for comparison purposes, using the formula suggested by Sekhar and Rajput (1968).

### **Results and discussion**

Table (1) reports the mean, range and CV values of fibre stress at the limit of proportionality (FSLP), MOR, MOE, MCS and density of the air-dried wood. It can be seen that rubberwood from the 35-year-old trees grown in the central region of Kerala had a mean MOR of 98.4  $Nmm^{-2}$ , MOE of 15.7  $kNmm^{-2}$ , MCS of 52.7  $Nmm^{-2}$  and density of 580  $kgm^{-3}$ . Even though wide variation was noticed in the FSLP and MOE values, this was not so in the case of MOR, MCS and density values. Coefficients of variation of MOR, MCS and density were very low compared to

the values suggested in the literature (USFPL 1974). In the case of MOR, CV was only 4.2% (against suggested value of 16%), MCS, 9.5% (against 18%) and density, 5.5% (against 10%). Coefficient of variation of FSLP (24.4%) was marginally higher than the suggested value of 22%. This clearly shows that the sample size was adequate.

**Table 1.** Strength properties of air-dried (12% MC) rubberwood

Property	Mean *	Range	CV (%)
FSLP, $N\ mm^{-2}$	60.45	41.70 - 80.64	24.4
MOR, $N\ m^{-2}$	98.35	91.22 - 102.20	4.2
MOE, $kN\ m^{-2}$	15.67	9.36 - 25.11	37.8
MCS, $N\ mm^{-2}$	52.73	43.23 - 55.95	3.5
Density, $kg\ m^{-3}$	580.0	536 - 624	5.5

\* Mean of 6 samples and in the case of density, of 12 samples.

Table (2) shows that the values of the mechanical properties obtained in the present study are higher than those of other studies reported from India and Malaysia (Sanyal & Dangwal 1981, MTIB 1982, Shukla & Mohan Lal 1985). Comparing the present study either with that of Sanyal and Dangwal (1983), or that of Shukla and Mohan Lal (1985), it is seen that strength values increase with age. In rapid-grown *Eucalyptus*, Jain (1969) found that there was no such difference in strength values between wood of 13- to 14-year-old trees and that of 20- to 21-year-old trees, which means that fast growing trees attain maximum strength much earlier than trees growing in natural forests. Rubber trees (*Hevea brasiliensis*) also have fairly fast growth. However, in *Hevea*, strength values continue to increase even up to 35 years. This has to be confirmed by testing wood from different age trees growing in the same locality.

**Table 2.** Physical and mechanical properties of dried rubberwood from different places

Location	Moisture content, %	Density $kg\ m^{-3}$	MOR $N\ mm^{-2}$	MOE $kN\ mm^{-2}$	MCS $N\ mm^{-2}$	Reference
Malaysia	17.2	550	66.8	9.42	32.9	MTIB (1982)
Kottavam	11.7	562	58.7	6.07	33.1	Sanyal & Dangwal (1983)
Quilon	12.0	557	75.6	8.20	37.4	Shukla & Mohan Lal (1985)
Trichur	12.0	580	98.4	15.67	52.7	Present study

The present study did not give any indication of possible influence of site factors. Whether locality plays a major role in variation in strength values or not can be ascertained only when trees of the usual felling age, that is 35 years, from different localities in Kerala are tested.

There is no strong correlation between density and MOR, and density and MOE. But the correlation coefficient between density and MCS is significant

(Table 3) and nearly 74% variation in MCS can be explained by density alone.

**Table 3.** Correlation coefficient for the relationship between density and other mechanical properties of air-dried rubberwood

X	Y	R <sup>2</sup>
Density	MOR	0.062
Density	MOE	0.538
Density	MCS	0.736

Table (4) compares the physical and mechanical properties of some common timbers of Kerala which are widely utilised for various end-uses. It can be seen that mechanical properties of rubberwood are in the comparable range of most of the conventional timbers.

**Table 4.** Physical and mechanical properties of some common timber species growing in Kerala (Nazma *et al.* 1981) in comparison with rubberwood

Species	Density kg m <sup>-3</sup>	MOR N mm <sup>-2</sup>	MOE kN mm <sup>-2</sup>	MCS N mm <sup>-2</sup>
<i>Tectona grandis</i> (teak)	650	95.9	12.0	53.2
<i>Albizia lebbek</i> (kokko)	640	88.7	12.3	53.4
<i>Albizia odoratissima</i> (kaku siris)	595-1010	143.8	14.5	78.7
<i>Artocarpus heterophyllus</i> (jack)	595	80.6	10.1	49.6
<i>Artocarpus hisutus</i> (aini)	595	96.9	12.2	61.6
<i>Cocos nucifera</i> (coconut palm)	761	92.7	15.9	57.2
<i>Grevillea robusta</i> (silver oak)	640	63.3	8.3	38.9
<i>Grewia tiliifolia</i> (dlhamau)	785	130.2	16.4	70.1
<i>Mangifera indica</i> (mango)	690	90.4	11.2	44.8
<i>Terminalia paniculata</i> (kindal)	785	111.8	14.3	63.9
<i>Xylia xylocarpa</i> (irul)	850	109.8	14.8	71.4
<i>Hevea brasiliensis</i> (rubberwood)*	580	98.4	15.7	52.7

\*Data from present study.

It is generally thought that rubberwood is weak. However, this study has shown that rubberwood from 35-y-old trees of the central region of Kerala has strength values comparable with those of many structural timbers. This study has indicated that strength values increase with age, and from the utilisation point of view, it is advisable to fell the trees around 35 y for replanting.

### Acknowledgements

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