



DRAFT TANZANIA STANDARD

Textiles – Determination of Density of flax fibre

DRAFT FOR STAKEHOLDERS' COMMENTS ONLY

TANZANIA BUREAU OF STANDARDS

0 FOREWORD

This Draft Tanzania Standard describes two types of methods to determine the density of flax fibre of any length attained by a specified percentage of fibres in a test beard which constitutes a uniformity index.

Fibre density is useful in the evaluation of new materials, determination of fibre strength and modulus of a single or bundle of fibres. For composite industries, fibre density is used as a property to determine reinforcement volume and void volumes based on reinforcement mass and laminate density.

In the preparation of this Draft Tanzania Standard, the assistance was derived from ASTM-D8171 – 18 Standard Test Methods for Density Determination of Flax Fibre published by the American Society for Testing and Materials.

In reporting the results of a test made in accordance with this Draft Tanzania Standard; if the final values observed or calculated is to be rounded off; it shall be done in accordance with TZS 4: (see clause 2)

1. SCOPE

This Draft Tanzania Standard specifies two types of methods for determination of the density of flax fibre of any length. The method is applicable to the fibres taken from raw or partially processed flax, but not to fibres taken from blends of flax yarns or fabrics.

- 1.1 The first test method A is by gas pycnometry which is considered to be more accurate where specimen conditioning (oven drying) can be followed or outgassing is mitigated by purge settings.
- 1.2 Test Method B uses similar equipment set up but is used as an alternative to test method A in which high precision is not required. It is preferred in cases where outgassing effects in Test Method A cannot be addressed by oven drying or purges settings and prevent Test Method A from producing valid results.
- 1.3 In test method B-Buoyancy (Archimedes) Method; the sample is weighed in air and weighed in certified soybean oil that will thoroughly wet the sample and is of a lower density. The difference in weight of the samples in the two media is the buoyancy force. This force is converted to sample volume by dividing it by the soybean oil density. The sample weight in air divided by the sample volume equals the sample density.

2. NORMATIVE REFERENCE

For the purpose of this Draft Tanzania Standard the following reference shall apply: -

- a) TZS 534 – Standard atmospheres for conditioning and testing of textiles.
- b) TZS 325 – *Textile fibres – some methods of sampling for testing.*

3. TERMS AND DEFINITIONS

For the purpose of this Draft Tanzania Standard the definitions related to flax fibre shall be as outlined in Glossary of terms (TZS...)

3.1 impurity

material (such as, but not limited to, water, shives or dirt) present in the sample that, if not removed or accounted for would obfuscate the density measurement of targeted material in the sample.

3.2 symbols;

M_1 = weight of suspension wire in air, g.

M_2 = weight of suspension wire in liquid (to immersion point), g.

M_3 = weight of suspension wire plus item whose density is to be determined (in air), g.

M_4 = weight of suspension wire plus item whose density is to be determined (in liquid), g.

$M_3 - M_1$ = weight of item for density to be determined in air, g.

$M_4 - M_2$ = weight of item for density to be determined in liquid, g.

ρ_f = density of fibre, g/cm³

ρ_i = density of the impurities, g/cm³

ρ_{if} = density of the combined fibre and impurities, g/cm³

ρ_s = density of standard, g/cm³

ρ_l = density of liquid, g/cm³

ρ_{sur} = density of surfactant, g/cm³

ρ_w = density of water, g/cm³

P_1 = equilibrium pressure of the sample chamber after closing the input valve, kPa.

P_2 = equilibrium pressure of the sample and expansion volume chambers after opening the connection valve, kPa.

s = standard deviation.

V_e = volume of the calibrated expansion volume chamber, cm^3

V_f = measured volume of the fibre specimen, cm^3

$V_{\min}$ = equipment specific minimum volume of solid material required in each test to produce accurate results.

V_s = volume of the calibrated sample chamber, cm^3

W_1 = weight of specimen before testing, g.

W_2 = weight of specimen after testing, g.

W_g = change in weight of the specimen during the test, g.

x_i = mass of impurity as a percentage of the total mass of the measured fibre, g.

4. SAMPLING

4.1 The sample of flax fibre for testing shall be taken according to TZS 325. However other methods of sampling may be used upon agreement between the interested parties.

4.2 The samples shall be free from impurities to prevent bias because the measured fibre density will be a combination of the average densities of the fibre and impurity. The formula below may be used to calculate the effect of the impurity on the density of the material.

$$p_{if} = \frac{(100 - x_i)\rho_f + x_i(\rho_i)}{100}$$

4.3 For the statistical purposes, a minimum of five specimens for each sample should be tested. The test specimen weight shall be a minimum of 0.5 g

5. CONDITIONING AND TESTING ATMOSPHERE

5.1 Before testing, the fibre should be conditioned to equilibrium at 0 % relative humidity (RH) at the temperature the testing will occur. This will remove moisture that can outgas during the testing.

5.2 The testing atmosphere shall be conditioned as specified in TZS 532 i.e. a relative humidity of $(65 \pm 2\%)$ and a temperature of $(20 \pm 2)^\circ\text{C}$. In tropical regions, a temperature of $(27 \pm 2)^\circ\text{C}$ may be used, subject to agreement between the interested parties.

5.3 If the sample is expected to be tested without conditioning, its moisture content should be included in the report and the sample should be evaluated for outgassing and, if required, outgassing mitigation methods used during testing.

5.4 The certified soybean shall be conditioned to a test temperature, typically 23°C .

6. TEST METHOD A: GAS PYCNOMETRY

6.1 This method is done under the release of gas such as water vapour or volatile organic compound, interfere with the measurement of the gas pressure and, if not mitigated, can invalidate the results. The changes in temperature can cause changes in volume. The equipment and specimens should be given sufficient time to reach a steady state condition.

6.2 APPARATUS

6.2.1 Available the commercial gas pycnometer instruments with specified instructions pertaining to loading/unloading and operation manual to meet the minimum requirements. The laboratory setup should include the following:

6.2.3 *Calibrated sample volume chamber*-a chamber having a volume suitable to the sample size and calibrated to the nearest 0.1 cm³. The chamber shall be accessible to the operator such that a sample can be loaded into the chamber and then sealed for testing. The chamber should have a known volume (V_s cm³).

6.2.4 *Calibrated expansion volume chamber*; a chamber of known volume (V_e cm³) that is calibrated to the nearest 0.1 cm³

6.2.5 *Pressure transducer*, capable of measuring 0 to 175 kPa with minimum volume displacement and linear within 0.1 %.

6.2.6 Pressure relief valve, used to avoid the over pressurization of the transducer.

6.2.7 *Filter*, prevents powder or dust from contaminating the transducer and valves.

6.2.8 *Input flow control valve*, controls the rate of pressurization.

6.2.9 *Output flow control valve*, controls the rate of depressurization.

6.2.10 *Connection valve*, connects the calibrated sample volume chamber with the calibrated expansion volume chamber.

6.2.11 Nonporous calibration standard(s), object(s) of known volume that can be used in different configurations to calibrate the chambers for different volumes of sample.

6.2.12 *Digital meter*, used for reading the pressure to 0.007 kPa.

6.2.13 *Analytical balance*, capable of weighing to 0.001 g.

6.3 The minimum volume of solid material (V_{min}) required to produce accurate measurements is equipment specific and shall be known before conducting testing. The volume can be based on manufacturer recommendations or determined through testing. If done through testing, V_{min} would be the volume in which increasing the volume of the solid material being measured does not change the value of the results beyond what would fall within sample or standard deviation.

6.4 PROCEDURE

6.4.1 Weigh the specimen immediately before testing and record the weight as W_1 in g.

6.4.2 Load the sample chamber with the specimen and seal.

6.4.3 Vent the sample chamber to atmosphere and zero the pressure transducer. All pressure references in the procedure and calculations should refer to gauge pressure.

6.4.4 Close all vents and the valve separating the sample chamber from the expansion chamber.

6.4.5 Perform a purge cycle to prepare the specimen before testing. It is recommended that the purge cycle be a series of pulses, a fixed purge time, or exposure to vacuum. For pulses, a minimum of nine pulses should be used and the system should pressurize to 20 kPa before venting and return to less than 3 kPa before starting the next pulse. For purge times, the sample should be exposed to a minimum of 2 min of continuous flow of the test gas. For vacuum purge systems, the sample should be exposed to a minimum of 5 min of vacuum before testing. Report the purge cycle and settings used.

6.4.6 Pressurize the sample chamber to the target test pressure. The target pressure selected should be between 105 and 125 kPa above ambient and remain consistent for all specimens.

6.4.7 Close the input valve to the sample chamber and wait a minimum of 10 s for stability. Record the pressure as P₁ kPa and the wait time in seconds. The wait time for stability should be consistent between all specimens.

NOTE 1 - if the pressure decreases or increases outside of usual measurement flutter, the system may have a leak, the gas may be penetrating the solid, or outgassing may be an issue. Discontinue testing and consult the manufacturer's documentation regarding leaks, gas penetration, or modifying purge routines.

6.4.8 Open the valve between the sample chamber and the expansion volume and wait for stability using the same wait time as previous. Record the pressure as P₂ kPa.

6.4.8 Open the vent valve and allow the chambers to fall to 0 kPa (ambient).

6.4.9 Repeat 13.7 – 13.10 a minimum of six times to collect multiple determinations on the specimen.

6.4.10 Remove the specimen from the sample chamber.

6.4.11 Weigh the specimen and record the weight as W₂ g.

6.4.12 Repeat the entire procedure for each specimen

7. CALCULATION AND EXPRESSION OF RESULTS

7.1 For each specimen in the sample:

7.1.1 Calculate and record the weight change (if any) as follows:

$$\Delta W_g = W_2 - W_1$$

7.1.2 Mass loss during testing may indicate outgassing and should be considered when determining the validity of the results.

7.1.3 Calculate the specimen volume for each of the runs (excluding the first three) as follows:

$$V_f = V_R + \frac{V_e}{1 - \left(\frac{P_1}{P_2}\right)}$$

Where: P₁ = Equilibrium pressure of the sample chamber after closing the input valve, kPa, and P₂ = Equilibrium pressure of the sample and expansion volume chambers after opening the connection valve, kPa.

7.1.3 During the first three runs, the equipment may not have reached a steady state condition and small thermal differences between the chambers and gas may cause results with greater deviation than the remaining runs.

7.1.4 Calculate the specimen density using the average specimen volume and the final weight of the specimen as follows:

$$\rho_f = \frac{W_2}{V_f}$$

7.1.5 Calculate the sample average density from the specimen densities.

8. TEST METHOD B—BOUYANCY (ARCHIMEDES) METHOD

8.1 Temperature—the temperature of the certified soy bean oil shall remain constant within a tolerance of 61 °C, since the soybean oil density will change with temperature.

8.2 Sample Wetting (Entrapped Air)—since this test method is very dependent on buoyancy, any entrapped air in the sample will change the measured density and not give a true material density. Ensure visually that the sample does not contain entrapped air bubbles.

8.3 Immersion Point— the sample is lowered into the soybean oil and overall liquid level should be the same throughout determinations for Procedure A. This may be done by putting a line for the desired soybean oil level on the outside of the container.

8.4 Apparatus

8.4.1 The kits or custom laboratory setups should include the comparable or better of the following:

8.4.2 *Thermometer*, capable of reading the test temperature during the test to 0.1 °C.

8.4.3 *Balance*, analytical, capable of weighing to 0.0001 g.

8.4.4 *Balance stand*, depending on the type of balance used; two recommended stands are shown in Figs. 1 and 2.

8.4.5 *Suspension wire*, nickel or stainless steel, approximately 0.4 mm in diameter, cut and shaped to match the system used.

8.4.6 *Density standard*, a solid piece of borosilicate glass (approximate density 2.2 g/mL) of known density to four significant figures as determined by water immersion (Terminology E12)

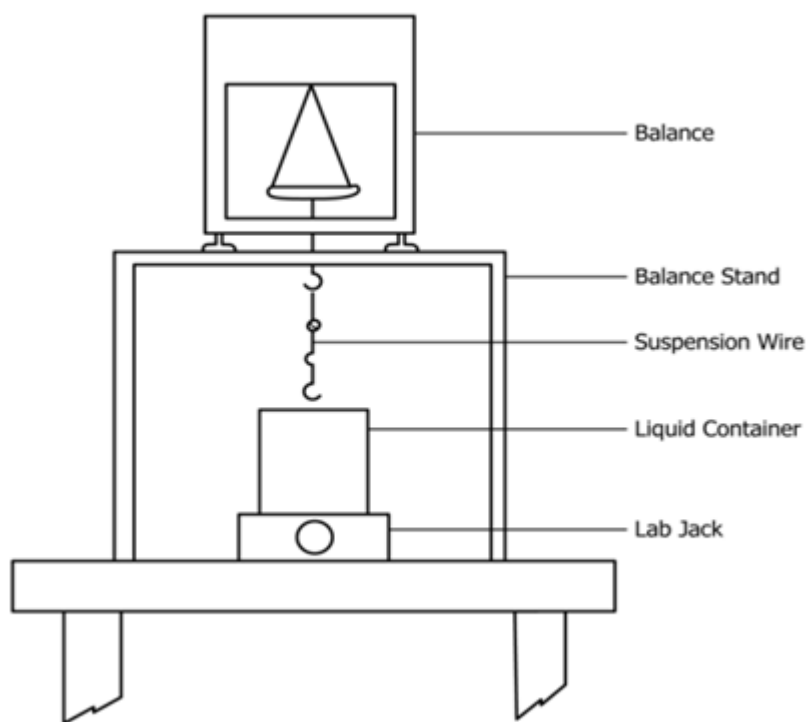


Figure 1: Density Apparatus

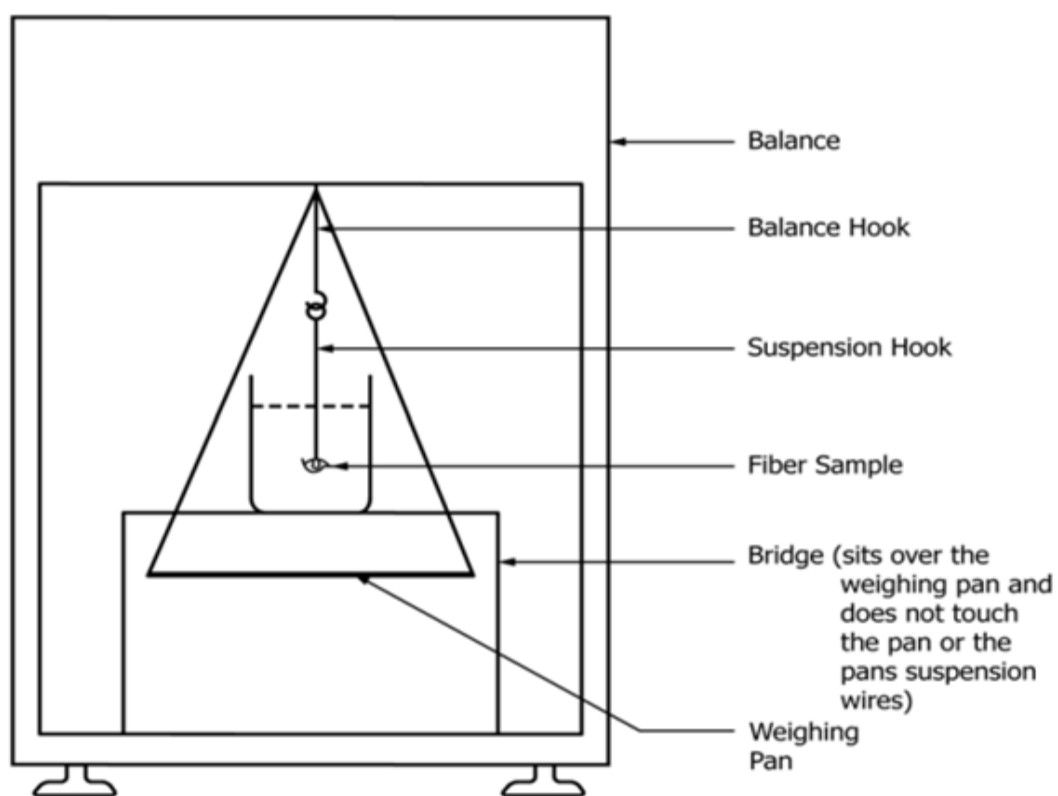


Figure 2: Density Apparatus (Alternative)

8.4.7 Vacuum desiccator (with pump), an airtight container in which a low vacuum (less than 75 kPa [560 Torr]) can be maintained.

8.4.8 Container, glass or other transparent container resistant to a liquid medium is recommended.

8.4.9 Immersion liquid, certified soybean oil should be used as the immersion liquid. The specific gravity of the certified soybean oil shall be determined shortly before and after each use.

8.4.10 Gloves, clean, non-linting (or lint-free) gloves for use when handling fibres.

8.4.11 Laboratory jack, heavy duty precision.

8.4.12 Hydrometer, capable of reading liquid density.

8.4.13 Certified soybean oil with the average density of 0.906 ± 0.05 g/ml

8.5 Reagents and Materials;

The reagent chemicals shall be handled by laboratory workers with general training in the safe handling of chemicals. Unless otherwise indicated, all reagent chemicals shall conform to **TZS.....** Other grades may be used provided that the chemical is sufficient to permit its use without lessening the accuracy of the determination.

8.6 Preparation of Apparatus

8.6.1 The apparatus is shown in Figure 1 and 2. The balance stand shall be firmly secured to a stable surface with the balance resting on the stand directly over the hole provided for the suspension system. Place the immersion fluid container on the laboratory jack directly under the suspension hook.

8.6.2 To prevent stray air currents between the bottom of the balance and the top of the stand, it is advisable to shield this area. If excessive vibration is observed while weighing, vibration damping pads shall be used.

8.7 Calibration and Standardization

8.7.1 All measuring equipment shall have certified calibrations that are current at the time of use of the equipment.

8.7.2 Density Standard Calibration; fill the immersion fluid container $\frac{3}{4}$ to $\frac{7}{8}$ full with distilled water. Place the container on a collapsed laboratory jack and zero the balance. Attach the suspension wire and weigh, record as M_1 . Raise the laboratory jack to the immersion point of the suspension wire and record the weight as M_2 . Rinse the wire with acetone and let air dry. Attach the wire plus glass standard and weigh, record as M_3 . Again, raise the jack to the immersion point and weigh, Record as M_4 . Lower the jack, remove the standard plus wire, and rinse them with acetone to dry. Measure the temperature of the water to 0.1 °C and record the water density at that temperature.

8.7.2 Immersion Fluid Standardization; this calibration step should be done at the beginning of each series of density determinations with a new set of specimens when a new batch of immersion fluid is to be used or there is temperature change greater than 61 °C. Fill the clean and dry fluid container $\frac{3}{4}$ to $\frac{7}{8}$ full with certified soybean oil and allow it to come to temperature equilibrium. Proceed by weighing the suspension wire, record as M_1 . Raise the laboratory jack to the immersion point of the

suspension wire and record the weight as M_2 . Rinse the wire with acetone and let air dry. Attach the wire plus glass standard and weigh, record as M_3 . Again, raise the jack to the immersion point and weigh, record as M_4 . Remove the wire and standard and rinse with acetone.

8.8 Procedure

8.8.1 Select and wrap the specimen (the end of a small winding cone is suitable) and intertwine the ends of the fibre tow to prevent unravelling. Proceed by weighing the suspension wire, record as M_1 . Raise the laboratory jack to the immersion point of the suspension wire and record the weight as M_2 . Rinse the wire with acetone and let air dry. Attach the specimen to the wire and weigh, record as M_3 .

8.8.2 Raise the laboratory jack to the immersion point of the suspension wire. Remove the wire and soak in the certified soybean oil. Put the container with the specimen and certified soybean oil into a vacuum desiccator and pull vacuum to approximately 10 mm Hg or until boiling of the solution begins. Hold vacuum for a minimum of 3 min or until all trapped air on the fibre surface is removed.

8.8.3 Re-attach the specimen and wire to the balance. Again, raise the jack to the immersion point and weigh, record as M_4 . Remove wire and standard and rinse with acetone and repeat each specimen.

9. Calculation and Interpretation of Results

9.1 Density of glass standard, g/mL:

$$\rho_s = \frac{(M_3 - M_1)\rho_w}{((M_3 - M_1) - (M_4 - M_3))}$$

Where:

ρ_w = density of water, and

ρ_s = density of the glass standard.

9.1.1 The density of the standard should remain consistent to 60.0001 g/mL.

9.1.2 Density of immersion liquid (Certified Soybean Oil), g/mL:

$$\rho_s = \rho_s \frac{((M_3 - M_1) - (M_4 - M_2))}{(M_3 - M_1)}$$

Where:

ρ_l = density of certified soybean oil, and

ρ_f = density of the glass standard.

9.1.3 Density of fibre specimen, g/mL:

$$\rho_f = \frac{(M_3 - M_1)\rho_l}{((M_3 - M_1) - (M_4 - M_3))}$$

Where:

ρ_l = density of certified soybean oil, and

ρ_f = density of the fibre specimen.

10. Report

10.1 The test report shall include the details of any operations not specified in this Draft Tanzania Standard, or incidents likely to have had an influence on the results.

10.1.1 The type of instrument used and the specific test method used (test Method A or B)

10.1.2 Complete test parameters including test temperature in degrees Celsius; and the specimen conditioning performed before testing.

10.2 For Test Method A, the report shall include the following additional information:

10.2.1 The source of the sample, such as raw stock in process stock, waste;

10.2.2 The number of specimens tested;

10.2.3 The immersion liquid (certified soybean oil) used, its temperature, each measured density and average, g/mL, and a measure of the degree of variation in the density such as standard deviation.

10.2.4 The moisture content of the sample (if the sample was not dried before testing);

10.2.5 The analysis gas and pressure used in testing;

10.2.6 The purge process and the settings selected;

10.2.7 The average density and standard deviation of the sample; the weight change before and after testing (each specimen), the calculated volume of each of the runs (except the first three), the average volume and standard deviation, the calculated density, and the standard deviation.

NOTE 3

To allow the results to be compared across manufacturers, only flax fibre samples that are clean, low dust, and contain no more than 5 % shive by weight or have had the effect of impurities removed through calculation can be reported as “fibre density” in the report and all other samples should have density reported as “sample density”.

11. Precision and Bias

11.1 At the moment, the data required for the development of a precision and bias statement are not available for these test methods (A or B). However, Table 1 can be used to evaluate the test methods which contain the accuracy needed by the testing agent for same flax samples.

TABLE 1: The Test Results of Density Measurements with Methods A and B for the Same Flax Sample.

SN	Method	Number of Specimens	Density of Flax Fibre, (g/cm ³)
1	Method A	10	1.49±0.002
2	Method B	10	1.47± 0.013