Establishment of Protocols for Natural Fibre Density Measurement

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Biomaterials Back to the Future
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Introduction

• Objective

• Project Structure
  • U of M / CIC collaboration

• Work Completed
  • Fibre density testing
    • Linear Density and Diameter Measurement
    • Helium Pycnometry
    • Archimedes Method
  • Evaluation of test methods

• Results
  • Recommendations for natural fibre quality assessment
  • Future work
Objective

- Recommend a method for the testing of natural fibre density
  - Current methods investigated are all very qualitative
- Industrial Applications:
  - Quality control for raw materials to be used in composites
  - Predict performance of manufactured parts utilizing natural fibres
  - Aid in part design and construction
    - e.g. part with required V_f %;
      - Measure total fibre mass going into a part
      - Measure fibre density
      - Calculate amount of resin required for a desired fibre volume fraction
Project Structure: Partnerships

• CIC
  • Project management
  • Technical support

• U of M Textiles Department
  • Research test methods
  • Perform laboratory trials
  • Document results

• Schweitzer-Mauduit Canada, Inc
  • Provide fibre samples
Project Structure: Work Plan

• Phase 1 – Evaluation Plan
  • Selection of potential test methods
  • Development of evaluation criteria

• Phase 2 – Laboratory Trials

• Phase 3 – Results Assessment
  • Final report
  • Work instructions
Work Completed: Evaluation Plan

• Existing test methods identified through a literature review
  • Academic publications
  • Current standards – ASTM & ISO

• Initial selection screened to produce short list for further investigation through laboratory trials:
  • Diameter and Linear Density Measurement
  • Helium Pycnometry
  • Archimedes Method
Diameter and Linear Density Measurement

• Material required is a single long fibre
• Equipment required;
  • Measuring Scale
  • Balance
  • Optical Fibre Diameter Analyzer
  • Stage Micrometer
• Linear Density and Diameter measured in accordance with ASTM standards D1577-01[3], D2130[2] and D6500[10]
• Fibre samples dried at 60°C for 72 hours then placed in conditioning room at 65 ± 2% relative humidity for 24 hours
Diameter and Linear Density Measurement

- Linear Density calculated by measurement of mass and length of specimen
  - Fibre mass measured on balance, $m$
  - Fibre length measured with ruler, $L$
  - Linear Density calculated; $\rho_L = \frac{m}{L}$
- Cross-sectional diameter of fibre specimen measured on projection screen, $D$
  - Cross-sectional area calculated; $A = \frac{\pi D^2}{4}$
- Fibre Density calculated; $\rho_f = \frac{\rho_L}{A}$
Helium Pycnometry

• Material required is a fibre bundle
• Required equipment;
  • Helium Pycnometer
• Testing performed at Pennsylvania State University, CISP Laboratory
• Fibre samples dried at 60°C for 72 hours then placed in conditioning room at 65 ± 2% relative humidity for 24 hours prior to shipping
Helium Pycnometry

• Test method;
  • Fibre mass measured on balance, m
  • Helium Pycnometer measures the fibre volume;
    • The skeletal volume (V) of a fibre specimen is determined by measuring the pressure difference of helium in a calibrated volume after the gas is released from the reference cell and allowed to absorb into the small voids of a fibre sample
  • Fibre Density is then calculated; \( \rho_f = \frac{m}{V} \)
Archimedes Method

- Material required is a fibre bundle
- Equipment required:
  - Micro-Balance
  - Vacuum Desiccator
  - Vacuum Pump
  - Density Determination Kit
  - Immersion Fluid
- Flax fibre density measured in accordance with ASTM standards D3800-99 and D792/D276.
- Fibre samples dried at 60°C for 72 hours
Archimedes Method

• Test method;
  • Density Standard Calibration

  Determination of density of glass plummet

• Immersion Fluid Standardization

  Determination of density of immersion fluid
Archimedes Method

• Density Measurement;

Minimum 0.5 grams of fibre required per specimen

Soak samples in immersion fluid for 1 minute

Specimens placed in vacuum desiccator for 5 minutes to remove trapped air from between fibre cells

Completely immerse specimen in immersion fluid and take mass reading

Fibre density calculated from immersion fluid density and ratio of recorded masses
Work Completed: Laboratory Trials

• Single sample of oilseed flax fibre used for evaluation

<table>
<thead>
<tr>
<th>Method</th>
<th># of Specimens</th>
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<tr>
<td>Archimedes</td>
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<tr>
<td>- Methanol</td>
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<tr>
<td>- Acetone</td>
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<tr>
<td>- Canola Oil</td>
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<td>- Water</td>
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<tr>
<td>- Ethanol</td>
<td>7</td>
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</table>
Work Completed: Results Assessment

- Tests compared against five broad categories:
  - Quality of Results
    - Accuracy
    - Repeatability
  - Cost
    - Capital Equipment
    - Consumables
    - Labour
  - Speed
    - Set-up and calibration
    - Testing
  - Safety
  - Ease of Use
Results: Quality of Measurements

![Density (g/cm³) vs. Different Measurement Methods](attachment:image.png)

- **Archimedes: Methanol**
- **Archimedes: Acetone**
- **Archimedes: Canola Oil**
- **Archimedes: Water**
- **Archimedes: Ethanol**
- **Diameter/Linear Density**
- **Helium Pycnometry**
Results: Quality of Measurements

Flax fibre density published values: 1.4 - 1.6 g/cm³
## Results: Overall Performance

<table>
<thead>
<tr>
<th>Method</th>
<th>Accuracy</th>
<th>Repeatability</th>
<th>Cost</th>
<th>Speed</th>
<th>Ease of Use</th>
<th>Safety</th>
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</tbody>
</table>

- Diameter and Linear Density (Diameter and Linear Density)
- Archimedes
  - Methanol
  - Acetone
  - Canola Oil
  - Water
  - Ethanol
- Helium Pycnometry

### Method Details

- **Accuracy**:
  - Poor
  - Fair
  - Good
  - Excellent

- **Repeatability**:
  - Poor
  - Fair
  - Good

- **Cost**:
  - Good
  - Excellent

- **Speed**:
  - Poor
  - Fair
  - Good

- **Ease of Use**:
  - Excellent
  - Fair
  - Poor

- **Safety**:
  - Excellent
  - Fair
  - Poor
Results & Recommendations

• Archimedes test using canola oil as an immersion fluid is a simple and effective method for general use in measuring flax fibre density

• Helium pycnometry may provide more reliable results and would be recommended where a greater amount of testing is required and a higher costs can be justified

• Additional test results are needed to provide a recommendation on the quantity of measurements required for reliable quality control

• Testing a variety of fibre types would provide data on the how the performance of the tests changes using different samples

• Research and development of a post-part construction natural fibre volume/mass test method
  • Glass fibre – resin burn off
  • Carbon fibre – acid digestion
University of Manitoba, Textiles Department

Schweitzer-Mauduit Canada, Inc.

Agricultural Policy Framework
Report in full can be requested at:

http://www.compositesinnovation.ca

→ Projects

→ Biofibre Initiative

→ Density Report (07-020-07)
Questions?