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VOLUMETRIC COMPOSITION IN COMPOSITES
AND HISTORICAL DATA

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ABSTRACT

The obtainable volumetric composition in composites is of importance for the prediction of mechanical and physical properties, and in particular to assess the best possible (normally the highest) values for these properties. The volumetric model for the composition of (fibrous) composites gives guidance to the optimal combination of fibre content, matrix content and porosity content, in order to achieve the best obtainable properties. Several composite materials systems have been shown to be handleable with this model. An extensive series of experimental data for the system of cellulose fibres and polymer (resin) was produced in 1942 – 1944, and these data have been (re-)analysed by the volumetric composition model, and the property values for density, stiffness and strength have been evaluated. Good agreement has been obtained and some further observations have been extracted from the analysis.

1.   INTRODUCTION

The volumetric composition in composites is of importance for their performance and it includes the fiber, matrix and porosity content. The interrelation between these parameters has been described and evaluated in a series of papers, partly by the present authors, (Madsen and Lilholt 2002, Madsen, Thygesen and Lilholt 2007, Madsen, Thygesen and Lilholt 2009). The models are based on a maximum obtainable fibre *volume* content, which can be related to the packing ability of a fibre assembly under a given pressure (applied typically during processing of the composite). At fibre contents below the maximum the composite can be fully compacted to, ideally, no porosity, while at fibre contents above the maximum the composite will (inevitably) contain structural porosity caused by the inability of the matrix content to fill the voids in the fully compacted fibre assembly. The optimum composition is the one where the fibre content just equals the maximum value (under the given pressure), and the matrix content is just sufficient to fill the voids of the fibre assembly. This optimum composition corresponds to the best obtainable properties, such as (highest) density, stiffness and strength. The modeling of these aspects is based on the fibre *weight* fraction of the composite, which can be varied from

zero (pure matrix) to one (only fibres, in an assembly).

The model has been developed in the above papers, and illustrated by data for the volumetric composition, as well as by data for density and stiffness, for a series of composites with different fibres, matrices and fibre orientations. The effect of (processing) pressure on the volumetric composition is also studied in some cases. A recent closer study of the effect of (processing) pressure on the volumetric composition is given in the paper Madsen (2013) in these proceedings.

In composites the traditional view is that the fibres give a reinforcement of an otherwise weak (less stiff) matrix, and therefore the concept starts with a pure matrix and the fibres are added to this matrix. It is thus natural to use the (added) fibre fraction as a parameter for the composition of the composite. This approach is taken in the above analyses, and in particular the fibre *weight* fraction is used as the guiding parameter.

Actually, in most composites the fibres are the strength- and stiffness- dominating constituents, and it might therefore also be argued that the starting position is the fibres, normally in the form of some sort of preform (aligned bundle, aligned tape, bi-directional woven fabric, randomly oriented fibre mat e.g. paper, or other geometrical arrangements). Then *matrix is added* to the fibre assembly to allow concerted action of the fibres, and to keep the fibres together in the form of a solid (composite).

This thinking and approach was taken (unknown to the present authors) many years ago (1942 – 1944) by a research group under the British Ministry of Aircraft Production. During the second world war the aircrafts were to a large extent based on natural materials like wood, glue, and canvas, and a very large number of successful Mosquito aircrafts were manufactured (Gordon 1968). The focus of the research was on "the preparation of reinforced plastics from paper and phenol-formaldehyde resin" (Cox and Pepper 1944). The research was carried out jointly by the Engineering Department, National Physical Laboratory, and the Chemical Research Laboratory in England, thus including both engineering and chemical knowledge and expertice. It is interesting to note the threefold objects of the research: " (a) to study the effect of method of manufacture on the mechanical properties of phenolic paper-base plastics, (b) to assess the relative contributions of the resin and the paper-fibres, and (c) to provide information whereby improvements in existing materials might be affected." (Cox and Pepper 1944). It is also interesting to note one of the conclusions of this first paper, as quoted in the abstract: "The optimum resin content is that which just fills the air voids in the paper at any given pressure. This optimum resin content is indicated by a maximum in the density – resin content curve". In the second paper (Pepper and Barwell 1944) the studies are extended with "attempts to elucidate further the dependence of the mechanical properties on such factors as resin content, voids in the paper, and particularly on the pressure applied during bonding". In the abstract the conclusions are quoted: "…… although the tensile strength of reinforced plastics increases with the proportion of the stronger component, the fibre, the improvement is sharply limited by the need to fill all the voids in the paper; when the resin content is insufficient to fill the voids, the strength of the material is seriously reduced. It is demonstrated that the main function of the pressure applied during bonding is to reduce the voids in the paper ……".

The present evaluation is aimed at analyzing the extensive experimental results of this war-time research effort in the light of the concepts introduced in the above modern-time research on volumetric composition of fibre containing composites. The focus will be on the following elements of the materials and of the model: (i) volumetric composition, (ii) density, (iii) stiffness, (iv) strength, (v) compaction of paper/fibre assemblies.

2. BASIC MATERIALS AND PARAMETERS

The research group of Ministry of Aircraft Production used papers of various types, and phenol-formaldehyde resin, which was used either in an alcoholic solution or in a aqueous solution for impregnation of the paper. The basic materials are listed in table 1.

Table 1. Basic materials.

Paper	Manila Hemp paper
	Kraft paper
	High Density Kraft paper
Resin	Phenol-Formaldehyde, alcoholic solution (PF alc)
	Phenol-Formaldehyde, aqueous solution (PF aqu)

The preparation procedure is through four steps: (i) stacking of paper sheets, (ii) impregnation of paper with phenol-formaldehyde solution, (iii) drying of the impregnated paper, (iv) pressing of the laminated sheets. The pressing is typically done with pressure of 2000 or 2500 lbs per sq.in., at a temperature of 145 $^{\circ}$C, and for a time 30 minutes.

The stacking of the paper sheets is done in two different ways. For Manila hemp paper the sheets are stacked with same orientation for all sheets. Although nominally with random fibre orientation, the production of paper sheets induces some alignment in the "machine" direction. The data for tensile strength in the "machine" direction and in the "cross-wire" direction, quoted by Cox and Pepper (1944) in their table 1, are used to calculate an effective fibre orientation factor in the "machine" direction for these composites (Lilholt and Bjerre 1997), the factor becomes $\eta = 0.389$. For Kraft paper and for High density kraft paper, the paper sheets are "crossed", such that the composite (on average) has random fibre orientation, and the fibre orientation factor for this case is $\eta = 0.333$.

The stiffness and strength data are quoted in imperial units in the original tables and diagrams (Cox and Pepper 1944, Pepper and Barwell 1944), and the conversion factors to SI units are the following:

stiffness in 10^6 lbs per sq.in.: $\qquad$ 1 x 10^6 psi = 7.03 GPa

strength in tons per sq.in.: $\qquad$ 1 tsi = 15.75 MPa

The composition of the different composites are quoted in resin weight fraction (percent) for data and diagrams on stiffness and on strength, while the composition is quoted in resin volume fraction (percent) for data and diagrams on density, (Cox and Pepper 1944, Pepper and Barwell 1944). The modern-time models are based on *fibre weight* fraction and the conversions are the following:

$W_f = 1 - W_m$

$W_f = 1 / [1 + (V_m / V_f) \cdot (\rho_m / \rho_f)]$

where W is weight fraction, V is volume fraction and ρ is density, and f and m are fibre and matrix, respectively. The second equation is valid for no porosity in composites.

The evaluation will be made by presenting (the copy of) the historical diagrams and comparing them directly with the analysis and diagrams according to the volumetric composition model. In

the historical diagrams the authors have analysed their data by trend lines.

3. EXPERIMENTS AND MODELS

The experimental data are presented by their original diagrams, which include the (many) experimental points. The data points are read off the diagram in their original (imperial) units and the data are then converted to SI-units. This allows the data to be plotted according to the volumetric composition model, in the form used by the present authors, examples of "modern" data plotted in this manner are given in Madsen (2013) in these proceedings.

3.1. Density of Manila hemp paper / phenol-formaldehyde (alcoholic). These data are presented in Fig. 1a, which is copied directly from Cox and Pepper (1944) as their figure 3, and gives density in g/cm^3 as function of resin content by volume in percent. The 15 data point are converted to SI units and plotted as red points in Fig. 1b. With their paper fibre (cellulose) density of 1.53 g/cm^3, (which agrees with their linearly extrapolated trend line to zero resin content), and their phenol-formaldehyde density of 1.27 g/cm^3, the maximum fibre volume fraction $V_{f,max}$ is estimated to be 0.70, with the corresponding fibre weight fraction at transition $W_{f,trans}$ = 0.738. With these parameters the volumetric composition model curve is calculated and is plotted as the blue curve in Fig. 1b.
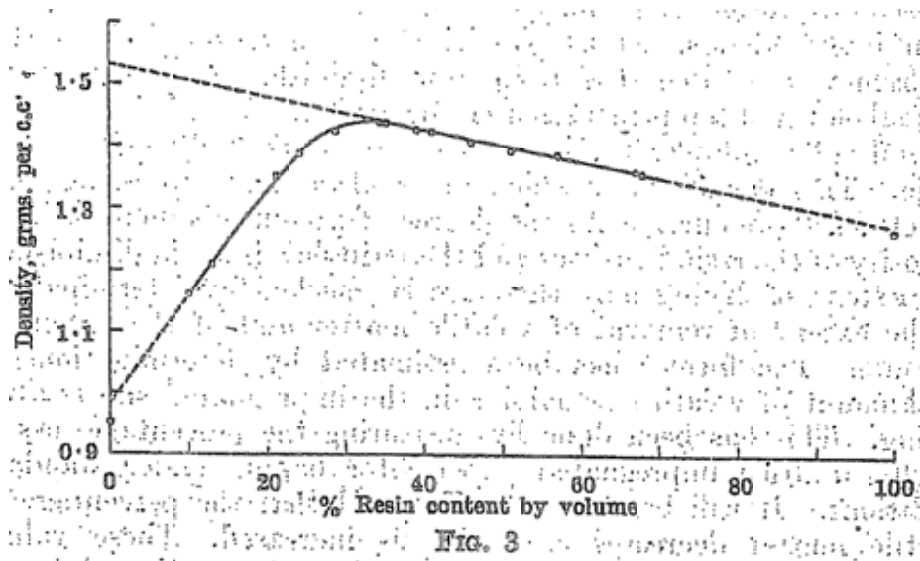

Fig. 1a. Density of Manila hemp paper / phenol-formaldehyde (alcoholic) in g/cm^3 as a function of resin content by volume in percent (Cox and Pepper 1944).
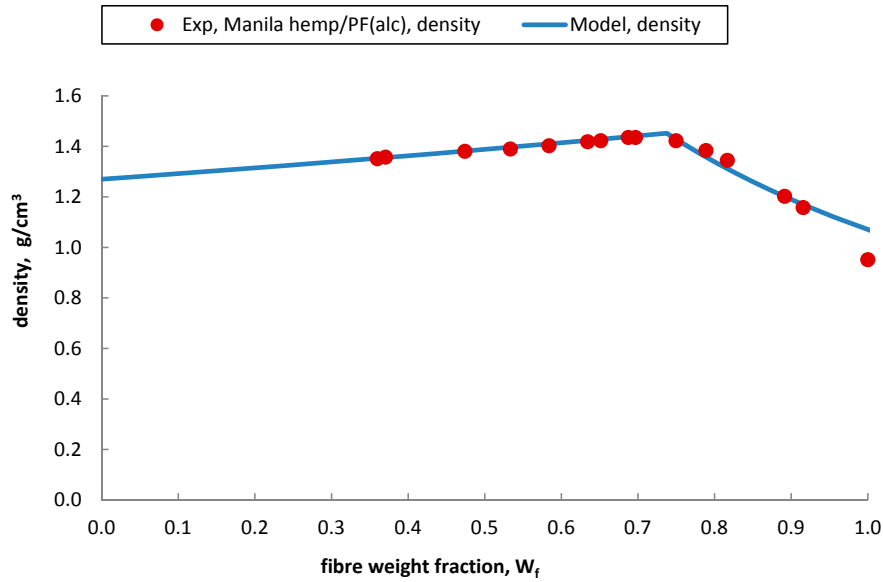
Fig. 1b. Density experimental data re-plotted as a function of fibre weight fraction, model calculated for $V_{fmax} = 0.70$.

The agreement of the experimental data with the model curve is satisfactory, especially at low fibre contents. The density value at $W_f = 1$ corresponds to the density of the dry (containing no matrix) fibre assembly compacted at the given pressure, in this case the experimental value is 0.952 g/cm^3, while the model implies a value of 1.084 g/cm^3. This probably indicates a situation where the fibre assemblies are compacted to higher density which is maintained during composite fabrication, while (part of) the compaction is released after compaction of a dry fibre assembly.

3.2. Density of High density kraft paper / phenol-formaldehyde (aqueous). These data are presented in Fig. 2a, which is copied directly from Pepper and Barwell (1944) as their figure 1, and gives density in g/cm^3 as function of resin content by volume in percent. The 12 data point are converted to SI units and plotted as green points in Fig. 2b. With their paper fibre (cellulose) density of 1.53 g/cm^3 and their phenol-formaldehyde density of 1.27 g/cm^3, the maximum fibre volume fraction $V_{f,max}$ is estimated to be 0.82, with the corresponding fibre weight fraction at transition $W_{f,trans}$ = 0.846. With these parameters the volumetric composition model curve is calculated and is plotted as the blue curve in Fig. 2b.
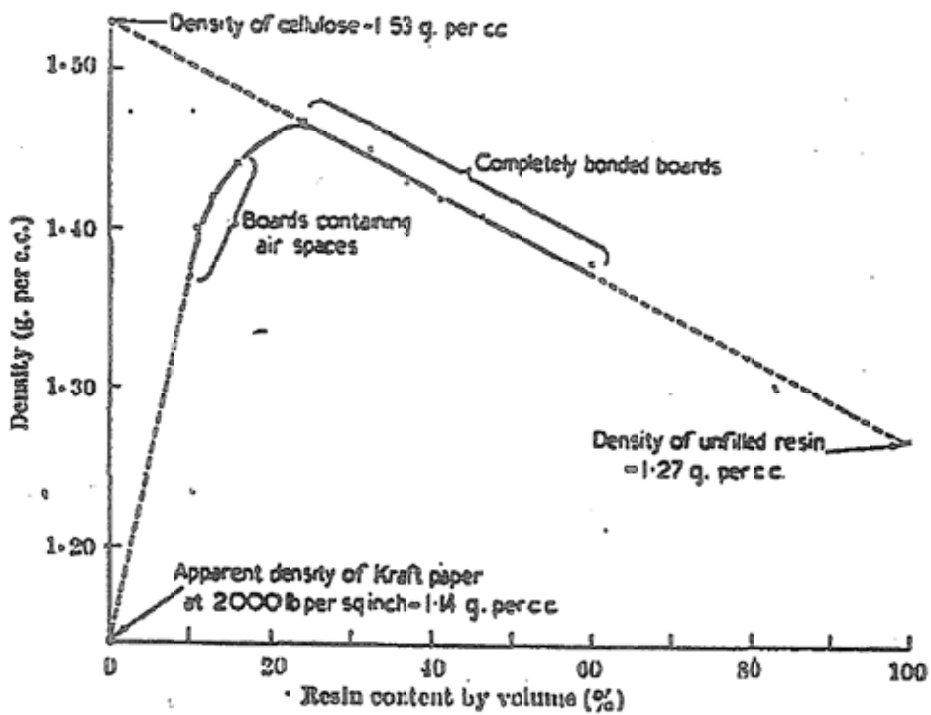

Fig. 2a. Density of High density kraft paper / phenol-formaldehyde (aqueous) in g/cm^3 as a function of resin content by volume in percent (Pepper and Barwell 1944).

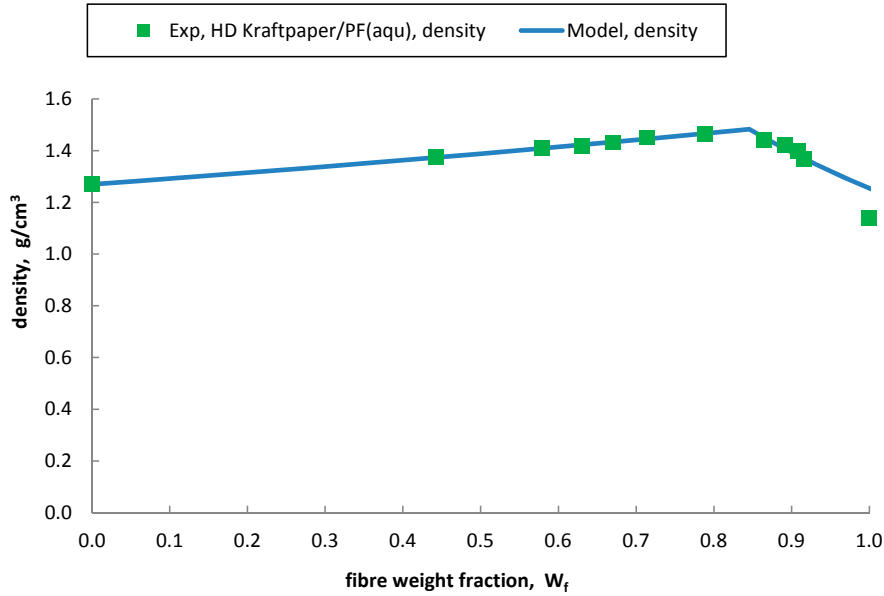Volumetric composition in composites and historical data

Fig. 2b. Density experimental data re-plotted as a function of fibre weight fraction, model calculated for $V_{fmax} = 0.82$.

The agreement of the experimental data with the model curve is satisfactory, especially at low fibre contents. The density value at $W_f = 1$ corresponds to the density of the dry (containing no matrix) fibre assembly compacted at the given pressure, in this case the experimental value is 1.140 g/cm^3, while the model implies a value of 1.260 g/cm^3. This probably indicates a situation where the fibre assemblies are compacted to higher density which is maintained during composite fabrication, while (part of) the compaction is released after compaction of a dry fibre assembly.

3.3. Stiffness of Kraft paper / phenol-formaldehyde (aqueous). These data are presented in Fig. 3a, which is copied directly from Pepper and Barwell (1944) as their figure 16, and gives stiffness in 10^6 lbs per sq.in. as function of resin content by weight in percent. The 7 data point are converted to SI units and plotted as green points in Fig. 3b. The calculation of the composite stiffness is made by the modified rule of mixtures (Madsen et al 2009):

$$E_c = (\eta \cdot V_f \cdot E_f + V_m \cdot E_m) \cdot (1 - V_p)^n \tag{1}$$

where E is stiffness, V is volume fraction, and f is fibre, m is matrix and p is porosity, η is the fibre orientation factor, and n is the exponent for porosity effect. The composites are made with "crossed" paper sheets, so the fibre orientation factor is $\eta = 0.333$, as described in section 2. The exponent n is set to 2.0, as used by Madsen et al (2009).

With the phenol-formaldehyde stiffness of 4.8 GPa (experimental value), the paper fibre (cellulose) stiffness is estimated to be 62 GPa and the maximum fibre volume fraction $V_{f,max}$ is estimated to be 0.77, with the corresponding fibre weight fraction at transition $W_{f,trans} = 0.801$. With these parameters the volumetric composition model curve is calculated and is plotted as the blue curve in Fig. 3b.

Fig. 3a. Stiffness of Kraft paper / phenol-formaldehyde (aqueous) in 10^6 lbs per sq.in. as a function of resin content by weight in percent (Pepper and Barwell 1944).
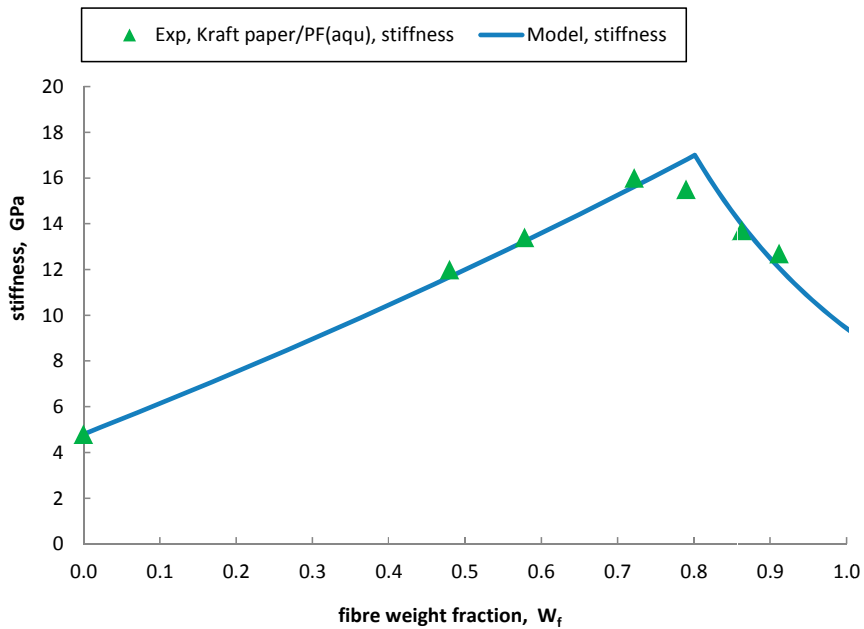
Fig. 3b. Stiffness experimental data re-plotted as a function of fibre weight fraction, model calculated for $V_{fmax} = 0.77$, fibre stiffness 62 GPa, matrix stiffness 4.8 GPa, fibre orientation factor 0.333, porosity exponent 2.0.

The agreement of the experimental data with the model curve is satisfactory. The stiffness value at $W_f = 1$ corresponds to the stiffness of the dry (containing no matrix) fibre assembly, i.e. the paper sheets, compacted at the given pressure, in this case the model implies a value of 9.65 GPa. This value might be compared to an experimental value for stiffness of the relevant Kraft paper sheets, (no data are available).

3.4. Stiffness of High density kraft paper / phenol-formaldehyde (aqueous). These data are presented in Fig. 4a, which is copied directly from Pepper and Barwell (1944) as their figure 4, and gives specific stiffness in km, as function of resin content by weight in percent. The specific stiffness is the stiffness divided by density, therefore the density data of Pepper and Barwell (1944) figure 1 are implied in the conversion to stiffness. The 9 data point are converted to SI units and plotted as green points in Fig. 3b. The calculation of the composite stiffness is made by the modified rule of mixtures (Madsen et al 2009), as eq. (1).

The composites are made with "crossed" paper sheets, so the fibre orientation factor is $\eta = 0.333$, as described in section 2. The exponent n is set to 2.0, as used by Madsen et al (2009).

The experimental value of phenol-formaldehyde stiffness is 3.5 GPa, the best fit of all data points is obtained with a matrix stiffness of 4.0 GPa, the paper fibre (cellulose) stiffness is estimated to be 63 GPa and the maximum fibre volume fraction $V_{f,max}$ is estimated to be 0.78, with the corresponding fibre weight fraction at transition $W_{f,trans} = 0.810$. With these parameters the volumetric composition model curve is calculated and is plotted as the blue curve in Fig. 4b.
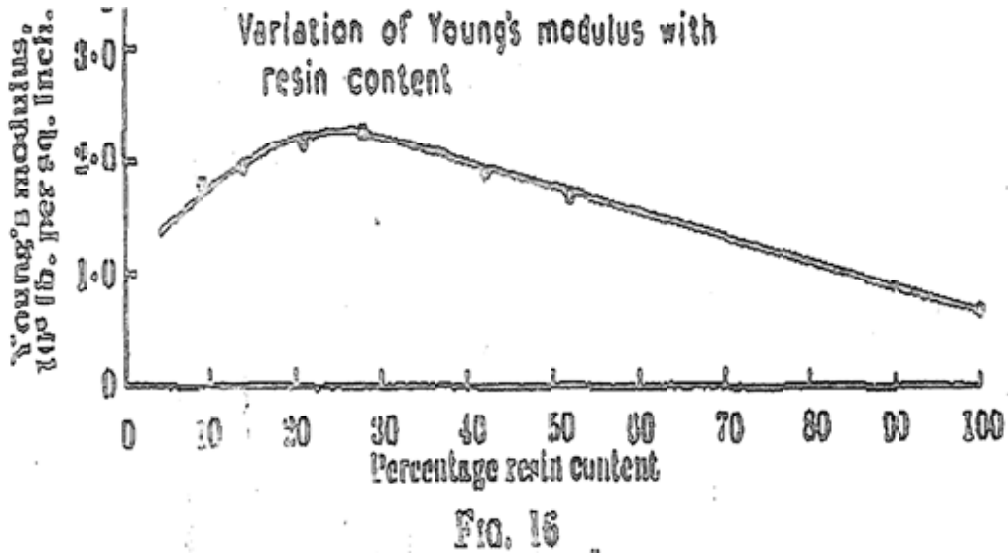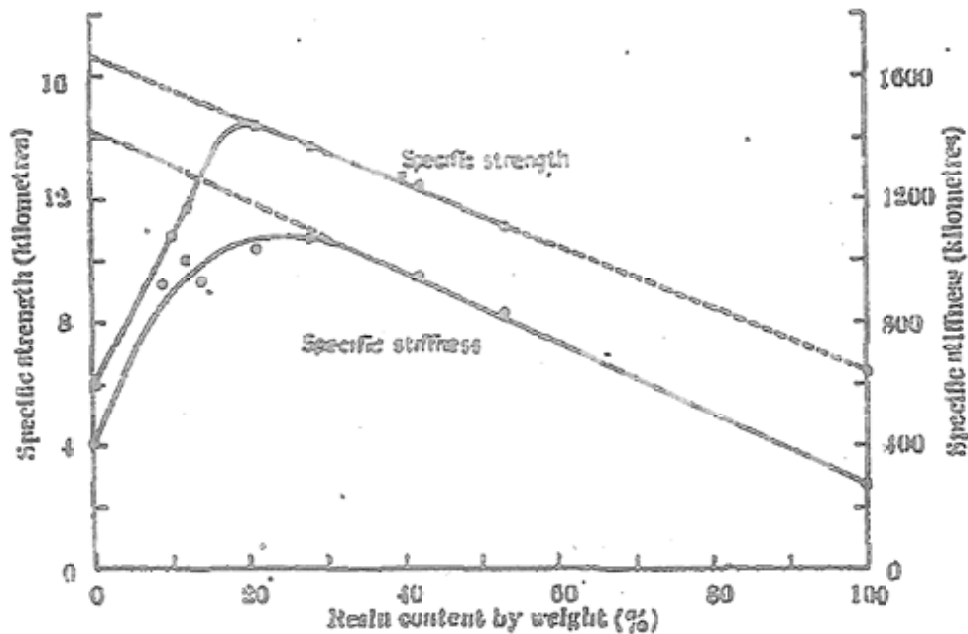

Fig. 4a. Specific stiffness (right-hand axis) of High density kraft paper / phenol-formaldehyde (aqueous) in km as a function of resin content by weight in percent, (Pepper and Barwell 1944).
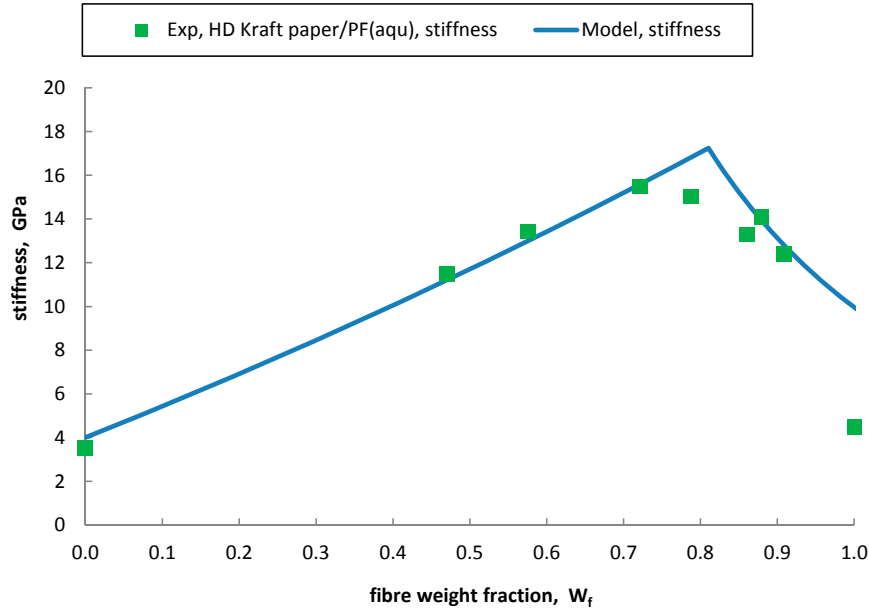
Fig. 4b. Stiffness experimental data re-plotted as a function of fibre weight fraction, model calculated for $V_{fmax} = 0.78$, fibre stiffness 63 GPa, matrix stiffness 4.0 GPa, fibre orientation factor 0.333, porosity exponent 2.0.

The agreement of the experimental data with the model curve is satisfactory. The stiffness value at $W_f = 1$ corresponds to the stiffness of the dry (containing no matrix) fibre assembly, i.e. the paper sheets, compacted at the given pressure, in this case the model implies a value of 10.2 GPa. This value might be compared to the experimental value of 4.49 GPa for stiffness of the High density kraft paper sheets (Pepper and Barwell 1944). As for density, the compaction of fibre assemblies are probably maintained, leading to higher stiffness, during composite fabrication, compared to the compaction of dry High density kraft paper sheets.

3.5. Strength of Manila hemp paper / phenol-formaldehyde (alcoholic). These data are presented in Fig. 5a, which is copied directly from Cox and Pepper (1944) as their figure 1, and gives strength in tons per sq.in. as function of resin content by weight in percent. The 12 data point are converted to SI units and plotted as red points in Fig. 5b. The calculation of the composite strength is made in analogy with the stiffness as given in eq. (1):

$$\sigma_c = (\eta \cdot V_f \cdot \sigma_f + V_m \cdot \sigma_m) \cdot (1 - V_p)^n \tag{2}$$

where σ is strength, V is volume fraction, and f is fibre, m is matrix and p is porosity, η is the fibre orientation factor, and n is the exponent for porosity effect. The composites are made with paper sheets all in the same orientation, so the fibre orientation factor is $\eta = 0.389$, as described in section 2. The exponent n is set to 2.0, as used by Madsen et al (2009).

The phenol-formaldehyde strength is 48.5 MPa (experimental value), the best fit of all data points is obtained with matrix strength of 65 MPa. With this value, the paper fibre (cellulose) strength is estimated to be 1100 MPa and the maximum fibre volume fraction $V_{f,max}$ is estimated to be 0.70, with the corresponding fibre weight fraction at transition $W_{f,trans} = 0.738$. With these parameters the volumetric composition model curve is calculated and is plotted as the blue curve in Fig. 5b.

Fig. 5a. Strength of Manila hemp paper / phenol-formaldehyde (alcoholic) in tons per sq.in. as a function of resin content by weight in percent (Cox and Pepper 1944). (Only the tension strength data are analysed).
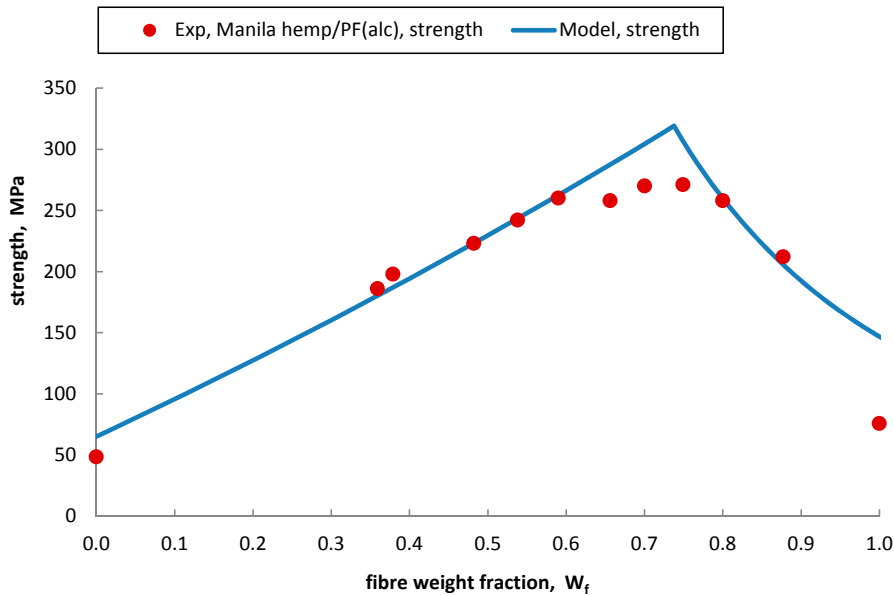
Fig. 5b. Strength experimental data re-plotted as a function of fibre weight fraction, model calculated for V_{fmax} = 0.70, fibre strength 1100 MPa, matrix strength 65 MPa, fibre orientation factor 0.389, porosity exponent 2.0.

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The agreement of the experimental data with the model curve is satisfactory. The strength value at $W_f = 1$ corresponds to the strength of the dry (containing no matrix) fibre assembly, i.e. the paper sheets, compacted at the given pressure, in this case the model implies a value of 151 MPa. This value might be compared to the experimental value of 75.7 MPa for strength of the Manila hemp paper sheets (Cox and Pepper 1944). As for density and stiffness, the compaction of fibre assemblies are probably maintained, leading to higher strength, during composite fabrication, compared to the compaction of dry Manila hemp paper sheets.

3.6. Strength of High density kraft paper / phenol-formaldehyde (aqueous). These data are presented in Fig. 6a, which is copied directly from Pepper and Barwell (1944) as their figure 3, and gives strength in tons per sq.in. as function of resin content by weight in percent. The 11 data point are converted to SI units and plotted as green points in Fig. 6b. The calculation of the composite strength is made in the same way as in section 3.5, using eq. (2).

The composites are made with "crossed" paper sheets, so the fibre orientation factor is $\eta = 0.333$, as described in section 2. The exponent n is set to 2.0, as used by Madsen et al (2009).

With the phenol-formaldehyde strength of 72 MPa (experimental value), the paper fibre (cellulose) strength is estimated to be 800 MPa and the maximum fibre volume fraction $V_{f,max}$ is estimated to be 0.78, with the corresponding fibre weight fraction at transition $W_{f,trans} = 0.810$. With these parameters the volumetric composition model curve is calculated and is plotted as the blue curve in Fig. 6b.
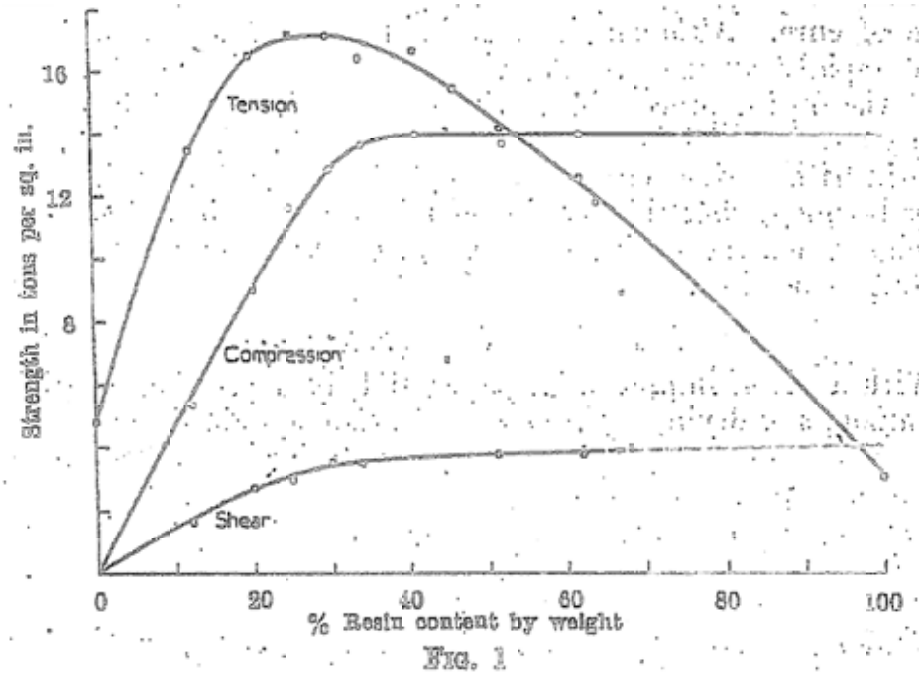
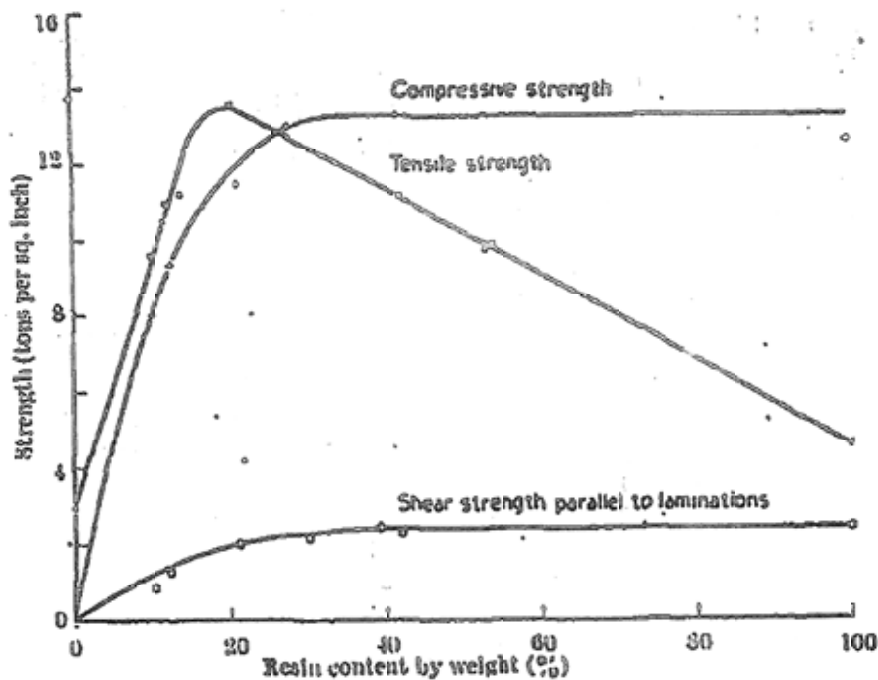

Fig. 6a. Strength of High density kraft paper / phenol-formaldehyde (aqueous) in tons per sq.in. as a function of resin content by weight in percent, (Pepper and Barwell 1944). (Only the tension strength data are analysed).
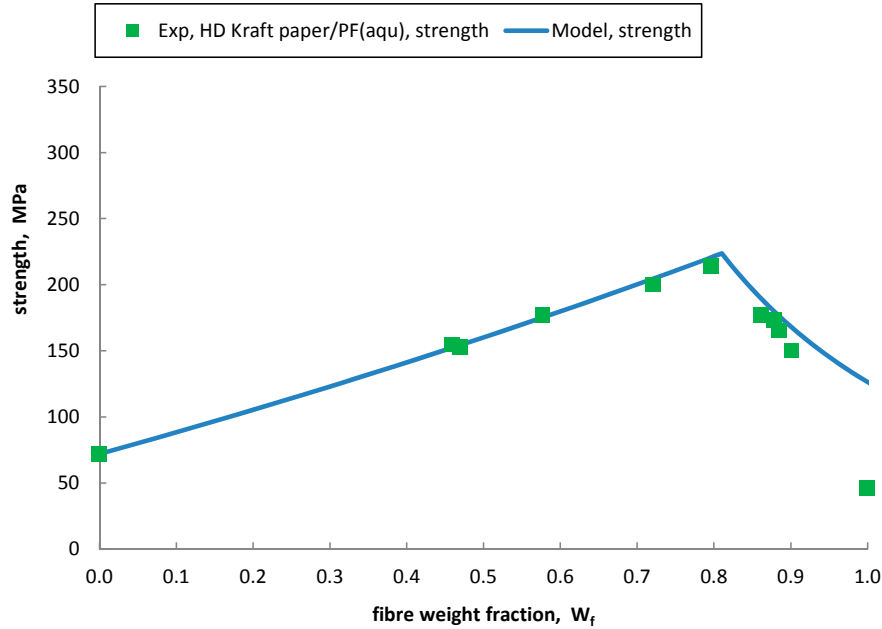
Fig. 6b. Strength experimental data re-plott ed as a function of fibre weight fraction, model calculated for $V_{fmax} = 0.78$, fibre strength 800 MPa, matrix strength 72 MPa, fibre orientation factor 0.333, porosity exponent 2.0.

The agreement of the experimental data with the model curve is satisfactory. The strength value at $W_f = 1$ corresponds to the strength of the dry (containing no matrix) fibre assembly, i.e. the paper sheets, compacted at the given pressure, in this case the model implies a value of 130 MPa. This value might be compared to the experimental value of 46 MPa for strength of the High density kraft paper sheets (Pepper and Barwell 1944). As for density and stiffness, the compaction of fibre assemblies are probably maintained, leading to higher strength, during composite fabrication, compared to the compaction of dry High density kraft paper sheets.

3.7.   Comparison of the paper sheet / phenol-formaldehyde composites.   The model curves established in the Figs. 1b to 6b, are all based on the best fitted values for those free parameter(s) which were not given experimentally.   In an attempt to compare the three composite materials systems, fitted parameters are established to achieve the best agreement between model and experimental data simultaneously for both density, stiffness and strength (not all three properties were recorded for all composites).   These values are collected in Table 2.

Table 2. Parameter to obtain *best agreement* with experimental data.

Property	Manila Hemp / PF(alc)	HD Kraft paper / PF(aqu)	Kraft paper / PF(aqu)
Fibre density, g/cm^3	1.53	1.53	1.53
Matrix density, g/cm^3	1.27	1.27	1.27
$V_{f\,max}$	0.70	0.82 & 0.78	0.77
$W_{f\,trans}$	0.74	0.85 & 0.81	0.80
Fibre stiffness, GPa	-	63	62
Matrix stiffness, GPa	-	4.0	4.8
Fibre strength, MPa	1100	800	-
Matrix strength, MPa	65	72	-
Fibre orientation, η	0.389	0.333	0.333
Porosity exponent, n	2.0	2.0	2.0

There is no clear tendency in the values of the materials parameters. The higher fabrication pressure (2500 psi) used for the Manila hemp composites does not lead to e.g. a higher maximum fibre volume fraction. The fibre stiffness of about 65 GPa is similar to the value estimated by the previous authors, from slightly different data and evaluations. The strength value for Manila hemp of about 1100 MPa is relatively high, and higher than the nominal fibre strength of Kraft paper(s) of about 800 MPa. The origin of the cellulose fibres of these papers is not clear, it may be based on wood, which generally has a lower cellulose content (about 40 %) than that of hemp (about 70%), and this may tentatively explain the lower strength for Kraft paper, because cellulose fibre strength is generally increasing with increasing cellulose content. The porosity exponent is about 2, but no clear indications can be derived from these data.

The density, stiffness and strength of the pure dry paper sheets were estimated above from the model, and compared to the available experimental data of Cox and Pepper (1944) and Pepper and Barwell (1944). These values are shown in Table 3.

Table 3. Density, stiffness and strength of dry paper sheets.

Density, g/cm^3	Fabr. pressure, psi	Value, model, $W_f = 1$	Exp. value
Manila hemp	2500	1.084	0.952
HD Kraft	2000	1.260	1.140
Stiffness, GPa			
Kraft	2000	9.65	-
HD Kraft	2000	10.2	4.49
Strength, MPa			
Manila Hemp	2500	151	75.7
HD Kraft	2000	120	46

The compaction of the fibre paper sheets during composite fabrication seems to be more efficient than compaction of dry fibre paper sheets, in terms of giving higher values for density, stiffness and strength, as discussed in the sections on the individual composite systems above. The effect on density is small, about a 10% increase, while the effect is much more significant for stiffness and strength, i.e. mechanical properties. This may be expected qualitatively, because mechanical performance of materials is rather sensitive to defects, in the present case the fact that the fibre paper sheets are not a solid but an assembly of fibres with air voids in between.

4. DISCUSSION AND CONCLUSIONS

The extensive study by Cox, Pepper and Barwell in 1942 – 1944 was emphasizing the need to just fill the air voids in the fibre assembly of the various papers with resin in order to make efficient composite materials. The volumetric composition model and the concept of a maximum fibre volume fraction addresses the same situation. The extensive amount of data for the three materials systems and the three properties investigated, were analyzed by the volumetric composition model and in general good agreement was established. The individual analyses for density, stiffness and strength give rather good fit between experiments and model. It is perhaps interesting to note that the situation for fibre contents at, and especially above, the maximum fibre volume fraction (fibre weight fraction transition point) the agreement is relatively good, as demonstrated in figures 1 to 6 with index b.

The model overestimates the value for density, stiffness and strength as the resin content approaches zero (fibre content approaches $W_f = 1$). For density the overestimate is about 10%, while the overestimate is by a factor of about 2 for stiffness and strength. This indicates, perhaps not surprisingly, that the mechanical performance drops extraordinarily, when the resin content is vanishing and the dry fibre assembly becomes not a true solid.

The combined modeling of the three materials systems allows reasonable best obtainable agreement with the common (same) parameters, listed in table 2, to describe density, stiffness and strength for each materials system.

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