Parametric optimization of the processing of all-cellulose composite laminae

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Abstract Single-polymer composites based on cellulose I and/or II (aka all-cellulose composites) are emerging as a class of high-performance bio-based composite materials with mechanical properties suited to structural applications. There are various synthesis routes for the preparation of all-cellulose composites. However, little has been reported on the optimization of the processing variables affecting the properties of all-cellulose composites. In the present work, a range of all-cellulose composites were produced as single laminae via solvent infusion processing using a precursor of cellulose II fibers that were assembled as a woven 2D textile. The effects of dissolution time, dissolution temperature, and compaction pressure during hot pressing on the properties of the laminae were then systematically examined using a Taguchi design of experiment approach in order to identify the critical control factors. The tensile properties, fiber volume fraction, and crystallinity of the laminae were determined. Statistical analysis of variance and the signal-to-noise ratio were used to rank the importance of key control factors.

Keywords All-cellulose composites, Solvent infusion process, Design of experiments, Taguchi method, Analysis of variance

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In spite of the large array of control factors, the development of a systematic approach to optimization of processing-property relationships of ACCs has not yet been reported in literature. Modern manufacturing frequently relies on design optimization (e.g. design of experiments (DOE)) in order to compete on the cost and performance of materials and products. An example of a robust design optimization approach is the Taguchi methodology that has been previously used in optimizing the processing of both conventional and bio-based composite materials. The construction of orthogonal arrays (OAs) as performed in a Taguchi experimental design enables the quantification of the effect of each control factor on various characteristic outputs (e.g. mechanical properties). The Taguchi methodology allows identification of the most dominant control factors on chosen performance characteristics so that the non-dominant control factors can be ignored. Hence, a design optimization approach such as the Taguchi methodology is useful for studying the complex interdependencies between the above control factors in the manufacturing of ACCs.

ACCs may be fabricated as thin films (<1 mm thick) or laminated constructs with thicknesses up to 8–10 mm, leading to a range of potential applications. However, an increase in the thickness of the ACCs also increases the likelihood of differential shrinkage and warpage of the final material. Generally, the solvent represents a relatively large volume (~80%) of...
the cellulose–solvent solution that is subsequently removed during cellulose regeneration. Hence, significant volumetric shrinkage can be observed during the process from the precursor solution to the consolidated composite material. The application of pressure during processing of ACCs has been used to ensure consolidation and dimensional stability in the final material, adding yet another control factor in both the regeneration and drying stages.10,22

In the present work, the Taguchi methodology is implemented to quantify the impact of various control factors on the mechanical properties of ACCs produced via a laminate manufacturing route termed solvent infusion processing.22 A preform (single or multiple woven or non-woven textile layers) is infused with a solvent through the application of a low vacuum pressure, similar in principle to vacuum-assisted resin transfer molding of conventional composites. Solvent infusion is then followed by temperature-triggered partial dissolution of the textile fibers in situ. The effect of the control factors on the microstructure was also investigated to gain greater understanding of the processing–structure–property relationships of ACCs.

### Experimental procedures

#### Materials

A rayon fiber textile (Cordenka GmbH, Obernburg, Germany) in the form of a K2/2 twill weave was used. The textile was based on a multifilament yarn (Cordenka 700, 1840 dtex, f 1000) with a filament diameter of 12 μm and final areal weight of 450 g/m². The textile layers (120 (l) × 120 (w) mm) were dried under vacuum at 80 °C for 24 h prior to use. The ionic liquid, 1-butyl-3-methylimidazolium acetate (BmimAc) (BASIONIC™ BC 02, supplied by Sigma-Aldrich, St. Louis, USA), was dewatered under vacuum at 80 °C for 5 days prior to use.

#### Preparation of all-cellulose composite laminae

The ACC lamina was made using solvent infusion processing (SIP) which procedure has been described by Huber et al.23 Briefly, a single layer of the textile was first placed on a flat plate mold covered with a bag, then it was vacuum and infused with BmimAc at room temperature (21 ± 2 °C). After this, the material–mold assembly was placed in a hot press for making a consolidated ACC lamina. The processing conditions investigated included dissolution time (t₉), dissolution temperature (T₉), and compaction pressure during hot pressing (σₚ) (Table 1). Once the hot pressing was completed, the material–mold assembly was cooled to room temperature (21 ± 2 °C) and the ACC lamina was then immersed in distilled water for a period of 3 days to regenerate the dissolved cellulose. The distilled water was replaced every 6 h over the first day to ensure rapid removal of the solvent, and replenished once daily over the second and third days. The resulting ACC lamina was then vacuum dried at 60 °C for 2 days under a pressure of 0.1 MPa.

#### Experimental design

The selection of control factors (t₉, T₉, and σₚ) and their levels as given in Table 1 were decided based on previous experimental findings.7,10,22–26 It has been reported that the mechanical properties of ACCs increase with dissolution time (t₉) until a critical point after which the mechanical properties of ACCs may decrease due to the excessive dissolution of cellulose, resulting in an increased volume fraction of the matrix phase. Conversely, the formation of insufficient matrix phase at shorter t₉ may lead to poor fiber–matrix adhesion that decreases the mechanical properties of the final ACC.7

It is also reported that an increase in dissolution temperature (T₉) would normally decrease the viscosity of the solvent, resulting in enhanced diffusion of the solvent into the cellulose textile that leads to more uniform dissolution of the cellulosic matrix and improved mechanical properties.23 However, degradation of the cellulose molecule may occur if T₉ is too high, resulting in a decrease in the mechanical properties of ACCs.24 For instance, cellulose is thought to be degraded by most of the cellulose-dissolving ionic liquids at temperatures above ~150 °C.27

Huber et al. observed that increase in hot-pressing pressure (σₚ) improves the mechanical properties of ACCs, presumably due to a more uniform distribution and consolidation of the matrix phase that surrounds the undissolved fibers and the elimination of voids.19 However, excessive pressure may force the cellulose fibers to separate that reduce the adhesion among fibers resulting in a decrease in the mechanical properties of ACCs.23

Using traditional design method, a full factorial experiment for three control factors and three levels would require 27 (3³) trials. By comparison, according to the Taguchi fractional factorial experimental design, only nine trials are required to complete the experimental matrix.28,29 An L₉ orthogonal array corresponds with the number of control factors and levels to be explored in this work (Table 2).

The Young's modulus (E) and ultimate tensile strength (UTS) are considered as "larger-the-better" quality characteristics in the context of a Taguchi analysis. Hence, the signal-to-noise ratio (S/N) is calculated using Equation (1).

\[
S / N = -10 \log \left( \frac{1}{n} \sum_{i=1}^{n} \frac{1}{x_i^2} \right)
\]

### Table 1 The selected control factors and their levels

| Control factors | Levels |
|----------------|--------|
| t₉ (min) | 30 60 90 |
| T₉ (°C) | 95 105 115 |
| σₚ (MPa) | 0.25 0.5 1.0 |

### Table 2 L₉ orthogonal array with the levels of the control factors used in each of the 9 trials

| Trial | t₉ (min) | T₉ (°C) | σₚ (MPa) |
|-------|---------|---------|----------|
| 1     | 30      | 95      | 0.25     |
| 2     | 30      | 105     | 0.50     |
| 3     | 30      | 115     | 1.00     |
| 4     | 60      | 95      | 0.50     |
| 5     | 60      | 105     | 1.00     |
| 6     | 60      | 115     | 0.25     |
| 7     | 90      | 95      | 1.00     |
| 8     | 90      | 105     | 0.25     |
| 9     | 90      | 115     | 0.50     |
Mechanical testing

Tensile testing was carried out according to ASTM D3039\textsuperscript{17} using an MTS Criterion Model C43.104 load frame equipped with a 2.5-kN load cell. Rectangular coupons (100 (l) × 10 (w) mm) were manually cut using a fresh razorblade. These samples were conditioned at 23 °C and 50% relative humidity (RH) for 24 h prior to testing at a constant crosshead speed of 2 mm/min. The samples were tested with a gauge length of 40 mm. Non-contact measurement of the strain was performed using a video extensometer (MTS FVX, TestWorks Axial/Transverse Video Extensometer with Video Traction software). The average $E$ and UTS were based on five replicates.

Results and discussion

Taguchi and statistical analyses

The highest $E$ and UTS are observed for Trial 6, while Trial 3 resulted in the lowest $E$ and UTS (Table 3). A larger value of S/N indicates a greater influence of the control factor on ACC production. The highest S/N ratio for $t_s$ and $T_s$ on $E$ are found at Level 2 (Figure 1). These results correlate with a $t_s$ and $T_s$ of 60 min and 105 °C, respectively (Table 1). The highest S/N ratio for $t_s$ on UTS is observed at the same level as $t_s$ on $E$. In contrast, the highest S/N ratio for $T_s$ on UTS is found at Level 1 which correlates with a $T_s$ of 95 °C (Table 1). The highest S/N ratio for $\sigma_p$ on $E$ and UTS is observed at the same level (Figure 1 and 2), corresponding with $\sigma_p = 0.25$ MPa (Table 1).

The optimal combination levels for maximizing $E$ was at $t_s$, $T_s$, and $\sigma_p$ of 60 min, 105 °C, and 0.25 MPa, respectively (Figure 1). Meanwhile, the optimal combination for maximizing UTS was at $t_s$, $T_s$, and $\sigma_p$ of 60 min, 95 °C, and 0.25 MPa, respectively (Figure 2).

The above results are consistent with those reported in literature. Huber et al. reported that both $E$ and UTS are affected by $t_s$, and $\sigma_p$ in a similar manner as shown in Figures 1 and 2. From the SEM images (Figure 3), it is found that a reduction

\begin{table}[h]
\centering
\caption{Summary of the $E$, UTS and S/N values for the 9 trials. The standard deviation is indicated in parentheses.}
\begin{tabular}{|c|c|c|c|c|c|c|}
\hline
\textbf{T}rials & $t_s$ (min) & $T_s$ (°C) & $\sigma_p$ (MPa) & $E$ (GPa) & S/N & UTS (MPa) & S/N \\
\hline
1 & 30 & 95 & 0.25 & 5.8 (0.04) & 15.3 & 75.3 (0.17) & -22.5 \\
2 & 30 & 105 & 0.5 & 5.9 (0.11) & 15.4 & 74.2 (0.20) & -22.6 \\
3 & 30 & 115 & 1.0 & 5.4 (0.15) & 14.7 & 73.9 (0.15) & -22.6 \\
4 & 60 & 95 & 0.5 & 7.1 (0.19) & 17.0 & 76.9 (0.25) & -22.3 \\
5 & 60 & 105 & 1.0 & 7.2 (0.19) & 17.2 & 77.2 (0.18) & -22.3 \\
6 & 60 & 115 & 0.25 & 7.3 (0.08) & 17.3 & 77.7 (0.11) & -22.2 \\
7 & 90 & 95 & 1.0 & 6.9 (0.14) & 16.8 & 72.5 (0.14) & -22.8 \\
8 & 90 & 105 & 0.25 & 6.8 (0.06) & 16.7 & 71.8 (0.19) & -22.9 \\
9 & 90 & 115 & 0.5 & 6.8 (0.12) & 16.6 & 71.2 (0.12) & -23.0 \\
\hline
\end{tabular}
\end{table}

where $n$ is the number of measurements in each trial, $x$ is the mechanical property value ($E$ or UTS), and S/N ratio is in decibels (dB). Therefore, based on Equation (1), higher values of $x$ will lead to a higher S/N ratio that consequently indicates greater influence of the control factor on the quality characteristic (i.e. $E$ or UTS). The ANOVA parameters were also calculated, including the F-ratio ($F$), and percentage contribution ($P$). The most significant control factor was shown by the highest $P$. In addition, the comparison of $F$ ratio of the factor variance to the error variance was used to highlight significant control factors in the experimental design, where $F_{max}$ is determined from the F distribution table.

\[
CrI = \frac{\text{Area}_{\text{crystalline}} - \text{Area}_{\text{amorphous}}}{\text{Area}_{\text{crystalline}}}
\]

\[
D = \frac{K\lambda}{\beta \cos \theta}
\]

where $D$, $\lambda$, $\beta$, and $\theta$ are determined using the Scherrer equation (Equation 3).\textsuperscript{17}
in $t_d$ results in an increased presence of microvoids (Figure 3(a)), where the diameter of the microvoids is in the range of ~1–2 μm. The presence of microvoids is also concomitant with a reduction in the matrix phase that will reduce the transfer of load from the matrix to the fibers (Figure 4). However, the reinforcing and matrix phases that are both composed of cellulose II should not have greatly differing elastic moduli and thus loading sharing is not expected to play a critical role in the final mechanical properties of the ACC lamina. It is more likely that the presence of voids reduces the overall elastic modulus of the material and the critical stress required for the onset of failure. Further detailed investigation is required to observe the effects of voids on the $E$ of ACC. The above observations are consistent with a 26 and 5.1% decrease in $E$ and UTS with a change of $t_d$ from 60 to 30 min, respectively (Table 3).

The microstructure in cross section comprised a uniform distribution of matrix phase that surrounds the fibers when processed with an optimal $t_d$ and $\sigma_p$ of 60 min and 0.25 MPa, respectively (Figure 3(b)). Under the above optimal processing conditions, $E$ and UTS of the ACC were increased to 7.3 GPa and 77.7 MPa, respectively, (Table 3) due to enhanced homogeneity and consolidation of the ACC microstructure (Figure 3(b)).

The observation of widespread fiber–matrix interfacial failure tends to suggest that an interface or interphase exists in ACCs in spite of the fibers and matrix being chemically similar. A 6.8 and 7.6% decrease in $E$ and UTS are observed with a change of $t_d$ from 60 to 90 min, respectively (Table 3). Increased dissolution of the fibers is observed as $t_d$ is increased as evidenced by an increase in $V_m$ (Figure 4). A decrease in $E$ and UTS is also observed in conjunction with an increase in $V_m$ at a $t_d$ of 30 or 90 min for the reasons as discussed above (Figure 4).

Although 10.0% matrix is often insufficient for wetting of fibers in conventional composites, the matrix is formed in all-cellulose composites in situ, resulting in the matrix phase being directly adjacent and in close proximity to the fiber surfaces, minimizing void formation due to insufficient wetting. It is asserted here that an interface and/or interphase do exist since failure does appear to emanate from the fiber–matrix boundary even in a microstructure in which most fibers are wetted by the matrix. It is also rare to observe a transverse failure of a fiber in spite of these ACCs consisting solely of cellulose II. The presence of an interphase and/or interface in ACCs remains to be clarified. The exact nature of the interface is still unknown in these materials. Attempts to characterize the interface/interphase region are hampered by experimental
It is possible that a compaction pressure of 0.25 MPa during dissolution decreases the void content. An increase in $\sigma_p$ increases the final mechanical properties of the ACC laminae, as previously discussed by Huber et al.\textsuperscript{10} A fully consolidated composite laminate is possible with the addition of compaction pressure.\textsuperscript{10} Further work that involves expanding the lower end range of the compaction pressure may be required.

**Effect of dissolution time on the cellulose II structure**

Generally, the $\text{CrI}$ of the ACC lamina was found to increase from that of the as-received cellulose II textile (42%\textsuperscript{10}) as shown by an increase in peak height and decrease in peak width in the $(1\bar{1}0)$ and $(200)$ planes, respectively (Figure 5). An increase of $\text{CrI}$ from 49.7 to 58.6% was observed with an increase in $t_d$ from 30 to 90 min as calculated via the integral method according to Wakelin et al.\textsuperscript{34} (Figure 6). An increase in $\text{CrI}$ is likely to be due to the solvent selectively dissolving amorphous cellulose that subsequently regenerates into a more crystalline (or paracrystalline) phase.\textsuperscript{22} In contrast, Soykeabkaew et al. report that $\text{CrI}$ decreases with increasing $t_d$ due to an increase in the fraction of amorphous phase under similar conditions to the present work.\textsuperscript{7} An increase in the crystallite size was also observed with increasing $t_d$ (Figure 6). The increase in crystallite size is difficulties due to the small fraction of matrix phase that requires probing by nanoscale characterization methods.

According to the ANOVA results, $t_d$ is the most significant control factor for both $E$ and UTS as indicated by the highest $P$ value (Tables 4 and 5) and $F > F_{\text{crit}}$. The relatively small influence of $T_d$ on $E$ and UTS could be due to the narrow $T_d$ range used, although at higher $T_d$ it would be necessary to verify that cellulose degradation does not take place. The lack of influence of $T_d$ over $E$ and UTS was indicated by the lowest $P$ value (Table 4 and 5) and $F < F_{\text{crit}}$.

The optimal level of $\sigma_p$ was identified as 0.25 MPa that corresponds closely with findings by Huber et al. (0.20 MPa).\textsuperscript{22} However, $\sigma_p$ appears to have a relatively small effect on $E$ and UTS as indicated by a lower $P$ (Tables 4 and 5) and $F < F_{\text{crit}}$.

![Figure 3](image1.png)

Figure 3: Scanning electron micrographs (SEM) of ACC laminae that were processed with dissolution times of (a) 30, (b) 60, and (c) 90 min

![Figure 4](image2.png)

Figure 4: Fiber, matrix and void volume fractions as a function of dissolution time

### Table 4 ANOVA results for Young’s modulus

| Control factor | $F$ | $P$ (%) | $S/N$ differences | Significance |
|---------------|-----|---------|------------------|--------------|
| $t_d$         | 41.09 | 96.67 | 2.04 | High |
| $T_d$         | 0.19 | 0.44 | 0.17 | Weak |
| $\sigma_p$    | 0.22 | 0.51 | 0.18 | Weak |

$^*F_{\text{crit at } 2,4} = 6.94.$

### Table 5 ANOVA results for ultimate tensile strength

| Control factor | $F$ | $P$ (%) | $S/N$ differences | Significance |
|---------------|-----|---------|------------------|--------------|
| $t_d$         | 84.38 | 95.2 | 0.63 | High |
| $T_d$         | 1.33 | 1.50 | 0.07 | Weak |
| $\sigma_p$    | 1.92 | 2.16 | 0.09 | Weak |

$^*F_{\text{crit at } 2,4} = 6.94.$

It is possible that a compaction pressure of 0.25 MPa during dissolution decreases the void content. An increase in $\sigma_p$ increases the final mechanical properties of the ACC laminae, as previously discussed by Huber et al.\textsuperscript{10} A fully consolidated composite laminate is possible with the addition of compaction pressure.\textsuperscript{10} Further work that involves expanding the lower end range of the compaction pressure may be required.
due to an increase in the lateral crystallite thickness perpendicular to the fiber axis. Generally, the relationship between crystallite size and mechanical properties of ACC is inversely proportional in this present study which can be observed at $t_d$ of 90 min. This finding is due to the increasing matrix fraction of ACCs at $t_d$ of 90 min (Figure 4), resulting in the decreasing mechanical properties of ACCs. However, further works have to be done in order to obtain a better relationship between crystallite size and mechanical properties of ACCs.

Conclusions

The present study shows that it is possible to approach the optimization of all-cellulose composites through the use of a Taguchi-type analysis. The results of the Taguchi analysis could be correlated with microstructural changes that are found to be sensitive to the processing conditions used to fabricate an ACC laminae. The main conclusions that could be drawn from this study of ACC laminae are as follows:

- $t_d$ imparts the greatest influence on $E$ and UTS of ACC laminae as shown by a Taguchi fractional factorial experimental design and ANOVA. In contrast, $\sigma_f$ and $T_f$ had a relatively small effect on the mechanical properties of ACC laminae;
- A $t_d$ of 60 min and $\sigma_f$ of 0.25 MPa are found to be optimal in maximizing $E$ and UTS of the final ACC laminae. The optimization of the mechanical properties was associated with a more homogeneous, void-free microstructure in the ACC laminae;
- An increase in CrI and crystallite size were observed with an increase in $t_d$ in ACC laminae; and
- An increase of $t_d$ up to 90 min results in a nonlinear, curved crack morphology due to an increase in $V_f$. The presence of microvoids is observed at a $t_d$ of 30 min, leading to a decrease in $E$ and UTS.

In future work, the authors will test the hypothesis that the optimization approach taken here may be applied to multiaxial all-cellulose laminates that are expected to be significantly different due to changes in failure mechanisms.

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Disclosure statement

No potential conflict of interest was reported by the authors.

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