TECHNOLOGICAL INVESTIGATION OF *PROSOPIS LAEVIGATA* WOOD FROM NORTHEAST MEXICO

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Artemio Carrillo-Parra

born in Linares, Nuevo León, México

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Examination committee: 1. Prof. Dr. Dr. h.c. František Hapla

2. Prof. Dr. Ursula Kües

3. PD Dr. Gerald Koch

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ABSTRACT

This study describes the anatomical properties of *Prosopis laevigata* trees found in northeast Mexico. The chemical composition and the topochemical distribution of lignin and phenolic compounds are described along with the deposition of extractives in pit canals, parenchyma cells, and the fiber S_2 layer using UV microspectrophotometry (UMSP). The main physical and mechanical characteristics of trees from four different areas of northeast Mexico are presented. The natural durability of wood samples from various regions is determined through use of the soil-bed test ENpr 807. The durability of extractive-free wood specimens toward basidiomycetes is investigated as is the growing inhibition of *Coniophora puteana* and *Trametes versicolor* caused by extractives obtained by using hot water, ethanol-water, acetone-water, and cyclohexane. The shear strength of the wood after being glued with melamine formaldehyde (MF) and Polyvinyl acetate (PVAc) is measured. The effect of artificial weathering is also discussed.

The *Prosopis* genus normally grows on arid and semi-arid land. It is used as a source of fodder for domestic animals, of flour for human consumption, and as a source of gums, mulch or compost. It also plays an important role in the production of honey. The wood is used to produce parquet lumber, furniture and decorative hand-crafted items; however, its main use is still as a source of fuel.

The importance of the *Prosopis* species, both within Mexico and around the globe, is presented in Chapter 1. Its use, distribution and ecological importance are also discussed. An anatomical description and an analysis of its chemical composition are given in Chapters 2 and 3, respectively. The size, proportion and distribution of the wood's fiber structure, of its vessels and of its ray parenchyma cells are discussed and compared with those of other *Prosopis* species. The chemical wood composition reveals a holocellulose content of between 61.7 - 64.5% and a Klason lignin content of between 29.8 - 31.4% within the heartwood tissue. A large percentage of extractive compounds (14.1 to 16.0 %) are found within the wood, including catechin, epicatechin and taxifolin.

The characteristics of the trees and, consequently, the properties of the wood are influenced by weather conditions in its natural habitats. Chapter 4 deals with the physical and mechanical properties, including density, swelling and shrinkage, as well as the modulus of elasticity, and the modulus of rupture and hardness. The results reveal that this wood is very stable with regard to dimensional changes and that it has medium to high wood strength. Differences in properties of wood grown in different areas are also presented.

P. laevigata wood is highly resistant to decay. As described in Chapter 5, its heartwood has a very low mass loss and a dynamic modulus of elasticity loss after 32 weeks of soil contact. Low mass loss (0.4 to 1.5 %) is also found after 16 weeks of exposure to the basidiomycetes *Coniophora puteana, Trametes versicolor, Irpex lacteus* and *Pleurotus ostreatus* in a modified EN 113.The natural durability is classified as Class 1 (very durable) according to European Standard EN 350-1. Extractives have a moderate to large effect on *C. puteana* and *T. versicolor* growth after dissolving in a malt-agar medium; the extractives are most effective at 1000 ppm concentration.

Artificial weathering and bonding properties are presented in Chapters 6 and 7. The wood has high stability with respect to dimensional changes and displays a great resistance to artificial weathering. The general appearance of *P. laevigata* changed from brown to white; Delta C (change of colour) increased from 5.6 to 9.6 and there were fewer crack formations than in *Fagus sylvatica* species but more than in *Tectona grandis*. Shear strength results obtained after gluing *Prosopis* wood (normally used for indoor applications) with Melamine Formaldehyde (MF) adhesives under wet condition demonstrate that Prosopis is suited for use in outdoor application.

In summary, it must be emphasised that the density, the wood stability with regard to moisture changes and artificial weathering, the high natural durability, and, finally, the high amount of shear strength after bonding are parameters which point to an almost limitless number of indoor and outdoor applications for this wood. The analyses of the properties of *P. laevigata* wood as well as those of feasible wood uses done in

this study have revealed some very important elements worthy of further research and development within the forestry and wood sciences.

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ABBREVIATIONS

%	Percentage
°C	Degrees Celsius
µm	Micrometer
A	Area of cross section (mm^2)
AFS	accelerated field simulators
ANOVA	Analysis of variance
ASE	Accelerated solvent extraction
AU	Absorption units
b	Width of specimen (mm)
CC	Cell corner
cm	Centimetre
CML	Compound middle lamella
CWE	Condense wood extractives
D	Diameter of steel ball (mm)
d	Diameter of steel ball impression (mm)
d_0	Diameter of control culture
d_1	Diameter of culture in presence of extractives
DBH	Diameter to breast height
EMC	Equilibrium moisture content
F	Load (N)
f	Frequency (KHz)
Gi	Growing inhibition effect in percent
h	Thickness of the specimen
HB	Brinell hardness (N/mm^2)
I	Moment of Inertia (mm^4)
K_1	Constant value (49.48)
kJ	Kilojoules
l	Span length
l_0	Length of sample
LP	Axial parenchyma cell,
m	Mass (g)
m	Metre
M	Molar
m_1	Constant value (4.72)
M-A	Malt-agar
MDI	Isocyanate or Diphenylmethane-4,4'-discocyanate
MF	Melamine-formaldehyde

ml	Millilitre
min	Minutes
MOE	Modulus of elasticity (N/mm^2)
MOE_{dyn}	Dynamic modulus of elasticity (N/mm^2)
MOE_{stat}	Static modulus of elasticity (N/mm^2)
MOR	Modulus of rupture (N/mm^2)
MUF	Melamine-urea-formaldehyde
N	Climate condition (20±1°C, 65±3%)
N/mm^2	Newton per square millimetre
nm	Nanometre
ppm	Parts per million
PRF	Phenol-resorcinol-formaldehyde
PVAc	Polyvinyl acetate
ρ	Density ($g/cm^{3)}$
RH	Relative humidity
RP-HPLC	Reversed phase high performance liquid chromatography
s	Second
S_1	Secondary cell wall layer 1
S_2	Secondary cell wall layer 2
S_3	Secondary cell wall layer 3
t/r	Ratio of tangential/radial
UF	Urea-formaldehyde
UMSP	Scanning UV microspectrophotometry
UV	Ultraviolet light
v	Vessel
v	Volume
W_1	Mass at beginning of test (g)
W_2	Mass at the end of test (g)
Wl	Mass loss in percent
Σ	Sum

Chapter 1

INTRODUCTION

1.1 General

It has been estimated that there are currently more than 50,000 plant species world wide. The largest number of native tree species found in a single country is 7,880 in Brazil. Astonishingly, only about 1000 different tree species are utilized globally (Sutton 1999; FAO 2006a). Thus, thousands of tree species are either, not utilized, under utilized, or used inappropriately. The present human population, estimated at approximately 6.5 billion in 2005 (Aktuell 2007), has wood consumption needs within the range of 0.3 to 0.6 m^3/year/habitant. As a result, the annual wood and wood based products consumption have been calculated to be around 3.5 billion m^3, approximately 66% of which are hardwoods used mainly as fuel; the rest are softwoods used principally in industry (Youngquist & Hamilton 1999).

In order to satisfy wood needs, forestry has been focussed on increasing wood production by improving forestry management. Plantations provide another option. In areas of Venezuela and Brazil between 5 to 90 m^3/ha/year of *Pinus caribea* and *Eucalyptus grandis* are produced, respectively (FAO 2006a); however, the material obtained from these plantations is "different" quality-wise in comparison to wood coming from natural forests (Zobel 1984). Plantation wood might show some unexpected characteristics, e.g., the anatomical structure and chemical composition can demonstrate fewer but wider annual rings. There is a different proportion of earlywood and latewood, a higher percent of juvenile wood and a different amount of extractives, all of which might effect such physical and mechanical properties as density, swelling, shrinkage, strength and hardness (FAO 2006b).

For the reasons mentioned above, one of the tasks of wood science and the wood industry must be to concentrate on increasing research to ensure a better utilization of lesser-known tree species from around the world. This should particularly apply to

trees grown on arid and semi-arid land which have shown desirable characteristics, making them good alternatives for a variety of wood, wood-based and non-wood products.

1.2 Forest resources in Mexico

Mexico is located between longitude 86°42'36'' W and 118°22'00'' W and latitude 32°43'06'' N and 14°32'27'' N (INEGI 2007); its overall area is 1,964,375 km^2, 60% of which are mountains and 40% of which is considered hilly to flat (Rodríguez & Maldonado 1996). According to an inventory of land use in Mexico completed in 2000, vegetation is dominated by xerophytes (27.0%), followed by agriculture lands (23.5%), forest lands (16.9%), rain forest (15.8%) (Palacio-Prieto *et al.* 2000). The variable vegetation types are a result of a variety of factors. These include the geographical location: Mexico lies between the Nearctic and Neotropical zones; the different types of climate; the geologic and orographic location which produces great differences in environmental conditions, habitats and microhabitats.

Even though the area covered by forest and rain forest lands was calculated as more than 0.6 million km^2 (32.75% of Mexico), the wood production in 1994 was only about 2.8 $x10^9$ m^3 stock; in 2003 the annual harvest was approximately 6.9 $x10^6$ m^3 (SEMARNAT 2007b). The wood industry in Mexico is not highly developed. In 2003 the production volumes of major wood products were as follows: saw wood (4.5 $x10^6$ m^3), cellulose (0.8 $x10^6$ m^3), veneer and plywood (0.4 $X10^6$ m^3), and firewood and charcoal (0.7 $x10^6$ m^3) (SEMARNAT 2007b). There are approximately 1230 sawmills; most of them are small with a daily production of less than 94 m^3 and an effectiveness of 60%. The veneer industry in 2000 consisted of 48 veneer and plywood factories and 17 particle board factories. In 2000 there were seven pulp factories with an annual production of $2x10^6$ and 57 paper factories (Torres-Rojo 2004)

For many years the wood industry has faced numerous problems. Those most worthy of note include:

- High cost for transport from the cut areas to the processing plants over an average distance of 200 to 250 km with bad road conditions.
- Use of old machinery and under-trained workers.
- Limited usage of the various species due to a lack of knowledge with respect to the physical and mechanical wood properties of new or alternative species (Torres-Rojo 2004).

All of these factors have a large impact on the market.

1.3 Importance of Prosopis species

1.3.1 Prosopis worldwide

The *Prosopis* genus comprises about 44 species of trees and shrubs; the number could be as high as 77 since similar species are now included in other genera like *Acacia* (Burkart 1976; USDA 2007). It occurs naturally in arid and semi-arid areas where it has been used by local populations as a good source of timber, fuel and fodder. The taxonomy is very complex (Burkart 1976); the species have been divided into five sections, distributed in North America, Central/South America, Africa, and Asia (Pasiecznic *et al.* 2001). The species from the *Prosopis* section are native to Asia and North Africa; the *Anonychium* section is composed of a single species *P. africana*, which is found on arid lands of North Africa. The species from the *Strombocarpa*, *Monilicarpa* and *Algarobia* sections are indigenous to Central and South America where the largest *Prosopis* forests are also found (Lopez *et al.* 2006).

Tropical Africa could be where *Prosopis* originated. As all species are closely related to *Adenanthera* L. and *Pseudoprosopis* Harms, all species may have evolved from these two genera (Burkart 1976). The name *Prosopis* comes from the ancient Greek word "*Prosopis*", which means "bark used for tanning sheep skins" (Rodríguez &

Maldonado 1996). This particular type of tree is known as “mesquite“ in Mexico. The word “mizquitl” comes from the native language Náhuatl and also means “bark for tanning” (Pennington & Sarukhan 1968; Rodríguez & Maldonado 1996).

The importance of *Prosopis* trees have been confirmed in many ecosystems around the world. These species have the capacity to positively influence soils, thus improving the environmental conditions for themselves as well for other plants and animal species. And, even under the poorest conditions, they are still able to produce multiple products. For that reason they have been grown on plantations in a number of habitats. Even though there are no exact records about the distribution of *Prosopis*, the common belief is that the first travellers across America used the sweet pods during their journeys. They could have also been spread indirectly by domestic animals consuming the sweet pods. In the last 200 years the *Prosopis* species have been introduced or reintroduced in certain areas of Argentina, Chile, Peru, Mexico and the USA (Pasiecznic *et al.* 2001), as well as in some regions of Asia, Africa, India and Australia.

There are contradicting opinions regarding the use of some species in reforestation programs. As a result of their fast colonizing behaviour, they have been considered as problematic trees. In fact, some users consider these tree species to be amongst the worst invasive weeds. *Prosopis* have already infested areas of Africa, Australia, Brazil, and Hawaii, where large amounts of money have been spent on eradication by mechanical, chemical or biocontrol means (Richardson 1998; Hughes 2001). In the USA an eradication program lasting more than 50 years has been employed to remove *Prosopis* from grasslands; however, neither herbicides nor mechanical means have proved successful. After a period of time the *Prosopis* has always returned (Pasiecznik 2002).

1.3.2 Prosopis in Mexico

Throughout history the Mexican people have associated themselves with the *Prosopis*. Several eras of Mexican history are in fact related to forest uses. The pre-Hispanic era before 1500 A.D. was characterized by rational use as a result of low demand and religious beliefs. During this period the forest was only used to supply the most important necessities. In contrast, the era of Spanish Conquest was characterized by an increase in the use of forest products due to mining activities. When Mexico became independent at the beginning of the 19th Century, forest resources were used without any regard to any technical criteria. Finally, prior to the revolution at the onset of the 20th century, the forest area was reduced drastically as the people felt they had infinite resources (Rodríguez & Maldonado 1996). Today, the *Prosopis* species are managed under forest programs which rationally determine the volume of the wood to be harvested.

Prosopis vegetation covers almost 3 million hectares from sea level to 2,200 m, corresponding to 1.51% of Mexico's area (Palacio-Prieto *et al.* 2000). 9 *Prosopis* species grow naturally forming the complex named North American or "Mexico-Texano". These species are *P. palmeri, P. reptans, P. pubescens, P. articulate, P. tamaulipana, P. vetulina, P. juliflora, P. laevigata, P. glandulosa* var. glandulosa and var. torreyana (INE 1994). *P. laevigata* is especially prominent in some localities of Guerrero, Queretaro, Estado de Mexico, Michoacan, Morelos, Oaxaca, Puebla, San Luis Potosi, Veracruz, Nuevo Leon, Aguascalientes, Durango, Guanajuato, Hidalgo, Jalisco and Zacatecas, Mexico (INE 1994).

Although various factors, such as cattle management, excess harvesting and general agriculture, have reduced tree numbers, the *Prosopis* species still play a very important role in the economy and the environment. The most recognised uses of *Prosopis* are illustrated in Fig.1.

Wood products

Firewood
Charcoal
Fence posts
Tool handles
Sawn timber
Furniture
Flooring
Craft items

Non-wood Products

Flour
Pod syrups
Coffee substitute
Seed gum
Animal feed
Honey
Wax
Exudate gum

Fig. 1: Uses of *Prosopis* species. Based on Pasiecznik *et al.* (2004)

Natural *Prosopis* stands have been use as fodder for domestic animals, e.g., cows and goats. In 1965 approximately 40,000 mt (metric tons) of *Prosopis* pods were used to feed cattle, sheep, goats, horses, donkeys and mules (Felker 1981a).

It is also possible to produce flour for human consumption and due to its sugar content even an alcoholic brew. Some fairly recent studies have found that *Prosopis* seeds are comparable to soybeans (Waggle *et al.* 1989). *Prosopis* flour absorbs 185% of its weight in water, which is quite similar to the results obtained for *Phaseolus sp.* (Barba de la Rosa *et al.* 2006).

As *Prosopis* trees produce an abundance of blossoms, they play an important role in quality honey production (Pasiecznik *et al.* 2004). Gums are also produced in large amounts from wounds to the bark; the gum quality has been compared to commercial arabic gum (from *Acacia senegal*), which is mainly used as an emulsion stabilizer, colloid protector and flavour encapsulating agent in the food, cosmetic, pharmaceutical and petrochemical industries (Beristain *et al.* 1996).

The leaves of some native *Prosopis* species which grow in India are rarely browsed by livestock; this is seen as an advantage during its initial establishment. Some

African and a few American species are valued as leaf fodder. Sometimes the leaves are gathered and used as a mulch or compost on cultivated fields; they display some noteworthy fungicidal and insecticidal qualities. The bark is a source of tannins, dyes and fibers. Various plant parts are used in the preparation of medicines, mostly for eye, skin and stomach ailments (Felker 1979; Galindo & García 1986; Gérardin *et al.* 2004).

Because of its high density, *Prosopis* has been widely recognized as a source of wood. Its wood has been used for agricultural tool handles, the hubs for cart wheels, poles for mining, in house construction, for fence posts, door and window frames, furniture, parquet flooring, fire wood, and charcoal. Without a doubt, these last two products are the ones most often utilized. This results from the fact that these trees have a small growth pattern, thus producing lumber of small dimension. In addition, these two products are relatively cheap to produce (Felker 1979; 1981a).

Mexico's charcoal exports increased from 2,000 to 20,000 mt from 1982 to 1992 (Meraz et al. 1998) with the United States being its main buyer. Five cubic metres of wood are needed to produce 1 metric ton of charcoal, which means that 100,000 m^3 of wood were used in only one year. In two traditional *Prosopis* harvesting municipalities of northwest Mexico the logging of only approx 50,000 m^3 was authorized from 1990 to 1997 (León-Luz *et al.* 2005). The official statistics regarding nation-wide *Prosopis* harvesting do not reflect the actual harvest, since this wood is grouped together with other species such as *Populus sp.*, *Liquidambar sp., Fraxinus sp.* and *Juglans sp.* Records for these show an overall wood production of 135,563 m^3 in 2003 (SEMARNAT 2007a).

1.4 State of the Art

Research on the *Prosopis* species has been very broad since it is a multi-purpose tree with many ecological interactions. Since the taxonomy with regard to the number of species and subspecies is still under discussion, initial taxonomic work

concentrated on phenological characteristics, e.g. leafe size (Pasiecznik *et al.* 2004). The great amount of cross-linking has produced various hybrids, making identification more difficult. A detailed description of *Prosopis* distribution in North America divides the population into three segments: the plain-mountain area “Altiplanicie”, the depression area “Balsas Depression” and the plain area “Northwest Cost Plain”, which are separated by humid mountains (Johnston 1962; Burkart 1976).

The geographic distribution has been changing. In some areas the coverage of several species has been increased by plantation, and in others reduced by overuse. This explains why *Prosopis* ecology has become a widely studied issue. Frequent topics are the floral patterns, germination, and fruit production as well as the ecological interactions between microclimatic conditions, water relations, soil modification, and nitrogen fixation.

In forest management, research topics have included seed collecting and scarification, germination, pests and diseases, density of plantation, silviculture pruning and species selection. The wood volume of a single tree has also been determined from the diameter and height using regression tables. *Prosopis* has been traditionally managed by using range management guidelines which require the cutting of the total stand rather than the application of silvicultural techniques. Nowadays, in some northern states of Mexico *Prosopis* is managed under short cut periods of 10 years (Sanchez & Leal 2003).

1.5 Objectives of the thesis

The environmentally sound use of trees and shrubs has become a necessity, making the use of trees growing under difficult conditions that much more urgent. Semi-arid and arid lands cover 38.3 x 10^6 km^2 worldwide. For many years the research on *Prosopis* in Mexico has played an important role. CONAFOR, the National Forestry Commission of Mexico, published an assessment of research needs in the forest sector in 2002. *Prosopis* research has priority status. Until recently research topics

focused on why *Prosopis* trees were dying out in some areas of the state of Nuevo Leon, Mexico. Developing new technologies is needed for the genetic conservation, for production as well as for the use of these trees. Attention needs to be paid to using *Prosopis* wood in a rational and sustainable manner as well as to finding new worthwhile products.

In an effort to reassess the available information and to establish a basis for the better utilization of *Prosopis laevigata* wood grown in northeast Mexico, the present work begins with an in-depth anatomical description of the wood, emphasizing the differences in size and distribution of cell types compared to other *Prosopis* species. This is followed by a determination of the wood's chemical composition, including a description of the topochemical distribution of lignin and the phenolic compounds within the cell walls. The quantity and identification of wood extractives are also determined. Physical and mechanical properties, such as density, swelling and shrinkage, compression, hardness, modulus of elasticity (by use of both the static and dynamic method) are then investigated. The modulus of rupture of wood stemming from four different localities is also determined. The natural durability of the wood are tested under laboratory conditions in soil containers and includes exposure to wood decay fungi. The effect of artificial weathering on lightness and cracking in *Prosopis laevigata*, *Tectona grandis* and *Fagus sylvatica* are examined and compared. In addition, the bonding properties of two different glues under five conditions are tested. Finally, an in-depth discussion, including proposed alternatives uses, is presented.

Chapter 2

WOOD ANATOMY

Summary

Anatomical heartwood characteristics of *Prosopis laevigata* species grown naturally in northeast Mexico were determined; the histometrical evaluations were carried out by light microscopy coupled with a digitized-image analysis system. It was found that the growth ring boundaries of semi-ring-porous or diffuse-porous wood are often marked by a marginal parenchyma band. The vessels are arranged in non-specific patterns and there are differences between the average (tangential) diameter of earlywood (116 μm) and latewood (44 μm). In these samples most of the vessels were filled with an amber-coloured gum; crystals were found in both ray cells and axial parenchyma cells but no silica compounds were observed. The average fiber length was 975 μm and the thickness of a single cell wall of a fiber was 13 μm on average.

2.1 Introduction

For centuries wood has been used as a building material. Wood is composed of many 'small' cell structures. The structure is determined by the cell's type, size, shape and arrangement. Wood tissue is anisotropic and has been described as an orthotropic material, producing different material in three main directions (Schachner *et al.* 2000; Reiterer *et al.* 2002). The characteristics of the wood structures are not only different within a genus and species but within a tree itself. It is possible that specimens of wood from one tree might differ if they are obtained at different heights or distances from the pith (Forest Products Laboratory 1999; Leal *et al.* 2006). The variance in wood formation effects the wood structure, chemical composition, physical and mechanical properties, resistance to decay, and ultimately the quantity and quality of the wood products (Butterfiel 2003). The determination of the wood anatomy is the first step toward establishing possible uses of a particular wood.

Several authors, including Iqbal & Ghouse (1983), Villalba (1985), Castro (1994), Villagra & Roig (1997), Lopez *et al*. (2005) and Scholz *et al*. (2005), have described the wood anatomy of several *Prosopis* species; their results have shown great differences in wood structures within these species. The growth patterns of the species studied are compared to that of species from a relatively wet environment. Especially *P. juliflora* displays a diffuse porous structure, whereas species such as *P. caldenia* and *P. chilensis* from low rainfall zones are ring-porous and semi-ring-porous, respectively (Gomes & Muñiz 1986).

The various species of *Prosopis* grow mainly in semi-arid areas and under poor soil conditions throughout the world (Juárez-Muñoz *et al.* 2002). *Prosopis* species have been able to survive drought by developing deep roots or adapting physiologically to ensure more efficient water uptake and minimal water loss (Pasiecznic *et al.* 2001). A microscopical examination of *P. laevigata* = "Mesquite" (trade name) wood produced in the northeast area of Mexico, reveals anatomical structures, such as vessel diameter, vessel distribution patterns, size and width of ray cells. The results will help

to characterize this wood species and to relate it to the environmental factors. This will also make it possible to study the effect of the wood anatomy on the physical and mechanical properties. Finally, the results could aid in developing other uses for the wood.

The objectives in this chapter are i) to characterise the microscopic wood structure of *P. laevigata,* ii) to determine the relation of some wood structures to the environmental conditions where *P. laevigata* grows and iii) to find other uses for this wood.

2.2 Wood description

This section provides a brief general description of wood as well as wood characteristics. Wood-producing trees can occur in both angiosperms and gymnosperms. The angiosperms, or hardwoods, are the most diverse group. This group includes ring-porous species of trees such as the oak, or diffuse porous species like beech, ash and birch. The gymnosperms or softwoods consist of about 600 perennial species (Lev-Yadun & Sederoff 2000). This group includes commercial timber such as pine, fir, and spruce.

The wooden tissues of trees have been divided into various sections. The pith is in the centre of the tree and is formed by dead cells; the outer section of the pith is the heartwood section, also formed by dead cells. In most cases, synthesis and the accumulation of extractives give heartwood a darker colour and make this wood section more resistant to decay. The sapwood provides a line for water movement and storage (living cells) in the tree trunk and is the area where the young tissues are found. The cambium = “meristematic tissue” is comprised of two kinds of cells: the fusiform initials and the ray initials. The bark is the outer section of the tree which provides for the transport and storage of carbohydrates (products of assimilation) as well as offering physical protection.

The anatomical characterization of wood generally consists of three major wood sections (transversal, tangential, and radial). In Fig. 2 it is possible to see the sections of a hardwood structure. 1) The transversal section, also called cross-section, is perpendicular to the longitudinal axis of the tree; its surface exposes the concentric growth of the rings. 2) The radial section is perpendicular to the annual growth ring and displays the parenchyma rays. 3) The tangential section is the longitudinal section of wood; it is parallel to the growth rings of the tree and is perpendicular to the annual rays' growth. In this section the rays and the vessels are visible and are oriented vertically.

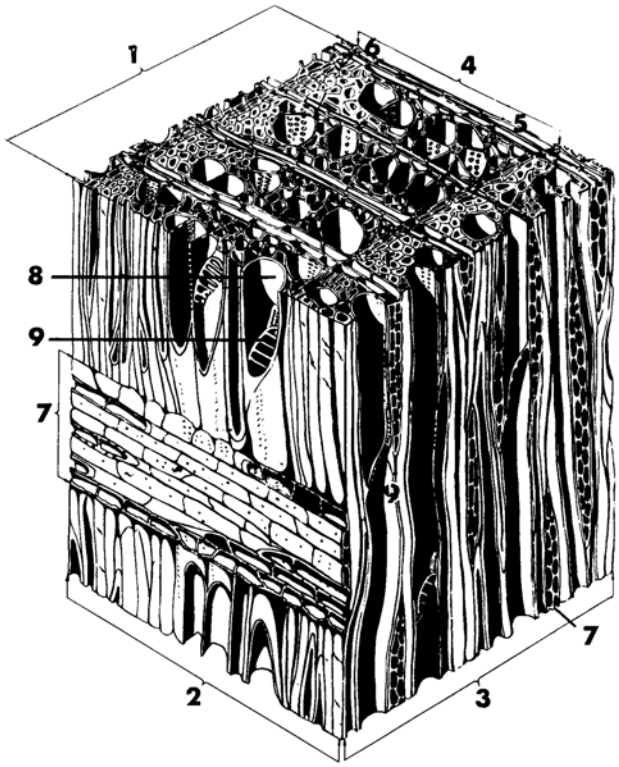

Fig. 2: Segment of hardwood tissue showing 1) transversal section, 2) radial section, 3) tangential section, 4) annual growth ring, 5) earlywood, 6) latewood, 7) wood ray, 8) vessels and 9) perforation plate. (Foulger 1969).

2.2.1 Wood structures

2.2.1.1 Vessels

Hardwoods are called porous wood because of the presence of vessels. The vessels in living trees conduct water and dissolved minerals from the roots to the leaves (Akachuku 1985; Eaton & Halle 1993). The proportion of vessels and their diameters is different within individual species and the vessels proportions have a high correlation between site indexes or area quality (Maeglin 1976). The vessels proportion and size are also a result of environmental conditions. Their fundamental function is to ensure water supply. In yellow-poplar, as in many other tree species, the vessel proportion from pith to bark varies (Taylor 1968). The wood density and, consequently, the mechanical properties are also determined by the vessels' diameter and the number of vessels per square millimetre (Leal *et al.* 2006). If the vessel proportion of a tree species is high and the vessel diameter is large, the wood produced by such trees has a lower density and, as a consequence, lower strength properties.

2.2.1.2 Rays

Further important wood elements which offer metabolic pathways for short-distance transport and storage are the xylem rays. These structures, also known as wood rays, have been described as parenchyma cells which extend radially inward from the cambium (Jane 1970). The rays are subdivided into uniseriate and multiseriate. The uniseriate are the rays which are only one cell wide; the multiseriate rays are two or more cells wide at the widest point (Carlquist 1988). The most important anatomic characteristics worthy of mention are ray width, ray length, and the kinds of cells (heterocellular or homocellular structure) which rays are formed by. Hardwood species with wide rays have 50% more proportional limit stress in radial compression (Kennedy 1968).

2.2.1.3 Fibers

Fibers are described as long and pointed elements with a simple pit. The structure and chemical composition of the fibers are responsible for most wood properties. A schematic representation of a fiber is shown in Fig. 3. The fiber is subdivided into several layers. The outer is known as the primary wall; it is thinner than the other layers. The inner layer is the secondary wall. It is thicker and is composed of three layers: the thin S_1 which is the first formed layer next to the primary wall; the thick S_2 and the inner S_3. The S_2 layer of the secondary wall contributes the most to the bulk of wall material, as well to its physical and mechanical properties. The cell lumen is the cavity close to S_3. The function of fibers is to provide mechanical support to the tree. Libriform cells and tracheids form two different kinds of fibers. The latter provide support and conduct water. Fibers are the most important material in the pulp and paper industry. This industry is the controlling force behind the demand for tree species with different attributes, including fiber length and diameter, and wall fiber thickness (Igartúa *et al.* 2000).

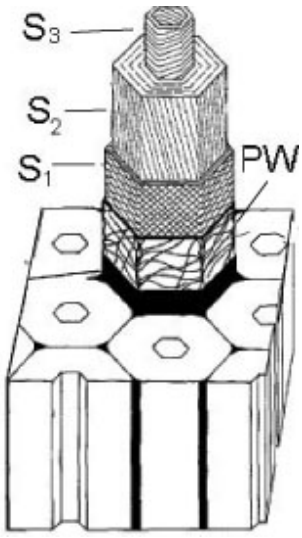

Fig. 3: Microscopic structure of a wood cell showing the primary wall (PW) secondary cell walls S_1 and S_2, and the S_3 wall. Based on imaged by Timell (1967).

2.2.1.4 Axial parenchyma

This structure generally appears as axially oriented strands or light coloured areas surrounding the vessels. It is composed of elongated cells. The axial parenchyma is normally observed in a cross-section of a piece of wood; it is formed by maturing living cells. They are derived from fusiform cambial initials. The cells that compose the axial parenchyma are usually thinner than the imperforated tracheid elements. Each individual cell is normally surrounded by a secondary wall.

2.2.1.5 Gums

Gums are mainly produced by exudation from the stem of the tree. They are solids consisting of polysaccharides and are considered a pathological response to injury to the tree, caused either by accident or by insects.

2.2.1.6 Crystals

The presence of crystals on plant tissues is common and is a distinctive characteristic in some groups of trees. The crystals are considered as “waste” products from the metabolism of plant cells (Rao & Dave 1983). Prismatic crystals, composed of calcium oxalate, are located in rays and axial parenchyma cells. Crystal-laden rays are upright and/or square and procumbent; upright and/or square ray cells are not chambered. Crystals in procumbent ray cells are not radially aligned. Crystals-laden axial parenchyma cells are chambered in various species.

The relative abundance of crystals varies. In some species crystals are consistently abundant; in others, they are consistently present, but not abundant, and in yet other species, they are present in some, but absent in other samples. The chambered crystal feature comprises a considerable diversity of chambered or subdivided cell types (cf. Parameswaran & Richter 1984). The same holds true with regard to the length of the chains of crystalliferous chambers or subdivisions. In some taxa there are only a few chambers in a series, in others there are long chains.

2.3 Material and methods

2.3.1 Origin of wood samples

The wood samples were obtained at a height of 0.3 m to 2 m from twelve *P. laevigata* trees from four areas in northeast Mexico (three per locality); the diameters of the trees were greater than 0.3 m at breast height (DBH). The location of the study area is given in Fig. 4. Further information on the trees and their origins are provided in Tab. 1.

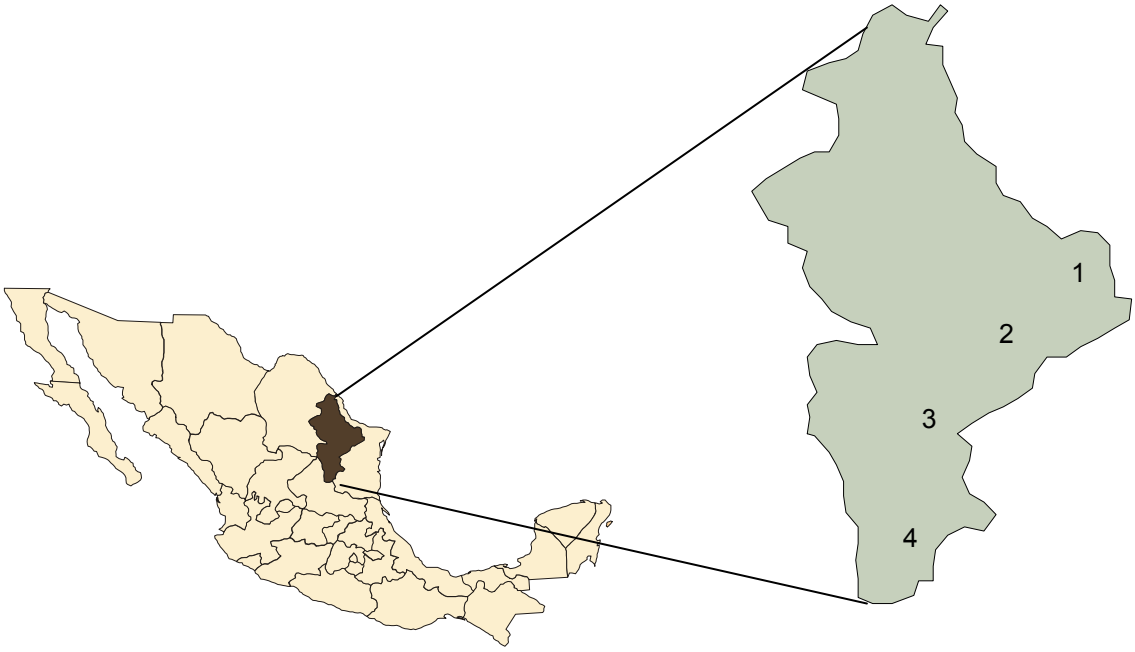

Fig. 4: Localisation of the sampling areas of *P. laevigata* wood in northeast Mexico. 1) local area Rancho Saltilleros, 2) local area Rancho San Lorenzo, 3) local area Ejido la Reforma and 4) local area Ejido Santa Gertrudis.

Tab. 1: Height and diameter of the trees at breast height, annual average temperature, precipitation, position, latitude and longitude of the areas where the trees were logged for sample elaboration.

Origin (No.)	Rancho Saltilleros 1	Rancho San Lorenzo 2	Ejido La Reforma 3	Ejido Santa Gertrudis 4
Municipality	China	General Teran	Linares	Doctor Arroyo
Latitude	25° 24'23"	25° 20'18"	24° 42'05"	23° 54'48"
Longitude	99°10'22"'	99°31'00''	99°32'05''	100°10'14''
Temperature °C *	22 - 24	22 - 24	20 - 22	16 - 20
Precipitation (mm)*	512	631	759	300 - 600
Tree height (m)	6.24	8.20	8.44	6.70
Tree DBH** (m)	0.35	0.34	0.36	0.50

* Source: INEGI (2000)
** DBH: diameter at breast high.

2.3.2 Preparation of wood samples for microscopical analysis

2.3.2.1 Wood softening and slicing

Wood samples to determine the anatomical characteristics were from trees in locality 3; the specimens were softened[1] according to the following procedure:

> Cubic-shape *P. laevigata* wood specimens were boiled in water for one hour. The samples were then sliced with a sliding microtome into 10 - 20 µm thick sections along their transversal, tangential, and radial axis. To determine the fiber size and length, the tissue was macerated with Jeffrey solution[2]. Very thin tooth-pick sharp specimens of wood were prepared and immersed in Jeffrey solution. During this process the middle lamella dissolved; neither the primary nor secondary walls were damaged.

[1] Microscopy studies were performed at the Department of Wood Biology, Hamburg University, Germany
[2] The Jeffrey solution is a mixture of nitric acid (HNO3) and chromic acid (CrO3) – 10% in water at 60°C. The time is dependent on wood density. 1 h was required for *Prosopis laevigata* wood.

2.3.2.2 Staining

For a histochemical characterisation of the wood tissue, the slices obtained with the microtome were stained according to the following procedure:

1. The sections (slices) were fixed 1 minute in 96% alcohol.
2. The slices were put in a safranin + alcohol 1% solution for 3 min.
3. The section was washed twice with ethanol.
4. The samples were immersed in astrablue and alcohol for 5 min.
5. They were once again washed in ethanol.
6. For microscopic examination three sections -radial, tangential and transversal- were embedded in euparal.

2.3.3 Laboratory equipment and tools

The microscopy investigations were carried out using two different microscopes. An Olympus AX70 was used which was equipped with a DC 300 digital imaging camera. The wood structures were histometrically evaluated with a digitised image analysis system (analySIS®, Olympus) at different magnifications (4x, 10x, 20x and 40x). The Nikon Eclipse E600 light microscope was equipped with a Dxm1200 digital imaging camera and image analysing software (LUCIA image version 4.82).

The data generated was exported to a Microsoft Excel spreadsheet. Subsequently, the data was analysed according to the International Association of Wood Anatomists (IAWA) standard list of characters for hardwoods. The basic standard statistics were evaluated including the average and standard deviation, the range and number of observations for vessel diameter, number of vessels per square millimetre, ray height, ray width, and fiber length. Other wood characteristics, e.g., the types of intervessel pits were also obtained.

2.3.4 Microscopical analysis

2.3.4.1 Diameter of the vessels and vessel per square millimetre

The diameter of the vessels and the number of vessels per square millimetre was determined after measuring 400 vessels using light microscope images of the transverse surface at 4x magnification. This level of magnification provided an appropriate resolution in the field of view. Fig. 5a shows the measurement procedure determined by the IAWA standards (IAWA Committee 1989) whereby the diameter of the vessel in the tangential direction is recorded at its widest point. The vessel walls were also measured in the same manner in 50 vessels. The number of vessels per square millimetre was determined by counting all individual vessels in a single field of view and then dividing that number by the area in mm^2. In the case of multiple vessels composed of groups of two or more vessels, each vessel was counted as an individual vessel. 50% of the incomplete vessels visualized in each image were counted as well. The data with respect to the vessel diameter and the number of vessels per square millimetre obtained using Lucia Software were first exported to Notepad, and then to Excel Microsoft. The average values, standard deviations, and frequency graphs were generated from this data.

2.3.4.2 Width and height of rays

The width of rays was determined from images on tangential surfaces as shown in Fig. 5b. The width of the ray value corresponds to the average number of cells from 150 rays, which were counted along the perpendicular axis at the widest part of the rays.

The ray height was measured at 4x magnification along the parallel axis in the tangential surface sections (Fig. 5c). The average values, standard deviations and

frequencies of ray width and height were calculated from 150 rays and summarized in figures and tables.

2.3.4.3 Fiber length

After maceration with the Jeffrey solution, fibers were put on microscopic slides and embedded with glycerine. The length of a fiber was determined by establishing four measurement points on each of the 50 fibers tested. Fig. 5d shows the data for several fibers.

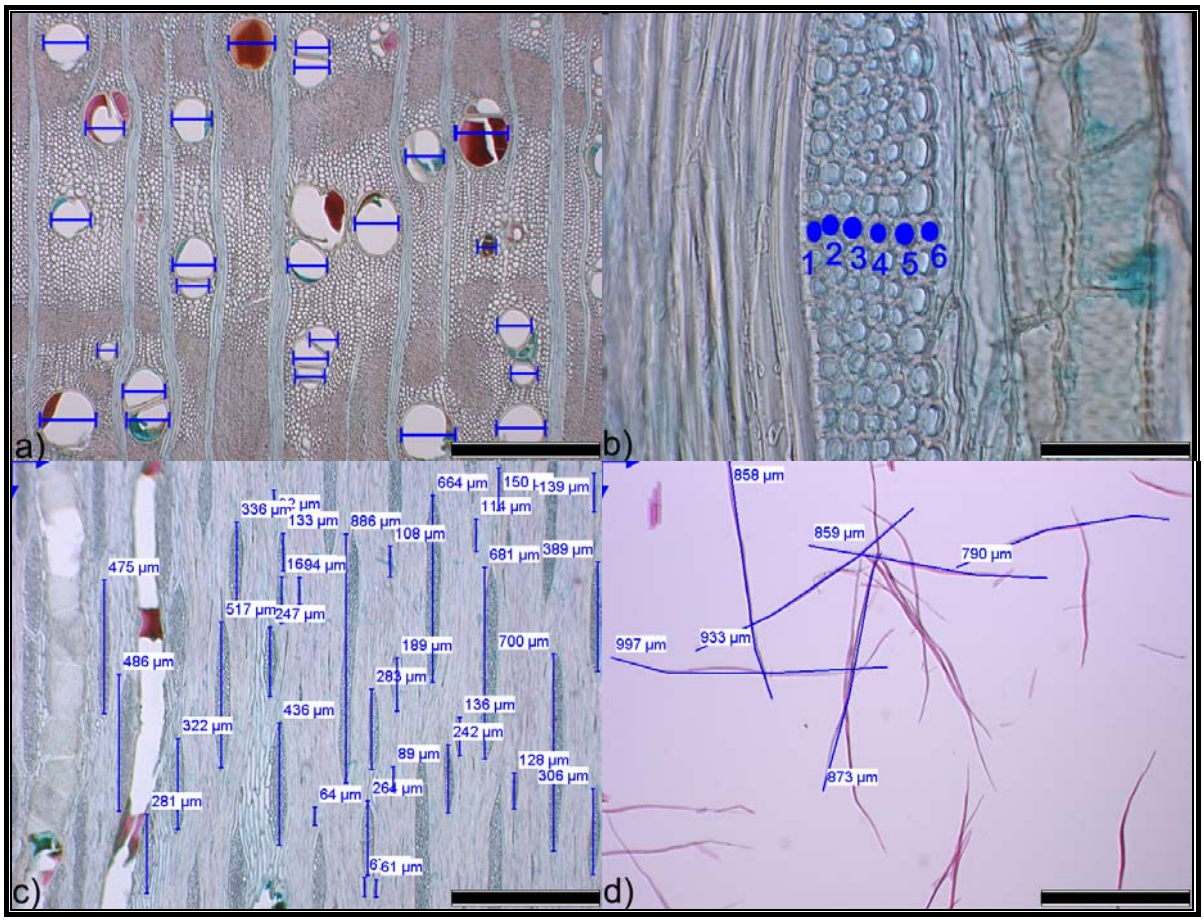

Fig. 5: Schematic representation showing the procedure used to record the data gathered through microscopic observation. a) measurement of the diameter of earlywood and latewood vessels and determination of the number of vessels per mm^2, b) determination of the width of the ray by counting the number of cells at the widest part (here width comprised of 6 cells), c) determination of ray height and d) a view field during fiber length measurement. Scale bars length: a = 500 µm, b = 50 µm, c = 500 µm and d = 500 µm.

2.3.4.4 Axial parenchyma

A characterisation of the axial parenchyma and the distribution of the earlywood and latewood were included in the wood anatomy description.

2.4 Results and discussion

2.4.1 Microscopical analysis

P. laevigata wood displays very pronounced differences between the sapwood and heartwood. The sapwood is yellowish in colour, whereas the heartwood is characterized by a light to dark brown colour containing streaks (Fig. 6). The annual growth rings of the tree are distinct and are demarcated by discontinuous marginal parenchyma bands composed of smaller cells. Fig. 7 shows the distribution of vessels with no specific pattern in vessel arrangement.

Fig. 6: Macroscopic view of *P. laevigata,* a) heartwood is brownish and sapwood (arrow) is very narrow and yellowish in colour, b) bark, c) cross section.

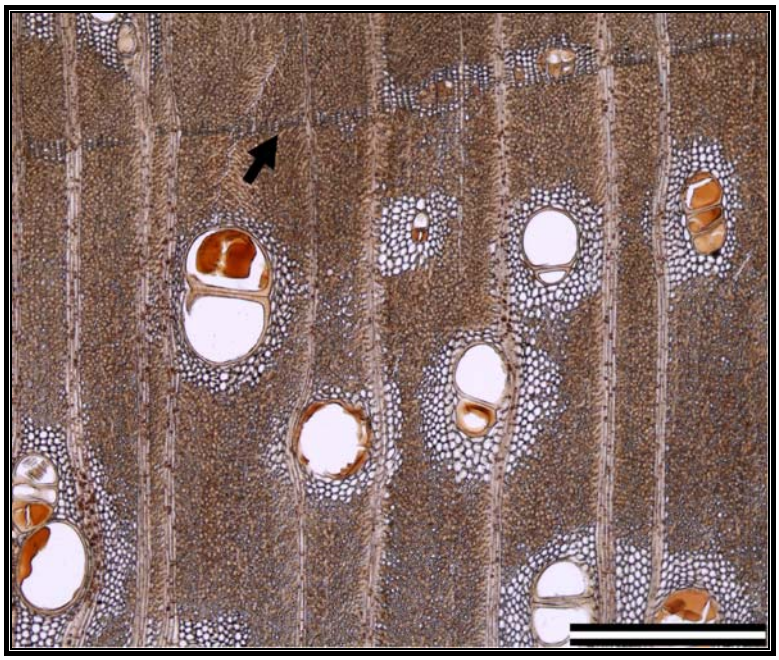

Fig. 7: Microscopic view of *P. laevigata.* The arrow shows the axial parenchyma band. Several vessels are filled with amber-coloured gum. Scale bar length = 500 µm.

2.4.1.1 Diameter of the vessels and vessels per square millimetre

The function of vessels in the living tree is to conduct water and minerals from the roots to the leaves (Akachuku 1985). The vessel diameter, vessel distribution and vessel length might have an effect on water conduction efficiency (Zimmermann 1982). The efficiency of water movement is reduced in hardwood species which show a decrease in the width of the vessels (Stamm 1972)

The environmental conditions in the areas throughout which *P. laevigata* trees are distributed are marked by a moderate to low rainfall. The tissues analyzed reveal a semi-ring-porous and diffuse-porous structure with no specific pattern in vessel arrangement. Two different patterns of porous arrangement related to rainfall have been generally described for *P.* species. *P. juliflora* trees growing in areas with a high rainfall display a diffuse porous structure while species from lower rainfall areas such as *P. caldenia* and *P. chilensis* are ring porous and semi-ring porous, respectively (Gomes & Muñiz 1986).

Most of the vessels in *P. laevigata* tissue are arranged in groups of two, three and four. As shown in Fig. 8, the diameter of the vessels varies between earlywood and latewood. At the beginning of the rainfall season the earlywood vessels in the test samples had an average diameter of 116 µm (maximum 224 µm, minimum 20 µm and standard deviation = 60). This is much wider compared to the latewood vessels whose average diameter was 44 µm (maximum 141 µm, minimum 13 µm and standard deviation = 27). The frequency of classes (histogram) of vessel diameters in earlywood is shown in Fig. 9. The diameter classes with the highest (absolute) frequency are those of 40, 100 and 140µm. In latewood the 40 µm class has the highest frequency of the fibres (Fig. 10). The average vessel diameter in earlywood and latewood of *P. laevigata* is indeed similar to other *Prosopis* species; however, the maximum diameter (224 µm) is higher than in the other species. See Tab. 2.

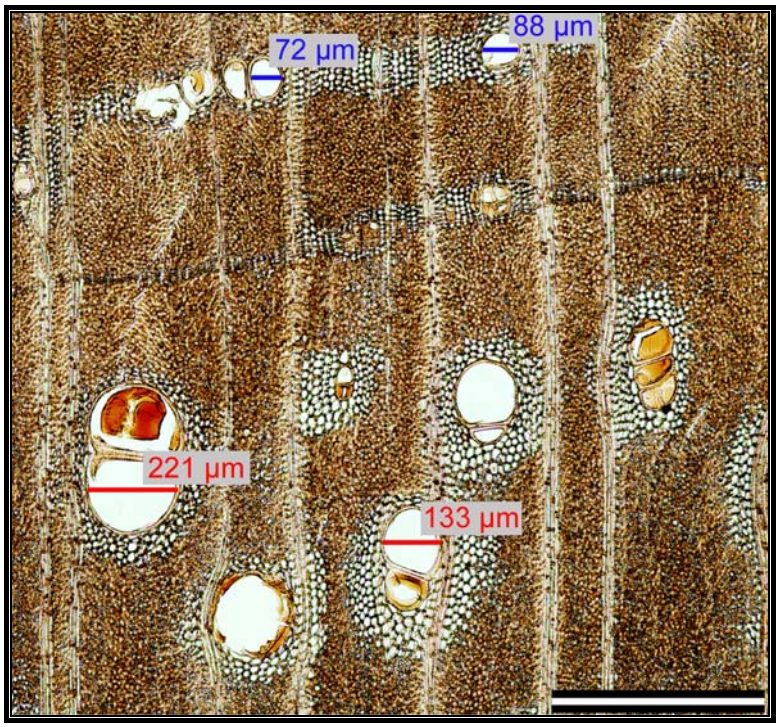

Fig. 8: *Prosopis laevigata* heartwood. The size of earlywood and latewood vessels differs. Scale bar length = 500 µm. Image by Carrillo *et al.* (2007).

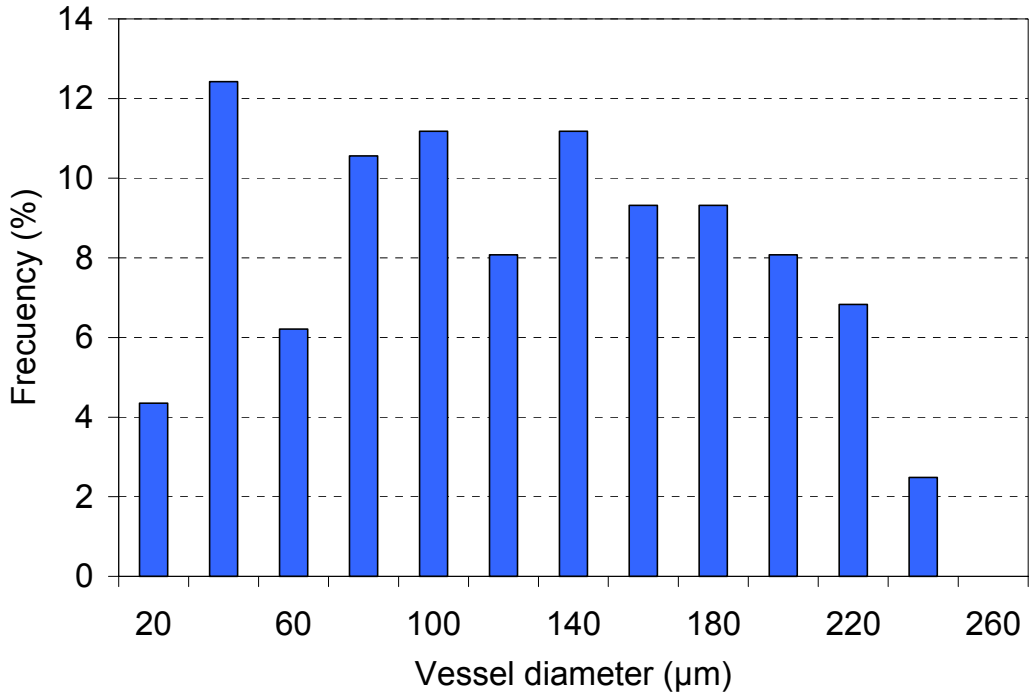

Fig. 9: Distribution of earlywood vessel diameter classes (histogram) of *P. laevigata.* The classes with highest frequency are 40, 100 and 140µm.

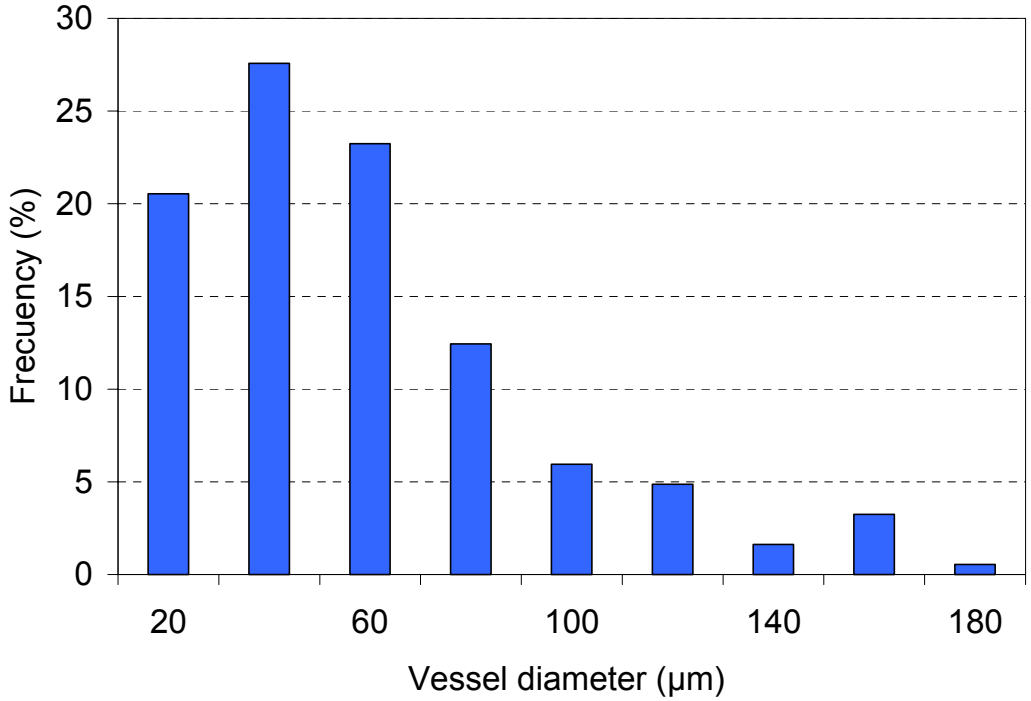

Fig. 10: Distribution of latewood vessel diameter classes (histogram) of *P. laevigata*. The class with highest frequency is 40 µm.

In dry climate zones the density of vessels is an important factor with regard to water transport in plants. *P. laevigata* exhibited an average value of 10 vessels/mm^2 (maximum 12 µm, minimum 7 µm and standard deviation = 2). Varying results have been reported for others species: *P. kunzei* 12 vessels/mm^2, *P. pallida* 5 vessels/mm^2, *P. alpataco* 52 vessels/mm^2, *P. argentina* 142 vessels/mm^2, *P. flexuosa* 30 vessels/mm^2, and *P. chilensis* 94 vessels/mm^2 and *P. strombulifera* 193 vessels/mm^2 (Castro 1994; Villagra & Roig-Juñent 1997; López *et al.* 2005; Scholz *et al.* 2005).

The average value for individual vessel wall thickness in *P. laevigata* is 3.0 µm with a range in values from 1.4 to 4.4 µm and a standard deviation of 0.9. The average vessel length is 99.4 µm (minimum 52.4, maximum 192.3 µm and standard deviation = 23.1).

2.4.1.2 Width and height of rays

The tissue formed by the rays is known as ray parenchyma. The rays are important for the horizontal transport and storage of carbohydrates and starch (food reserves); they are also involved in the biosyntheses of extractives. The profile of the rays is visible in the tangential surface; the lateral walls are visible in the radial section. Fig. 11 shows the ray profiles in tangential sections of *P. laevigata* at low magnification. The microscopic studies reveal that the rays are not aggregated. Most of the rays were classified as multiseriate with medium width since the ray width was formed by 3 to 6 cells. A lower percentage of multiseriate rays formed by more than 6 cells was detected.

With regard to the cellular composition of ray tissue, rays of *P. laevigata* are homocellular and/or heterocellular (Heterocellular rays are sporadic.). The heterocellular rays are square upright cells which are restricted to marginal rows; in most instances there is one marginal row of upright or square cells. The homocellular ray cells are procumbent. There is an absence of sheath cells as well as of tile and perforated ray cells. Disjunctive ray parenchyma end walls are also indistinct or absent.

Rays had an average width of 5 cells (maximum 6, minimum 3 and standard deviation = 1) and an average height of 283 µm (maximum 884 µm, minimum 43 µm and standard deviation = 176). The statistical distribution of the ray height classes is presented in Fig. 12. The higher frequencies are displayed by the classes of 50 and 150 µm ray height; the lowest found in the 850 µm class.

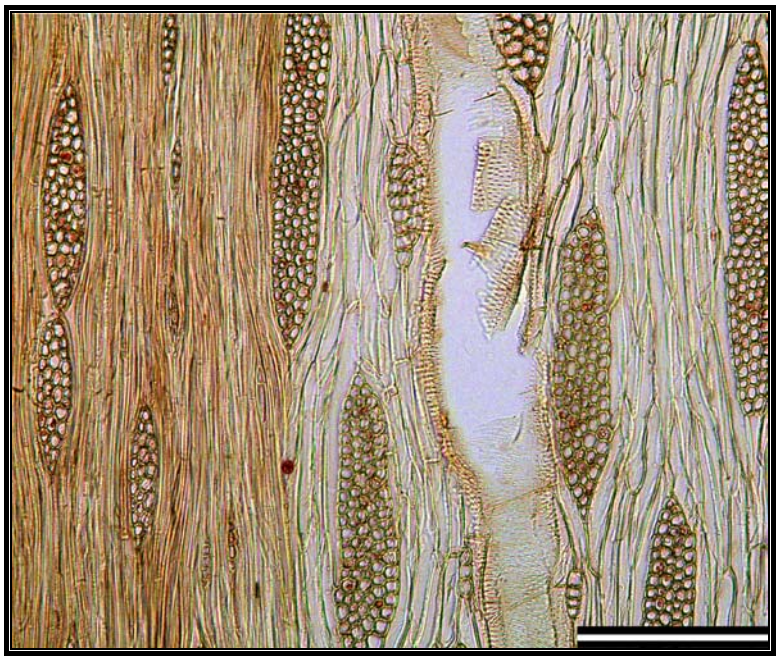

Fig. 11: Tangential view of a section of *P. laevigata* wood. The rays do not form aggregates. Scale bar length = 200 µm. Image from Carrillo *et al.* (2007).

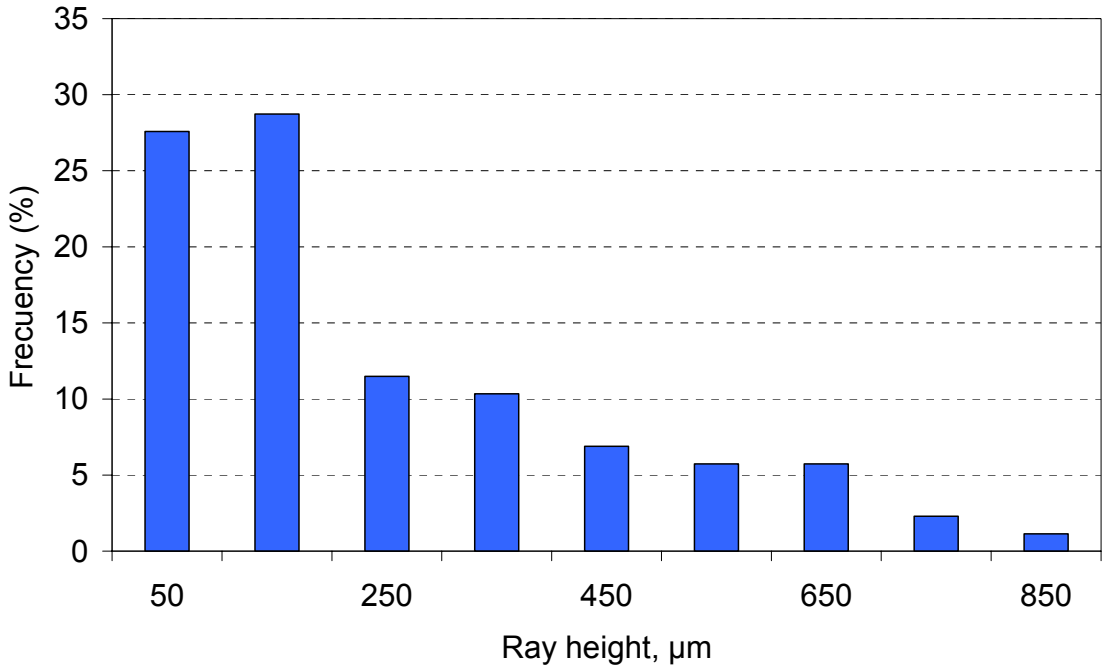

Fig. 12: Distribution of the ray height classes (histogram) of *P. laevigata*.

2.4.1.3 Fiber length

Wood-fiber characteristics of hardwood tissues, including length, diameter, and special indices such is the L/D ratio, have often been tied to wood and paper properties (Horn 1978). The results of the fiber characteristics of *P. laevigata* described in this section show that the fibers are non-septate and thick-walled. The average thickness of single wall fibers is 13 µm. The average fiber length is 975 µm (maximum: 1312 µm, minimum: 589 µm and standard deviation = 158). For other *Prosopis* species average fiber lengths vary from 532 µm for *P. argentina* (Villagra & Roig-Juñent 1997) to 1257 µm for *P. kuntze*i (Scholz *et al.* 2005). The frequency distribution classes of the parameter fiber length are shown in Fig. 13. The statistical evaluation reveals that more than 80% of the fibers are distributed in the class of 1000 µm, 1200 and 1400 µm in length.

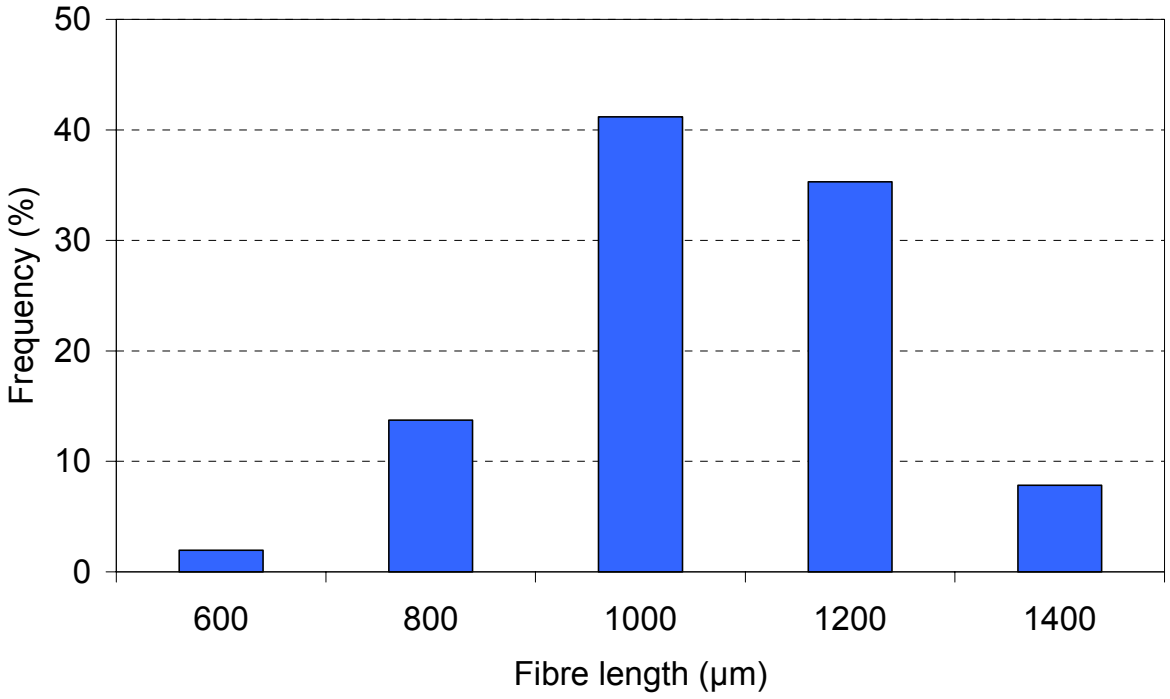

Fig. 13: Distribution of the fiber length classes of *P. laevigata*. More than 80% of the fibers measured fall into the higher classes (1000, 1200 and 1400 µm).

2.4.1.4 Axial parenchyma

The axial parenchyma of *P. laevigata* is mainly apotracheal. Paratracheal parenchyma associated with the vessels also exists. The apotracheal axial parenchyma occurs in diffusing aggregates or in longer bands. The bands are marginal or seemingly marginal. The paratracheal axial parenchyma might be vasicentric, aliform, or confluent. Aliform parenchyma displays a lozenge form. Axial parenchyma occurs as a fusiform and as strands. The average number of cells per axial parenchyma strand is 2 - 4. Unlignified axial parenchyma is absent.

2.4.1.5 Gums

Gums are formed as a result of wounds caused by mechanical injury or physiological stresses (in the living tree). Microscopic studies have revealed that numerous vessels of *P. laevigata* are filled with amber coloured gums. The black arrow in Fig. 14a points to a representative vessel of *P. laevigata* with deposits of gums; the white arrows point to the same compounds in ray parenchyma cells. The presence (deposition) of gums in *P.* species synthesised from vascular cambium serves as a means of protection for the tree from water loss and microbial attack (Greenwood & Morey 1979). Chapters 3 and 5 contain more detailed information on wood extractives and their chemical composition and their role in the resistance of *P. laevigata* to wood decay.

2.4.1.6 Crystals

Sharp prismatic calcium oxalate crystals are present in the tissue of *P. laevigata*, as shown in Fig. 14b. The crystals are located in both ray cells and axial parenchyma cells. The rays containing the crystals are upright and/or squared and procumbent. The crystals occurring in procumbent ray cells are not in radial alignment. The crystal-laden axial parenchyma cells are chambered. There is a single crystal per cell or chamber. The crystals are of normal size; cystoliths are absent. In addition, compound crystals (twins) occur in procumbent and square ray cells; the size of

these crystals and crystalliferous cells vary. Silica was not observed in *P. laevigata* tissue.

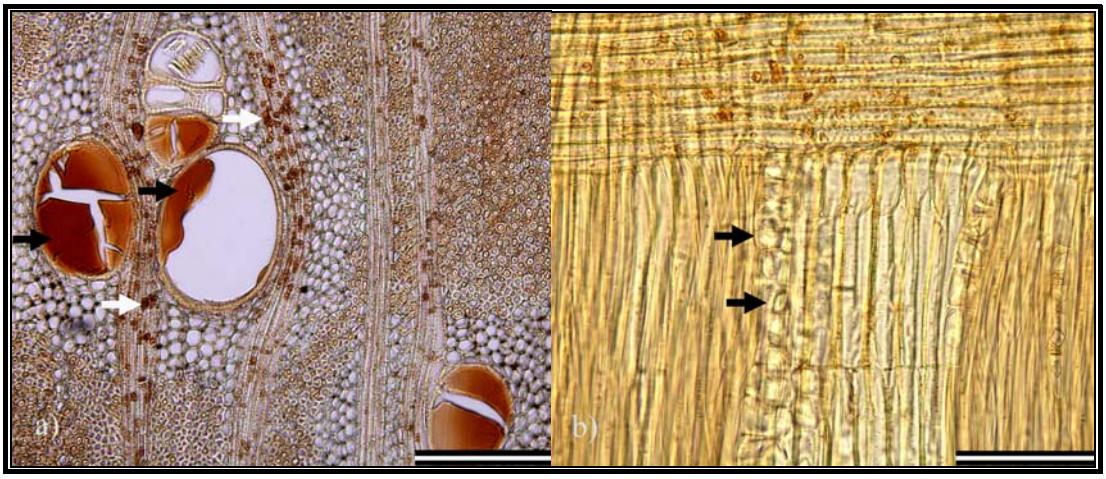

Fig. 14: *Prosopis laevigata* heartwood: a) earlywood cross section, phenolic deposits in the lumina of vessels (black arrows), in parenchyma (white arrows) and fiber cells, b) radial section with crystals in axial parenchyma cells (arrows). Scale bars length: a = 200 µm and b = 100 µm. Images from Carrillo *et al.* (2007).

2.5 Overview of the anatomic structures within Prosopis species

The histometrical data of individual wood structures (parameters) of *Prosopis* species is summarized in Tab. 2. According to this table different species of *Prosopis* reveal a great variability with respect to the size of wood anatomical structures. The publications reviewed gave very few results on the diameters of various vessels types (earlywood and latewood vessels). Villalba (1985) reports average vessels diameters of 130 µm for earlywood and 40 µm for latewood in *P. flexuosa*. In this study the difference between earlywood and latewood in *P. laevigata* are similar. Earlywood vessels have an average diameter of 116 µm and the latewood vessels an average diameter of 44 µm

The average diameter reported by other authors ranges from 40 µm for *P. argentina* (Villagra & Roig-Juñent 1997) to 140 µm for *P. pallida* (López *et al.* 2005). The

density of vessels in *P. laevigata* was determined to be 10 vessels/mm^2. Varying values have been reported: *P. kunzei* 12 vessels/mm^2, *P. pallida* 5 vessels/mm^2, *P. alpataco* 52 vessels/mm^2, *P. argentina* 142 vessels/mm^2, *P. flexuosa* 30 vessels/mm^2, and the highest value *P. strombulifera* 193 vessels/mm^2 (Iqbal & Ghouse 1983; Castro 1994; Villagra & Roig-Juñent 1997; López *et al.* 2005; Scholz *et al.* 2005)

The fibers in *P. laevigata* are non-septated and thick-walled. The average thickness of a single wall fiber is 13 µm. Average fiber length is 975 µm (maximum: 1.312 µm, minimum: 589 µm). For other *Prosopis* species average fiber lengths vary from 532 µm for *P. argentina* (Villagra & Roig-Juñent 1997) to 1.257 µm for *P. kuntze*i (Scholz *et al.* 2005).

Most of the characteristics observed in this study in *P. laevigata* are analogous to other *Prosopis* species already described by Iqbal & Ghouse (1983), Villalba (1985); Castro (1994), Villagra & Roig (1997), Richter & Dallwitz, López *et al.* (2005) and Scholz *et al.* (2005).

Tab. 2: Anatomic wood structures values in *Prosopis* species. Vessel diameter (µm), number of vessel mm^{-2}, vessel element length (µm), intervessel pit size (µm), fibre length (µm), number of rays mm, ray height (µm), ray width (number of cells), presence of crystals and tyloses.

Structure	a				b	c	d	d	e	f	f	g	h
	P. laevigata				*P. kuntzei*	*P. nigra*	*P. pallida*	*P. alpataco*	*P. argentina*	*P. flexuosa*	*P. chilensis*	*P. strombulifera*	*P. spicigera*
	average	min	max	std[1]									
Vessel diameter (µm)*	**116**	**20**	**224**	**60**	63 (11-93)		140 +/- 5	58 (10-152)	40 (8-127)	80 (20-140)	94 (27-200)	104 (10-191)	
Number of vessels mm^{-2}	**10**	**7**	**12**	**2**	12 (5-18)		5.19 +/- 2.42	52 (14-80)	142 (69-230)	30 (13-47)	94 (20-256)	193 (120-304)	
Vessel element length (µm)	**100**	**52**	**192**	**23**	200 (82-322)			72-248	76-294	140 (100-170)	172 (80-243)	136 (64-216)	116-220
Intervessel pit size (µm)	**3**	**2**	**5**	**1**		5…7							
Fiber length (µm)	**975**	**589**	**1312**	**158**	1775)			752 (404-1015)	532 (279-838)	920	100 (648-1680)	667 (391-1606)	448-1600
Number of rays mm	**8**	**6**	**10**	**1**			6.5 +/- 1.2	8.5	7.6	5	48 per mm^2	90 mm^2	
Ray height (µm)	**283**	**43**	**884**	**176**	244 (129-380)	500…1000		282 (56-856)	438 (51-1000)	300 (150-450)			
Ray width (number of cells)	**5**	**3**	**6**	**1**	4 (1-6)	3…5							
Crystals			**		$Ca_2C_2O_4$								
Tyloses			—		—	—							

Sources: a) from this research, b) Scholz *et al.* (2005), c) Richter & Dallwitz (2000), d) Lopez *et al.* (2005), e) Villagra & Roig (1997), f) Villalba (1985), g) Castro (1994) and h) Iqbal & Ghouse (1983). [1]Standard deviation, * earlywood vessels** Calcium oxalate crystals

2.6 Conclusion

Based on the results discussed in this chapter with regard to the anatomy of *P. laevigata* wood, several aspects concerning the wood structures of this important semi-arid and arid land tree are worthy of special note. The information obtained for each specific wood structure provides a sound database for comparison to other *Prosopis* species which have already been studied.

With respect to the environmental factors effecting the anatomical wood characteristics of *P. laevigata* from northeast Mexico, differences were observed among anatomic wood structures. Growth ring boundaries of the semi-ring-porous or diffuse-porous wood were often marked by a marginal parenchyma band. The differences in size and distribution of vessels between earlywood and latewood and within a specific growth year indicate that wood formation is also dependent on climatic conditions (rainy seasons), since these structures reveal distinctive anatomical features of tree species growing in semi-arid or arid conditions.

The anatomical characteristics of wood and its chemical composition have a great influence on its physical and mechanical properties. The earlywood —composed of wider vessels and thin-walled cell fibers— in semi-ring porous or diffuse porous species such as *P. laevigata* has the same area even when weather conditions result in varied ring widths. In contrast, the area of latewood —composed of narrow vessels and thick-walled cell fibers— is dependent on growth patterns (FAO 2006b). In areas where weather conditions lead to wider annual rings, wood density is increased. Species displaying long wide rays are stronger and have greater dimensional stability along the radial axis even when subjected to changes in moisture content.

The structural elements of *P. laevigata* reveal several differences to *Prosopis* species already described. The average fiber length is 975 µm and the fibers are also quite thick (13 µm). The percent of fibers of this length or longer is high (> 80%). Crystals are found in both ray cells and axial parenchyma cells as has already been reported for *P. kuntzei* and *P. juliflora*. Other results concerning the deposits of gums which fill

the vessels and concerning vessel wall and fiber wall thickness are noteworthy as they might negatively effect the glue capacity of the wood. As far as the colour of the gums is concerned, the amber, brown tones still fall within the quality parameters of the furniture industry.

Chapter 3

CHEMICAL WOOD COMPOSITION

Summary

The heartwood tissue of *Prosopis laevigata* was studied to determine the chemical composition and the topochemical distribution of lignin and phenolic compounds. The deposition of extractives in vessels, pit canals, parenchyma cells, fiber lumina, and the S_2 fiber layer was detected using scanning UV microspectrophotometry (UMSP). Borate complex anion exchange chromatography was used to determine the quantity and quality of monosaccharides. The results show that holocellulose content is between 61.7 - 64.5% and Klason lignin content between 29.8 - 31.4% in the heartwood tissue. Subsequent extractions of the soluble compounds were performed with petrolether, acetone-water and methanol-water by accelerated solvent extraction (ASE). Total extractive content in the heartwood ranges between 14.1 to 16.0% on a dry weight basis. Major compounds in acetone-water extracts were identified as (-)-epicatechin, (+)-catechin, and taxifolin and were quantified by liquid chromatography (RP-HPLC-UV).

3.1 Introduction

Prosopis laevigata is one of 44 described species of the *Prosopis* genus; it is naturally distributed in semi-arid to arid lands from central to northeast Mexico. The timber of *P. laevigata* is used for elaborate a range of products. The bark is used for tanning; the wood is used for parquet flooring and for furniture because of its decorative properties and durability (Felker 1979; 1981a; 1981b). Still, the main utilisation of *P. laevigata* is for firewood, fence posts, and charcoal. Other uses have most likely remained small scale because it is difficult to find enough large logs to produce long straight timber and because firewood and charcoal production is relatively cheap.

The physical and mechanical properties as well as the natural durability of *P. laevigata* depend on its anatomic structure and chemical composition. The percentage of cellulose, hemicelluloses, lignin, and of the extractive content in wood cells are important factors in wood properties for solid wood products (Bertaud & Holmbom 2004).

A number of studies on *P. laevigata* have been carried out on fuel wood, charcoal production, fodder, food, fruit dispersion, association between insects and fruits, forest management, timber, and soil retention (Graham 1960; Felker *et al.* 1981a; Ffolliot & Thames 1983; Cantú 1991); moreover, there has been research done investigating *P. laevigata* wood as a feasible material to produce excellent charcoal. Such charcoal has a specific weight of 0.41g/cm^3 and a caloric value of 29.7kJ/g. (Maldonado-Aguirre 2000). There have, however, been far fewer studies concerning the heartwood chemistry, the chemical composition of the extractives, and the topochemical distribution of lignin and phenolic compounds of the *Prosopis* species (Gomes & Muñiz 1986).

The objectives of this chapter are to characterize the chemical wood composition of *P. laevigata* heartwood, to describe the topochemical distribution of lignin and phenolic compounds and to identify the wood extractives.

3.2 Chemical wood composition and distribution within individual cell layers

Wood is a complex and non-uniform material; the chemical compounds of wood are distributed throughout the cells wall in varying amounts. The varied chemical composition within trees depends on local origin, age, climate, and soil conditions (Han & Rowell 1996). Cellulose, hemicelluloses and lignin are the three main constituents of wood (Timell 1967; Fengel & Wegener 1989; Willför *et al.* 2005). Lignin makes up 18 to 35% of dry wood; the two major polymeric (aliphatic) materials, cellulose and hemicelluloses, constitute 65 to 75% (Han & Rowell 1996). Extractives occur in minor amounts (4 – 10%); these include organic compounds and inorganic minerals also known as extraneous materials. On an elemental level, wood is composed of about 50% carbon, 6% hydrogen, 44% oxygen, and trace amounts of several metal ions (Fengel & Wegener 1989).

3.2.1 Carbohydrates

The most abundant compounds in nature are carbohydrates (Bemiller 1989; Sjöström 1993). Cellulose, the mayor carbohydrate component of wood, is known as the structural component of the cell wall (Fengel & Wegener 1989). It makes up to 40 - 45% of the dry weight of a wood. It is a polymer with high molecular weight and consists exclusively of β-1,4-glycosidic linked D-glucopyranose units (Fig. 15). The hemicelluloses are the second most prominent carbohydrate compounds; they are branched and have a polymer with a lower molecular weigh than cellulose. The hemicelluloses are mixtures of polysaccharides and are synthesized in wood almost

entirely from glucose, mannose, galactose, xylose, arabinose, 4-*O* methylglucuronic acid, and galacturonic acid residues. Some hardwoods contain trace amounts of rhamnose (Sjöström 1993). Fig. 16 shows hemicellulose structures.

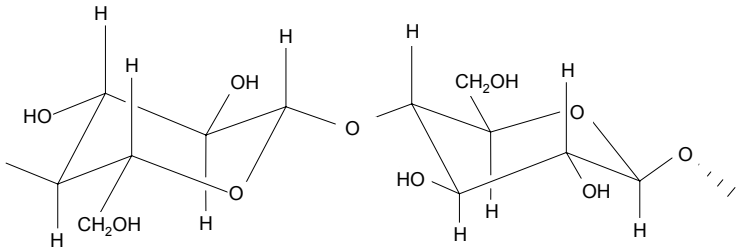

Fig. 15: Chemical structure of cellulose; two D-glucose monomers forming a "glucan". Image based on Rowell (2005).

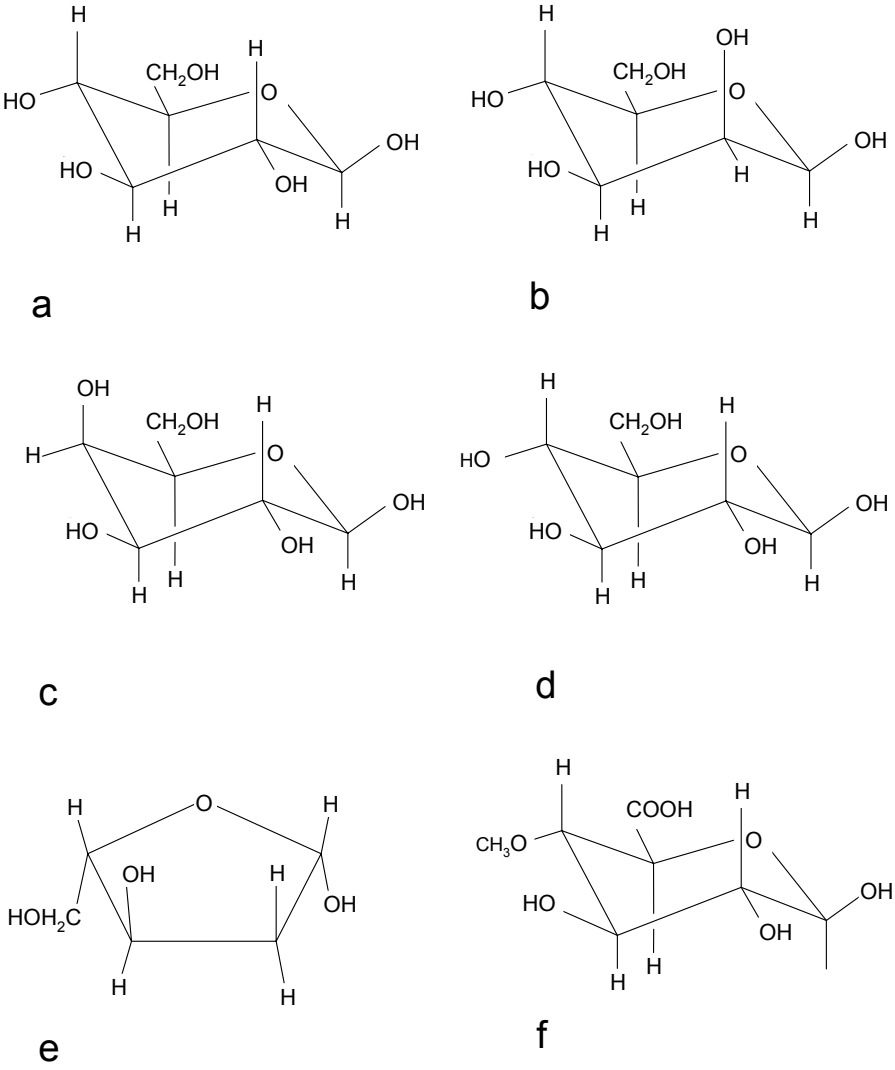

Fig. 16: Sugar monomer components of wood hemicellulose. a) ß-D-Glucose, b) ß-D-Mannose, c) ß-D-Galactose, d) ß-D-Xylose, e) α-L-Arabinose and f) 4-0-Methylgucuronic acid. Images based on Rowell (2005).

3.2.2 Lignin

Lignin is an amorphous polyphenolic polymer, consisting of an irregular array of variously bonded hydroxy- and methoxy-substituted phenylpropane units (Timell 1967). The lignin serves as a 'cementing' substance between cells. For this reason it occurs mainly in cell corners, compound middle lamella and the secondary wall S_1 (Fergus *et al.* 1969). Many properties of wood and its reactivity under chemical treatments are the result of the lignin composition and the ultrastructural distribution within cell walls (Koch 2004). Fig. 17 shows the lignin precursors.

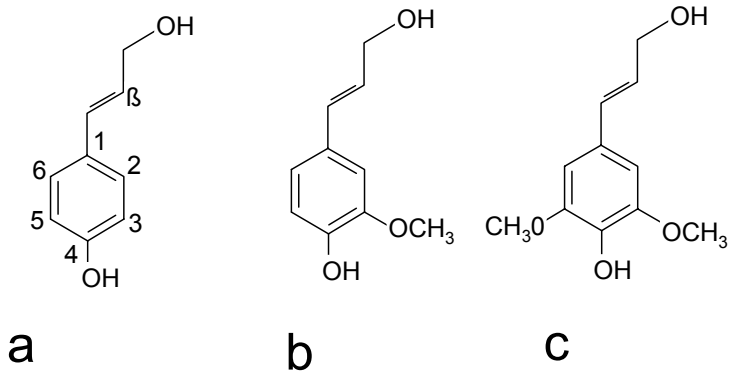

Fig. 17: Structures of the lignin precursors: a) *p*-coumarylalcohol, b) Coniferylalcohol and c) Sinapylalcohol. Images based on Rowell (2005) and Fengel & Wegener (1989).

3.2.3 Extractives

Extractives are present in varying amounts in wood. They influence a variety of properties, including colour, smell, ranges in swelling and shrinkage and decay resistance (Hillis 1968; Gutiérrez-Oliva *et al.* 2006). Extractives are organic compounds or inorganic elements and do not contribute to the cell wall structure. In the wood of species which grow in temperate climates they make up 4 - 10% of the wood composition. In tropical species this can rise to as much as 20% in normal wood. The extractives encompass a wide variety of organic compounds such as fats,

waxes, alkaloids, proteins, simple and complex phenolics, simple sugars, pectins, mucilages, gums, resins, terpenes, starches, glycosides, saponins and essential oils. Although the extractives contribute only a small proportion to the wood mass (Fengel & Wegener 1989), they are essential to trees as defence mechanisms against microbial attack. They are also important with regard to wood properties such as swelling and shrinkage and to processing wood qualities such as colour, odour and pitch factors in pulp production.

Gums are important wood extractives. They are considered metabolic end-products generated in response to injures to prevent the loss of moisture and to provide protection against infections (Hillis 1987). Gums found in the wood of several *Prosopis* species have been related to the natural resistance of these species; however, little research has been done on this topic. Gum arabic is one of the most often studied extractives; it is obtained from the *Acacia senegal* species. It has a mixture of compounds with a molecular distribution similar to that of the other plant polysaccharides, except for the fact that it has very few linear polymers. The *Prosopis* gums which are highly branched arabinogalactan acidic exudates resemble the *Acacia* gums.

3.2.4 Distribution of chemical compounds on cell layers

The chemical composition within the cell wall layers of the wooden tissue varies from one structure to the next as shown in Fig. 18. The middle lamella is mainly composed of lignin and of smaller amounts of cellulose, hemicelluloses and pectin. The primary wall has a very small amount of lignin but more cellulose and hemicelluloses than do middle lamella, yet less than the secondary wall. The percentage of lignin decreases from the S_1 layer through to the S_3 layer. The S_1 layer is composed mostly of lignin and hemicelluloses. The major component of the S_2 layer is cellulose followed by hemicelluloses and lignin. The S_3 layer contains mostly cellulose, but a larger fraction of hemicelluloses; it contains little lignin.

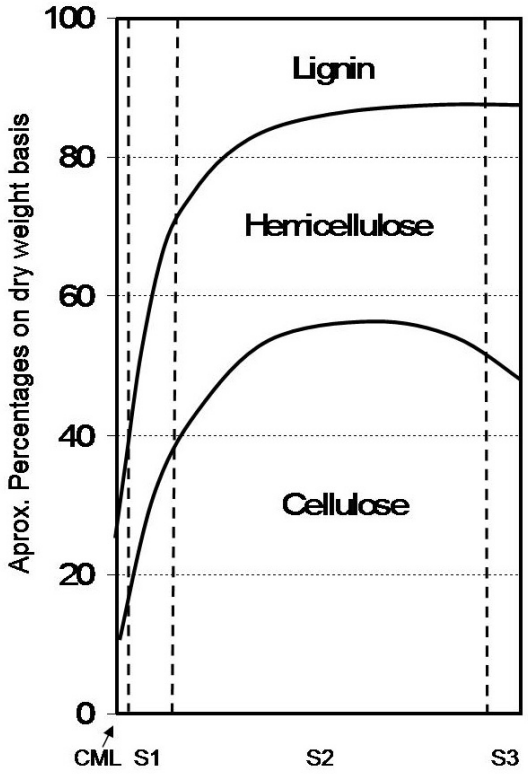

Fig. 18: The distribution of the main chemical wood compounds in the cell layers. Image based on Panshin & Zeeuw (1970).

3.3 Material and methods

3.3.1 Origin of wood samples

The wood samples for chemical and topochemical analyses were obtained at a height of 0.3 to 2 m from one *P. laevigata* tree collected from locality three. The diameter of the tree was greater than 0.3 m at breast height (DBH). The location of the study area is given in Section 2.3.1.

3.3.2 Quantitative determination of the chemical wood components

Before analysing the polysaccharide content and the lignin content of the tissue, a gradual extraction was performed to remove the soluble extractives. Wood shavings from selected *P. laevigata* heartwood tissue were used. Two sections of a tree trunk disk free of knots were prepared according to Fig. 19. Wood fractions were obtained from middle heartwood (Section A) and Section B was obtained very close to the pith in the inner heartwood. The samples were freeze dried and ground in a mill with rotating knives (Retsch) using a 3 mm screen. The gradual extraction was performed on 2 g freeze dried wood powder using an accelerated solvent extraction Dionex ASE 200 (Dionex, Sunnyvale, CA, USA): (a) solvent petrol-ether, temperature 50 °C, pressure 100 bar, heating time 5 min, static time 10 min, flush volume 100%, purge time 120 s, static cycles: 1; (b) solvent acetone-water (9:1), temperature 60 °C, pressure 100 bar, heating time 5 min, static time 10 min, flush volume 100%, purge time 120 s, static cycles: 1; (c) solvent methanol-water (3:1), temperature 60 °C, pressure 100 bar, heating time 5 min, static time 10 min, flush volume 100%, purge time 120 s, static cycles: 1.

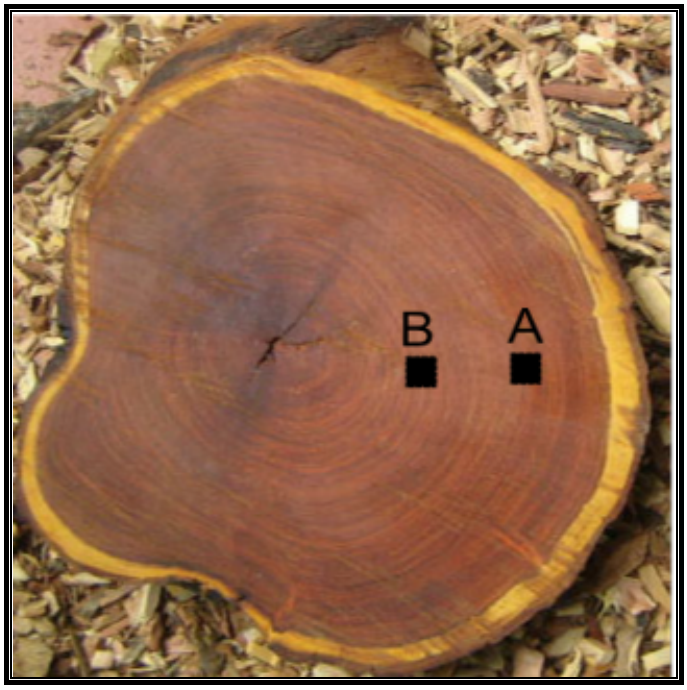

Fig. 19: Schematic representation of areas from which samples were taken for chemical analysis of *P. laevigata* wood.

After hydrolysation with 72% sulphuric acid, the Klason lignin content was determined for an acid-insoluble residue. Monosaccharides were qualitatively and quantitatively determined by borate complex anion exchange chromatography (Puls 1993). The tests were performed in triplicate.

3.3.3 Topochemical distribution of lignin and phenolic extractives in wood tissues

Cellular UV microspectrophotometry (UMSP) has been established as useful technique for the topochemical detection (analysis) of lignin and phenolic extractives within individual cell wall layers (Koch 2001).
The lignin absorption spectrum shows three weak asymmetrical bands. They are the resulting absorbance of five types of chromophore systems based on *p*-hydroxyphenylpropane groups (Lozovik & Kaflyuk 2005). The spectrum presented between 270 - 280 nm is the second band and depends on the delocalisation of the π-electron in aromatic ring structures (Koch & Kleist 2001).

An improved UV microspectrophotometry-scanning-device developed by Zeiss (Oberkochen, Germany) was used to determine, in more detail (resolution 0.25 μm^2), the distribution of lignin and aromatic phenolic compounds within cell walls in *P. laevigata* wood. This technique is based on the differential UV absorbance of lignin and phenolic compounds.

For the topochemical analyses, transverse sections of 1 µm thickness from selected heartwood samples were prepared using a diamond knife. The sections were transferred to quartz microscope slides, immersed in a drop of non-UV absorbing glycerine and covered with a quartz cover slip. The analyses were carried out with a ZEISS UMSP 80 micro spectrophotometer equipped with a scanning stage, which makes it possible to image profiles at constant wavelengths with the (ZEISS) scan software APAMOS® (Automatic-Photometric-Analysis of Microscopic Objects by Scanning, Zeiss). This scan programme digitises rectangular fields on the tissue with

a local geometrical resolution of 0.25 μm^2 and a photometrical resolution of 4096 grey scale levels. These are then converted into 14 basic colours making absorbance intensities visible.

3.3.4 Quantitative determination of the extractive content

The amount of extractives content can be determined in three ways: gravimetric determination of total extractives, determination of different compound groups and analysis of individual compounds (Sjöström & Alén 1998). The extractive content of *P. laevigata* was determined gravimetrically; the solvent was subsequently removed from the extracts with a rotary evaporator under vacuum.

3.3.5 Reversed-Phase High Performance Liquid Chromatography (RP-HPLC)

Reversed phase HPLC (RP-HPLC) consists of a non-polar stationary phase and a moderately polar mobile phase. The retention time is longer for molecules which are more non-polar in nature, allowing polar molecules to elute more readily. Retention time is increased by the addition of polar solvent to the mobile phase and decreased by the addition of additional hydrophobic solvent.
RP-HPLC functions on the principle of hydrophobic interactions which result from repulsive forces between a relatively polar solvent, between the relatively non-polar analyte, and between the non-polar stationary phase. The driving force in the binding of the analyte to the stationary phase is the decrease in the area of the non-polar segment of the analyte molecule which is exposed to the solvent. This hydrophobic effect is dominated by a decrease in free energy through entropy associated with the minimization of the ordered molecule-polar solvent interface. The hydrophobic effect is decreased by adding more non-polar solvent to the mobile phase. This shifts the partition coefficient so that the analyte spends a certain portion of time moving down the column in the mobile phase, eventually eluting from the column.

To identify the chemical composition of *P. laevigata* extractives, 5 ml of acetone-water extract from inner heartwood fractions were directly injected without derivatisation into a *Jasco* system (Japan Spectroscopic Company). An Aquasil 5 µ C18 column (250 x 4.6 mm) was used. The temperature of the column was set at 30°C. Solvent A (0.001 M H_3PO_4) and Solvent B (Acetonitrile 100%) served as the mobile phase in a gradient mode described in Tab. 3.

Tab. 3: Operation condition during the Reversed-Phase High Performance Liquid Chromatography.

Time (min)	Solvent A (%, v/v)	Solvent B (%, v/v)	Flow rate (ml/min)	Wave length (nm)
0.0	92.5	7.5	1	280
30.0	85.0	15.0	1	280
40.0	80.0	20.0	1	280
60.0	60.0	40.0	1	280
65.0	0.0	100.0	1	280

The separate compounds were analysed with a photo-diode array detector (Jasco). The detection wavelength was set at 280 nm; UV spectra from 200 to 650 nm were also recorded for peak identification. Peak identification was performed by comparison of retention times and UV spectra with purchased standards (Sigma Aldrich Co.). For quantification, calibration curves with four calibration points for each substance were set. Quantification was performed in triplicate.

3.4 Results and discussion

3.4.1 Chemical composition of Prosopis laevigata wood

The monosaccharide composition of *P. laevigata* is given in Tab. 4. As is the case with regard to the hemicellulose composition in other hardwoods, a high value for xylose (about 12%) content and a low value for mannose (0.2%) content were

determined in *P. laevigata*. The glucose level, mainly derived from cellulose, increases from 45.7% (outer heartwood) to 48.6% (inner heartwood). Klason lignin content ranges from 29.8% (inner heartwood) to 31.4% (outer heartwood). The total carbohydrate content is higher in inner heartwood (64.5%) than in outer heartwood (61.7%). For comparative purposes, the chemical composition of *P. juliflora* determined by Patel & Safaya (1986) was as follows: 25 to 30% hemicellulose, 40 – 50% cellulose and 11 – 28% lignin. An independent study by Rajput & Terari (1986) found the levels to be 54% cellulose and 31% lignin.

Tab. 4: Monosaccharide and lignin content of *P. laevigata* heartwood (the values are based on percentage of oven dry weight). Table based on Carrillo *et al.* (2007)

Chemical compound	Unit	Section	
		Outer heartwood	Inner heartwood
Xylose	%	12	12.4
Arabinose	%	0.9	0.6
4-O-methyl-glucuronic acid	%	0.9	0.9
Rhamnose	%	0.3	0.3
Galactose	%	1.7	1.5
Mannose	%	0.2	0.2
Glucose	%	45.7	48.6
Σ Carbohydrates	%	61.7	64.5
Klason-Lignin	%	31.4	29.8

3.4.2 Distribution of lignin and phenolic extractives in wood tissues

The topochemical distribution and semi-quantitative determination of lignin and phenolic extractives were ascertained through scanning UV microspectrophotometry. The applicability of this technique for the topochemical detection of lignin within the individual cell wall layers of several hardwoods and phenolic deposits has been demonstrated by various authors (Koch & Kleist 2001; Koch & Grünwald 2004).

Fig. 20 shows representative UV scanning profiles of heartwood tissue of *P. laevigata* at a defined wavelength of 278 nm. Secondary walls (S_2) of fibers offer a non-uniform UV absorbance within a range of 0.20 to 0.65 AU (absorption units). Compound middle lamella (CML) is characterized by UV absorbance within a range of 0.45 - 0.65 AU, whereas absorbance above 1.0 AU is detectable in cell corners. The different absorbance values correlate strongly with various levels of lignin in the individual cell wall layers.

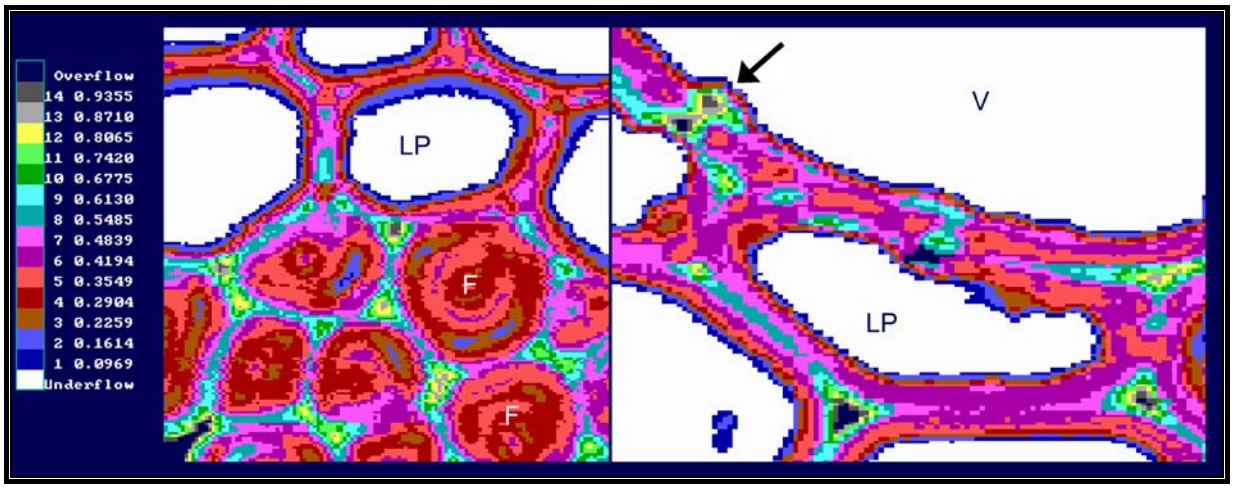

Fig. 20: UV microscopic scanning profiles of heartwood tissue of *P. laevigata*. The colour pixels represent different UV absorption values of the cell wall layers and phenolic extractives measured at 278 nm. The arrow marks extractives deposited within a pit canal. LP = axial parenchyma cell, F = fiber, V = vessel (Scanning field: left 37.0 µm x 38.5 µm, right 26.75 µm x 36.75 with a geometrical resolution of 0.25 µm x 0.25 µm). Image from Carrillo *et al.* (2007).

The deposition of extractives in the fiber lumen is also made evident by applying the UV scanning technique. The deposits are characterised by high absorbance values within the range of up to 0.85 AU, which is significantly higher than in adjacent cell wall layers (Fig. 21). In addition, UV line scans across fiber cell walls with non-uniform absorbance were carried out on representative S_2 fiber layers. The scan presented in Fig. 21 shows the fiber cell wall viewed horizontally across the cell. Evaluation of this scan reveals a high local UV absorbance within the S_2 layer, which is visible as a clear peak with a numerical value of 0.64 AU. This peak demonstrates the local impregnation of S_2 with phenolic extractives. Within the cell wall of vessels, local spots of high absorbance (0.8 - 1.0-overflow) can also be detected (Fig. 20, arrow). These areas are localised in the region of pit canals and pit membranes. Similar findings were achieved earlier by Koch *et al.* (2006) for merbau (*Intsia* spp.)

and afzelia (*Afzelia* spp.) and verified as an impregnation of the pit membranes and canals by phenolic extractives synthesised by pit membrane-associated enzymes.

Fig. 21 displays a UV scanning line across the cell wall with the extractive, illustrating deposits; their presence is visible in the S_2 cell wall layer of fibers of *P. laevigata* heartwood. Fig. 22 shows the UV microspectrophotometrical analysis of lignin and phenolic deposits distribution in an axial parenchyma cell.

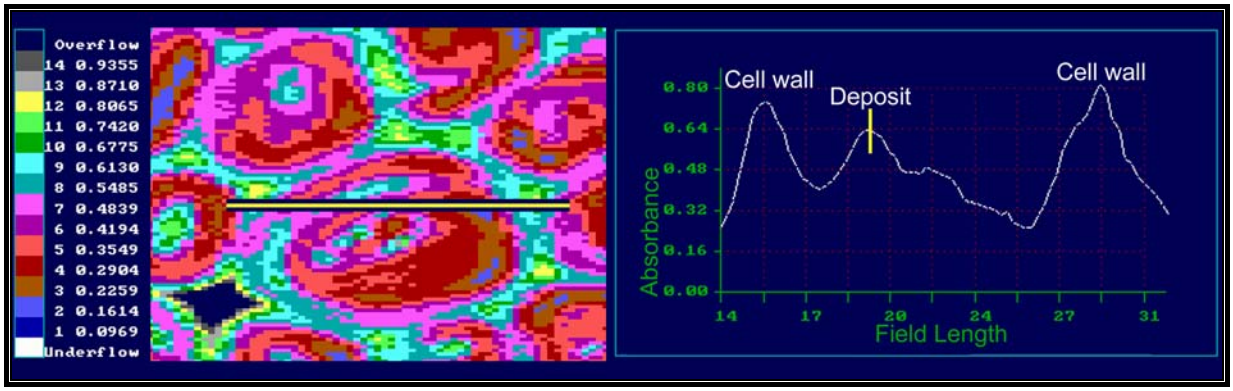

Fig. 21: UV microspectrophotometrical analyses of lignin and phenolic deposit distribution in fibers from a cross section (1 µm thickness) of *P. laevigata* heartwood. Left: two-dimensional UV scan (280 nm); right: line scan (280 nm) across the fiber wall and the deposits in the S_2 layer (scanning field: 15.25 µm x 20.75 µm with a geometrical resolution of 0.25 µm x 0.25 µm). Image from Carrillo *et al.* (2007).

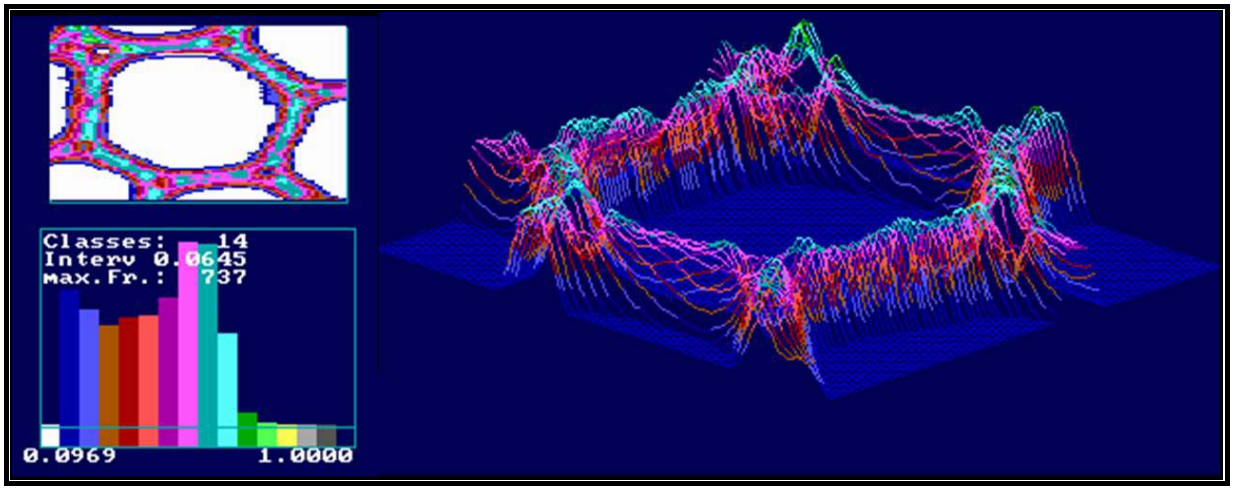

Fig. 22: UV microspectrophotometrical analyses of lignin and phenolic deposits distribution in an axial parenchyma cell from *Prosopis laevigata* heartwood. Left: histogram of frequency of absorbance; right: absorbance of parenchyma cell; the high picks are the corner and secondary middle lamella (scanning field: 15.25 µm x 20.75 µm with a geometrical resolution of 0.25 µm x 0.25 µm).

The lignification of individual cell wall layers can also be studied by evaluating the UV absorption spectra within a wavelength range of 240 to 400 nm (Koch 2001). In Fig. 23 typical UV absorption spectra of individual cell wall layers and phenolic extractives in *P. laevigata* heartwood are presented. The UV spectra of the cell corner and compound middle lamella show typical absorbance behaviour of a hardwood lignin with a distinct maximum at 278 nm and a local minimum at about 250 nm (Fergus *et al.* 1969). The cell corners of the fibers are generally characterised by higher absorbance values (0.75 AU) as compared to the compound middle lamella (0.48 AU). The phenolic extractives detected in the fiber wall display a bathochromic shift to a wavelength of 284 nm and a slight shoulder at a wavelength range of 320 nm. This spectral behaviour can be explained by the presence of chromophoric groups, e.g., conjugated double bonds (Hon *et al.* 1986; Feist & Hon 1990).

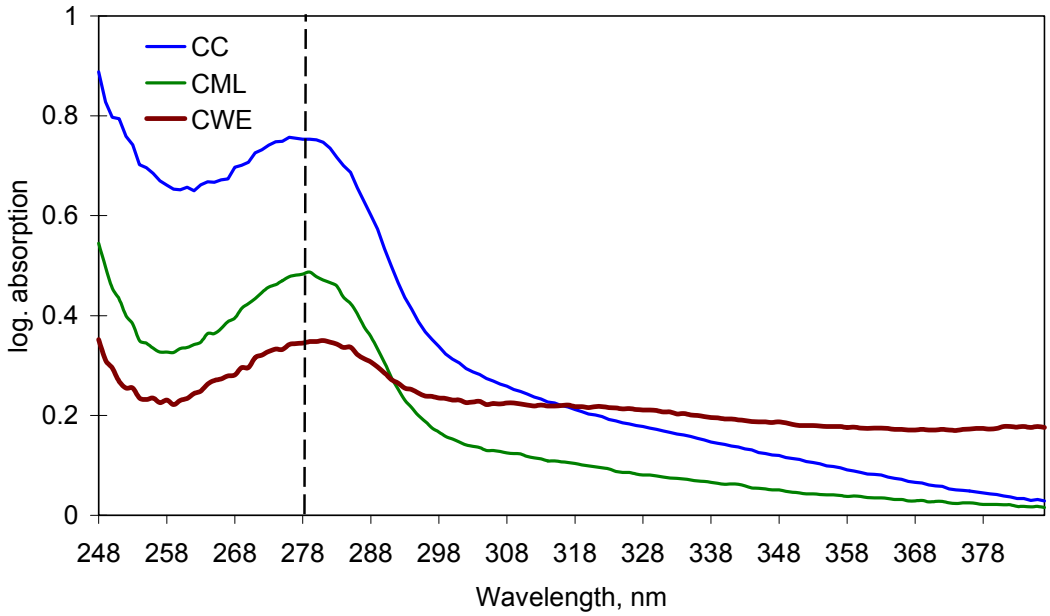

Fig. 23: Absorption spectra in different sections of cell wall layers from *P. laevigata* wood. CC: cell corner; CML: compound middle lamella; CWE: condense wood extractives.

3.4.3 Quantitative determination of extractive content

Fig. 24 displays the different extracts of *P. laevigata*. The content of organic accessory compounds (extracted gradually with petrolether, acetone-water and methanol-water) is rather high in *P. laevigata* heartwood: 14.1% in the inner heartwood and 16.0% in the outer heartwood in relation to oven dry weight (Tab. 5). The amount of extractives determined in the acetone-water fractions was 11.6% and 12.8%, respectively. Similar amounts of extractive content were determined in the acetone extracts of *P. africana* heartwood (Gérardin *et al.* 2004), namely 11.7%.

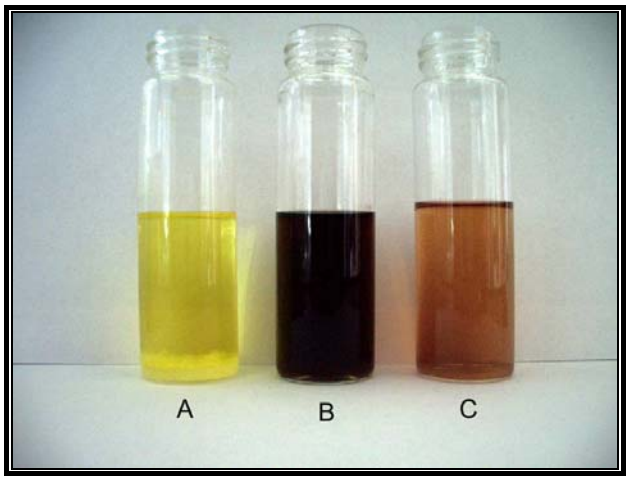

Fig. 24: Extracts of *P. laevigata* heartwood. A: Petrol-ether extract; B: Acteone-water (9:1); C: Methanol-water (3:1)

Tab. 5: Extractive content of *P. laevigata* heartwood (values are given in percentage on a dry weight basis of the original wood sample). Table from Carrillo *et al.* (2007)

Section	Petrolether	Acetone-water (9:1)	Methanol-water (3:1)	Σ Extractives
Inner heartwood	0.3	11.6	2.2	14.1
Outer heartwood	0.4	12.8	2.8	16.0

3.4.4 Characterisation of soluble phenolic compounds

Few major compounds were detected in acetone-water-extracts of *P. laevigata* heartwood. Three flavonoid compounds were identified as flavan-3-ols (+)-catechin (retention time 14.7 min), (-)-epicatechin (retention time 16.26 min), and the flavonol

taxifolin (retention time 35.5 min) (Fig. 25). The content of the identified compounds in heartwood of *P. laevigata*, calculated from their concentration in the acetone-water extracts, were established as follows: (-)-epicatechin: 5.33%, (+)-catechin: 0.51%, taxifolin: 0.05% on a dry weight basis. Fig. 26 shows the compounds identified.

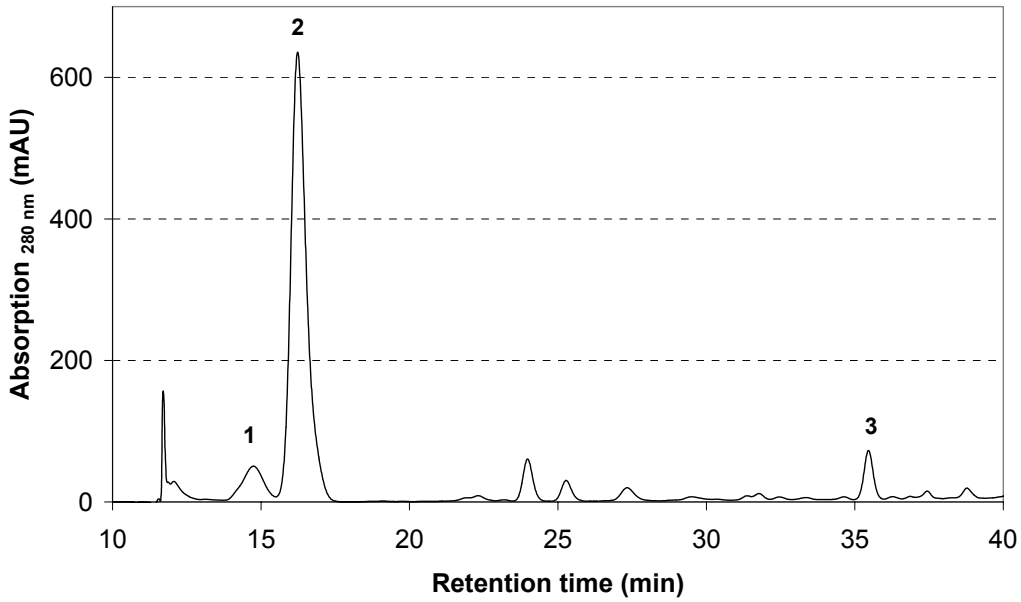

Fig. 25: HPLC chromatogram monitored at 280 nm of heartwood extract of *P. laevigata*. 1: (+)-catechin, 2: (-)-epicatechin, 3: taxifolin. Image from Carrillo *et al.* (2007).

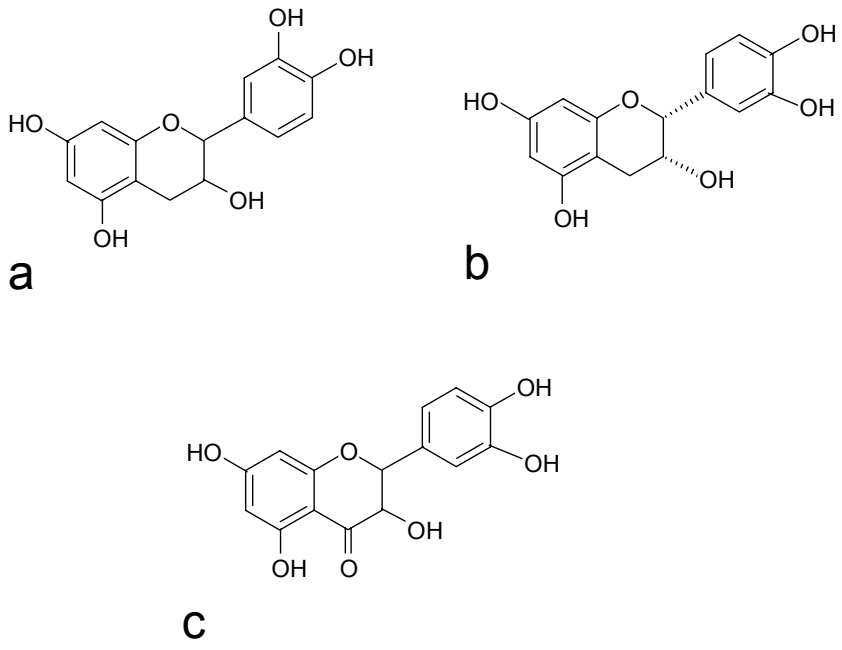

Fig. 26: Molecular structures identified in wood extractives of *P. laevigata* heartwood. a) (+)-catechin, b) (-)-epicatechin and c) taxifolin.

3.5 Conclusion

The chemical wood composition of *P. laevigata* investigated in two sections of heartwood reveals a high proportion of carbohydrates in both the outer (61.7%) and inner heartwood (64.5%). A reduction of glucose concentration (the only building molecule of cellulose) was also found between inner and outer heartwood (48.6 to 45.7%). In the case of hemicelluloses the xylose content is similar in both sections: 12.0 - 12.4% as well as 4-O-methyl-glucuronic acid (0.9%), rhamnose (0.3%), and mannose (0.2%). A reduction in the concentration of arabinose (0.9 to 0.6%) and galactose (1.7 to 1.5%) was, however, observed. The concentration of lignin is 31.4% in the outer and 29.8% in the inner heartwood.

Prosopis laevigata heartwood in acetone-water extractions contains a high concentration of extractives in the inner and outer sections (11.6 and 12.8%). The main phenolic compounds identified following acetone-water extraction are (+)-catechin, (-)-epicatechin and taxifolin.

Topochemical analyses applying UV microspectrophotometry scanning show that lignin is mainly localised in the cell corners and the compound middle lamella of fibers. Wood extractives were topochemically detected in the lumen of vessels, parenchyma cells and in pit canals; they were locally impregnated in the S_2 of fibers. These compounds and their distribution might be contributing factors to the decay resistance of *P. laevigata.*

Chapter 4

PHYSICAL AND MECHANICAL PROPERTIES OF *PROSOPIS LAEVIGATA* WOOD

Summary

Mesquite wood (*Prosopis laevigata*) is used for a wide range of constructive and decorative purposes and also serves as a source of energy; however, basic research of the physical and mechanical properties are still needed to establish additional uses of the wood and thereby increase its value to the timber industry. This chapter gives a detailed technological characterisation of *P. laevigata* and reports on the main physical and mechanical wood characteristics of trees from four different areas of northeast Mexico. These wood specimens have an average density values of equilibrium moisture content (EMC) of 11.2%, ranging from 0.79 to 0.91 g/cm^3, and from 0.72 - 0.84 g/cm^3 under oven dry conditions. The tangential shrinkage varies from 2.2 - 3.3%, radial shrinkage and the ratio (t/r) of the wood were 1.6 - 1.9% and 1.2 - 2.0, respectively. The average values of the modulus of rupture (MOR) lie between 97-126 N/mm^2; the static and dynamic modulus of elasticity (MOE_{stat}, MOE_{dyn}) range from 6580 to 9669 N/mm^2 and 6678 to 9984 N/mm^2, respectively. The coefficient of correlation between MOE_{stat} and MOE_{dyn} is 0.96. The range of average values for compression strength parallel to the fiber is 63 - 68 N/mm^2. The statistical differences found between different physical and mechanical properties within local areas provide useful feedback which forestry biologists and the timber industry should consider in selecting a) the best parts of the tree for a variety of proposes, b) the local areas for further plantation programs and c) in implementing new forestry production measures.

4.1 Introduction

Trees are influenced by many factors such as insects, wildlife, climate, soil conditions, and land management. Each of these lends to a great variability in the properties of a particular wood (Forest Products Laboratory 1999). Different ecological conditions and anthropogenic intervention can induce the development of varied forms of trees, varied leaf size and thickness, and varied bark colour. They can also influence the durability as well as other physical and mechanical properties of the wood (Graham 1960; Johnston 1962; Galindo & García 1983; Hapla *et al.* 2000; Juárez-Muñoz *et al.* 2002; Pasiecznik *et al.* 2004; Raiskila *et al.* 2006). The *Prosopis* genus is known to be very adaptable genetically, which fact accounts for its widespread populations (Peacock & McMillan 1965; Rzedowski 1988). It is comprised of more than 44 species naturally distributed in arid and semi-arid climates of North America, Central and South America, Africa and Asia (Burkart 1976; USDA 2007).

Prosopis laevigata can be found in a variety of environmental areas. In Mexico it is most predominate in Guerrero, Michoacan, Morelos, Oaxaca, Puebla, San Luis Potosi, Veracruz, Nuevo Leon, Durango, Guanajuato, Hidalgo, Jalisco, and Zacatecas (INE 1994). In these areas the timber is used for furniture, wagons, tool handles, utensils in rural households and as firewood or charcoal (Ffolliott & Thames 1983; Rodríguez & Maldonado 1996; Meraz *et al.* 1998).

Recently, studies have been carried out to determine the physical and mechanical properties of several *Prosopis* species, especially *P. juliflora, P nigra* and *P. pallida* (Tortorelli 1956; Berni *et al.* 1979; Universidad Nacional del Nordeste 1979; Ffolliott & Thames 1983; Galindo & García 1986; Perpiñal & Pietrarelli 1995; Tewari *et al.* 2000). The results reveal that the physical properties are very homogeneous; its wood is characterised by high density and low levels of swelling and shrinkage. It is highly durable with regard to compression; it is very hard, but displays low strength values for modulus of elasticity and modulus of rupture (Tortorelli 1956; Berni *et al.*

1979; Universidad Nacional del Nordeste 1979; Ffolliott & Thames 1983; Galindo & García 1986; Perpiñal & Pietrarelli 1995; Tewari *et al.* 2000). Inspite of the economic and ecologic importance of *Prosopis* and the increasing interest to plant and harvest these tree species, information describing the quality of the wood from different *P. laevigata* forest areas is very limited. The main objective of this chapter is to describe the physical and mechanical properties of *P. laevigata* wood from different areas of northeast Mexico. These properties include density, rate of swelling and shrinkage, hardness, static modulus of elasticity (MOE_{stat}), dynamic modulus of elasticity (MOE_{dyn}), modulus of rupture (MOR) and compression strength.

4.2 Material and methods

Wood specimens were collected from four areas in northeast Mexico. Information about the trees and their habitats was provided in Section 2.3.1. Tab. 6 shows the types of tests performed, the standards applied and the number of specimens and specimen dimensions used to determine physical and mechanical properties.

Tab. 6: Physical and mechanical standards performed, overall number of specimens and specimen dimensions used to determine the physical and mechanical properties of *P. laevigata.*

Test type	No. specimens	Specimens dimension[1] (mm)	Standard
Density (g/cm^3)	120	20X20X20	DIN 52 182
Swelling (%)	120	10X20X20	DIN 52 184
Shrinkage (%)	80	10X20X20	DIN 52 184
Modulus of elasticity (static) (N/mm^2)	120	100X5X10	DIN 52 186
Modulus of elasticity (dynamic) (N/mm^2)	120	100X5X10	*
Modulus of rupture (N/mm^2)	120	100X5X10	DIN 52 186
Compression strength (N/mm^2) **	120	30X10X10	DIN 52 185
Janka hardness (N/mm^2)	40	50X50X50	ASTM D143-94
Brinell hardness (N/mm^2)	40	50X50X50	EN 1534

[1] longitudinal x radial x tangential; * Hearmonn 1966; ** Parallel to the fiber

4.2.1 Physical properties

4.2.1.1 Wood density

Density defined as the mass per unit volume is a good indicator with respect to the resistance of wood and the amount of cell wall substance. The value of density depends on many endogenous and exogenous, factors including rate of growth as well as cellulose and lignin content. There is thus a strong correlation between density and the mechanical properties (Kollmann & Cote 1968; Forest Products Laboratory 1999). The density of 120 *P. laevigata* specimens from four different local areas (30 replicates per local area) was determined under two conditions: oven-dry (103± 3°C) and at 20±1°C, 65±3% relative humidity (RH). To calculate the density Formula 4-1 was used.

Formula 4-1

$$\rho_N = \frac{m_N}{v_N}$$

Where:

ρ_N = density under climate condition (20±1°C, 65±3% RH) in g/cm^3

m_N= mass under climate condition (20±1°C, 65±3% RH) in g

v_N = volume under climate condition (20±1°C, 65±3% RH) in cm^3

4.2.1.2 Swelling and shrinkage

All hygroscopic materials swell and shrink to a lesser or greater degree depending on climatic conditions, but the proportions are different in each (Kollmann & Cote 1968; Mantanis *et al.* 1994). Wood is exposed to changes in humidity and temperature of the surrounding air. As wood is an anisotropic material, the changes in humidity produce different values for swelling and shrinkage in each of the three main directions. The absorption and the release of the different percentages of humidity within wood produce larger changes tangentially than radially and longitudinally.

There are many factors involved in the proportion of swelling and shrinkage; these include anatomical characteristics, cellulose content, wood density, cell wall thickness and the proportion of earlywood and latewood (Sekhar & Rajput 1967; Cave 1972; Eligon *et al.* 1992). Some problems are related to the swelling and shrinkage of the wood; among these are warping, checking, splitting, and, in the case of tool handles, loosening (Forest Products Laboratory 1999).

The swelling of 120 *P. laevigata* specimens (30 replicates per local area) was calculated according to Formula 4-2. This formula related the change in percent of a certain anatomical direction of wood from the oven-dry condition to a predetermined moisture condition (20±1°C, 95% RH). The shrinkage was calculated in 80 specimens (20 replicates per local area) using Formula 4-3 to determine the change in percent of a certain wood-anatomical direction from a determined moisture condition (20±1°C, 95% RH) to oven dry condition. The wood swelling, shrinkage and the ratio between tangential/radial (t/r) directions were determined for the tangential and radial directions.
All calculations were performed at a constant weight; the equilibrium moisture content was also determined by applying Formula 4-4.

Formula 4-2

$$\alpha = \frac{l_w - l_o}{l_o} \cdot 100$$

Where:

α = maximum swelling in %

l_w = dimension of the specimen at saturation point

l_0 = dimension of the specimen under oven-dry condition

Formula 4-3

$$\beta = \frac{l_w - l_o}{l_w} \cdot 100$$

Where:

β = maximum shrinkage in %

l_w = dimension of the specimen under saturation point

l_0 = dimension of the specimen under oven-dry condition

Formula 4-4

$$EMC = \frac{m_c - m_0}{m_0} \cdot 100$$

Where:

EMC= equilibrium moisture content in %

m_c = mass of the specimen at specific moisture content

m_0 = mass of the specimen under oven dried condition

4.2.2 Mechanical properties

Mechanical properties are used to describe the wood strength and the ability of the wood to resist applied or external forces (Record 2004). The final use of the wood is dependent on these properties. The mechanical properties of *P. laevigata* wood were tested on wood specimens free from defects under controlled climatic conditions (65±3% RH and 20±1°C, DIN 52 180). The tests were focused on determining static and dynamic modulus of elasticity, modulus of rupture, compression strength and Janka and Brinell hardness. The results were evaluated to determine differences between the local areas.

4.2.2.1 Modulus of elasticity (MOE)

Elasticity is defined as the property which enables a loaded material to recover its original form after the load is removed; if the load is greater than a certain value, the material will displays a plastic deformity or even failure. The elasticity properties, as well as the density, are fundamental in determining the quality of wood (Ilic 2003). The value of elasticity of *P. laevigata* was obtained by applying both the dynamic

MOE (MOE_{dyn}) and the static MOE (MOE_{stat}) bending strength tests. The coefficient of correlation between the MOE_{dyn} and MOE_{stat} was also measured.

Dynamic modulus of elasticity (MOE_{dyn})

The MOE_{dyn} is a non destructive test which is quick and easy to perform and which does not require installed equipment to determine elasticity (Hearmon 1966; Machek *et al.* 1998b). The test technique is used to determine the mechanical properties of the wood without destroying the samples (Ying *et al.* 1994; Bucur 2006). It is possible to test the same wood specimens more than once; moreover, tests can be carried out to establish whether differences occur over time or whether treatments have any effect. The strong relationship between static and dynamic MOE shows, in most of the cases, a coefficient of correlation greater than 95% (Pellerin 1964; Görlacher 1984; Machek *et al.* 1998b; Ilic 2003; Grinda & Göller 2005).

The MOE_{dyn} of *P. laevigata* was determined at the fiber saturation point on 120 specimens with resonant frequencies. Two flexible sponges located at a distance of 0.224 x length from each end supported the specimens; the vibrations were produced by hitting the mid-point on the upper surface of the specimens with a hammer (Machek *et al.* 1997; Machek *et al.* 1998a; Machek *et al.* 1998b; 2001). The vibration test is presented schematically in Fig. 27, showing the antinode (an), the impact point (ip) and the position of the transducer (tr). The latter sends a signal to the Grindo Sonic device which displays the fundamental resonant frequency (kHz). These values were used in Formula 4-5.

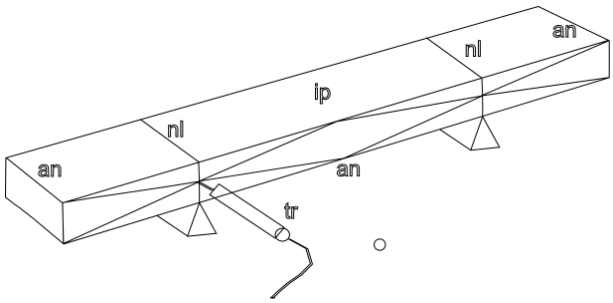

Fig. 27: Schematic representation of the MOE_{dyn} test on the *P. laevigata* specimen. In the flexure mode: antinode (an), node line (nl), impact point (ip), transducer (tr). Image based on Machek *et al.* 2001.

Formula 4-5

$$MOE_{\text{dyn}} = \frac{4 \cdot \pi^2 \cdot l^4 \cdot f^2 \cdot \rho \cdot A}{m_1^4 \cdot I} \cdot \left(1 + \frac{I}{l^2 \cdot A} \cdot K_1\right) \qquad (\text{N/mm}^2)$$

Where:

MOE_{dyn}= dynamic modulus of elasticity (N/mm^2)

I = moment of Inertia (mm^4)

A = area of the cross section (mm^2)

f = frequency (kHz)

ρ = mass density (g/mm^3)

l = length (mm)

K_1 = 49.48

m_1= 4.72

Static modulus of elasticity (MOE_{stat})

Elasticity is the physical property which describes the deformation caused by a low level stress from which recovery can be complete once the stress is suspended (Kollmann & Cote 1968). The stiffness and strength of the wood is normally determined in a static-bending test (Formula 4-6). The wood specimen is supported at both ends by rollers and the resistance is measured as a slow load is applied at the centre of the specimen. The annual rings are horizontally orientated. Fig. 28 shows the three point static bending test.

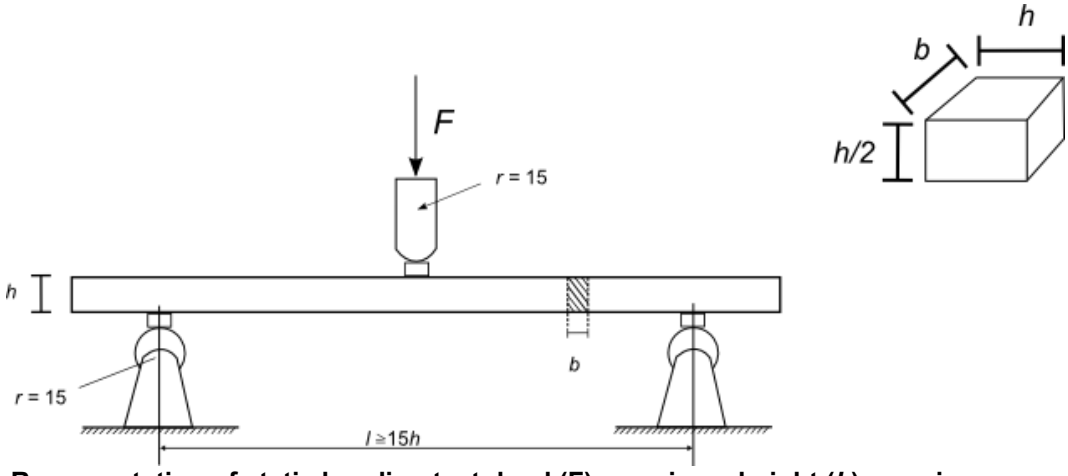

Fig. 28: Representation of static bending test: load (F), specimen height (*h*), specimen area (*b*), span length (*l*). Image based on DIN 52 186.

Formula 4-6

$$MOE_{stat} = \frac{l^3}{4 \cdot b \cdot h^3} \cdot \frac{\Delta F}{\Delta f} \qquad (N/mm^2)$$

Where:

MOE_{stat} =static modulus of elasticity of three point bending test (N/mm^2)

ΔF = load (N)

l = span length of the specimen (mm)

Δf= deflection (mm)

b = width of the specimen (mm)

h = thickness of the specimen (mm)

The normal behaviour of wood specimens during the test is shown in Fig. 29. During the first part of the test a straight-line is produced, which indicates that the deflection of the specimen is directly proportional to the load. If the load is withdrawn at this point in time, the sample returns to its original state with out any damage. Otherwise, if the load is not stopped but is increased, the limit point of proportionality will be reached. This increase continues, the material loses its elasticity and becomes plastic. Then even, if the load is removed, the deformation caused by deflection will be permanent.

At the point of maximum load, ultimate load or ultimate strength the material begins to yield and will fracture unless the load is substantially reduced. In *P. laevigata*, the modulus of elasticity was determined on 120 specimens by testing static bending strength; the test was performed with a universal machine (ZWICK / Roell, Ulm, Germany, Software TestExpert). The load (F) was applied at a uniform rate (5 mm/min) in the direction of the narrow side at the centre of the sample span.

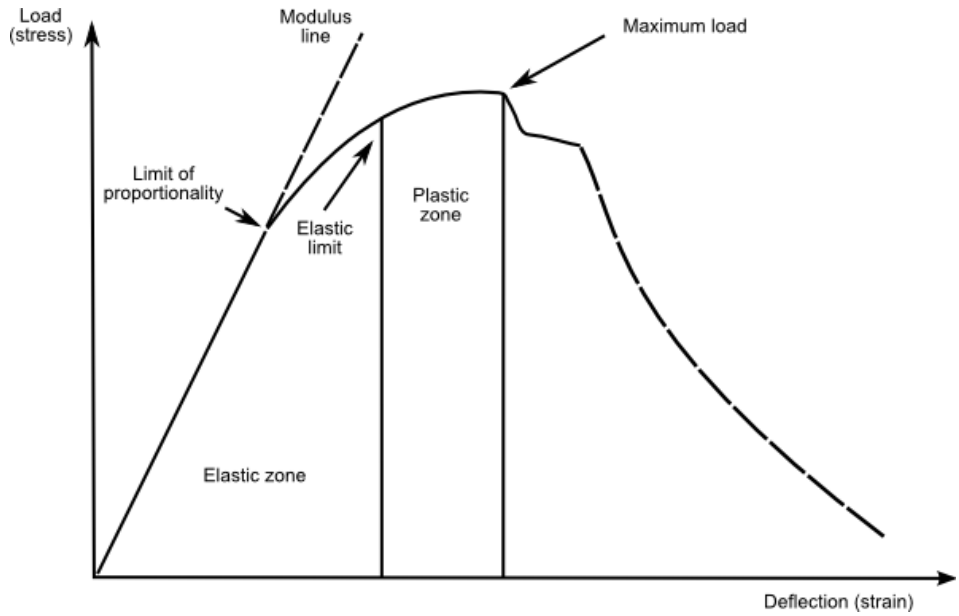

Fig. 29: Stress and strain curve of wood specimens. Represented are the elastic zone, limit of proportionality, plastic zone, elastic limit and maximum load. Image based on Brandon (2005).

4.2.2.2 Modulus of rupture (MOR)

Modulus of rupture or bending strength is defined as the maximum load capacity of a member; it is proportional to the maximum moment borne by the specimen (Kollmann & Cote 1968). This modulus was computed for 120 specimens by applying Formula 4-7. *P. laevigata* wood specimens were also examined to determine the type of failure after their rupture. Drawings are shown in Fig. 30.

Formula 4-7

$$MOR = \frac{3 \cdot F \cdot l}{2 \cdot b \cdot h^2} \quad (\text{N/mm}^2)$$

Where:

MOR= modulus of rupture (N/mm^2)

F = load (N)

l = span length (mm)

b = width of the specimen (mm)

h = thickness of the specimen (mm)

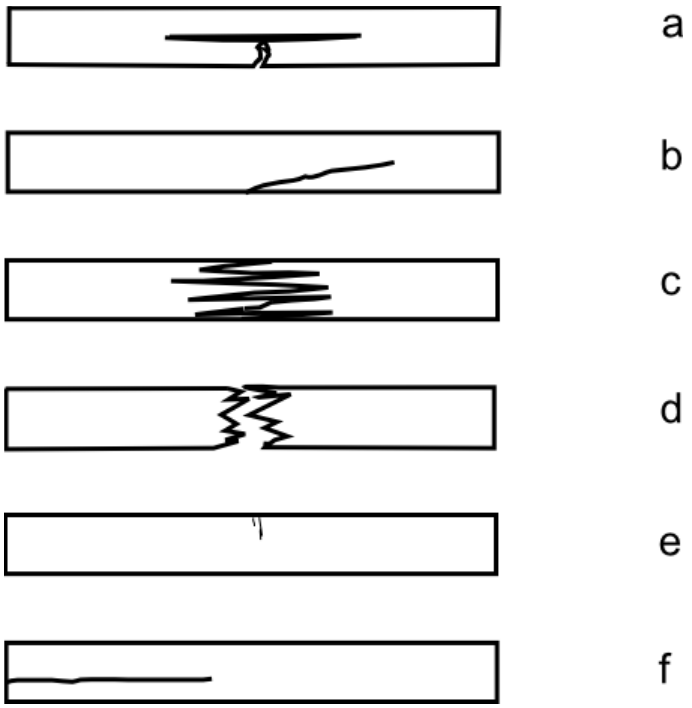

Fig. 30: Type of failures in static bending, according to ASTM 143-94: a) simple tension, b) cross-grain tension, c) splintering tension, d) brash tension, e) compression and f) horizontal shear. Image based on ASTM 143-94.

4.2.2.3 Compression strength

The compressive behaviour of wood is measured parallel to the fiber; this determines the amount of weight that structural wood systems can support prior to failure (Gong & Smith 2004). Fig. 31 shows the typical slip plane formation in late wood cells after compression stress. The compression strength analysis in a parallel direction to the fiber of *P. laevigata* was evaluated with 120 replicates 30 x 10 x 10 mm (l x r x t) according to DIN 52 185.

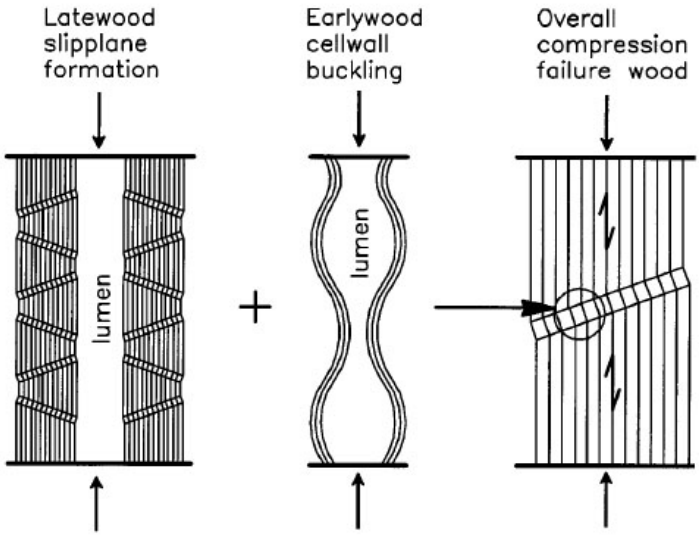

Fig. 31: Formation of a slip plane in latewood and in earlywood following failure of compression strength. The arrow shows the failure zone. Figure by Clorius *et al.* (2000).

4.2.2.4 Brinell and Janka hardness

Hardness is defined as the resistance of one solid body to the force of another solid body. In wood this mechanical property is related to tensile strength and toughness (Fig. 32). The Brinell hardness test uses a hardened steel ball 10 mm in diameter to produce an indentation on the surface of the wood material; the applied load is known and the resulting indentation value is used to determine the hardness per square millimetre.

Janka hardness was proposed in 1906. In this test a steel ball with a diameter of 11.284 mm is indented into a test piece to a depth equivalent to that of the hemisphere, producing a projection area of 1 cm^2. The applied load thus equals the hardness value (Hirata *et al.* 2001). In this test the load (F) was applied until the indentation equalled half of the diameter of the steel ball (Kollmann & Cote 1968; Green *et al.* 2006).

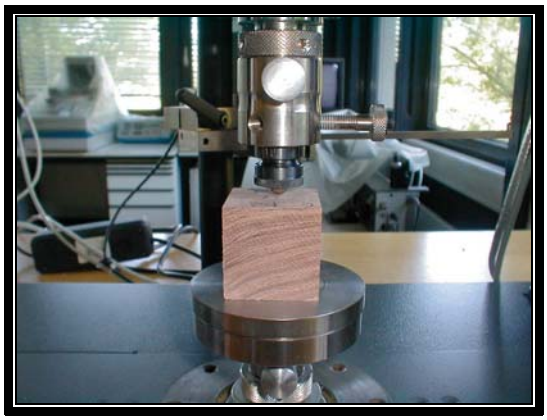

Fig. 32: Determination of Janka hardness according to ASTM D143-94.

The hardness of *P. laevigata* wood was determined on 40 specimens applying both the Janka and the Brinell tests in all three directions (r, t, l) to 50 mm specimens. The measurements for the Brinell test were preformed with a load of 3000 N (Formula 4-7). The maximal load (F) was reached within 15 sec, kept constant over a period of 30 sec and then reduced to zero within another 15 sec.

Formula 4-7

$$HB = \frac{2F}{\pi D(D-\sqrt{D^2-d^2}}$$

Where:

HB = Brinell hardness (N/mm^2)

F = load (N)

D = Diameter of steel ball (mm)

d = diameter of impression (mm)

4.2.3 Statistical analysis

The results obtained from the physical and mechanical tests were subjected to a variance analysis (ANOVA); the average of each property from each local origin was then compared to the corresponding analyses of other origins by applying the Tukey test to determine statistical differences between the habitats (Tables showing the results are in the Appendix). The coefficients of correlation (r) between both MOE_{stat}-MOE_{dyn} and MOR-MOE_{dyn} were also calculated. Two graphs depicting these relationships were plotted (Fig. 33 and Fig. 34).

4.3 Results and discussion

4.3.1 Physical properties

4.3.1.1 Wood density

The density of wood is the result of various factors including growth rate, proportion of earlywood and latewood and cellulose content (Simpson 1993; Forest Products Laboratory 1999). The wood densities of *P. laevigata* specimens (oven-dry and standard climate) originated from the four areas listed in Tab. 7. The average density under oven-dry conditions was 0.76 g/cm^3, and for 65±3% RH conditions 0.84 g/cm^3 at 20±1°C (11.2% EMC). As shown in Tab. 12, the values obtained are relatively high compared to three well-known and commercially important European species: *Fagus sylvatica, Quercus robur* and *Fraxinus excelsior*. The high density in all of the specimens tested might be a result of the wood structure which is characterised by thick fiber cell walls. Based on the ANOVA test, both conditions (oven dry and 11.2% EMC) were statistically different (P <0.0001); the density under oven dry conditions shows that all areas of origin differ. Areas 1 and 4 are statistically different with respect to the other two (11.2% EMC).

Tab. 7: Average density (g/cm^3) and standard deviation values of *P. laevigata* wood specimens from four different local areas.

Local area	No. Specimens	(11.2% EMC)[1] g/cm^3	(0% EMC)[2] g/cm^3
1	30	0.79 ± 0.01	0.72 ± 0.01
2	30	0.91 ± 0.04	0.84 ± 0.04
3	30	0.84 ± 0.01	0.78 ± 0.01
4	30	0.81 ± 0.02	0.73 ± 0.05
Average		0.84 ± 0.05	0.76 ± 0.06

[1] 20±1°C, 65±3% RH
[2] Oven dry

4.3.1.2 Swelling and shrinkage

The swelling and shrinkage behaviours of this wood are used as comparative parameters to indicate the degree of wood stability during utilisation. These values represent the amount of deformation through the absorption and desorption of external humidity. It was found that the equilibrium moisture content (EMC) of *P. laevigata* wood at 20±1°C, 65± 3% RH is 11.2%. When the temperature is 20±1°C and the relative humidity is increased to 95±3%, the EMC is 18.8%.

The values for swelling in the radial and tangential directions and the ratio (tangential/radial) from oven-dry to 18.8% EMC are summarized in Tab. 8. The same table shows the shrinkage values from 18.8% EMC to oven-dry. The results for swelling and shrinkage in the tangential direction display relatively low average values (2.8 and 2.6% respectively). Similar results have already been presented for other *Prosopis* species: 2.7 and 4.8% of shrinkage in the radial and tangential directions, respectively, for *P. glandulosa* (Pasiecznik *et al.* 2001). The same research pointed out that the accordant values for *P. juliflora* from Pune, India were 2.3 and 4.0%.

Research examining the swelling and shrinkage of *P. juliflora* wood planted in different areas verifies the low values of wood shrinkage for specimens from the driest area (Sekhar & Rawat 1960). In contrast, it was found in this study that *P.*

laevigata from local origin No. 4 (the driest area) has the highest tangential values for shrinkage. The ANOVA tests ($\alpha \leq 0.05$) reveal that the swelling as well as the shrinkage of *P. laevigata* woods are statistically similar in the radial direction in all areas; however, the same test reveals differences between local origins in the tangential direction. Consequently, there are also differences ($P<0.0001$) in the t/r ratios.

Wood density and extractive content are factors which might be involved in high dimension stability as discussed for *P. laevigata* in Chapter 3. The chemical analyses revealed a high concentration of extractives (14.1 to 16.0% related to the dry mass) in the wooden tissue of *P. laevigata*. The effect of extractives on swelling and shrinkage is explained by the fact that extractives occupy the spaces which are normally used by water, thus reducing the water absorption and desorption (Tortorelli 1956; Stamm 1964; Mantanis *et al.* 1994).

Tab. 8: Average and standard deviation of swelling and shrinkage in the tangential and radial directions and ratio (t/r) of *P. laevigata* wood from different local areas.

Local origin	No. specimens	[1]Shrinkage %			[2]Swelling %		
		Tang	Rad	t/r	Tang	Rad	t/r
1	20	2.2 ± 0.2	1.8 ± 0.3	1.3 ± 0.3	2.8 ± 0.2	1.6 ± 0.3	1.6 ± 0.3
2	20	2.4 ± 0.2	1.9 ± 0.2	1.2 ± 0.2	2.3 ± 0.2	1.8 ± 0.2	1.3 ± 0.2
3	20	2.8 ± 0.5	1.8 ± 0.6	1.6 ± 0.6	3.6 ± 0.5	1.8 ± 0.6	2.0 ± 0.5
4	20	3.3 ± 0.6	1.6 ± 0.2	2.0 ± 0.4	2.6 ± 0.5	2.0 ± 0.2	1.2 ± 0.2
Average		2.6 ± 0.6	1.8 ± 0.4	1.6 ± 0.4	2.8 ± 0.6	1.8 ± 0.4	1.6 ± 0.44

[1] From 20±1°C, 95±3% RH to oven dry.
[2] Form oven dry to 20±1°C, 95±3% RH.

4.3.2 Mechanical properties

4.3.2.1 Modulus of elasticity (MOE)

The results of the static bending test and the frequency resonance performance of *P. laevigata* specimens from different local areas are given in Tab. 9. The average value for MOE_{stat} is 8504 N/mm^2 and the average for MOE_{dyn} is 8835 N/mm^2. The values

are relatively low compared to other commercially utilized timber species presented in Tab. 12.

The fact that the strength decreases as bending in the MOE static and dynamic test as well as in the MOR is increased might be the result of the cross spiral grain (interlocked grain) which occurs in *P. laevigata* wood. The exclusive selection of those specimens containing the interlocked grain (34%) displayed a breaking pattern in accordance with ASTM 143-94. The static MOE increased to 9112 N/mm^2 and the dynamic MOE to 9473 N/mm^2.

No investigations have been carried out with regard to the relationship between MOE_{dyn} and MOE_{stat} in *P. laevigata*. Based on the results found in this investigation, it is feasible to deduce the strength properties of *P. laevigata* wood as measured by MOE_{dyn}. A good coefficient of correlation (r= 0.96) was found between MOE_{stat} and MOE_{dyn} (Fig. 33). Coefficients relating MOE_{stat} and MOE_{dyn} parameters range from poor (r = 0.71) to very high r= 0.95 in different species (Pellerin 1964; Larsson *et al.* 1988; Ilic 2001; 2003).

The ANOVA test produced a statistical difference (P<0.0001) between areas with respect to two moduli. Values of the local area No. 4 were always statistically different than those for other habitat areas.

4.3.2.2 Modulus of rupture (MOR)

Modulus of rupture is the maximum load carrying capacity of a member in bending strength; it is proportional to the maximum moment borne by the specimen. In *P. laevigata* the MOR test reveal an average value of 114 N/mm^2 (Tab. 9). This value is relativised when compared to other commercially utilized timber species (Tab. 12). The ANOVA test shows statistical differences (P<0.0001) between local origins in the MOR. In the later test local area No. 4 displays a lower value. The coefficient of correlation (r) between MOE_{dyn} and MOR is 0.89 (Fig. 34).

Tab. 9: Average and standard deviation values of static and dynamic MOE and MOR for *P. laevigata* from four different local origins.

Local area	No. samples	MOE_{stat} N/mm^2	MOE_{dyn} N/mm^2	MOR N/mm^2
1	30	8526 ± 1711	9252 ± 1312	112 ± 19
2	30	9242 ± 2165	9427 ± 2246	122 ± 26
3	30	9669 ± 1885	9984 ± 2332	126 ± 26
4	30	6580 ± 1708	6678 ± 1828	97 ± 19
Average		8504 ± 2201	8835 ± 2329	114 ±25

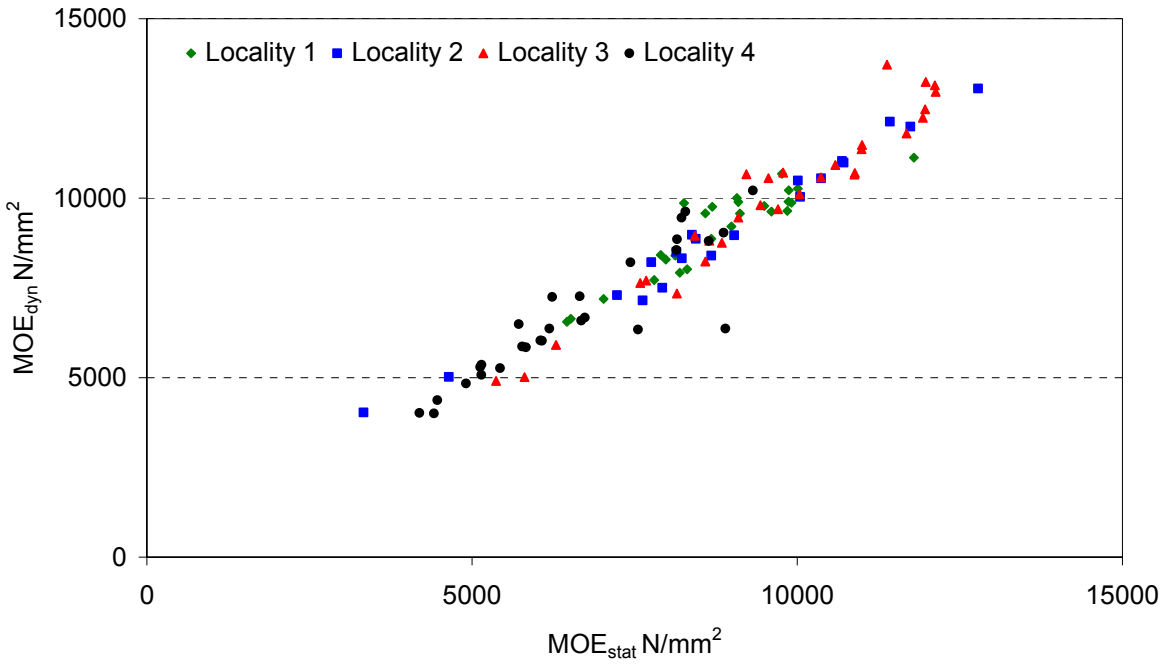

Fig. 33: Relationship between MOE_{dyn} and MOE_{stat} at 95% confidence intervals of *P. laevigata* wood from different localities; the different colours represent the values from each locality (r = 0.96).

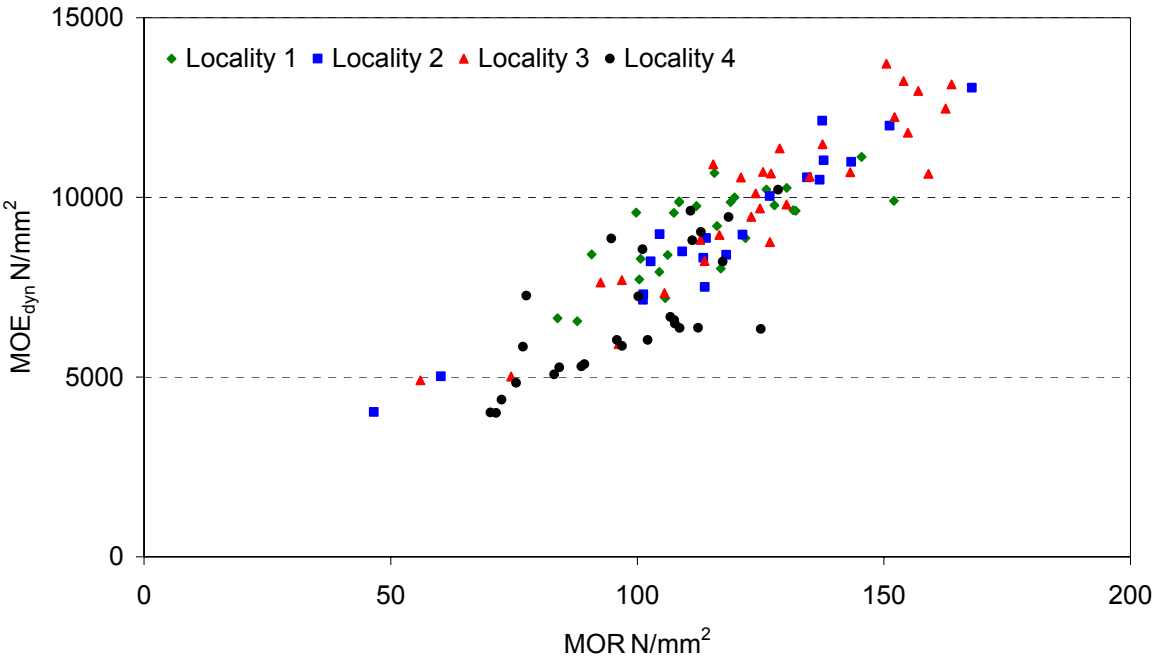

Fig. 34: Relationship between MOE_{dyn} and MOR at 95% confidence intervals of *P. laevigata* wood from different localities, the different colours represent the values from each locality (r = 0.89)

Depending on the kind of failure evaluated according to ASTM 143-94, it was found that typical patterns which occur in *P. laevigata* wood are cross-grain tension and compression (Fig. 35). These types of failure are normally found in wood of tree species with grain which changes direction.

Fig. 35: Type of failures in static bending of *P. laevigata* wood according to ASTM 143-94. 1 and 2 are typical failures from cross-grain tension and 3 from wood tension.

4.3.2.3 Compression strength

The compression strength applied parallel to the fiber direction is higher than that applied perpendicularly. The average compression strength value for *P. laevigata* is 66±6 N/mm^2 (Tab. 10).

It was found that the lower value occurred in specimens from local area No. 4 (63 N/mm^2) and the highest were in those from No's 2 and 3 (68 N/mm^2). An analysis of the results using the ANOVA test revealed significant differences between the localities. Localities 1, 2 and 3 are similar; No 4 is statistically different than local areas 2 and 3 but similar to No. 1.

Tab. 10: Average and standard deviation values of compression strength parallel to the fiber of *P. laevigata* wood from different local areas.

Local origin	No. Specimens	Compression (N/mm^2)
1	30	65 ± 4.3
2	30	68 ± 7.0
3	30	68 ± 5.2
4	30	63 ± 5.2
Average		66 ± 5.8

4.3.2.4 Janka and Brinell hardness

The hardness values for *P. laevigata* in cross sections, radial sections and tangential sections are presented in Tab. 11. The values for the cross section expressed as Janka hardness (91 N/mm^2) and Brinell hardness (74 N/mm^2) are similar to values reported for *P. juliflora* (Sekhar & Rawat 1960). The average values for Janka and Brinell hardness reveal statistical differences between local origins as well as sections. The Janka and Brinell values for locations 1 and 4 are similar as they are for locations 2 and 3; however, compared to each other these two groups are statistically different.

Tab. 11: Average and standard deviation values of Janka and Brinell hardness in cross, radial and tangential directions of *P. laevigata* from four different local areas.

Local origin	No. Specimens	Janka hardness (N/mm^2)			Brinell hardness (N/mm^2)		
		cross	Radial	tangential	cross	radial	tangential
1	10	84 ± 8.4	68 ± 3.9	69 ± 2.7	70 ± 2.3	40 ± 3.5	43 ± 3.1
2	10	101 ± 4.0	89 ± 6.1	92 ± 5.2	78 ± 6.7	44 ± 3.9	50 ± 6.1
3	10	96 ± 3.7	86 ± 5.4	84 ± 4.7	78 ± 8.3	52 ± 7.4	44 ± 4.6
4	10	82 ± 4.8	67 ± 3.2	68 ± 3.3	69 ± 2.6	50 ± 8.5	44 ± 2.6
Average		91 ± 9.7	78 ±11.3	78 ± 10.8	74 ± 6.9	46 ±7.8	46 ± 5.2

Tab. 12: The results for physical and mechanical properties of *P. laevigata* obtained in this research in comparison to three well known, commercially important hardwood species.

Wood characteristic	*Prosopis laevigata*	*Fagus sylvatica*	*Quercus robur*	*Fraxinus excelsior*
Density $_{r0}$ (g/cm^3)	**0.76**	0.68	0.65	0.65
Density $_{r12\text{-}15}$ (g/cm^3)	**0.84**	0.72	0.69	0.69
Shrinkage[1] $_{radial}$ (%)	**1.8**	5.8	4.0…4.6	4.6…5.0
Shrinkage[1] $_{tangential}$ (%)	**2.6**	11.8	7.8…10.0	8.0…8.4
Compression strength[2] N/mm^2	**66**	62	61	52
Hardness Brinell[3] N/mm^2	**74**	72*	66*	65
Hardness Janka[3] N/mm^2	**91**	83	47…78	74
MOE$_{stat}$ N/mm^2	**8504**	16000	11700	13400
MOR N/mm^2	**114**	123	88	105

[1] Shrinkage from 20±1°C, 95±3% RH to oven dry
[2] Parallel to the fiber
[3] Cross section
* Rough value.

4.4 Conclusion

The results for the physical and mechanical properties of *P. laevigata* from different local habitats show noteworthy values, e.g. high density, low shrinkage and swelling, low equilibrium moisture content, and high compression and bending strength. Due to this compression strength parallel to the fiber, *P. laevigata* wood is suitable for

uses in which such strength is needed or for applications in which high changes in moisture conditions prevail. Its use in high strength applications is, however, only recommended if the selection of timber excludes the interlocked grain pattern. Wood with interlocked grain, resulting from the natural growth characteristics of *P. laevigata,* has a relative low bending strength and therefore tends to break.

The statistical differences found within *P. laevigata* wood between its local origins and the variance in its physical and mechanical properties (density, swelling and shrinkage, static and dynamic modulus of elasticity and hardness) should remind the potential user that a given material should be selected for a specific use according to its origin. A characterization of the physical and mechanical properties of *P. laevigata* from the country as a whole will eventually provide more information about the influences of environmental conditions.

Chapter 5

NATURAL DURABILITY

Summary

Natural durability of *Prosopis laevigata* (Mesquite) wood from different regions was investigated using a soil bed test (ENpr 807) and a resistance to basidiomycetes test (modified EN 113). In the latter the durability of extractive-free wood specimens toward basidiomycetes was tested. The inhibition in *Coniophora puteana* and *Trametes versicolor* caused by extractives obtained by applying hot water and ethanol-water, acetone-water and cyclohexane solutions at concentrations of 100 ppm and 1000 ppm was also determined. The results for natural durability after 32 weeks of soil contact showed a very low mass loss (17.2%) and MOE_{dyn} loss (40%) compared to the control, *Fagus sylvatica* (mass loss 84% and MOE_{dyn} loss 90%). The mass loss of specimens exposed for 16 weeks to *Coniophora puteana, Trametes versicolor, Irpex lacteus* and *Pleurotus ostreatus* in a modified EN 113 ranged from 0.3 to 1.5%. The results classify *P. laevigata* wood as Class 1 (very durable) according to EN 350-1. The growth inhibition caused by ethanol-water extractives at 1000 ppm suspended in a malt-agar medium was 33.3% for *Coniophora puteana*.

5.1 Introduction

Wood is the most widely used construction material in the world. It can be used with excellent results under almost any type of environmental conditions; however, in its natural state wood and wood products are compromised by deterioration through a number of sources. Its service life can be reduced by physical, mechanical or chemical means (Kollmann & Cote 1968). Natural durability is defined as the resistance of wood to biological degradation (Eaton & Halle 1993). Several factors, including its microscopic structure, certain physical properties and, above all, wood extractives, influence the natural wood resistance within wood. Wood extractives −those low-molecular-weight substances found in very low amounts (2 – 10%) (Mantanis *et al.* 1995)− are responsible for a large number of wood properties (Fengel & Wegener 1989).

Extractives are present in heartwood and sapwood; the largest proportions are in heartwood. They are mostly located in parenchyma cells but they can also be found in vessels, fibers and specialized cells (Hillis 1971). The extractives are produced during the wood maturation process, in which the cambium creates differentiated sapwood and heartwood cells. Some extractives are produced as part of defence mechanisms after a tree has been mechanically injured by insects or has suffered a microbial attack (Rowell 2005). The way in which the extractives act has been described in several studies. The durability of some trees has been ascribed to bioactive extractives (Schultz *et al.* 1995). A wood which has a worldwide reputation for its extraordinary durability with regard to fungi and insects, including termites, is teak (*Tectona grandis* L.f.). This wood, which is classified as Class 1 according to EN 350-1, can have a service life of to 40 years. The anti-decay compound is tectoquinone (Haupt *et al.* 2003; Thulasidas & Bhat 2007).

The *Prosopis* genus, a very common tree-like shrub or shrub-tree known as *Prosopis laevigata* in Mexico. has a wide-spread population (Burkart 1976; Alden 1995; Juárez-Muñoz *et al.* 2002). Some species are considered undesirable because they can become invasive in agricultural and pastoral areas competing for soil nutrients

and space (Pasiecznik *et al.* 2004). Other species are considered a valuable natural resource for use in food production, fuel, furniture, and environmental protection (Ffolliott & Thames 1983; INE 1994; Meraz *et al.* 1998; Giménez *et al.* 2000).

The physical, mechanical and the natural durability properties of *P. laevigata* wood have been highlighted in several research projects; however, neither the natural durability, nor the agent responsible for producing that durability has been sufficiently investigated (Donoso *et al.* 1984; Rodríguez & Maldonado 1996; Ríos *et al.* 2001; Gérardin *et al.* 2004).

The main objectives of this chapter are: a) to determine and classify the natural durability of *P. laevigata* heartwood from different areas of northeast Mexico, b) to test the natural durability of *P. laevigata* wood extracted with water, acetone-water (9:1), ethanol-water (8:2), and cyclohexane, and c) to determine whether the extracted solutions affect the growth of *Coniophora puteana* and *Trametes versicolor* fungi.

5.2 Material and methods

The *P. laevigata* wood specimens were obtained from four localities already described in Section 2.3.1. In addition, Scot pine sapwood specimens (*Pinus sylvestris* L.) and European beech specimens (*Fagus sylvatica*) were obtained from the Institute of Wood Biology and Technology in Göttingen, Germany. All wood specimens and wood-degrading fungi used during the tests were cultivated and maintained according to European standards. The sample sizes and modifications made during the tests will be described in their respective sections.
The natural durability of *P. laevigata* wood was determined in reproducible laboratory tests and through a rapid initial screening of the decay process (Nilsson & Edlund 1995).

5.2.1 Soil-bed test (ENpr 807)

Unsterile soil, soil bed test, terrestrial microcosmos (TMCs), accelerated field simulators (AFS), or fungus cellars are the different names used to describe the European pre-standard test, ENpr 807. This test is used to determine the effectiveness of wood preservatives with respect fungi (ascomycetes and fungi imperfecti) responsible for soft rot. The unsterilised soil is also colonized by several microorganisms, including bacteria, basidiomycetes and the fungi responsible for moulds.

In this study, the test was used to determine the natural durability of *P. laevigata* from different local origins. Twenty-five samples of *P. laevigata* wood (100mm x 10mm x 5mm, l x r x t) per local origin were selected. 30 reference specimens of *F. sylvatica* and 30 reference specimens of *P. sylvestris* were randomly distributed 20 mm apart and then buried so that approximately 20% of each piece protruded above the surface of the unsterile soil-beds in 2 plastic containers (32cm x 40cm x 22cm) (Fig. 36). The containers were supplied with three different soil layers: 20mm gravel, 20mm river sand, 150mm of loam-based horticultural soil. The samples were incubated in a culture chamber at 20±2 °C with 65±5% relative humidity (RH). The soil was kept at 95% of its water holding capacity (WHC). After periods of 8, 16, 24 and 32 weeks the degradation of the wood specimens was recorded as mass loss calculated by Formula 5-1. The loss in the dynamic modulus of elasticity (MOE_{dyn}) was calculated by applying MOE_{dyn} after incubation. These results were then related to MOE_{dyn} of sound specimens before they were exposed to decay. The MOE_{dyn} of specimens which had been cleaned and then water-saturated with a vacuum pump were determined according to Formula 4-5.

Fig. 36: Plastic container used to expose *P. laevigata* test specimens to unsterile soil.

Determination of mass loss:

Formula 5-1

$$W_l = \frac{w_1 - w_2}{w_1} \times 100$$

Where:

W_l = mass loss in percent

w_1= mass at beginning of the test in g

w_2= mass at the end of the test in g

5.2.2 Resistance to basidiomycetes

To determine the natural durability of *P. laevigata* with respect to basidiomycetes, a modification of the EN113 test was developed; in this modified version the sample size was changed to 100mm x 10mm x 5mm (l x r x t) to quantify the decay as loss of strength which was determined by MOE_{dyn} with Formula 4-5. Nine replicates of *P. laevigata* wood from each local origin were exposed to the four basidiomycetes: *T. versicolor*, *C. puteana*, *I. lacteus* and *P. ostreatus.* In addition, 12 samples of *F. sylvatica* were used as a control; 12 replicates of *F. sylvatica* were used as a virulence control. 32 *P. laevigata* replicates from each locality and 40 replicates of *F. sylvatica* specimens were tested to determine the leaching effect. All specimens were autoclaved for 20 minutes at 121°C in a Tuttnauer Systec Type 5075 ELVC. As shown in Fig. 37, each 50 ml-Kolle flask contained eight wood specimens; six *P.*

laevigata specimens from any of the four local origins and two specimens of *F. sylvatica* were distributed randomly. The specimens were exposed to decay for 16 weeks in a culture chamber at 20±2°C and 65±5% RH. After the test period the loss of MOE_{dyn} caused by fungi was determined on *P. laevigata* specimens, on control specimens as well as on virulence specimens using Formula 4-5. The mass loss was also calculated by applying Formula 5-1 and the data were corrected by subtracting the average mass loss for the specimens from agar control. Finally, the natural durability of *P. laevigata* wood was classified in accordance to EN 350-1.

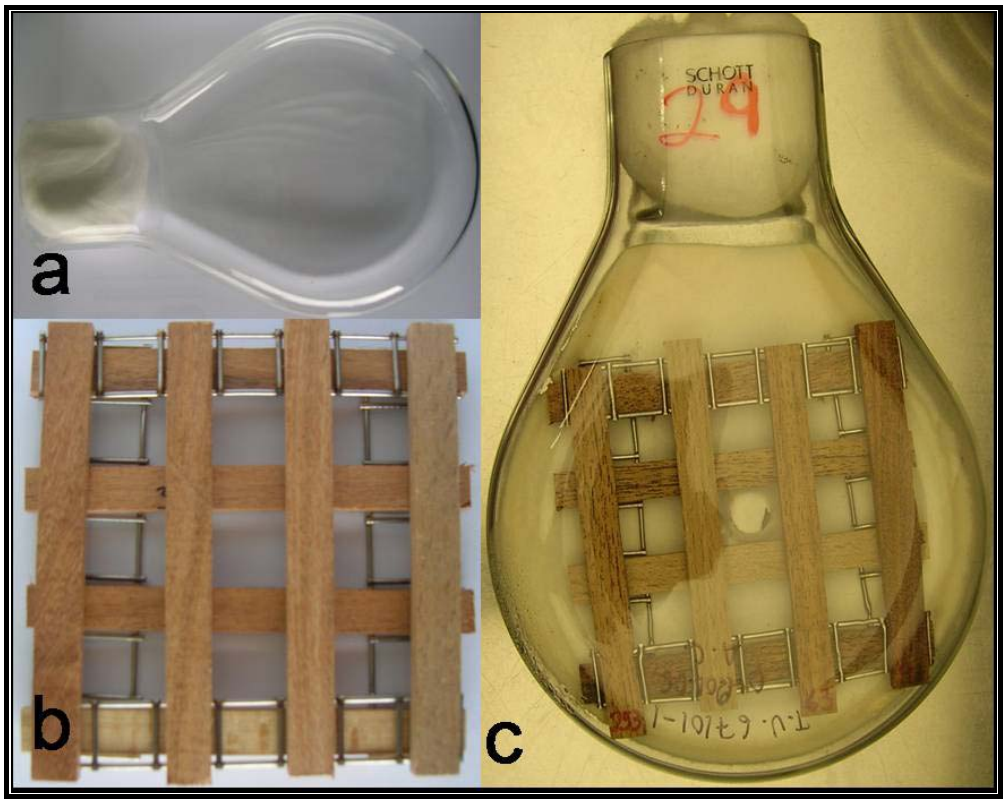

Fig. 37: Preparation of wood specimens, a) Kolle flask, b) sample distribution on two levels, c) Kolle flask with malt-agar medium and wood samples after their inoculation.

5.2.3 Growth inhibition caused by extractives suspended in malt-agar medium

The extractives present in heartwood have a large influence on the natural durability of wood (Donoso *et al.* 1984; Gérardin *et al.* 2004). In order to determine the effect of extractives on the natural durability of *P. laevigata*, the wood was extracted with a Soxhlet extraction apparatus (Fig. 38a). This is a standard method imployed to

determine the extractive content and chemical composition of wood (Demirbaş 1991; Schwanninger & Hinterstoisser 2002). Air-dry sawdust from *P. laevigata* wood (20 g) was put through 0.4 mm (40 mesh) sieves. The *P. laevigata* sawdust was added to Soxhlet extraction flasks and filled with 200 ml solvent. Four different solvents with increasing polarity were used: hot water, ethanol-water (8:2), acetone-water (9:1) and cyclohexane. The oil oven was kept at 135°C providing a minimum boiling rate of 24 cycles during a 6 hour period. The extractives were then isolated from the solvent in a vacuum rotary evaporator (Rotavapor Type R-114 Büchi) at 40°C as shown in Figs. 38b and 38c.

The extractives were dissolved in acetone at 100 and 1000ppm concentrations. To ensure that the mixture remained stable, the solution was stirred with a mixer (Heidolph Type Mr 3003 Control G.) for 10 min. Subsequently, the extractives which had been diluted with acetone were transferred to 140 mm petri dishes. Then 50 ml of malt-agar medium at 60°C was added to the petri dish. The plates were inoculated by placing a 10 mm diameter plug in the centre of each plate. The plugs were obtained from the edge of a colony in the growth phase of the brown rot fungus, *C. puteana*, and the white rot fungus, *T. versicolor*. Five replicates of each extractive were used; five of the acetone-control (only acetone in the medium) and five with the medium served as a control. The cultures were kept in a growth chamber at 25°C until the mycelium of the control had reached the rim of the plates. For *C. puteana* it took 8 -10 days and for *T. versicolor* 10 - 12. The inhibition effect was determined at the end of the period by calculating the average of diametric growth in the two perpendicular directions as shown in Fig. 39. The growth inhibition was determined using Formula 5-2 (Gérardin *et al.* 2004).

Formula 5-2

$$Gi = 100 \cdot \left(1 - \frac{d_1}{d_0}\right)$$

Where:

Gi = Growing inhibition effect in percent

d_1= average diameter of the culture in the presence of extractives in mm

d_0= average diameter of the control culture in mm

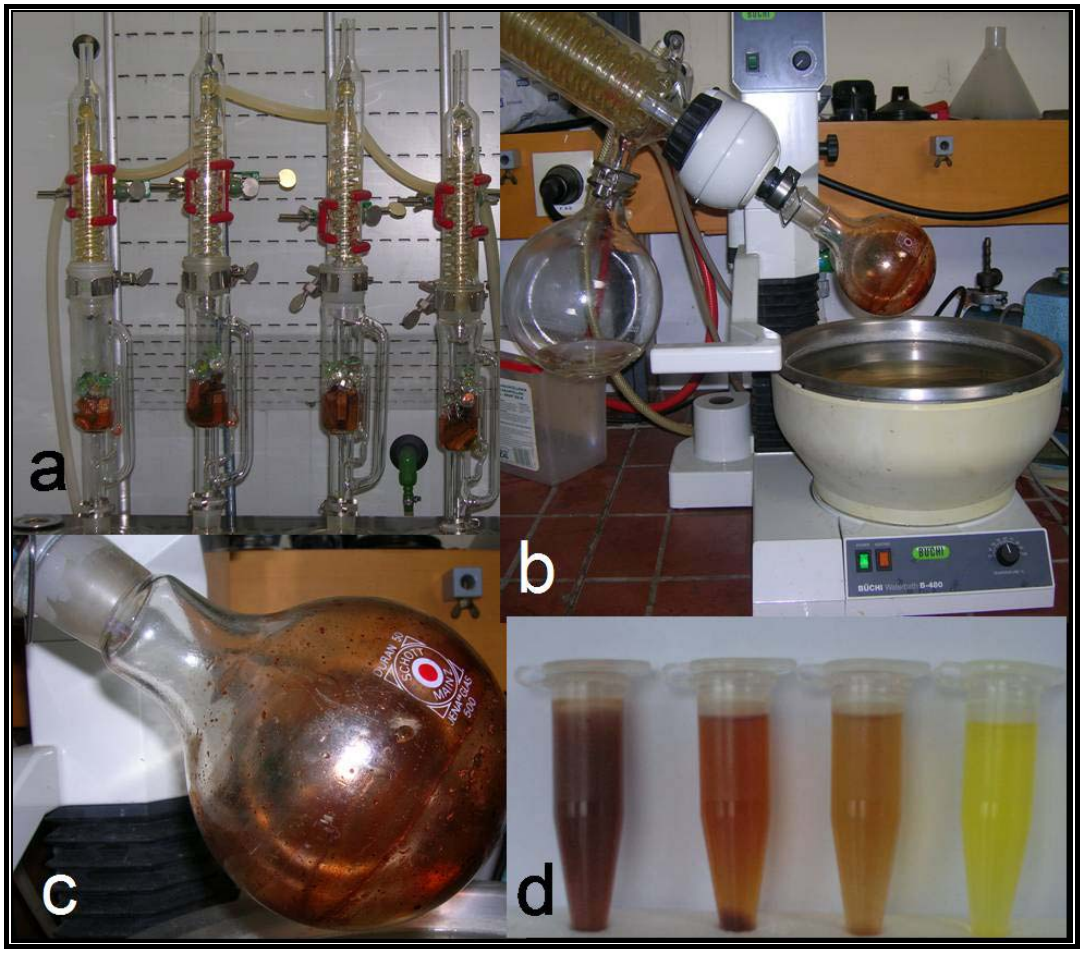

Fig. 38: a) Soxhlet apparatus used to extract *P. laevigata* wood, b) vacuum rotary evaporator, c) acetone extractives after evaporation of solvent, d) wood extractives diluted in acetone, (from left to right: extracted with water, acetone-water, ethanol-water and cyclohexane).

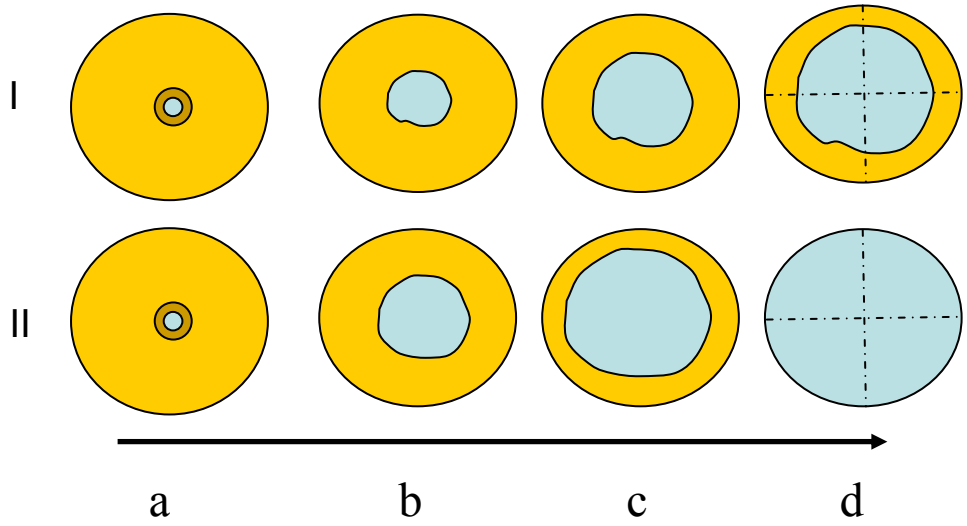

Fig. 39: I) Schematic representation of fungi growing on *P. laevigata* extractives diluted in malt agar, II) Fungi growing on malt agar (control): a) first day after inoculation, b) 2-4 days after inoculation, c) 4-6 days after inoculation, d) 10-12 days after inoculation. The perpendicular lines in "d" represent the measurement direction.

5.2.4 Growth inhibition caused from extractives impregnated on cellulose discs

Ten replicates of 99%-pure cellulose discs (15mm, 750 g/m^2 Munktell, Ederol) were used as a matrix for *P. laevigata* wood extractives (Lennette 1985). Extractions of *P. laevigata* wood using the Soxhlet apparatus were performed according to TAPPI T 204 cm-97. These were similar to the extractions described in Section 5.2.3. The resulting solution was used to impregnate the cellulose discs for 1h by immersion. Subsequently, the discs were dried for 2 days at 103 ± 2 °C. The discs were placed in malt-agar plates freshly prepared with two-week-old circular fungal plug inoculates (*C. puteana* and *T. versicolor*). Six discs were arranged around the fungal inoculums on a 140mm petri dish: 2 untreated discs (control), 2 acetone-impregnated discs (acetone control) and 2 discs with extracted solution (Fig. 40). Five petri dishes used as replicates were incubated for 15 days in the case of *T. versicolor* and for 18 days in the case of *C. puteana*. Thereafter, the plates were examined for inhibition zones and ranked as follows:

0 = no covering by mycelia nor inhibition zone visible
1 = up to half of the specimen covered with fungal mycelium
2 = disc totally covered with fungal mycelium.

Fig. 40: Distribution of cellulose discs in petri dish (diameter 140 mm): I) cellulose discs impregnated with *P. laevigata* extractives, II) cellulose discs impregnated with acetone (acetone control), III) cellulose discs not impregnated (control discs). The circle in the center corresponds to the fungus.

5.2.5 Durability of extracted specimens with respect to basidiomycetes

P. laevigata wood specimens (30mm x 10mm x 5mm (l x r x t)) were extracted using a Soxhlet apparatus and by applying the aforementioned method and the solvent systems described in Section 5.2.3. Prior to extraction the specimens were conditioned at 20±2°C and 65±5% RH. After extraction and oven drying for 24 h at 103°C the dry mass and the amount of extractives were determined. Following autoclavation as described in Section 5.2.2, the specimens were exposed to *C. puteana* and *T. versicolor* in 140 mm petri dishes on a malt agar medium (Fig. 41). Twelve *F. sylvatica* replicates served as controls; 12 replicates of extracted *P. laevigata* specimens and 12 non-extracted *P. laevigata* specimens were used to determine the degree of decay inhibition. 12 replicates of *P. laevigata* were used to determine the moisture content and leaching and 12 *F. sylvatica* replicates to determine the virulence of the fungi tested. The natural durability of *P. laevigata* wood was calculated with regard to the mass loss after 16 weeks.

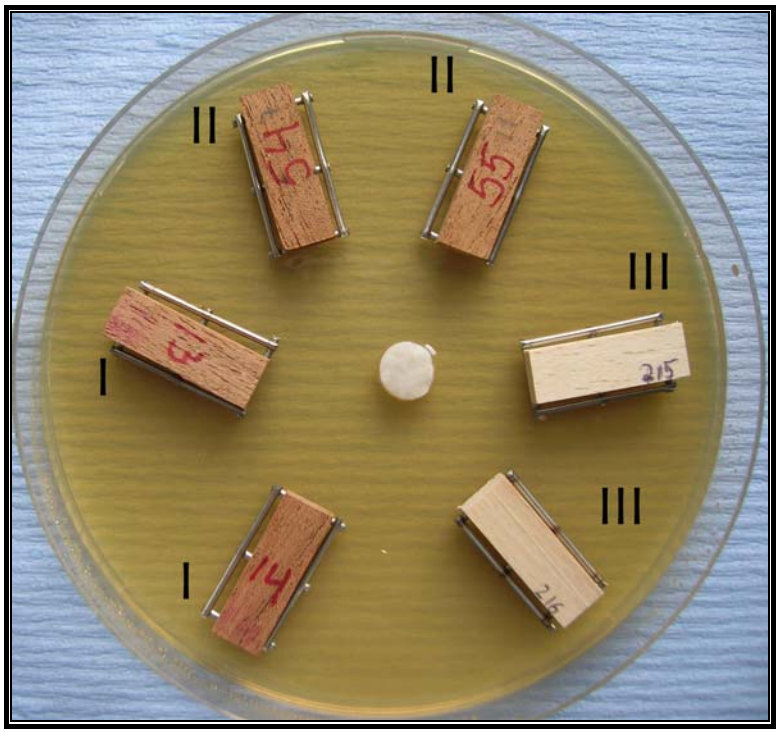

Fig. 41: Distribution of wood specimens: I) *P. laevigata* wood specimens extracted, II) *P. laevigata* wood specimens not extracted, III) F. sylvatica wood specimens (control) and centre fungus inoculums.

5.3 Results and discussion

5.3.1 Soil bed test (ENpr 807)

The soil bed test ENpr 807 was used to determine the natural durability of *P. laevigata* wood with regard to soft-rot, which is mainly caused by ascomycetes, and deuteromycetes in soil under favourable moisture conditions; a succession of detoxifying wood extractives is hereby formed (Scheffer & Cowling 1966). As indicated in Fig. 42, the mass loss in *F. sylvatica* specimens after 32 weeks was 84%, in *P. sylvestris* specimens 35% and in *P. laevigata* from all origins from 9.9 to 17.2%. Local origin No. 4 displayed the highest mass loss of the *P. laevigata* sources, whereas none of the other *P. laevigata* wood origins had mass loss values higher than 12.2% within the same time period. In contrast, Fig. 43 depicts losses in MOE_{dyn} of approx. 90% and 60% with *F. sylvatica* and *P sylvestris* specimens, respectively. It also shows an early loss in MOE_{dyn} after 16 weeks. The losses in *F.*

sylvatica and *P. sylvestris* were 80% and 43%. Similar results (86 and 27%) were detected after 12 weeks (Machek *et al.*). The loss of MOE_{dyn} in *P. laevigata* was 10 - 25% following the 16th week and 18 - 40% after 32 weeks. The results for mass loss and MOE_{dyn} loss in *P. laevigata* exposed to decay for 32 weeks in a soil-bed verify a high level of natural durability for this wood species. The difference in natural durability within specific origins might be due to different rates in durable heartwood formation. The variation in the natural resistance of species from different origins is explained in the literature: some teak trees begin to develop a mature heartwood earlier than other trees, thus influencing differential natural durability (Bhat & Florence 2003).

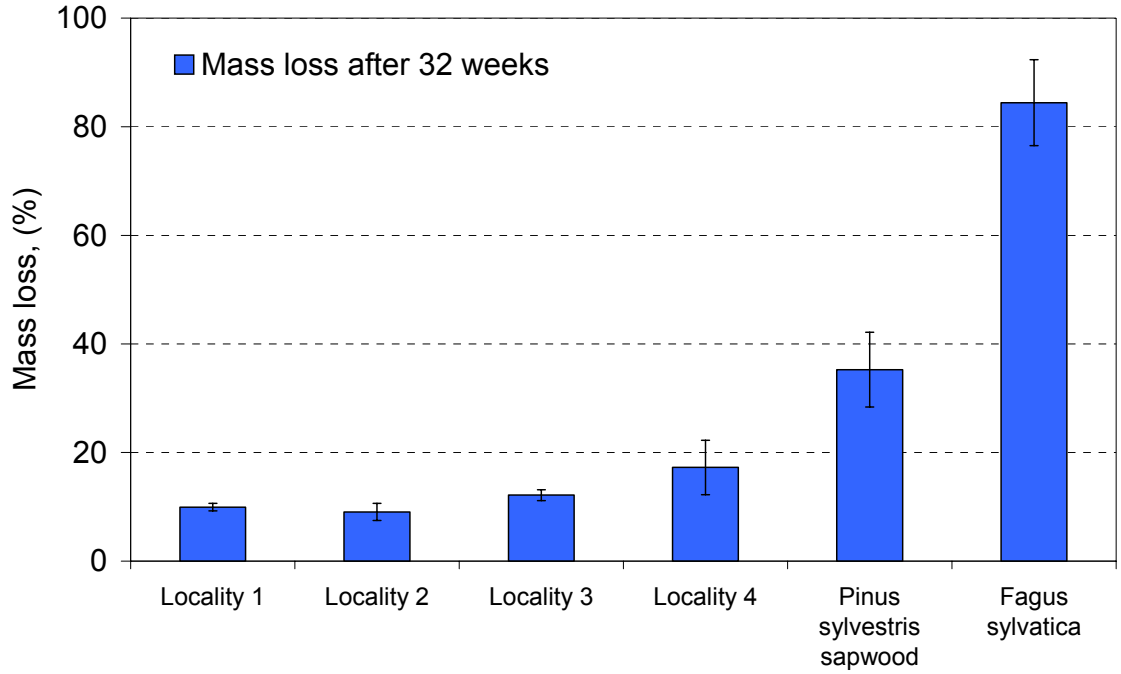

Fig. 42: Average of mass loss of *P. laevigata* wood from different localities after 32 weeks.

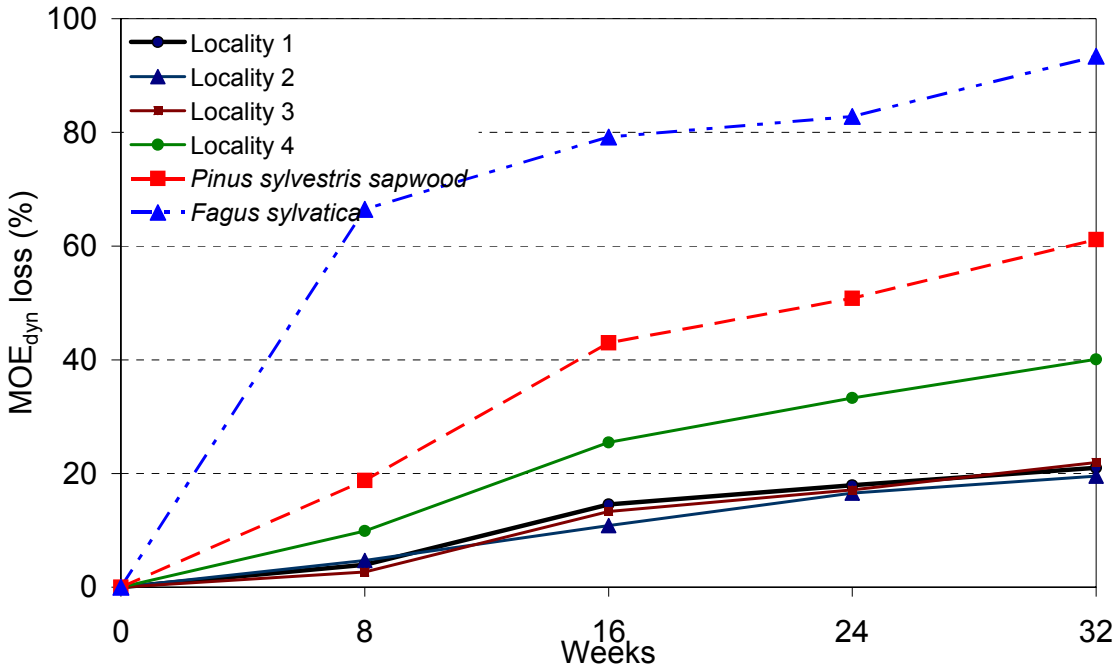

Fig. 43: MOEdyn loss of *P. laevigata* wood from different local origins and the controls *P. sylvestris* and *F. sylvatica*.

5.3.2 Resistance to basidiomycetes

The natural resistance of *P. laevigata* wood to basidiomycetes was ascertained, compared, and classified because this group of organisms produced the most important types of decay (Scheffer & Cowling 1966). Tab. 13 shows the outcomes for mass loss of *P. laevigata* after subtracting 1.68% to compensate for mass loss due to the leaching effect. The virulence of the fungi tested was confirmed by the average mass loss of 40 to 61% for *F. sylvatica* wood. *P. laevigata* wood revealed losses in mass of 0.3 to 1.5% caused by the white-rot fungi *P. ostreatus, I. lacteus* and *T. versicolor* and by the brown-rot fungus *C. puteana*. Local origin 1 was the most resistant (in all fungi tested) with mass losses of 0.4 to 0.8%. High levels of resistance (mass loss of 4%) to *Trametes versicolor* and *Lentinus lepideus* were established in the heartwood of *P. tamarugo* and *P. alba* (Donoso *et al.* 1984). With respect to the classification methods of EN 350-1 mass loss criteria shown in Tab. 14 where the natural durability is expressed according to a class grading scale, all *P.*

laevigata wood origins were classified as Class 1 (high durability) due to its low mass loss (Tab. 15).

Tab. 13: Average values and standard deviation of mass loss of *P. laevigata* from different localities and *F. sylvatica* after 16 weeks of fungal incubation.

Wood species (locality)	Mass loss (%)			
	P. ostreatus	***I. lacteus***	***C. puteana***	***T. versicolor***
P. laevigata (1)	0.4 ± 0.8	0.8 ± 0.3	0.4 ± 0.5	0.5 ± 0.4
P. laevigata (2)	1.2 ± 0.6	1.3 ± 0.8	1.2 ± 0.5	1.2 ± 0.4
P. laevigata (3)	0.9 ± 0.8	1.5 ± 0.9	0.7 ± 0.5	0.8 ± 0.5
P. laevigata (4)	0.3 ± 0.5	1.1 ± 0.6	0.5 ± 0.7	0.6 ± 0.5
F. sylvatica	40.4 ± 7.5	43.7 ± 16.4	61.1 ± 2.7	49.2 ± 19.8

Tab. 14: Wood durability classification system according to mass loss criteria and EN 350-1

Durability class	Description	Criteria	
		Mass loss (%)	EN 350-1 (x value) [1]
1	Very durable	ML ≤ 5	$x \leq 0.15$
2	Durable	5 < ML ≤ 10	$0.15 < x \leq 0.30$
3	Moderately durable	10 < ML ≤ 20	$0.30 < x \leq 0.60$
4	Slightly durable	20 < ML ≤ 30	$0.60 < x \leq 0.90$
5	Not durable	ML > 30	x > 0.90

[1] value x = average corrected mass loss of test specimens/average mass loss of reference specimens.

Tab. 15: *x* values calculated from mass loss of *P. laevigata* from different localities divided by the average of mass loss of *F. sylvatica* after 16 weeks of fungal incubation.

Prosopis laevigata (locality)	x value				Durability class (EN 350-1)
	P. ostreatus	***I. lacteus***	***C. puteana***	***T. versicolor***	
1	0.01	0.02	0.01	0.01	1
2	0.03	0.03	0.02	0.02	1
3	0.02	0.03	0.01	0.02	1
4	0.01	0.02	0.01	0.01	1

The durability of *P. laevigata* wood specimens expressed as MOE_{dyn} loss from different origins after 16 weeks are shown in Tab. 16. The control specimens of *F. sylvatica* displayed high losses of MOE_{dyn} of 67.6 to 87.2%, *P. laevigata* from local origin No. 2 revealed the highest MOE_{dyn} losses (15.0 to 28.0%) followed by specimens from locality No. 3 with losses of 2.0 to 13.2%. The most resistant local origin was locality No. 1.

Tab. 16: Average and standard deviation values for MOE_{dyn} losses of different *P. laevigata* origins and *F. sylvatica* control specimens after 16 weeks of exposure.

Wood species (locality)	MOE_{dyn} loss (%)			
	P. ostreatus	***I. lacteus***	***C. puteana***	***T. versicolor***
P. laevigata (1)	0.2 ± 5.2	9.6 ± 8.8	1.4 ± 8.0	4.0 ± 12.2
P. laevigata (2)	15.0 ± 17.0	28.0 ± 11.8	18.1 ± 34.4	18.8 ± 10.4
P. laevigata (3)	13.2 ± 22.4	9.4 ± 6.0	3.4 ± 16.3	2.0 ± 4.8
P. laevigata (4)	6.2 ± 13.6	5.9 ± 5.6	4.1 ± 23.6	3.7 ± 32.8
F. sylvatica	80.0 ± 9.2	87.2 ± 9.6	67.6 ± 7.8	78.3 ± 14.2

5.3.3 Growth inhibition caused by extractives diluted in malt-agar medium

The biocidal effect of *P. laevigata* extractives obtained in four different polarity solvents of two different concentrations was determined with regard to the growth of *C. puteana* and *T. versicolor.* Tab. 17 shows higher growth inhibition at 1000 ppm after 10 and 12 days of growth. Growth of the brown decay fungus *C. puteana* was inhibited from 28.0 - 33.6% and 13.8 - 26.8% with respect to the white decay fungus *T. versicolor*. Table 17 also displays high standard deviation values due to irregular fungi growth behaviour. Fig. 44 shows the effect of wood extractives in two different solvents at two concentrations. These inhibition effects are relatively high considering that 1000 ppm is 0.1% m/m. Acetone-control solvents used as a control do not inhibit the growth *of C. puteana*; however, the growth of *T. versicolor* was inhibited by 1.1% in acetone-control solvents. The growth of *C. puteana* was always reduced by more than 28% by all extractives at the higher concentration. Different patterns evolved for *T. versicolor*. Its growth was more highly effected by ethanol-water extractives at 1000 ppm; lesser effects were observed for extractives dissolved in water, acetone-

water and cyclohexane. In comparison, an inhibition effect of up to 50% was found for water extractives and acetone extractives from *P. africana* heartwood diluted at concentrations of 1000 ppm. The use of diethylether extractives as an inhibition agent effected the growth up to a level of 80% at 1000 ppm (Gérardin *et al.* 2004). A real inhibition effect is only observed at concentrations of up to 5000 ppm (Reyes-Chilpa *et al.* 1998).

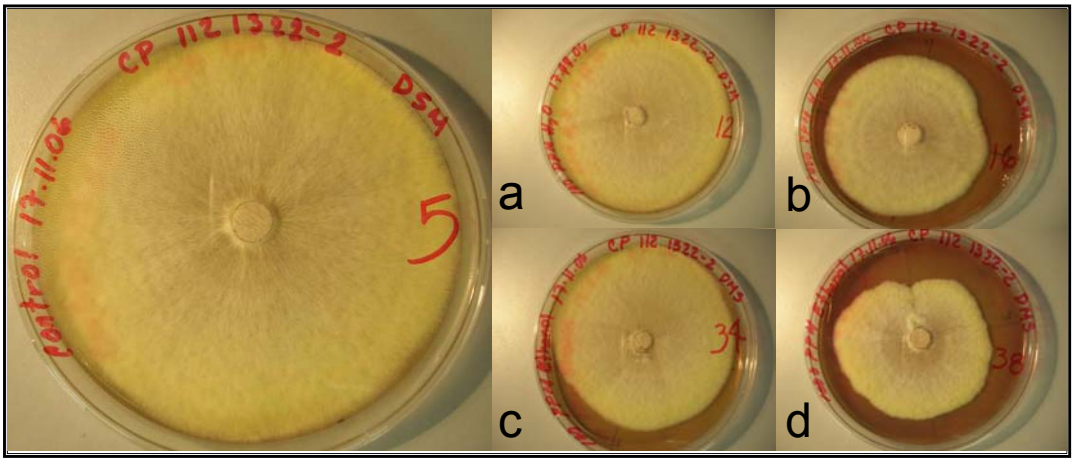

Fig. 44: Growing inhibition of *P. laevigata* extractives on *C. puteana* fungus after 8 days of inoculation: a) water-extractives at 100 ppm and b) water-extractives at 1000 ppm, c) ethanol-water-extractives at 100 ppm and d) ethanol-water-extractives at 1000 ppm and.

Tab. 17: Average and standard deviation values of antifungal activity expressed as percentage of growth inhibition produced by different concentrations of *P. laevigata* wood extractives on *C. puteana* and *T. versicolor*.

Solvent	Concentration [ppm][1]	*C. puteana*	*T. versicolor*
Water	100	17.5 ± 12.4	1.7 ± 2.0
	1000	33.6 ± 12.9	17.8 ± 13.8
Ethanol-water	100	13.6 ± 3.5	10.3 ± 7.5
	1000	33.2 ± 13.4	26.8 ± 11.2
Acetone-water	100	12.9 ± 2.4	9.3 ± 3.6
	1000	31.8 ± 28.4	18.2 ± 7.6
Cyclohexane	100	7.3 ± 6.0	2.8 ± 1.2
	1000	28.0 ± 12.4	13.8 ± 3.0

[1] parts per million.

5.3.4 Growth inhibition caused by extractives impregnated on cellulose discs

Cellulose discs were used to determine the inhibition effect of *P. laevigata* wood extractive on fungal growth. As shown in Tab. 18, the growth of fungi tested is affected by the concentration of the wood extractives. The acetone controls do not have an effect on the growth of the fungi tested; all of them were ranked 2. The ranking value shows that all extractives had an inhibition effect on the growth of *C. puteana* and *T. versicolor*. On *C. puteana* ethanol-water extractives (1000 ppm) had a high inhibition effect (low rank values), as did acetone-water and cyclohexane wood extractives on *T. versicolor*. The grading procedure used here was inadequate to carefully evaluate the inhibition effect of *P. laevigata* wood extractives after 8 or 12 days.

Tab. 18: Average and standard deviation values of growth inhibition ranking system in *C. puteana* and *T. versicolor* caused by different wood extractives of *P. laevigata* wood.

	T. versicolor				***C. puteana***			
[ppm][1]	**100**		**1000**		**100**		**1000**	
	a[2]	**b[3]**	**a**	**b**	**a**	**b**	**a**	**b**
Water	1.3 ± 0.4	2 ± 0.0	1.5 ± 0.5	2 ± 0.0	1.8 ± 0.4	2 ± 0.0	1.6 ±0.5	2 ± 0.0
Ethanol-water	1.4 ± 0.5	1.9 ± 0.3	1.7 ± 0.4	2 ± 0.0	1.6 ± 0.5	2 ± 0.0	1 ± 0.0	2 ± 0.0
Acetone-water	1.7 ± 0.4	2 ± 0.0	1.4 ± 0.5	2 ± 0.0	1.5 ± 0.5	2 ± 0.0	1.6 ± 0.5	2 ± 0.0
Cyclohexane	1.2 ± 0.4	2 ± 0.0	1.4 ± 0.5	2 ± 0.0	1.6 ± 0.5	2 ± 0.0	1.3 ± 0.4	2 ± 0.0

[1] parts per million
[2] Cellulose discs impregnated
[3] Cellulose discs non-impregnated.

5.3.5 Durability of extracted specimens with respect to basidiomycetes

In order to study the effect of extractives on the natural resistance of *P. laevigata* wood, extractive-free *P. laevigata* wood specimens were exposed to *C. puteana* and *T. versicolor* during a period of 16 weeks (Bravery 1978). The results, which are expressed as mass loss of extracted specimens after using different polar solvents, are presented in Tab. 19. The test was validated through the mass losses observed for the *F. sylvatica* control specimens; these show a mass loss of 39 - 59%. All

extracted wood specimens displayed low mass loss values; the extracted *P. laevigata* specimens showed even lower mass losses than non-extracted specimens. The same pattern was also observed with respect to extracted and non-extracted *P. africana* specimens when toluene-acetone (2/1), diethyl ether and acetone were used as solvents (Gérardin *et al.* 2004).

The residual decay resistance of extracted *P. laevigata* wood specimens might be a result of the following factors:

a) The extraction of solid *P. laevigata* specimens was not completed since only 3.8 to 10.3% of extractives were removed, whereas the total extractive content is 14.1 - 16.0% (Section 3.4.3). The heartwood still displays a high fungicidal effect after exhaustive extractions (Schultz *et al.* 1995).
b) The extractives which cause fungal inhibition are bonded to the cell wall and do not permit total extraction (Smith *et al.* 1989).
c) The fungal decay of non-extracted specimens is overrated because of a leaching effect; losses in mass were not detected during fungal exposition of non-extracted agar specimens.

Tab. 19: Average and standard deviation of mass loss of extracted *P. laevigata* wood specimens, non-extracted *P. laevigata* specimens and *F. sylvatica* exposed to *C. puteana* and *T. versicolor*.

Solubility	Fungus	*P. laevigata*		*Fagus sylvatica*
		Extracted	Non-extracted	
Water	*C. puteana*	1.9 ± 0.9	4.8 ± 1.4	40.0 ± 24.6
	T. versicolor	2.0 ± 1.5	3.9 ± 1.1	52.4 ± 14.4
Acetone-water	*C. puteana*	3.6 ± 1.2	4.2 ± 1.8	39.7 ± 27.4
	T. versicolor	3.6 ± 1.2	4.1 ± 1.2	56.1 ± 11.8
Ethanol-water	*C. puteana*	3.1 ± 0.8	4.6 ± 1.1	50.5 ± 26.4
	T. versicolor	3.2 ± 0.6	4.0 ± 1.2	59.5 ± 8.2
Cyclohexane	*C. puteana*	4.8 ± 1.0	4.4 ± 2.0	47.8 ± 25.9
	T. versicolor	4.8 ± 1.0	3.8 ± 1.6	59.5 ± 8.2

5.4 Conclusion

The results for the natural durability of *P. laevigata* wood specimens with regard to decay caused by brown-rot and white-rote fungi and with respect to decay in a soil bed used in this study demonstrated that *P. laevigata* is highly durable and that its heartwood can be considered Class 1 (very durable) according to EN 350-1. Its durability is not effected by ecological differences in the origins of the wood. The natural durability of wood was not affected after extraction with different solvents; the residual antifungal agents were still active in the wood in sufficient amounts to impart resistance to decay fungi.

The inhibition pattern of wood extractives could not be clearly identified. The effect with regard to *C. puteana* and *T. versicolor* fungi was present at higher concentrations (1000 ppm) for all kinds of wood extractives. The biocidal effect of ethanol-water extractives in both concentrations displayed highest efficacy for *T. versicolor*.

The natural durability of *P. laevigata* wood might be the result of various factors, including high density and low swelling and shrinkage; however, they are primarily due to the antifungal effect of extractives. With respect to the latter factor (-)-epicatechin, (+)-catechin, and taxifolin in concentrations of 5.33, 0.51, and 0.05%, respectively, were identified in *P. laevigata* wood (Section 3.4.4). Taxifolin, a substance also found in species of the genus *Larix*, *Cedrus* and *Pseudotsuga* (Eaton & Halle 1993), has been described as an antifungal agent (Kennedy 1956); yet, taxifolin is mainly responsible for the high durability of the species mentioned above (Rudman 1962).

Chapter 6

THE BONDING PROPERTIES OF *PROSOPIS LAEVIGATA* WOOD

Summary

Two adhesives, polyvinyl acetate (PVAc) and melamine formaldehyde (MF), were used at a spread rate of 250 g/m^2 and 350 g/m^2, respectively, to determine the bonding properties of *Prosopis laevigata* under five different conditions according to EN 302-1. The bonding properties were measured by a shear tensile strength test on a Zwick/Roell Z100 testing machine. These results were then compared to the minimum failing load determined by the EN 301 test of bonded *Fagus sylvatica* specimens. The results were analyzed using ANOVA and then the averages were compared by applying the Tukey test. High shear strength values were detected in specimens glued with MF under all of the five conditions tested. The specimens glued with PVAc also produced high values; however, under wet conditions A4 (6 h in boiling water and 2 h soaking in water at 15 ± 5°C) they had lower values. Statistical differences were observed between adhesives as well as between conditions. Gluing *P. laevigata* wood with MF adhesive is recommended for structural use and for use under outdoor conditions because its shear strength values are quite high. *P. laevigata* wood glued with the thermoplastic adhesive PVAc is recommended for indoor applications; the use of PVAc is recommended for use in the furniture industry where the thermoplastic qualities are not required.

6.1 Introduction

In the wood industry the use of solid wood for lumber and veneer is decreasing. The main reason for this is the increasing volume of trees with small dimensions taken from natural stands and from the first cuts on plantation sites. *P. laevigata* trees are a good example. They come from arid and semi-arid lands and are short (6 -10 m) and have small diameter (0.3 - 0.5 m); in addition, they are sometimes damaged by insects or other microbial agents. As only quality logs are selected and sawn, the wood is not used efficiently. As a result, only a small portion of the tree is used to manufacture lumber and the remaining portion is used as fuel. Using adhesives to reduce structural problems and to increase efficiency is playing an increasingly larger role in the wood industry. Similarly, the use of adhesives in *Prosopis* could increase the range and number of possible applications of this wood.

Adhesives have played an important role as far as wood-based products are concerned. Today, up to 70% of the wood industry uses adhesives (Marra 1984). Applying adhesives to engineered wood products (EWP) helps use forest resources more efficiently. Sawmill efficiency is around 40%; oriented strand boards and timberstrand have an efficiency level of 75 and 76%, respectively (Schuler 2000). Adhesives and EWP make both the use of short dimension logs possible as well as the use of species that have not been previously utilized (Mckeever 1997). The wood industry has been able to lend their products more consistent properties than those of sound wood.

Information related to non-structural glue use on *Prosopis* species is scarce. Among the few examples is a study done on particle boards made from *P. nigra* trees which had been damaged by insects. Wood particles of *Prosopis* were glued with urea formaldehyde (Medina & Martínez 1988). Good results have also been obtained with *P. juliflora* (Khali *et al.* 2005). Nevertheless, information regarding the structural use and bonding properties of *Prosopis* species is either not available or non-existent. For this reason, it is essential for the elaboration of EWP's from *P. laevigata,* to test its bonding properties. This could lead to an increase in the amount of wood used

from this tree and at the same time minimise waste. A more effective production process would provide benefits to the people living in arid and semi-arid areas. In this chapter the bonding properties of *P. laevigata* were measured to help establish a basis in an effect to find more uses for this wood.

6.2 Material and methods

6.2.1 Experimental design and description of adhesives

Prosopis laevigata specimens were glued with melamine formaldehyde (MF) and polyvinyl acetate (PVAc). Melamine formaldehyde is formed by the reaction between melamine and formaldehyde; MF must be dissolved in water prior to utilization. The curing process can be carried out under several conditions: A hardener such as ammonium sulphate can be added. An acid condition can be created by adding, e.g., maleic acid; formic acid or phosphoric acid (Cognard 2005).

PVAc is one of the most important adhesives in the wood industry; it is known as “white glue”. This type of adhesive has many advantages. It is fast setting and easily applied and it has high bonding strength and minimal environmental impact. Since it is a water-based emulsion and thermoplastic glue, PVAc has several disadvantages, including a low resistance to humidity and heat. The specifications of this adhesive are given in Tab. 20. Sixteen replicates were used in each of the five conditions showed in Tab. 21.

Tab. 20: Adhesive description.

Adhesive	Polyvinyl Acetate	Melamine Formaldehyde
Resin/hardener ratio (parts)	100/5	100/50
Spreading rate (g/m^2)	250	350
Open assembly (min)[1]	7 to 10	5
Pressure time (min)	120	75
Pressure (N/mm^2)	0.5	0.7
Viscosity (mPas)[2]	8000	Resin 15000, Hardener 2200
Density (g/m^3)	1.04	Resin 10 Hardener 1.7
pH	5.2	-
Hardener (resin)[3]	102.26/195.35	1247/2526

[1] Minutes
[2] Viscosity in millipascal
[3] Trade name

Tab. 21: Description of condition specimens *of P. laevigata* wood tested after gluing according to EN 302-1.

Treatment	Description
A1	7 days in standard atmosphere[1]
A2	7 days in standard atmosphere 4 days soaking in water at (15 ± 5) °C
A3	7 days in standard atmosphere 4 days soaking in water at (15 ± 5) °C 7 days in standard atmosphere
A4	7 days in standard atmosphere 6 h in boiling water 2 h soaking in water at (15 ± 5) °C
A5	7 days in standard atmosphere 6 h in boiling water 2 h soaking in water at (15 ± 5) °C 7 days in standard atmosphere

[1] 20°C and 65% RH (Relative humidity)

6.2.2 Prosopis laevigata wood specimens

The bonding properties of *P. laevigata* heartwood were tested by joining specimens from natural habitats of northeast Mexico (Section 2.3.1). Lumber samples were sawn and planed to dimensions of 600 mm x 50 mm x 8 mm (l x r x t). They were then conditioned at standard atmosphere (20±1°C, 65±3% RH) until constant weight was reached. Before the gluing process, the wood was planed once again to dimensions of 600 mm x 50 mm x 5 mm (l x r x t) to create a fresh surface and thus ensure a good bond with the adhesive. A sponge-roller was used to apply the adhesive to both wood surfaces. After bonding, the specimens were cut to their final dimensions 150 mm x 20 mm x 10 mm as shown in Fig. 45. A general view of the specimens used in the test is shown in Fig. 46.

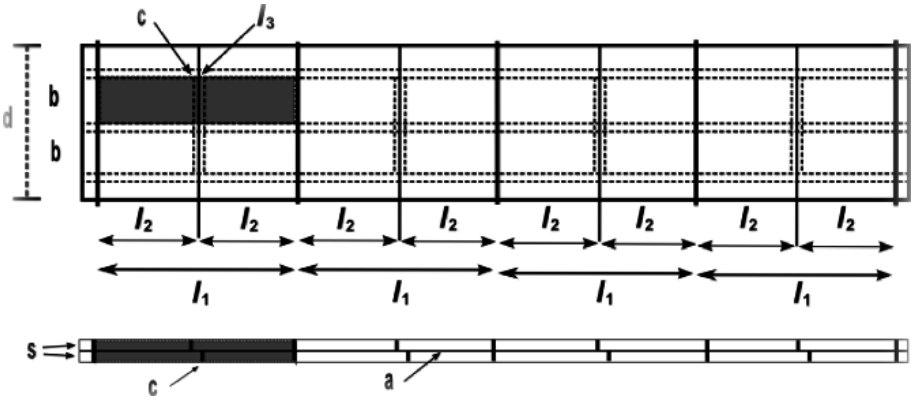

Fig. 45: Schematic representation of a *P. laevigata* assembly: a) glue line thickness, b) width of the sample (20mm), c) cut groove, l_1) length of the specimen (150mm), l_2) 75 mm, l_3) bonding area (10mm), s) thickness of a single panel (5mm). The dotted lines represent the cut lines; the shady areas represent a specimen. Figure based on EN 301.

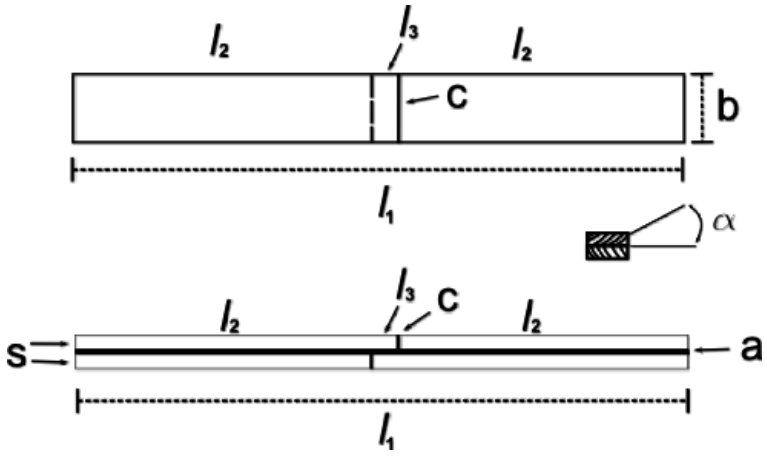

Fig. 46: Sample according to EN 302-1: a) glue line, b) width of the sample, c) cut groove, l_1) 150mm, l_2) 75 mm, l_3) 10mm, s) thickness of a single panel, α) angle between annual rings and surfaces (30° to 90°). Figure based on EN 301.

The bonding properties were measured with respect to shear strength according to EN 302-1. The reference parameter for evaluating the bonding properties of the adhesive was tensile shear force. Tensile shear force was used because it is the most common interfacial stress under service conditions and because it also provides a useful criterion for estimating the mechanical compatibility between the wood and the adhesive (Lavisci *et al.* 2001; Pizzo *et al.* 2003a; Pizzo *et al.* 2003b).

The maximum tensile force needed to shear the wood specimens was determined on a Zwick/Roell Z100 testing machine. The loading rate was 1 mm/min. Once the crack begins to extend, the load is reduced. When a 5% decrease in load is detected, the test machine maintains that condition for 45 seconds. An example of the shear test is pictured in Fig. 47.

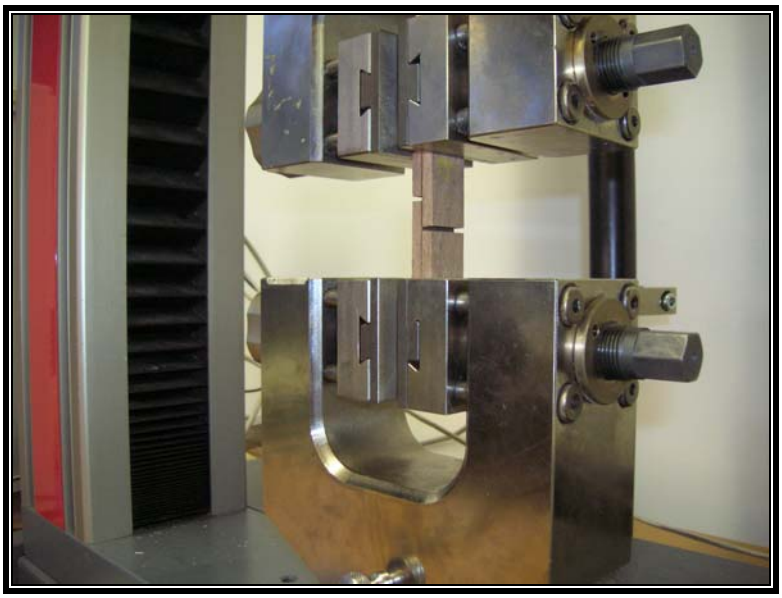

Fig. 47: A general view of tensile shear test according to EN 302-1; Zwick/Roell Z100 testing machine.

6.2.3 Statistical design and analysis

Sixteen bonded replicates from each adhesive and each condition were tested (160 specimens overall); the wood failure was also determined under dry conditions (20±1°C, 65±3% RH). The results were examined by applying the analysis of

variance test (ANOVA) and the difference between the averages was determined with the Tukey test.

6.3 Results and discussion

The results of the bonding properties of *P. laevigata* glued with two different adhesives are presented in Tab. 22; this table reveals that the two glues tested displayed the highest values of shear tensile strength under Condition A3. Both adhesives produced the lowest shear strength values under Condition A4; MF produced higher values in comparison to PVAc except under Condition A1 (due to the thermoplastic characteristics of PVAc).

Tab. 22: Average and standard deviation of shear tensile strength of bonding properties of *P. laevigata* under five different conditions.

Adhesive	Unit	Condition				
		A1	A2	A3	A4	A5
MF	N/mm^2	11.2 ± 1.9	10.4 ± 1.7	13.2 ± 3.4	5.8 ± 1.8	9.8 ± 1.4
PVAc		11.4 ± 0.8	4.6 ± 0.6	12.0 ± 2.6	2.4 ± 1.2	9.7 ± 2.6

Based on the results and the nature of the adhesive, the use of *P. laevigata* wood glued with MF is recommended for outdoor and damp conditions. The PVAc application is limited to indoor conditions because of low shear strength values under damp conditions.

As shown in Fig. 48 the wood failure under most of the conditions ranged from 50 to 100%.

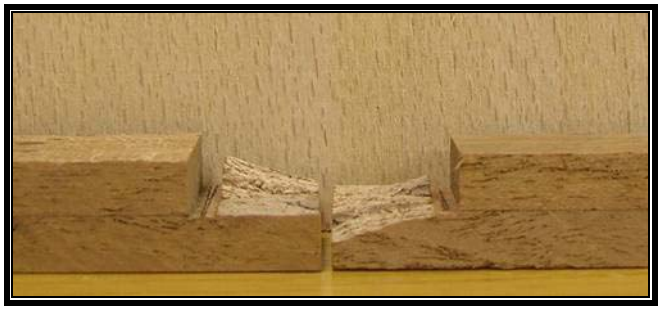

Fig. 48: *Prosopis laevigata* wood specimen showing the percentage of wood failure after shear tensile strength test.

Fig. 49 presents the minimum tensile shear failing loads for close contact joints which should be reached by standard bonding specimens of *F. sylvatica* according to EN 301. Except under Condition A4, the average of the failing loads of *P. laevigata* was higher than the minimum load required.

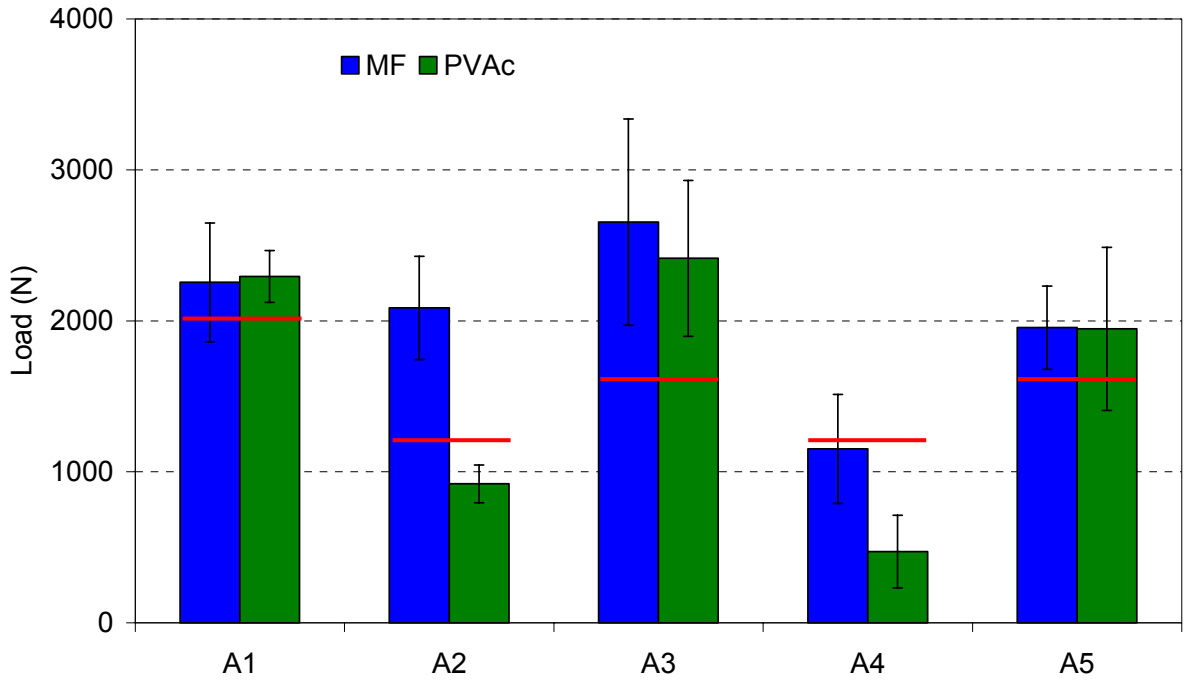

Fig. 49: Average of shear tensile strength of *P. laevigata* specimens glued with two adhesives under five different conditions. The red line shows the minimum standardized failing load of *F. sylvatica* according to EN 301.

The analysis of variance of the shear tensile strength values produced statistical differences between adhesives and conditions ($p < 0.0001$). The higher results for MF under A1, A2, A3 and A4 conditions could be attributed to the fact that it is a thermosetting adhesive, whereas PVAc is a thermoplastic adhesive.

6.4 Conclusion

The results of tests determining the bonding properties of *P. laevigata* wood show that this wood can be glued with structural adhesives such as melamine formaldehyde as well as with the non-structural adhesive polyvinyl acetate. According to EN 301, which tests adhesive efficiency, the shear strength obtained through the MF bonding of *P. laevigata* specimens was higher than the minimum load required under four of the five conditions tested.

Utilising the high natural resistance of *P. laevigata* species for bonding products is feasible. Due to its adhesive properties, the use of melamine formaldehyde in structural application is recommended over polyvinyl acetate for outdoor proposes. The results also confirm that *P. laevigata* heartwood can be used as a source material for wood-based products.

Chapter 7

EFFECTS OF ARTIFICIAL WEATHERING ON *PROSOPIS LAEVIGATA*

Summary

The effects of artificial weathering on *Prosopis laevigata* wood were studied on eight replicates (150 x 72 x 15mm (l x t x r)) free of knots, cracks and resin. The samples were exposed tangentially to UV light and to water spray during three cycles (1 cycle corresponded 1 week of artificial weathering). The conditioning time between each cycle was thirty days. The effects were measured according to visual appearance, crack formation, and colour changes; the results were then compared to two other well known timber species, namely teak (*Tectona grandis*) and beech (*Fagus sylvatica*). The specimens displayed changes in colour after three cycles of exposure. *P. laevigata* changed from brown to white. Delta C (Delta colour) increased from 5.6 to 9.6. There was less crack formation than in *F. sylvatica* but more than in *T. grandis*. Lightness was reduced from 61 to 37 after the first cycle; the lightness value of 35 was maintained at the end of the second and third cycles. The *P. laevigata* specimens showed several changes in colour after artificial weathering. The Delta C was higher due to the photodegradation of lignin and phenolic compounds caused the UV light and the leaching caused by water.

7.1 Introduction

The appearance of weathered wood depends on exposure conditions (Feist & Mraz 1978). *Prosopis laevigata* wood displays high natural durability to basidiomycetes as well as to soil contact under laboratory conditions. The structural bonding properties are also high. In order to expand the possible uses of *P. laevigata* wood in outdoor applications, weathering tests need to be carried out.

It is well known that environmental conditions cause significant damage to wood and wooden materials (Rowell 2005). Besides sunlight, other weather factors can cause degradation of the wood; these include moisture, hot and cold conditions, and wind (abrasion). The effect of sunlight is limited to a 200 µm thick surface layer because of the low degree of penetration of light into wood (Browne & Simonson 1957; Kataoka & Depth 2001). Natural weathering causes a chemical degradation of lignin, cellulose and hemicelluloses. Rain increases this effect by washing out the degraded compounds (Feist 1982; Kalnins & Feist 1993).

The effect of weathering differs among wood species. The most easily detectable effect is change of colour; wood erosion is more difficult to observe. The sunlight effect differs for each wood species; some species adopt different shades or display light colour changes such as bleaching or greying (Sandermann & Schlumbom 1962; Sell & Leukens 1971; Fengel & Wegener 1989). In general, light-coloured woods become darker, and dark-coloured woods become lighter. If the weathering continues, the change to grey results from the growth of mycelia fungi (Feist & Mraz 1978). The erosion process has been described as a slow mechanism; 5 – 8 mm of wood surface is lost per 100 years (Feist & Mraz 1978).

This chapter describes the changes of appearance in colour and lightness caused by artificial weathering on *P. laevigata* heartwood from natural areas of northeast Mexico and compares the results with two well-known timber species.

7.2 Material and methods

7.2.1 Wood specimens and exposition parameters

Eight replicates (150 x 72 x 15 mm, (l x t x r)) free of knots, cracks, and resin from *P. laevigata*, *T. grandis*, and *F. sylvatica* were obtained from logs as shown in Fig. 50. The tangential section of straight grained specimens was exposed in a UV cabinet with a spray option (QUV, Q Panel, Lab Products, Cleveland, USA). The specimens were alternately stressed with UV (A)-irradiation and water spray for a total of three cycles using the parameters presented in Tab. 23. At the end of each exposure cycle, the specimens were conditioned for three weeks at 20±1°C, 65±3% relative humidity (RH) until they reached constant weight. The three weeks of conditioning was necessary since *P. laevigata* reaches its equilibrium moisture content (EMC) slowly.

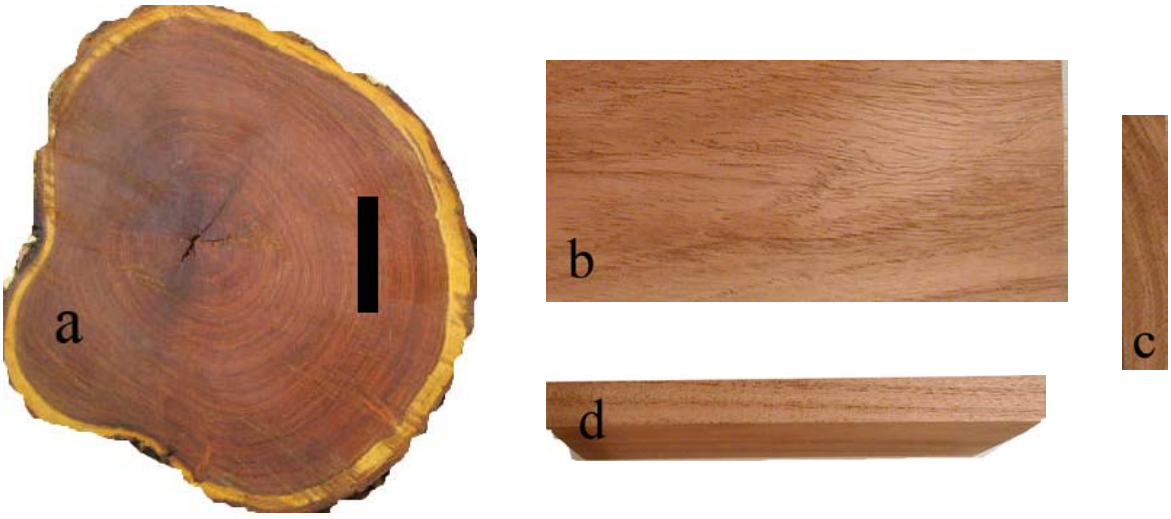

Fig. 50: Representation of samples of *P. laevigata* wood: a) cross section of a log (black area corresponds to sampling section "c"), b) tangential section (area exposed to artificial weathering), c) cross section of the sample, d) radial section of the sample.

Tab. 23: Parameters used during artificial weathering of *P. laevigata* wood and of two control species.

Step	Function	Temperature	Duration	Condition
1	Condensation	45±3°C	24h	100% RH
2	Subcycle step 3+4		48x	
3	UV	60±3°C	2.5h	0.77 W/(m^2nm) at 340 nm
4	Spray		0.5 h	61 - 71, UV off

7.2.2 Evaluation of the effect of artificial weathering

Weathering has a slow degrading effect on wood; it is caused by abiotic factors. Such an effect can be measured by various means. The following effects were evaluated: visual appearance, crack formation and colour change

7.2.2.1 Visual appearance

Visual assessment is a subjective way of determining weathering effects; nevertheless, it is one of the most used means. Some visual differences appear in the form of bleaching or greying or in other changes in colour (Sandermann & Schlumbom 1962). Comparison of the effects with respect to other species leads to more realistic results. The visual appearance of *P. laevigata* wood in comparison to other species was ranked according to the parameters shown in Tab. 24.

Tab. 24: Ranking system for visual appearance ENpr 927-6

Class	Classification
0	Unchanged
1	Very slight
2	Slight
3	Moderate
4	Considerable

5	Severe

7.2.2.2 Crack characterization

The loss of surface wood cells is influenced by defibrization; this effect starts with the degradation of lignin and water absorption. Consequently, stress fields form micro cracks which become visible on the wood surface (Sandberg & Söderström 2006). These cracks can be measured and ranked according to their size. The effect of artificial weathering on *P. laevigata* wood has been determined by surface crack characterisation (Schulte *et al.* 2004). The classes and the classification description of each class are presented in Tab. 25.

Tab. 25: Crack classification, according to the length of the crack.

Class	Classification	Length of the cracks
0	No cracks	0
1	Fine and small cracks	<1/3 of L[1]
2	Long and wide cracks	1/3 to 2/3 of L
3	Continuous cracks	<2/3 of L

[1] Exposed area

7.2.2.3 Colour change

The change of surface colour was determined according to coordinates which were established by the International Commission on Illumination (CIE L*a*b* coordinates) and which are based on the lab colour space. The data for “L”, “a” and “b” were obtained using Adobe Photoshop 7.0 software after scanning specimen surfaces with a Canon CanoScan 3000 scanner (image solution: 300 dpi). The “L” axis represents lightness and runs from 0 (black) to 100 (white); the coordinate +a* stands for red, -a* for green, +b* for yellow, and - b* for blue. Any colour can thus be characterized (Brock *et al.*, 2000).

The chroma change (ΔC) was calculated from the data following the initial scan (a_1, b_1) and after each successive scan (a_2, b_2) for weathering according to Formula 7-1.

Formula 7-1

$$\Delta C = \sqrt{(a_1 - a_2)^2 + (b_1 - b_2)^2}$$

Where:

ΔC = Change of colour
a_1= coordinate value before exposition representing red to green colour
a_1= coordinate value after exposition representing red to green colour
b_1= coordinate value before exposition representing yellow to blue colour
b_2= coordinate value after exposition representing yellow to blue colour

7.3 Results and discussion

7.3.1 Visual appearance

Appearance changes were detected in wood specimens as depicted in the following figures. After the first exposure cycle, all species changed their appearance as shown in Fig. 51. *P. laevigata* as well as *F. sylvatica* and *T. grandis* displayed bleaching. The ranking after this cycle was from 1 to 2 (Fig. 52). The effect was more pronounced after the third cycle.

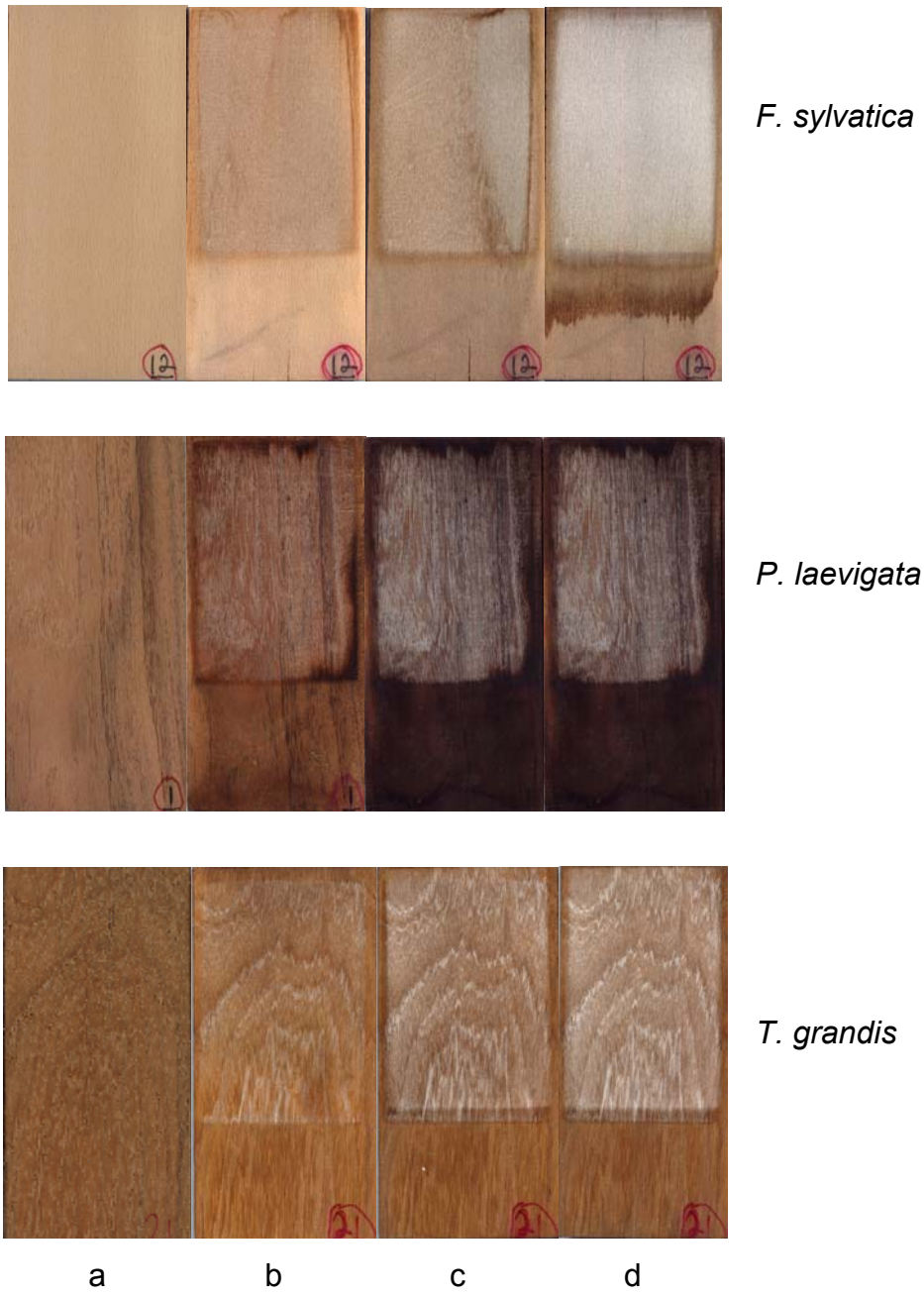

Fig. 51: The artificial weathering effect in *P. laevigata, F. sylvatica* and *T. grandis* after three cycles of exposure, a) before cycle 1 - cycle 0 -, b) after cycle 1, c) after cycle 2 and d) after cycle 3.

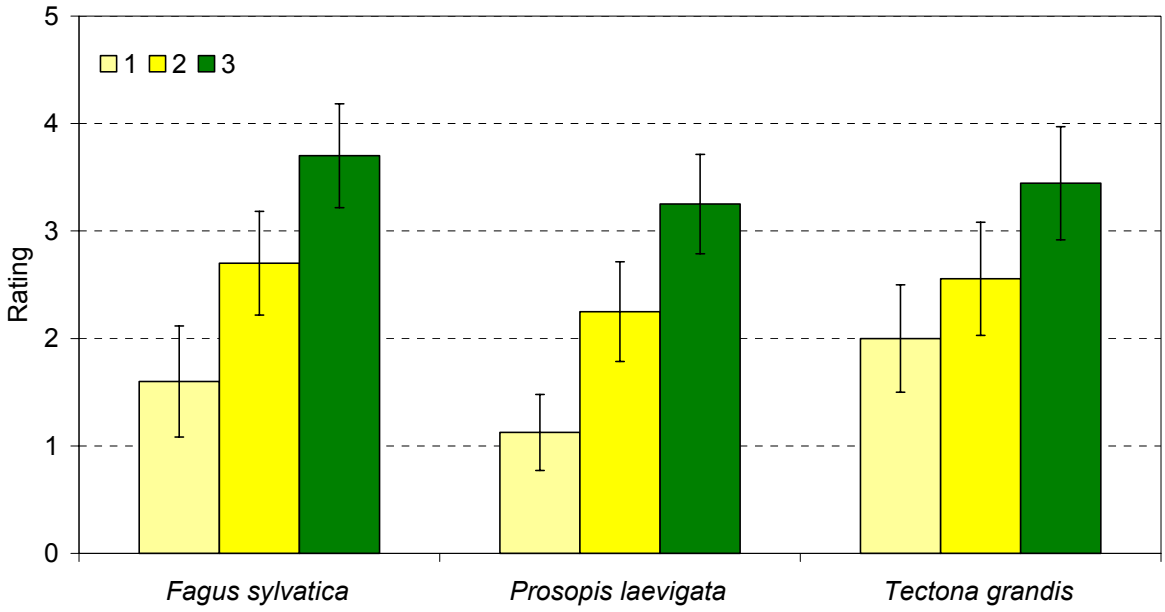

Fig. 52: Artificial weathering effect determined by a visual observation ranking of *P. laevigata, F. sylvatica* and *T. grandis* after three cycles of exposure. The numbers correspond to cycles 1, 2 and 3.

7.3.2 Crack characterization

The cracks were evaluated at the end of each of the 3 conditioning cycles. Fig. 53 shows the cracking effect induced by artificial weathering for the three wood species. *F. sylvatica* was used as a control; it exhibited a crack classification of 0.4 in the initial stage of exposure; and 1.1 and then 2.2 after the second and third cycles. In contrast, *P. laevigata* received a classification of 0.25 after the third cycle. Tectona grandis did not reveal any effect after the third cycle.

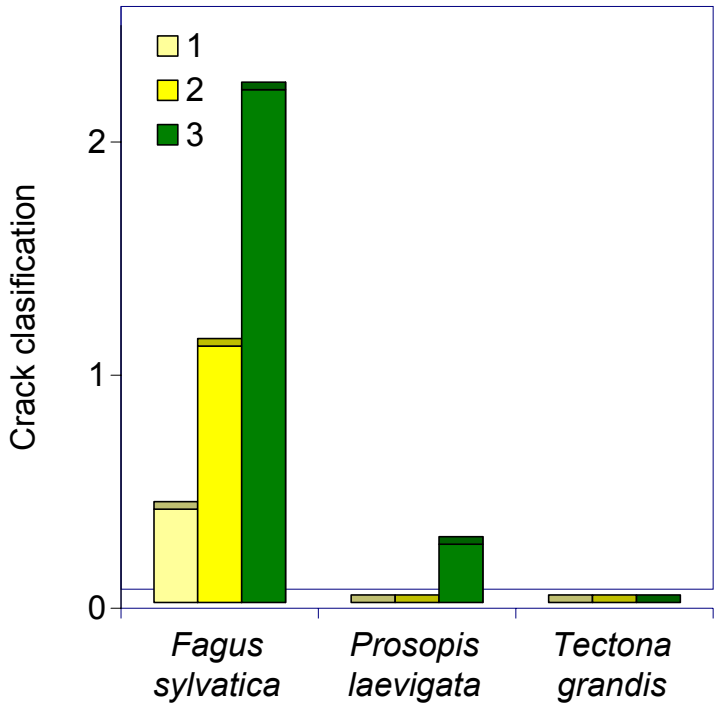

Fig. 53: The effect of artificial weathering determined by crack evaluation in *P. laevigata*, *F. sylvatica* and *T. grandis* after three cycles of exposure. The numbers correspond to cycles 1, 2 and 3.

7.3.3 Colour Change

Colour change induced by artificial weathering after each of the three cycles is shown in Fig. 54. The results exhibit a great change in colour after the first and second cycles for *P. laevigata* (5.6 to 9.6) as well as for the control, *F. sylvatica* (4.0 to 8.6). The colour change in the second control, *T. grandis*, was lower than for the other two species (3.0 to 6.7).

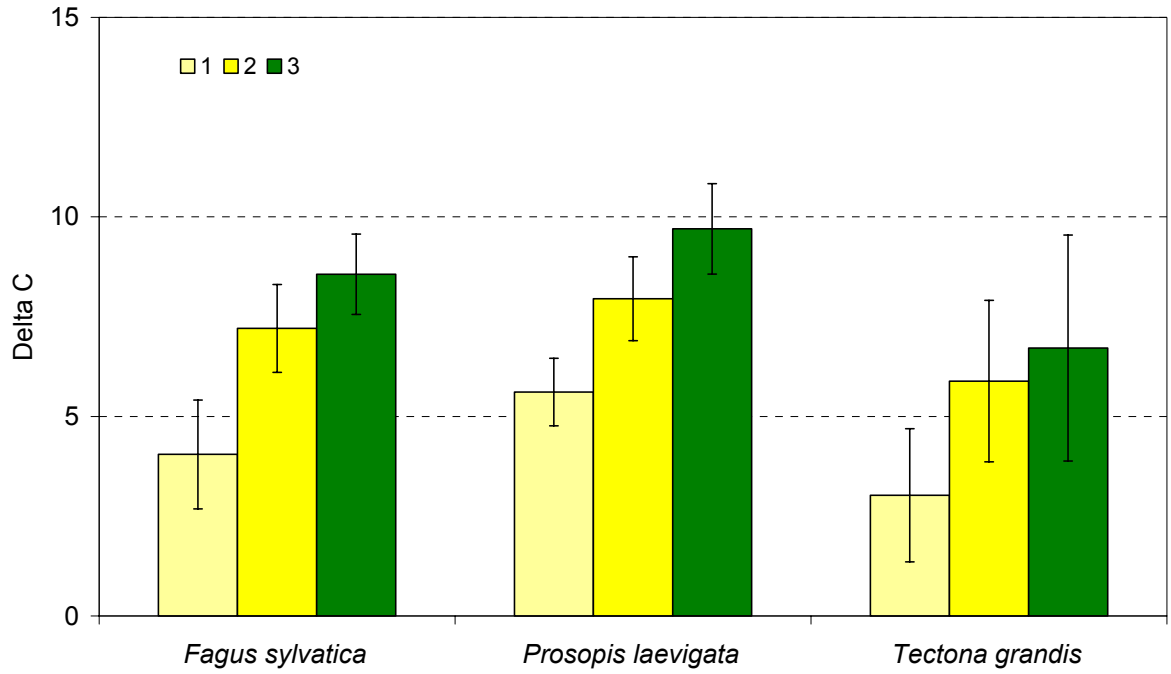

Fig. 54: Colour change according to the CIE-Lab system after 1, 2 and 3 cycle(s) of artificial weathering in OUV device. The numbers correspond to cycles 1, 2 and 3

It was observed that the change in lightness was dependent on the species treated (Fig. 55). After the first cycle all the species were darker, after the second cycle *F. sylvatica* was lighter. Following the third cycle the results were very similar to the first. *T. grandis* was also darker compared to the beginning even though it had begun to lighten after the second cycle. After the third cycle the lightness level was higher than it was initially. *P. laevigata* wood, which had darkened after the first cycle, continued to darken through the third cycle. The results show that the lignin content was oxidised after the first cycle in all wood samples. In *F. sylvatica* and *T. grandis* three cycles of weathering washed out the wood constituents. Different effects were observed in *P. laevigata*, whose high content of phenolic compounds had not been washed out by the end of the third cycle.

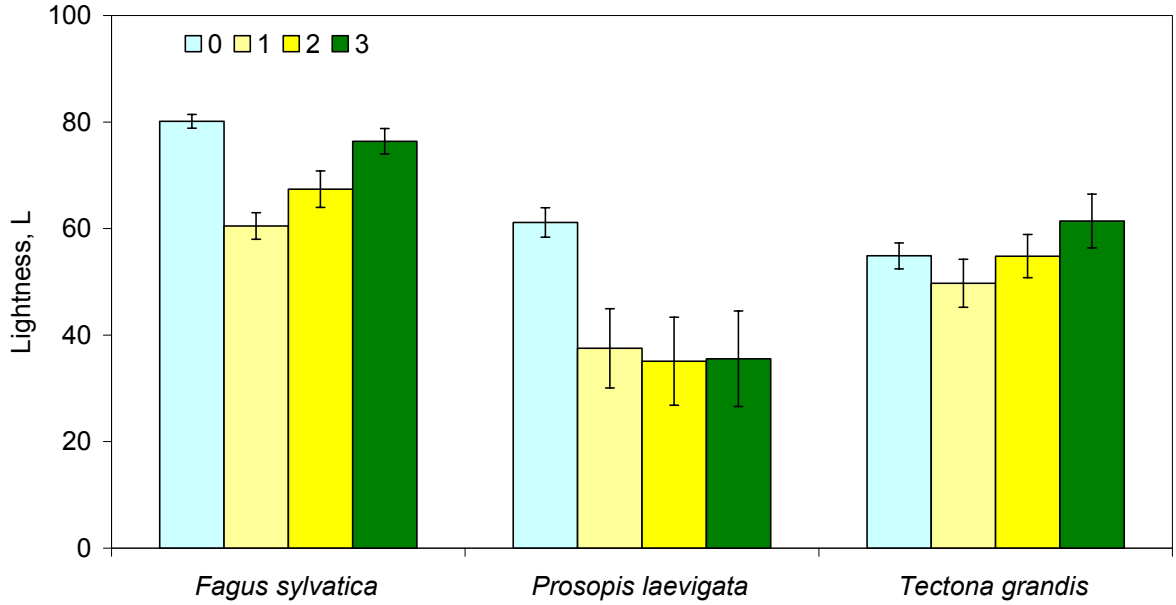

Fig. 55: Comparison of lightness according to the CIE-Lab system initially (0 = before cycle 1) and after each of the three cycles of artificial weathering in a QUV device.

7.4 Conclusion

Based on artificial weathering results, *P. laevigata* wood can be classified as a species with moderate to high resistance. The lower crack formation in comparison to *F. sylvatica*, corresponds to the high dimensional stability retained despite humidity changes (low swelling and shrinkage).

The effects of UV light were observed with regard to the oxidation of phenolic compounds in *P. laevigata*. A colour change from brown to white was detected after the first cycle; a leaching effect from the upper areas to the lower areas was observed. The relatively high density of *P. laevigata*, the finely textured diffuse porosity and the gums which fill the vessels lend proof to the weathering effects.

Chapter 8

GENERAL DISCUSSION

8.1 Wood anatomy

The anatomical wood characteristics of *Prosopis* species are varied; its diversity can be related to its worldwide distribution. Viewed macroscopically, a typical cross section of the stem of a *P. laevigata* tree contains an outer region which is comprised of a yellowish, very narrow (2 to 3 cm wide in a radial direction) sapwood section. The heartwood section displays a darker colour than sapwood and occupies a major area of the cross section. The growth ring pattern is narrow and is related to environmental zone conditions. In areas with relatively low rainfall (Average precipitation per year in the habitat areas studied varies from 300 to 760 mm.), the growth rings exhibit vessels arranged in ring-porous or semi ring-porous forms. Vessels of greater diameter are found in earlywood and vessels of lesser diameter are concentrated in latewood tissue (Gomes & Muñiz 1986; Bowyer *et al.* 2003). According to Zimmermann (1982), the differences between the diameters of vessel in earlywood and latewood and their distribution within the annual rings have an effect on water conduction efficiency. The number of vessels per square millimetre, their distribution, and the average vessel diameter within the growth ring are all characteristics which contribute to the ability of *Prosopis* trees to establish themselves in arid and semi-arid areas.

The vessel diameter found in *P. laevigata*, ranging from 13 to 224 µm, is the largest vessel diameter among the *Prosopis* species. Water conduction efficiency is, however, greater in tree species with vessels of lesser diameter (Stamm 1972). The range of *P. laevigata* vessel length elements (52 - 192 µm) is similar to *P. flexuosa* (100 - 170 µm); both species exhibit shorter vessel length elements in comparison to *P. kuntze*i (82 - 322 µm) and *P. chilensis* (80 - 243 µm) (Villalba 1985; Castro 1994; Scholz *et al.* 2005).

The pattern of vessel length elements and vessel diameter can be described as follows: While *P. laevigata* has the largest vessel diameter, *P. kunzei* has the smallest. The inverse is true with regard to the length of a vessel element since *P. kunzei* has the longest vessel length element and *P. laevigata* the second shortest.

Rays in hardwood are normally comprised of more than one individual ray parenchyma cell. They provide horizontal movement of nutrients from the periphery towards the centre of the tree. Rays also contribute to carbohydrate storage. *P. laevigata* displays multiseriate rays ranging from 3 to 6 cells. Rays composed of more than 6 cells are seldom present. Similar numbers from ray parenchyma cells are also found in *P. nigra* and *P. kunzei*. The rays in these species range from 3 to 5 and from 3 to 6 cells wide, respectively.

The main function of fibers in hardwood trees is to provide strength and support. The average fiber length of *P. laevigata* is 975 µm (range: 589 to 1.312 µm). There is a great difference in fiber length between *Prosopis* species. For instance, the fiber length of *P. kuntzei* ranges from 557 to 1775 µm (average 1257 µm); *P. argentina* ranges from 279 to 838 µm with an average of 532 µm (Villagra & Roig-Juñent 1997; Scholz *et al.* 2005). The range among fiber lengths within the same tree species is also great.

The proportion of different types of wood tissues affects chemical wood composition, as well as physical and mechanical wood properties. The effect of the climate, e.g., arid and semi-arid, on the growth pattern of hardwood trees is distinctive. Since *P. laevigata* wood has a ring-porous or semi ring-porous pattern, it follows that the growth rate affects wood density. Those local habitats in which environmental conditions increase wood formation produce denser and stronger wood. This is due to the fact that the earlywood section of one annual year ring (Earlywood is more porous and of lower density than latewood.) is less prevalent than the latewood section, which is formed by small diameter vessels and thick cell walls.

8.2 Wood chemistry

Chemical wood composition varies within the tree's individual parts (roots, trunk or branches), between the types of wood (sapwood or heartwood) and between geographic and climatic conditions (Pettersen 1984). Composed of cellulose and hemicellulose polymers, holocellulose comprises the major carbohydrate portion of wood (Fengel & Wegener 1989; Rowell 2005). The chemical wood composition of heartwood from *P. laevigata* reveals that holocellulose content ranges from 61.7 to 64.5%. The usual amount of holocellulose is from 65 to 70% of the dry weight (Han & Rowell 1996). The range of glucose, the main component of cellulose, in *P. laevigata* is from 45.7 to 48.6%. In comparison, the hemicelluloses, which are associated with cellulose and which contribute to the structural component of the tree, contain xylose (12.0 - 12.4%), arabinose (0.6 - 0.9%), 4-O-methyl-glucuronic acid (0.9%), rhamnose (0.3%), galactose (1.5 -1.7%) and mannose (0.2%). Reports on the chemical wood composition of other *Prosopis* species reveal that 70% of the wood tissue of *P. juliflora* is composed of holocellulose (Pasiecznic *et al.* 2001; Scholz *et al.* 2005).

Lignin is considered an encrusting substance; it is highly complex and is mainly composed of aromatic polymers in phenylpropane units (Rowell 2005). The lignin content of heartwood trees is usually 25 to 35%. *P. laevigata* lignin content determined by Klason-lignin lies between 29.8 and 31.4%. The lignin concentration in *P. glandulosa* has been reported at 25%, which is in accordance with the results in this study. In *P. juliflora* a wide range of lignin content (11.5 - 31%) was reported by Pasiecznic *et al.* (2001).

Extractives are comprised of a large number of compounds and can be classified according the solvent used to extract them. The function of extractives in trees is not completely clear; however, it is known that the extractives increase the resistance of the tree to microbial attack (Hillis 1987). Overall extractives of *P. laevigata* heartwood range from 14.1 to 16.0%. The range of petrol-ether extractives is 0.3 - 0.4%, acetone-water 11.6 - 12.8%, and methanol-water 2.2 to 2.8%. The extractive concentration in *P. juliflora* ranges from 3 to 15% (Patel & Safaya 1986). A concentration of 9% was established in *P. glandulosa* (Pasiecznic *et al.* 2001). The

content of acetone extractives in *P. africana* heartwood is 11.7% (Gérardin *et al.* 2004); this value is similar to that for *P. laevigata* acetone-water extracts (11.6 - 12.8%). The extractive compound, taxifolin, makes up 0.05% of hardwood acetone-water extractives. (-)-epicatechin was detected at a level of 5.33% through ethanol-water extracting; (+)-catechin comprises 0.51% of *P. laevigata* extractives. Both of the two aforementioned are phenolic compounds from the flavonoid class and are condensed tannins (Hillis 1987). They are also known for their antioxidant function. A topochemical characterization of *P. laevigata* wood reveals that the high phenolic compound content (including lignin) is distributed in axial parenchyma cells and within the pit canals.

8.3 Physical and mechanical properties

The physical and mechanical properties of *P. laevigata* wood vary within local habitats. The average density (0.76 g/cm^3, oven dry condition) which is considered a good estimator of physical and mechanical properties (Kollmann & Cote 1968) was greater than in *F. sylvatica* and *Q. robur* (0.68 and 0.65 g/cm^3, respectively). Local origins No.'s 1 and 4 produced lower oven dry density values than did other localities. Local origin No. 4 also revealed lower values than other localities in physical and mechanical tests.

Volume changes in wood while absorbing or desorbing humidity is described as the swelling and shrinkage effect. The size change occurs before the fiber saturation point is reached because the bonded water is absorbed from the environment into the hemicellulose and the chains of cellulose (Bowyer *et al.* 2003). *P. laevigata* wood demonstrates a higher dimensional stability than other wood; this tree species has a very low swelling and shrinkage rate compared to *F. sylvatica* and *Q. robur*. As the results in this study indicate, the low swelling and shrinkage values for *P. laevigata* are comparable to the results for other *Prosopis* species. Radial and tangential shrinkage values of *P. chilensis* are 0.8 and 2.3%, respectively. Similar results have been presented for *P. tamarugo* (2.8 and 5.6%) (Pasiecznic *et al.* 2001). Low to

medium equilibrium moisture content of 11.2 and 18.8% was found at 20±1°C, 65±3% RH and 20±1°C, 95±3% RH, respectively.

The physical and mechanical properties of *P. laevigata* wood result from its anatomy and chemical composition. Vessel patterns (semi-ring or diffuse porous) influence the physical results. Extractive content also plays an important role, as some of them are antioxidant and thus reduce the water uptake. They can also form physical barriers and occupy free space in to which water could otherwise flow.

Average values for bending strength obtained by static modulus of elasticity are moderately lower than for well known commercial timbers. The factors negatively affecting the bending strength values are the cross grain and the spiral grain. The average value obtained according to modulus of rupture, however, reveals that grain pattern does not considerably affect the modulus of rupture. The MOR value for *P. laevigata* is higher than for *Q. robur* and *F. excelsior* and similar to *F. sylvatica*. The wood hardness values for the three directions (parallel, radial and tangential) and the compression values parallel to the grain are high in comparison to other well known timber species because of the high density of oven dry *P. laevigata* (0.76 g/cm^3 oven dry condition).

8.4 Natural durability

Wood as a biological material is subject to damage imparted in different ways. The effect of fungi on wood can limit a wood's use, as well as decrease its aesthetic value and impair its mechanical properties (Schmidt 2006). The determination of the natural durability of timber species is a step forward providing information for adequate uses of the wood.

In soil bed test ENpr 807, in which the resistance *P. laevigata* wood to basidiomycetes was investigated, the *P. laevigata* specimens demonstrated a high level of natural durability. The portion of each specimen buried in soil revealed low

strength loss and a low loss of mass (18 to 40% and 9.9 to 17.2%, respectively). The results obtained from a modified EN113 standard test classified *P. laevigata* wood as Class 1 (very durable) according to EN 350-1, because the amount of decay caused by basidiomycetes led “x” values of less than 0.15. Similar resistance were determined for *P. tamarugo* and *P. alba* heartwood (Donoso *et al.* 1984).
The natural durability of *P. laevigata* wood could result from numerous factors producing multiple effects. Its anatomical characteristics could be one such factor. There is a low proportion of vessels per square millimetre. The vessels filled with gums form a physical barrier, which effects the water uptake. Cell wall thickness also represents an influential factor, for it is more difficult to degrade a thick cell wall than a thin one. The low moisture content as well as a low level of swelling and shrinkage reduce cracking and thus create fewer opportunities for fungi to start a colony.

Extractives in *P. laevigata* reduce the decay caused by fungi in different ways. This has been confirmed through analyses of its chemical composition, topochemical characterization, and microscopical observations. One way is through a high concentration of extractives (up to 16.0%). Another is the localisation of phenolic compounds (including lignin) which are mainly found in vessels, pit canals and impregnated on the S_2 layer. The nature of extractive compounds also plays a role. Three of these were identified chemically during the HPLC test as (+)-catechin, (-)-epicatechin and taxifolin. All are known as antioxidant substances which can reduce the hygroscopicity of the wood (Zulaica-Villagomez *et al.* 2005). Taxifolin extractives together with three other compounds extracted from *Pseudotsuga menziesii* are known to demonstrate an antifungal effect (Rudman 1962).

Subjecting extracted *P. laevigata* specimens to fungi decay has supported the notion that a cumulative effect occurs. The mass losses were similar to or less than those in the non-extracted specimens. Extraction with water, ethanol-water, acetone-water, and cyclohexane did not prove efficient enough to extract deposited elements because the inhibition “agents” (cell thickness, lignin content, low equilibrium moisture content) could be continuing to provide resistance to decay. Wood extractives dissolved in a malt agar medium inhibit the growth of fungi. The same results were produced with extractives on extractive-impregnated cellulose discs.

Similar findings were also observed with *P. africana* extractives which inhibited the growth of *T. versicolor* (Gérardin *et al.* 2004).

8.5 Bonding properties

Test in which the bonding properties of *P. laevigata* were investigated demonstrated that this particular species can be used for wood-based products. According to the Forest Products Laboratory (1999), wood from species with wide cell wall thickness, low vessel proportion, high density values, as well as high extractive content normally present problems during the bonding process; however, in the case of *P. laevigata* the bonding strength is relatively high. After testing two different glues under five different conditions, it was established that *P. laevigata* has a high shear strength value. A high level of resistance was recorded with the thermosetting adhesive (MF) on *P. laevigata* under conditions A4 and A5 (outdoor and structural applications). The values for *P. laevigata* under all conditions (except A4) were higher than the minimal shear load values for *F. sylvatica* measured by the EN 301 test (standard adhesive test). Shear strength values under all test conditions with PVAc, a semi-thermoplastic adhesive or “white glue” which is used to bond *P. laevigata,* is appropriate for wood-based products in indoor applications.

8.6 Artificial weathering

The effect of weathering on the surface of wood is extremely slow. It has been estimated that a 100 year exposure period leads to a loss, due to erosion, of 5 to 8 mm on the wood’s surface (Feist & Mraz 1978). A change in colour is a more noticeable effect; such changes vary depending on the species. Some light-coloured wood species become darker and some dark-coloured wood species become lighter. Ultimately, the surface of all wood species turns to some shade of grey (Feist & Mraz 1978).

Results obtained for artificial weathering which were analysed by applying three different testing systems provide complementary information. For example, the colour change of wood specimens affected by artificial weathering, resulted from the degradation of phenolic compounds.

At the beginning of this research project, the surface of *P. laevigata* wood specimens was brown; it became white after three cycles of exposure. The degraded wood compounds were washed and bleached with water. There was an increase in the lightness values in the exposed areas. After three exposure cycles, *P. laevigata* still retained a very high degree of structural stability. Low amounts of swelling and shrinking were observed and, as a result, fewer cracks were found in comparison to *F. sylvatica* but a somewhat higher number than in *T. grandis.* This effect occurs as a result of lignin bonds breaking down when exposed to UV light. The residue was also washed away with water. The lack of cracks reduced water penetration into deeper sections of the wood. This is a consequence of the physical barriers formed by extractives in vessels and pit canals as previously explained.

8.7 Application

Timber obtained from *P. laevigata* trees normally has small dimensions as a result of the growing conditions. The height of trees ranges from 4 to 10 m, but the lumber is generally only 1 to 2 m in length. The growth pattern also produces cross and interlocked grain affecting the strength and elasticity of the wood products. Lumber pieces for structural application should be chosen according to graded qualities. Up to now, *P. laevigata* timber has only been used for indoor applications, e.g., furniture production.

The applications of *P. laevigata* need not be limited to specific forest industries; its density values show that the development of additional structural uses is possible. The wood also has other positive attributes; it has great natural durability, a low

equilibrium moisture content, good dimensional stability (low swelling and shrinking), few cracks and good bonding strength.

Prosopis laevigata is of limited use in the pulp and paper industry (high extractive content and high density); however, it could be a feasible source of material for applications demanding short to medium long fibers as well as thick cell walls. The thickness of the fibers could provide the strength necessary for some kinds of paper.

Based on results obtained in testing bonding properties, the production of laminated wood from relatively short *P. laevigata* logs is possible, as is the production of laminated wood (glulam) after the gluing and finger-jointing of relatively short-timber boards. Tree branches from silvicultural operations, wood residues, and sawdust from the sawmill industry can be used in wood-based products such as particleboards (mixed with other species).

Information which has been collected with regard to various properties of this wood such as density, wood stability −with regard to moisture changes− and artificial weathering, high natural wood durability of trees from different habitats, and the high amount of shear strength after bonding, are all parameters which point to an almost limitless number of indoor and outdoor applications. The analyses of the properties and possible used of *P. laevigata* wood done in this study have led to some very important insights worthy of further research. Such research could lead to the development of wood-based products from *P. laevigata* as well as to the use of extractives within the forestry and wood sciences.

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LIST OF STANDARDS

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APPENDIX

Analysis of variance (ANOVA) of *Prosopis laevigata* wood from four local areas.

Physical properties

Wood density

Source	DF	SS	MS	F Value	P
Density (20±1°C, 65±3%)(locality)	3	0.2546998	0.08489993	84.45	0.0001
Residual	116	0.0533947	0.0004603		
Total	119	0.3080945			

Density (Oven dry)	3	0.25419745	0.08473248	78.37	0.0001
Residual	116	0.12541548	0.00108117		
Total	119	0.37961293			

Swelling

Source	DF	SS	MS	F Value	P
Radial direction (locality)	3	1.32042448	0.44014149	2.59	0.0593
Residual	76	12.9361112	0.17021199		
Total	79	14.2565357			

Tangential direction (locality)	3	17.1171887	5.70572957	31.41	<.0001
Residual	76	13.8077087	0.18168038		
Total	79	30.9248974			

Shrinking

Source	DF	SS	MS	F Value	P
Radial direction (locality)	3	0.8683591	0.289453	2.19	0.0959
Residual	76	10.038242	0.1320821		
Total	79	10.906601			

Tangential direction (locality)	3	13.761047	4.58701	24.54	<.0001
Residual	76	14.203443	0.18688		
Total	79	27.964489			

Analysis of variance (ANOVA) of *Prosopis laevigata* wood from four local areas.

Mechanical properties

Compression

Source	DF	SS	MS	F Value	Pr > F
Compression (locality)	3	40.89827	136.96609	4.28	0.0066
Residual	116	3709.84037	31.981382		
Total	119	4120.73864			

Modulus of elasticity

Source	DF	SS	MS	F Value	Pr > F
MOE_{stat} (Locality)	3	168132917	56044305.7	15.91	<.0001
Residual	116	408639627	3522756		
Total	119	576772544			
MOE_{dyn} (Locality)	3	195008058	65002685.9	16.73	<.0001
Residual	116	450810424	3886296.8		
Total	119				

Modulus of Rupture

Source	DF	SS	MS	F Value	Pr > F
MOR (Locality)	3	14874.6388	4958.21295	9.3	<.0001
Residual	116	61831.447	533.02972		
Total	119	76706.0859			

Hardness

Source	DF	SS	MS	F Value	Pr > F
Brinell Hardness (locality)	3	988.08731	329.3624	11	<.0001
Direction	2	20391.755	10195.88	340.67	<.0001
Local origin*direcction	6	1068.6703	178.1117	5.95	<.0001
Janka Hardness (locality)	3	10528.1328	3509.378	146.76	<.0001
Direction	2	4123.0824	2061.541	86.22	<.0001
Local origin*direcction	6	175.8144	29.3024	1.23	0.2989

CURRICULUM VITAE

Artemio Carrillo Parra

Date of birth: May 26, 1968

Place of birth: Linares, Nuevo León. México.

I Degrees:

1. PhD. Wood Biology and Wood Technology, Institute of Wood Biology and Wood Technology, Göttingen University. 2007
2. M.Sc. Forest Sciences, University of Nuevo León, México. 2002
3. B.Sc. Forest Sciences, University of Nuevo León, México. 1990

II Professional Activities:

1. Lecturer in Forestry Operations I, Forestry Operations II and Forest Science, University of Nuevo León 2004.
2. Coordinator in Natural Resources and Environmental Agency (SEMARNAT) responsible for planting of native tree species in State of Nuevo León1997.
3. Technician in the Programming Department in the Rural Land Measurement Program (PROCEDE). National Institute for Statistics, Geographic and Informatics (INEGI) 1993.
4. Inspector of Forest and Wildlife. Agriculture and Hydrological Resources Agency (SARH) 1990.

III Publication:

1. Carrillo, A., I. Mayer, G. Koch, & F. Hapla. "Wood Anatomical Characteristics and Chemical Composition of *Prosopis Laevigata* (Humb. & Bonpl. Ex Willd.) M.C. Johnst. Article accepted for publication by IAWA Journal.

IV Honors:

1 Highest grade average in Forest Science at University of Nuevo León 1990.
2. Scholarship from the Mexican National Council for Science and Technology (CONACyT) to do a Master of Science degree in Forest Sciences at the University of Nuevo León, México 2000.
3. Highest degree average in Forest Science Master's Program at University of Nuevo León, during the M.Sc. of Forest Sciences 2002.
4. Scholarship from Program for the Improvement of Teachers (PROMEP) and the University of Nuevo León (UANL) to do a PhD in the Institute of Wood Biology and Wood Technology, Göttingen University 2003.