# 3
# Materials and methods

This chapter presents the processing and experimental procedures implemented to generate results on micro-structural, meso-structural and macro-structural characteristics of peach palms trunk. The microscopic observation was carried out to visualize the micro-structure; digital images processing was performed to capture the meso-structure and the macro-structure characterization was carried out in order to determine its physical and mechanical properties. Macro-structure characterization is required for description and analysis using of the existing standards that support conventional materials.



## 3.1.
## Origin of material, handling and processing

Two peach palm stems of two different ages, 6 and 20 years, were selected for the analysis. Both analyzed Peach palm stems were 18 m long. They were cut into pieces of 4.5 m and dried at room temperature (Figure 3.1). The 20 year old stem was taken from the Amazon region of Colombia (South America) in the department of Putumayo, a municipality of Mocoa (1°8'59"N - 76°39'21"O); altitude of 650m above the sea level. The 6 year old stem was taken from Rio de Janeiro in the South East region of Brazil, municipality of Silva Jardim (22°34'48.86"S - 42°26'9.40"O); 530m above the sea level. The basic features of cultivation are shown in table 3.1.

Table 3.1. Material Characterization.

| Peach Palms Date | | |
|---|---|---|
| Age (years) | 6 | 20 |
| Origin | Brazil | Colombia |
| Altitude | 530 MSL | 650 MSL |
| Temp | 21° - 26° C | 18° - 28° C |
| Soil | Clay loam to clay | Low fertility soils |
| Texture soil | Medium | Heavy |
| PH soil | Acid | Acid |

Figure 3.1. Peach Palm cut *In situ*.

Once the palm was cut in sections of 4.5 m, the material was dried at room temperature for 1 month. Subsequently, specimens were prepared for micro-structural, meso-structural and macro-structural characterization, by cutting and preparing cross-sectional specimens of 40x40x10 +/- 1 mm for microscopy, fiber volume fraction measurements and physical characterization (Figure 3.2). Also according to the results of the fibre volume fraction measurements, specimens were cut and prepared specimens of 300x40x10 +/- 1 mm for the mechanical characterization of material (Figure 3.3).

Figure 3.2. Cutting and preparing the “peach palm”, to fiber-matrix characterization and physical characterization.

Figure 3.3. Cutting and preparing the “peach palm” for mechanical characterization.

## 3.2.
## Micro-structural methods

### 3.2.1.
### Machine, Equipment and Procedure

The microstructural characterization required the identification of the cross section with microscope assistance. Initially, specimens were cut of the main peach palm trunk using a metal or alumina strip impregnated with bakelite, boron nitride, silicon carbide or diamond to reduce deformation in the specimen surface. To provide a sample surface suitable for operations in a microscope, specimen dimensions area was cut into 50 to 150 $mm^2$ and 10 to 20 mm of thickness. The orientation of the specimen surface was registered.

Specimen polishing was done by hand using abrasive discs mounted on a wheel with successive degrees of progressively finer grit. A silicon carbide was used to graded thicker alumina and polished diamond for a finer finished. Silicon carbide was 58 to 15 microns of grit. The specimen did not require any other polishing treatment to identify the fiber-matrix in microscope.

The polished specimen was placed in a Zeiss Axioplan optical microscope (OM) coupled with a Zeiss AxioCam HR digital camera (Figure 3.4). A 100X magnification objective was used to give focus by using the coarse fine screws, until the object of the specimen was clearly seen. The image was taken when the object was on focus.

The results show an digital image that identifies the parts of the cross section of the palm at the micro level.

Figure 3.4. Optical microscope (OM) Zeiss Axioplan coupled with Digital Camera Zeiss AxioCam HR.



## 3.3. Meso-structural methods

After cutting and preparing cross-sectional specimens of 40x40x10 ± 1 mm, series of materialography techniques were used for surface preparation. This method consists on polishing specimen surface in a sander belt (Figure 3.5). Then an abrasive paper of different grades from 240 to 1200 was used to remove marks and regions generated by cutting the material that do not correspond to the composition of the material. The material structures were observed with sufficient contrast, so the characteristics of interest could be described and characterized in detail. This procedure was performed previous to the procedure used by Ghavami, *et al*., 2003 for the observation of the meso-structure of bamboo. The sequence of events is shown in Figure 3.6.

Figure 3.5. Polishing specimens surface in a sander belt.

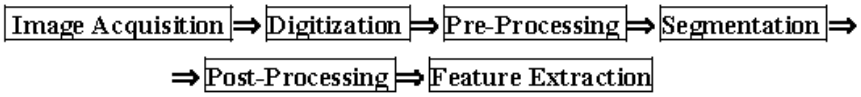


Figure 3.6. Imaging procedure of the meso-structure of peach palm through images obtained by scanner.

### 3.3.1. Machines and equipment

To select the correct method to obtain images for fibre measurement, a microscope segmentation comparison was performed by using a digital scanner with digital processing software.

Once the material was prepared, a conventional scanner was used to obtain images. The scanner is available to many researchers - becoming one of the contributions of this study to demonstrate the applicability of a simple method to obtain images. Figure 3.7 shows the conventional scanner HP3230 used and the image obtained.

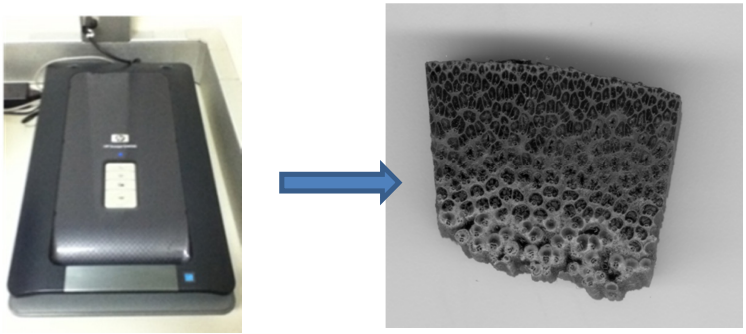

Figure 3.7. Conventional scanner HP3230 used and Peach Palm image of specimen obtained.

To analyze the images and quantify the percentage of fibre-matrix use ImageJ, an open access software for digital imaging processing. ImageJ uses a Java scripting language and was developed at the National Institute of Health for the free use. It runs as an online executable applet or on any computer with a Java 5, Microsoft Windows, Mac or Linux operating systems making it a popular platform for image processing.

The software allows the user to develop a customized plugins scanning, analysis and image processing by using the editor and Java compiler. The compiler allows to display, edit, analyze, process, save, and print images of 8-bit (256 colors), 16 bit (thousands colors) and 32-bit. ImageJ reads different image

formats such as TIFF, PNG, GIF, JPEG, BMP, DICOM, FITS, allowing time-consuming operations to be performed in parallel in order to calculate the area and statistics pixel value of user-defined selections. The plugins enable user-written that allows solving any problems of image processing and analysis. (Ferreira & Rasband, 2011).

### 3.3.2. Fiber volume fraction ($V_f$) measurements

ImageJ automatically detects the measure of interest of cross sectional images, by segmenting the binary images.

After obtaining percentage values of fibre and matrix, the volume curves fibre percentage against their position with respect to thickness were calculated. The results were compared to the ones obtained by Ghavami & Rodrigues (2003) on their study of bamboo properties.

Besides presenting the volumetric equation of the fibres, with the results of the fibre-matrix characterization of the material, make a cross-sectional analysis to determine the "representative volume element (RVE) or unit cell" (Figure 3.8) which will be used to perform physical and mechanical tests.

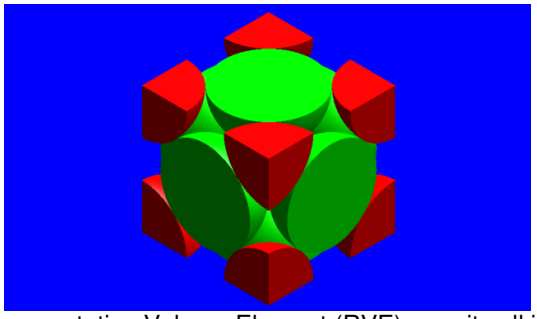

Figure 3.8. Representative Volume Element (RVE) or unit cell in 3d solid.

## 3.4. Macro-structural methods

To establish the physical and mechanical properties for classification as structural material, other types of tests were performed:

Direct Moisture Content.

Specific gravity.

Tension.

Flexion.

Torsion.

### 3.4.1.
### Test method for direct moisture Content (MC)

Moisture has been understood as the ratio between the mass of water and the mass of solids present in organic and inorganic materials, although water may be under different conditions. This means water could be incorporated in the specimen. The mechanical properties of natural materials depend on this physical property (Acha and Ghavami, 2011).

This practice is done based on the method described in ASTM D4442, which consists in seeking the water content within a peach palm specimen expressed as a percentage of dry mass, The Standard Test ASTM D4442 used has several methods. This study used the "A-method" corresponding to reference method. The methodology used is based on differences in dry mass and original mass.

ASTM D4442 recommends for wood and wood base materials a drying temperature of 103 +/- 2 ° C and a time period greater than 24 hours with subsequent intervals of 2 hours to obtain a weight difference between intervals of less than 0.01 grams. Also, a temperature of 104 ° C +/- 1 ℃ and a period of 24 hours without subsequent intervals are suggested in a previous protocol in order to prevent samples incineration.

To control relative humidity, specimens were stored in free moisture areas and protected from direct sunlight



#### 3.4.1.1.
#### Machines and equipment

Oven: The oven used, Sterlifer SX1.3, kept a stable temperature of 104 ± 1 ° C during the time required for samples to dry completely. It was determined that the relative humidity of the laboratory was below 70% and the oven is not vented to evaporate accumulated moisture inside.

So that the drying process is necessary to locate reliable 8 samples at a time in the oven thirds relative to the height, depth and width as shown in Figure 3.9.

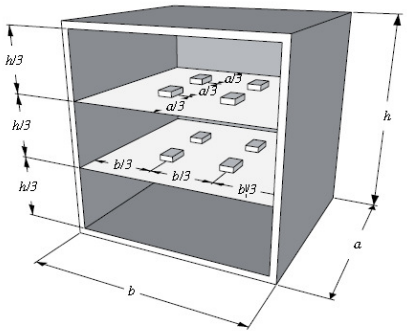


Figure 3.9. Placing samples for oven drying.

Balance: the balance used had a 0.01 g precision. Specimens weigh should be 1% by mass, therefore, the selection of the specimens had to be accurate to this weight, before recording measurements.



### 3.4.1.2. Procedure

Cross-sectional specimens of 40x40x10 ± 1 mm were stored in a place protected from sunlight and water. The wet specimens were weighed using the balance described above and values were recorded (*A* original mass).

After the material was dried to constant weight (24 hours at 104 ° C), the specimen was removed from the oven. It was necessary to allow the material to dissipate the heat energy that can be handled safely, preventing it from absorbing moisture from the atmosphere. Then, specimens were weighed.

The specimens were located in the oven at 104 ± 1 ℃ for a period of about 3 hours, or until the weight change was less than 2 times the accuracy of the balance.

### 3.4.1.3. Calculation

Specimens were weighed and dried, recorded B (oven-dry mass) and the moisture content was calculated by the eq. (3):

$$MC, \% = \frac{A-B}{B} X\ 100 \qquad (3)$$

Where:

A = original mass, g,

B = oven-dry mass, g, and

MC is the percentage of water in the sample relative to the dry mass of the specimen.

### 3.4.2.
### Test method for Specific Gravity (SG)

The practice for determining the SG was done based on the method described in the Standard Test ASTM D2395. The standard contains several methods, in this study used the A-method (Volume by measurement), prepare the specimen in regular shape with high precision. As SG is the most important single physical characteristic of woody material it may serve for the workability of the material, or the strength characteristics of a specimen or a species.

#### 3.4.2.1.
#### Machines and equipment

Caliper: A caliper was used in order to measure the specimen in all directions with an accuracy of ± 1% of measured distance (typical accuracies of the order of ± 2.5 microns).

Balance: A balance with 0.01 gram precision should be use. The mass of the specimens must be accurate to 1% by weight, therefore it is necessary to choose carefully the specimens before recording measurements.

It uses a measuring instrument with the capacity to determine the dimensions of the tested specimens with an accuracy of 0.1 mm, a scale capable of weighing to an accuracy of 0.01 g and an apparatus for determining the moisture content, as indicated in the previous section on the moisture content.

#### 3.4.2.2.
#### Procedure

Shape of Specimen: The specimens were regular in shape with right-angle corners for determination of volume by lineal measurement. The procedure is adaptable to any size of specimen or to specimens of any moisture content. If the surfaces of the specimen are smooth and sufficient measurements are taken, the volume can be obtained with considerable accuracy. Special care was taken to measure very small or thin specimens.

Measurement: length (L), width (w), and thickness (t) of the specimen were measured in a sufficient number of places to ensure an accurate indication of volume. In small specimens, uniform in size, one or two measurements of each dimension was enough; in larger specimens the number of measurements depended on the uniformity of the specimen, but at least three measurements of each dimension were required. The dimensions of test specimens were measured with a precision of ± 0.3 % or less, and the mass was determined with a precision of ± 0.2 % or less.

Mass: the final (oven-dry) mass of a specimen was determined from the protocol and sample used for moisture content (MC). The drying of specimens was done in an oven maintained at 104 ± 1℃.

### 3.4.2.3. Calculation

Specific gravity (SG) was calculated based on oven-dry mass and volume at test using the following eq. (4):

$$S = Km_o/V = (Km_o)/(Ltw) \quad (4)$$

Where:

$m_o$= final (oven-dry) mass of specimen

$V = L\ t\ w$, volume of specimen as measured at the time of the test;

$L$ = length of specimen;

$t$ = thickness of specimen;

$w$ = width of specimen; and

$K$ = constant whose value is determined by the units used to measure mass and volume:

$K$ = 27.68 when mass is in lb and volume is in $in^3$,

$K$ = 453.59 when mass is in lb and volume is in $cm^3$,

$K$ = 453 590 when mass is in lb and volume is in $mm^3$,

$K$ = 0.061 when mass is in g and volume is in $in^3$,

$K$ = 1.00 when mass is in g and volume is in $cm^3$, and

$K$ = 1000 when mass is in g and volume is in $mm^3$.

By calculating the average of the results obtained from individual test samples, reporting those values as the average for the SG of the samples tested, the results could express the nearest g / $cm^3$.

### 3.4.3.
### Tensile Properties test.

This practice is based on ISO314 and ASTM D3039 standards to find the tensile properties: Strength (TS) and Modulus of elasticity (TMOE). Initially the test specimens required constant rectangular section according to standard ASTM3039 and other specimens with reduced centrally according to standard ISO314, at which a load is applied longitudinally until material failure occurs. The force and displacement data were collected throughout the test in order to develop the constitutive relations for natural materials.



#### 3.4.3.1.
#### Machines and equipment

Tensile test use a hydraulic servo testing - universal machine I5960 (Instron USA) with a capacity of 500 kN. The tests are controlled by moving the crosshead at a speed of 1 mm / min with boundary conditions fixed-fixed.

Caliper: A caliper was used in order to measure the specimen in all directions with an accuracy of ± 1% of measured length (typical accuracies of the order of ± 2.5 microns).

The data collector (Clip gage): A clip gage was used with an accuracy of ± 1% in order to avoid the specimen alteration with external sticking elements.

The grip or specimen holding jaws: Friction workforce was applied to provide the lateral force, sufficient to prevent slippage of the specimen during testing.

The specimens had a tolerance of ± 1% on all its dimensions and the recommended size is shown in the following Figure 3.10, although it is possible to modify the dimensions so that the specimen represents the overall composite.

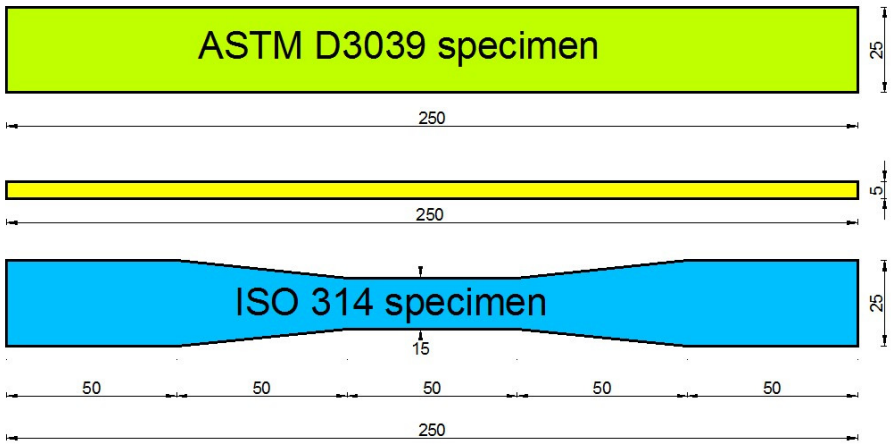


Figure 3.10. Schematic of specimen for tensile tests on composite materials according to ASTM D3039 and ISO314.



### 3.4.3.2. Assembly and Test conditions

The number of specimens with their main characteristics as fibre orientation, moisture and specific gravity, among others, should be recorded before start, besides external data temperature and relative humidity. If those parameters are not used within the recommended conditions, it may affect the test results.

The length (L), width (b) and thickness (t) of the specimen, was measured with the accuracy wanted and any deformation observed was recorded. The width and the thickness was measured at three positions to average cross-sectional area

Figure 3.11 shows the final assembly test with the clip installed to record the specimen elongation with the application of load with a constant cross speed of 1mm/min, so that the fault is reached in a time of 1min to 10min, recording applied load and strain throughout the test.

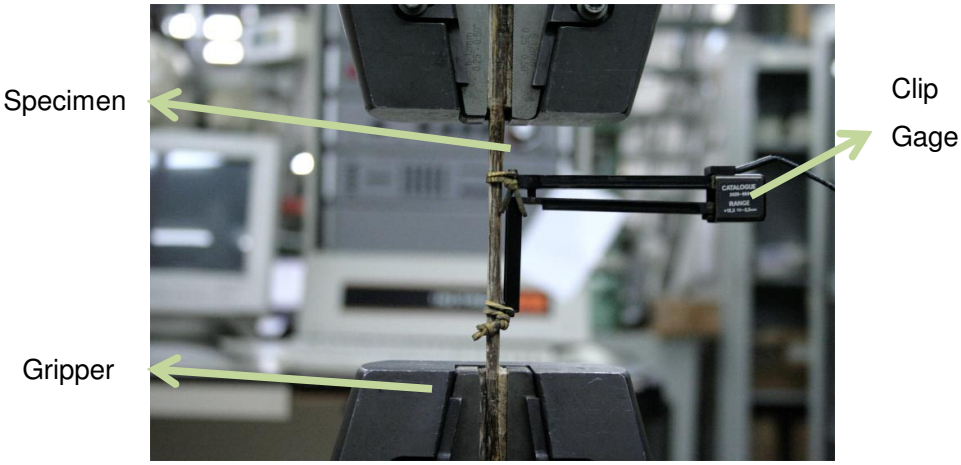


Figure 3.11. Tensile Test Set up

### 3.4.3.3. Calculations

The results reported by the laboratory gather the information necessary to perform the following calculations.

The tensile stress is calculated using the eq. (5):

$$\sigma = \frac{P}{A} \tag{5}$$

Where, P is the force applied at the instant "i" and A is the cross sectional area of the specimen. The tension set was calculated as eq. (6):

$$\epsilon = \frac{\delta}{L} \tag{6}$$

Where, $\epsilon$ is the strain, $\delta$ is a displacement and L the length of the clip gage.

When performing the material constitutive relation (stress – strain), the modulus of elasticity in tension was calculated as the slope of the stress-strain curve where the stress and strain differences must cover approximately straight sections of the graph. When two or more regions with different slopes were seen, the module had to be reported for each region.

### 3.4.3.4.
### Statistical Analysis

Test results in natural materials characterization is a random variable data of which population distribution, in general, is unknown. Therefore, it was necessary to identify the probability of distribution that better goodness-of-fit the experimental data, to estimate the parameters of interest. In that identification, the graphic methods can be used, being the probability plot the most common. The points of this graph were determined using a combining parametric and non-parametric method. The fitted line for this graph is the representation of the experimental data percentiles, which were obtained using order statistics, maximum likelihood estimate of probability distribution with better goodness-of-fit to the experimental data and the inverse cumulative distribution function.

## 3.4.4.
## Bending and torsion properties

### 3.4.4.1.
### Experimental approach

A testing machine was used for loading a cantilevered specimen at a central point and eccentric point to estimate the values of the flexural modulus, and the modulus of rigidity of the peach palm. Data applied force and the arrow was collected during the test in order to insert them in the theory of bending and twisting of prismatic bars of square cross section, which was used in the test.

### 3.4.4.2.
### Machines and equipment

A testing machine with a capacity of 100 kN and a load cell LC703-150 (Omega Engineering, Inc., USA) was used. The tests were controlled by the movement of the head of the machine at a speed of 1 mm / min with boundary conditions fixed-cantilever for bending and fixed-simple support for torsion. Also, an OM2-162 (Omega Engineering, Inc., USA) was used to filter and amplify the signals and a PMD acquisition card LS-1208 (Measurement Computing Corporation, USA) was used to digitize the signals at a frequency of 21 Hz, applied load with a displacement control at a speed of 1 mm / min. A specimen clamp is used at one end of the cylinder, leaving the other end free to be tested

for pure bending, and torsion. The specimen is clamped at one end and the other a spherical head support is used to allow torsion.

Caliper: A caliper was used in order to measure the specimen dimensions in all directions with an accuracy of ± 1% of measured distance (typical accuracies of the order of ± 2.5 microns).

The collector of information: it was performed with an accuracy of ± 1% deformation of the specimen. For this test an LVDT LD610-15 (Omega Engineering, Inc., USA) was used.

### 3.4.4.3. Assembly and Test conditions

Length (L), width (b) and thickness (t) of the specimen were measured and any imperfection was recorded. Width and thickness were measured at three positions to average cross-sectional area.

For the flexion and torsion test, the specimen was mounted in the holder so that the test specimen could have an aligned support with the line of action of the LVDT and load was applied with a constant speed of approximately 1mm/min. The process was repeated cyclically three times from 1min to 10min. The data from the applied load was recorded on the load cell and given by the LVDT displacement throughout the test. Figure 3.1 shows the test setting.

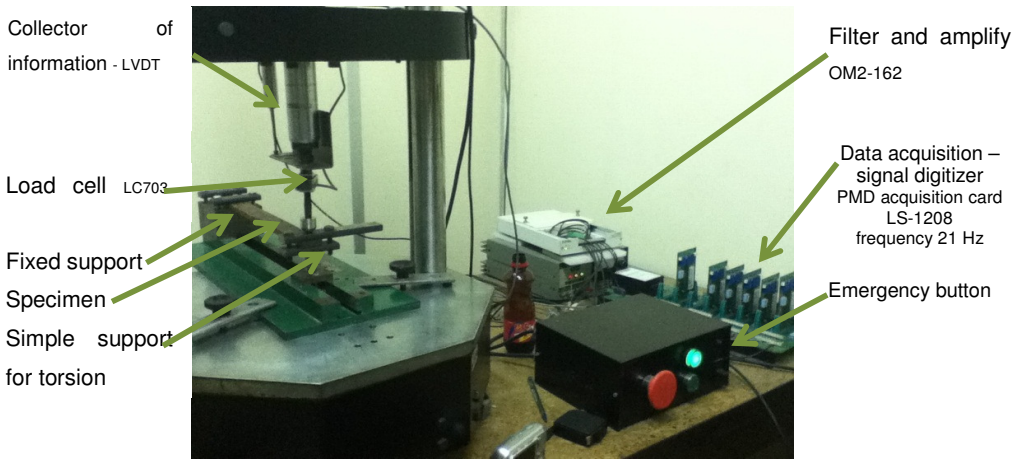


Figure 3.12. Bending and torsion test machine and Assembly.

### 3.4.4.4. Calculations and Theory of operation of the cantilever beam test apparatus

Consider a specimen fixed at one end and free at the other, also known as a cantilever beam, to which a concentrated F force is applied on the free end and a P force is uniformly distributed along the length of the beam. Figure 3.13 shows a cantilever beam of length L, constant rectangular section, P weight evenly distributed along its length and supporting a concentrated F load at the free end. The bending moment is a function of the $x$ coordinate given is the moment regarding the neutral axis of any section.

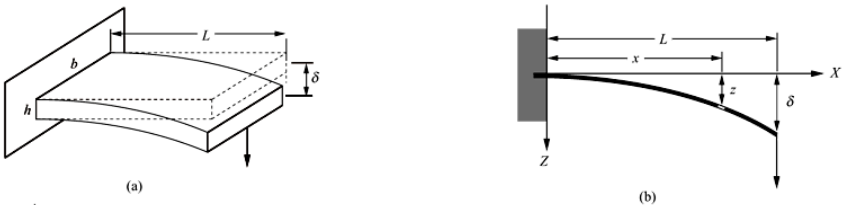


Figure 3.13. a. Bending of a cantilever beam with a rectangular section when a concentrated load is applied. b. Elastic cantilever beam.

Considering the cantilevered sample, the bending moment (Mf) due to the loading point (F) applied to the end of the beam with respect to the section located at a distance x can be calculated using Eq.

$$M_F(x) = F(L - x) \tag{7}$$

While the bending moment due to distributed load along the length of the beam has the eq. (8):

$$M_P(x) = \frac{P}{2L}(L - x)^2 \tag{8}$$

The total bending moment eq. (9) is the sum of the eq. (7) + eq. (8),

$$M_P(x) = F(L - x) + \frac{P}{2L}(L - x)^2 \tag{9}$$

Substituting eq. (9) in the differential bending equation in the elastic range for small displacements (small slopes) leads to eq. (10):

$$\frac{d^2z}{dx^2} = \frac{1}{(EI_n)^n}\left[F(L - x) + \frac{P}{2L}(L - x)^2\right]^n \tag{10}$$

Where for the cantilever beam boundary conditions are z = 0 and $\frac{d\ z}{dx} = 0$

In a general eq. (10) is a second order nonlinear differential equation and is solved numerically. However, it is possible to consider some particular cases of great interest to demonstrate that integration is immediate.

To calculate the displacement and then the bending elastic modulus it is enough to consider P = 0 in eq. (10), which yields the eq. (11) differential equation of the elastic:

$$\frac{d\ z}{dx} = \left(\frac{F}{(EI_n)}\right)^n (L-x)^n \tag{11}$$

This can be integrated immediately considering the boundary conditions at the fixed x = 0 (z = 0 and $\frac{d\ z}{dx} = 0$), yielding the eq. (12) for the elastic cantilever beam:

$$z = \frac{1}{(n+1)(n+2)}\left(\frac{F}{(EI_n)}\right)^n [(L-x)^{n+2} + (L-x)L^{n+2} - L^{n+2}] \tag{12}$$

The deflection d (see fig. 3.13b) at the free end is obtained by substituting x = L in eq. (12) leads to eq. (13):

$$\delta = \frac{L^{n+2}}{n+2}\left(\frac{F}{EI_n}\right)^n \tag{13}$$

In the particular case, where it is assumed that peach palm specimen is a linear isotropic elastic material, n = 1, the value of Elastic Modulus E can be computed as eq. (14):

$$\delta = \frac{FL^3}{3EI_1} \tag{14}$$

Where:

F= Force. (N)

L= Long. (m)

E= Elasticity Modulus. (MPa)

I = Inertia. ($m^4$)

In the case of the element subjected to torsion, the tested object was a prismatic bar of square cross section under a uniform torsion as shown in Figure 3.14 where planar cross sections normal to the axis of the bar did not remain

plane during the deformation (displacement experiment warping) and suffered distortion in its own plane. This implied that it was not possible to establish a simple theory as an elementary theory of torsion.

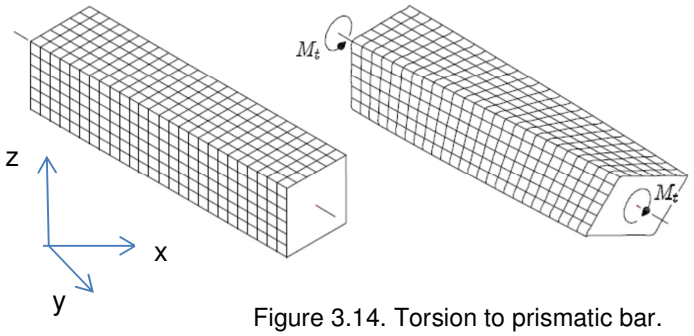


Figure 3.14. Torsion to prismatic bar.

Saint-Venant obtained the exact solution to the problem of non-uniform torsional arbitrarily prismatic parts, assuming that the deformation is uniform, and consists of:

- A rotation as a solid rigid in its plane sections, and
- A warping of the sections out of its plane.

The problem must be formulated by using the elastic continuum model L. Prandtl proposed in 1903. Tensions were expressed as a function of tension also called Prandtl function, so that (eq. (15)):

$$\tau_{xy}(x,y,z) = \frac{\partial \Phi(y,z)}{\partial z} \qquad \tau_{xz}(x,y,z) = \frac{-\partial \Phi(y,z)}{\partial y} \tag{15}$$

The problem was reduced by finding a function that satisfies the compatibility equations and boundary conditions of uniform torsion. This implied that the function satisfied the differential eq. (16) with the considered boundary condition for pure torsion.

$$\nabla^2 \Phi(y,z) = \frac{\partial^2 \Phi(y,z)}{\partial y^2} + \frac{\partial^2 \Phi(y,z)}{\partial z^2} = -2G\theta \tag{16}$$

The analytical solution of uniform torsion of prismatic bars with non-circular section of the above approach is generally very complex. However, different expressions have been obtained as a solution to different types of twisted sections. Figure 3.15 shows the rectangular sections, which marked the maximum shear stress point as it is suggested in the Timoshenko´s book that solves torsion bars uniform prismatic square and rectangular cross sections

according to the equations in Table 3.2 expressed by the coefficient values of Table 3.3.

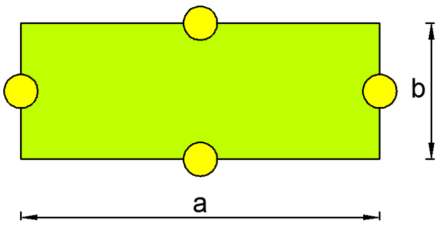


Figure 3.15. Points of maximum shear stress in square and rectangular cross sections, with uniform torsion.

Table 3.2. Expressions of values the maximum shear stress and rotation angle per unit length.

| Section | $T_{max}$ | θ |
|---|---|---|
| Square | $\frac{4.81M_t}{a^3}$ | $\frac{7.1M_tL}{a^4G}$ |
| Rectangular | $\frac{M_t}{\alpha ab^3}$ | $\frac{M_t}{G\beta ab^3}$ |

Table 3.3. Coefficient values (alpha and beta) depending on the ratio between the width (a) and thickness (b) of the rectangular section, used in expressions (Tau max and theta).

| **a/b** | **1** | **1.5** | **2** | **2.5** | **3** | **4** | **6** | **10** | ∞ |
|---|---|---|---|---|---|---|---|---|---|
| **α** | 0.208 | 0.231 | 0.246 | 0.256 | 0.267 | 0.282 | 0.299 | 0.312 | 0.333 |
| **β** | 0.141 | 0.229 | 0.229 | 0.246 | 0.263 | 0.281 | 0.299 | 0.312 | 0.333 |

In the particular case of a rectangular section and a / b = 4 the sheared modulus (G) shown eq. (17):

$$G = \frac{M_t}{\theta \beta ab^3} \tag{17}$$