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Recommended flax fibre density values for composite property predictions

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ABSTRACT

Density is one of the fundamental properties of fibres which reinforce polymer matrix composites, and is used both to estimate composite weight and to evaluate fibre content for property predictions. For traditional composites, reinforced by glass or carbon fibres, unique density values are well known for a particular fibre grade and provide reliable fibre content estimations and composite property predictions. However, this is not the case for natural fibres. This paper first reviews published density values for flax (*Linum usitatissimum* L.) fibres and describes the limitations of techniques used to measure fibre density. Significant variations in published densities are found, which can be related to the measurement method. New data quantify the influence of measurement technique, fibre extraction route, moisture content, and reinforcement geometry. Values obtained by buoyancy are around 10% lower than those obtained by pycnometry. Finally the consequences of measured density variations on property estimations for long fibre composites are discussed; volume fraction values calculated from buoyancy provide more accurate tensile modulus values compared to experimental values than those from gas pycnometry; the former are recommended for predictive use.

1. Introduction

Flax fibre density values can be found in various text books, Table 1, in a similar way to the values given for other composite reinforcements such as glass and carbon fibres.

The range of values given in Table 1 for glass fibre densities is directly related to differences in compositions of the different fibre grades. For example, for E-glass fibres a common value is 2.58 whereas high strength and corrosion resistant glass are a little lighter and heavier respectively (Dwight, 2000).

For carbon fibres there is a significant difference between the two families of precurseur; for polyacrylonitrile (PAN) based fibres, which represent the majority of carbon fibre reinforcements for composites, density of most commercial high strength fibres is around 1.8 (Morgan, 2005). For pitch based carbon fibres the densities are higher, generally around 2.1 (Morgan, 2005).

If the glass or carbon fibre grade is known, then the values indicated by suppliers can in general be used with confidence to estimate composite fibre contents. For natural fibres the situation is rather different. If potential users of flax fibres refer to textile fibre handbooks they will find a typical value for flax fibre density (Table 1), usually around 1.5. However, the recent development of natural fibre composite applications has resulted in a large increase in the number of studies of these materials, Table 2, and if results from these are examined the single-value approach used for glass and carbon (once the grade has been specified) is more difficult to justify. Published values range from 1.36 to 1.57, a difference of around 20%.

The first observation is that the origins of the values quoted are not always given; when they are provided then either immersion or gas pycnometry tend to be favoured. Different techniques are used traditionally to measure fibre density, and these may not be suitable for natural fibres. A study by Truong et al. (2009) identified five measurement methods for high performance fibre density, and discussed their application to natural fibres:

- Linear density and diameter calculation,
- Archimedes (buoyancy)
- Helium pycnometry
- Liquid pycnometry, and
- Density gradient column.

The first is included in an ASTM standard (ASTM, 2007). It was used by Soykeabkaew et al. (2004) and involves measuring the average diameter of 100 fibres and the weight of 10 fibres of known lengths, and calculating the density by assuming a circular cross-section. Clearly the latter assumption is not correct for natural fibres. Fig. 1 shows the cross section of a flax fibre bundle after fine polishing, indicating a polygonal cross-section for fax fibres. The values obtained by Soykeabkaew et al.

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Table 1
Density of fibre reinforcements.

Fibre reinforcement	Density values	Reference
Glass (range)	2.46–2.74	Hearle (2001)
e.g. E-glass	2.58	Dwight (2000)
e.g. S2-glass	2.46	Dwight (2000)
e.g. E-CR (Electrical/Chemical Resistance)	2.72	Dwight (2000)
Carbon PAN-based (range)	1.68–1.91	Morgan (2005)
e.g. T700	1.80	Toray datasheet (2018)
Carbon pitch-based (range)	1.90–2.20	Morgan (2005)
e.g. K63712	2.12	Mitsubishi datasheet (2018)
Flax (typical data)	1.54	Gordon Cook (2001)

Table 2
Published flax fibre density values.

Fibre type	Density values	Measurement method	Reference
Flax	1.43–1.55		Lewin (2010)
Flax	1.4–1.52		Müssig and Hughes (2012)
Flax	1.53	Immersion, ethanol	Baley (2002)
Flax scutched,	1.34	Immersion, ethanol	Martin et al., (2013)
Flax tow	1.37		Martin et al., (2013)
Flax, cut	1.54	Helium pycnometry	Chafei et al., (2014)
Flax	1.42–1.52		Dicker et al., (2014)
Flax yarn	1.54	Immersion, water, corrected for lumen	Madsen and Lilholt (2003)
Flax yarn	1.57	Helium pycnometry	Shah (2013)
Linseed flax	1.41–1.47	Immersion, different oils	Amiri et al. (2017)
	1.49	Helium Pycnometry	Amiri et al. (2017)

(2004) were very low, around 0.29 for a fibre bundle diameter of 210 µm. An improvement could be made by measuring the true sections on polished cross-sections, as Summerscales et al. (2010) have done for jute fibres, but again this is a very time-consuming approach, and the natural shape variability along the fibres will not be taken into account unless a large number of observations are made.

Buoyancy methods are simple to perform and widely used for composite density measurement but care is needed in choosing the immersion fluid. For example, ASTM D792 (2013) recommends weighing in water, but cellulose-based fibres such as flax are quite hydrophilic. They normally contain a few percent by weight of moisture and this can increase significantly during immersion. Le Duigou et al., (2017) have shown using environmental scanning electron microscopy that flax fibres absorb water and swell as relative humidity increases. This method is therefore not adapted to natural fibres unless an alternative immersion medium is used. Facca et al. (2006) suggested mercury as an alternative, while a recent study by Amiri et al. (2017) examined the influence of the immersion medium and recommended soybean oil rather than distilled water. This is not a new debate; there has been considerable controversy over the values of density for cellulose fibres. Early measurements by Davidson are often cited, dating from 1927 (Davidson, 1927). He measured the density of cellulose in toluene, helium and water. The density values in helium for cotton fibres (1.57) were intermediate between those in toluene (1.55) and water (1.61), though differences between the three are smaller than those noted in Table 2.

Hermans and Vermaas (1946) argued that density is a macroscopic concept and that if a macroscopic body contains pores only an apparent value can be determined. Stamm provided additional discussion (Stamm, 1950), presenting results from density measurements in

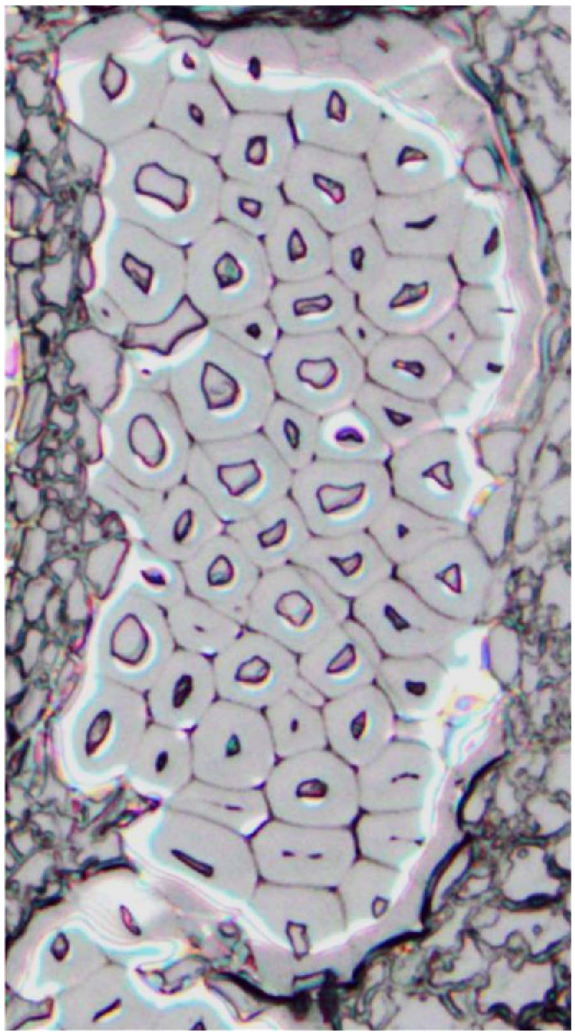

Fig. 1. Example of section through flax fibres in a bundle in a young plant, showing large lumens. Image corresponds to a section (160 × 300) µm^2.

benzene which were close to values from X-ray analysis.

The condition of the sample will also affect the density measurements, although this is a physical difference rather than an artefact of measurement. Nevertheless careful attention must be paid to moisture content in natural fibre specimens when measurements are made. Baley et al. (2005) showed that drying of fibres at 105 °C for 12 h resulted in a weight loss of 8.7%.

Gas and liquid pycnometers have been used for some time to determine density of polymers, and a standard test method exists (ISO, 1999). Pycnometers measure the volume of a sample of known weight, by measuring the volume of either displaced inert gas or liquid; the pressures measured on filling the sample chamber then discharging the gas or liquid into a second empty chamber enable computation of the sample solid phase volume.

Finally, gradient columns have been used traditionally to determine the density of bulk polymers, and again standard methods are available (ASTM, 2010), but the same concerns about the immersion medium noted above will apply.

Truong et al. (2009) showed results from measurements made using the first three methods on oilseed flax fibres. Data showed very large differences in density, from 1.5 with a helium pycnometer to over 2.5 using the linear density and diameter method. The authors recommended two methods, based on a number of criteria including accuracy (with respect to a published density range of 1.4–1.6), safety, cost and convenience; they favoured either buoyancy, using canola oil