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Effect of Steam Autoclaving on Laser Sintered Polyamide 12

Abstract

Purpose – The use of Laser Sintering (LS) in the medical sector has increased dramatically in recent years. With the move towards direct use of these parts in clinical applications, there is a greater need to understand the effects of standard processes, such as sterilisation, on the mechanical properties.

Design/methodology/approach – The research presented here focuses on the effect of **a single** steam sterilisation **cycle** on the mechanical properties of polyamide 12 parts manufactured using LS. The influence of water content on the properties was investigated, with additional drying steps trialled to establish the potential to reverse any changes observed, and to determine their root cause.

Findings – The results show that steam sterilisation has a significant effect on the mechanical properties of LS polyamide 12 parts, with a 39% reduction in elastic modulus, a 13% decrease in ultimate tensile strength, and a 64% increase in the elongation at break. These properties were also all found to correlate with the water content, suggesting that this was the cause of the difference. The original properties of the parts were able to be recovered after oven-drying.

Practical implications – These results show that with an additional drying step, LS polyamide 12 parts can be steam sterilised with no effect on the mechanical properties.

Originality/value – This is believed to be the first investigation into the effects of steam sterilisation in isolation on LS polyamide 12 parts, the first instance of drying parts to recover mechanical properties, and the first instance of multiple water content measurements being directly linked to the mechanical properties.

Keywords: Laser Sintering, Polyamide 12, Sterilisation, Water Content

1. Introduction

Over the last 30 years, Laser Sintering (LS) has established itself as one of industry's preferred methods of manufacturing functional, end-use products using Additive Manufacturing (AM). As with the majority of AM processes, material is joined layer by layer to make parts from 3D model data (ASTM, 2015); **with LS selectively melting (or sintering) consecutive cross-sections of a powder bed to build parts. For polymer materials, LS has the added benefit of unsintered powder acting as support material. This means that** LS has the capability of producing multiple personalised and unique parts in the same build, with little appreciable increase in manufacturing cost due to the geometric complexity (Goodridge *et al.*, 2012; Schmidt *et al.*, 2017). Combined with the relatively short lead times offered by AM, this makes LS an attractive option for the medical sector, where there is an inherent need for personalised, patient-specific medical devices.

Often used in the creation of 3D models for the visualisation and planning of complex surgeries, AM parts are also used directly to treat patients by acting as surgical guides or even implants (Leiggener *et al.*, 2009; Gibson, 2005). All these are able to be produced far more effectively, economically and with greater complexity than by using traditional manufacturing methods, resulting in shorter recovery times and reducing the need for repeat surgeries. However, there are certain procedures that any parts must be proven to withstand before entering a highly controlled environment such as an operating theatre. Guidelines in place for over 50 years, state that critical items (such as surgical guides) which pose a high risk of infection if contaminated with any microorganisms, must be sterilised before use (Spaulding, 1968; Rutala *et al.*, 2017; Rutala and Weber, 2004).

As the use of AM in medical devices has increased in recent years, specific guidance for the testing of parts is starting to be produced addressing the unique challenges associated with AM. For example, with the potential for multiple post-processing steps, specifying that the mechanical properties must be measured after parts have been "subjected to all post-processing, cleaning, and sterilisation steps" (U.S. Food & Drug Administration, 2017) could mean that different values are found. The effect of these steps therefore needs to be more thoroughly understood, particularly for devices such as surgical guides, which rely on the stiffness and dimensional accuracy in order to maintain their shape while in use. For polymer parts fabricated with other AM technologies, this has occasionally been seen as a limitation, both due to their stiffness and the need for more lengthy and expensive low-temperature sterilisation methods (Dahake *et al.*, 2016; Marei *et al.*, 2019). Some studies focusing on the effects of steam sterilisation have been carried out (Marei *et al.*, 2019; Török *et al.*, 2020); however the broad range of AM processes, materials, and sterilisation methods available, means that many more combinations still to be investigated.

The term sterilisation covers "any process by means of which all forms of microbial life (bacteria, spores, fungi, and viruses), contained in liquids, on instruments and utensils, or within various substances, are completely destroyed" (Perkins, 1969). The most common, and preferred, method of achieving this is through steam sterilisation in an autoclave (Rutala *et al.*, 2017; Rutala and Weber, 2004), where direct contact with high temperature steam is used to kill microbes (Dion and Parker, 2013). This method is widely used in hospitals due to its effectiveness, ease of use, low cost, and lack of any chemical residue.

While LS polyamide 12 is often marketed as being able to withstand the conditions in steam sterilisation, there is a remarkable lack of literature supporting this, with specific regards to the mechanical properties. With such parts now being used for critical applications, such as surgical guides (Krishnan *et al.*, 2012; Leiggener *et al.*, 2009), there is a need for a deeper knowledge of how these parts are affected by the steam sterilisation process. Some research has been carried out in this area (Haerst *et al.*, 2015); however, this only focused on the multiple re-use of items and used a combination of different methods to sterilise the parts, making it impossible to differentiate the effects of each process. As the largest use of these parts is likely to be custom made, single-use applications, the effects of a single sterilisation cycle need to be understood more deeply. **This research focuses on these single-use applications such as surgical guides, where only initial sterilisation is needed.**

When exposed to the conditions present in an autoclave, changes could be caused by either the exposure to high temperature, exposure to moisture / water, or a combination of the two. The first of these, exposure to high temperatures, was not expected to have a large effect on the mechanical properties of the LS parts. This is based on the extensive research carried out on the effect of temperature on LS powders during the printing process, whose main focus is on powder re-use (Dadbakhsh *et al.*, 2017; Wudy and Drummer, 2019; Goodridge *et al.*, 2012). While thermal degradation of polyamide 12 can be experienced during printing, the temperatures the unsintered powder are exposed to (typically 160–180°C) exceed those of steam sterilisation (typically 121–134°C), and the time these are held at is much greater (even exceeding 24h depending on the machine and build setup). Despite this, it is common practice to reuse this unsintered powder **with either 100% virgin (unused), or a mixture of virgin and used (unsintered) powder generally chosen** to build parts with comparable mechanical properties. Some research into the effects of the temperatures experienced in steam sterilisation have been carried out on LS polyamide 11, with no significant difference found after one cycle (George and Crawford, 2010).

It has previously been shown that the mechanical properties of polyamides are affected by their water content, as the absorbed water molecules break hydrogen bonds in the material, lowering the glass transition temperature and potentially degrading the polymer (Batzer and Kreibich, 1981; Jia and Kagan, 2001; Rajesh *et al.*, 2002; Razumovskii *et al.*, 1985; Kurokawa *et al.*, 2003; Fan *et al.*, 2009; Radheshkumar and Münstedt, 2005). While some papers have alluded to this affecting the properties of LS polyamide parts (Goodridge *et al.*, 2010; Cooke *et al.*, 2011; Gibson and Shi, 1997; Moeskops *et al.*, 2004), few have explicitly measured this (Seltzer *et al.*, 2011; Salazar *et al.*, 2014) and conflicting effects on the mechanical properties have often been found. Focusing on the tensile properties, it is generally agreed that the ultimate tensile strength and the Young's modulus decrease with an increase in water content (Gibson and Shi, 1997; Goodridge *et al.*, 2010; Cooke *et al.*, 2011; Salazar *et al.*, 2014; Haerst *et al.*, 2015; Seltzer *et al.*, 2011), however the effect on the elongation at break has been reported to both increase (Goodridge *et al.*, 2010; Cooke *et al.*, 2011) and decrease (Salazar *et al.*, 2014; Seltzer *et al.*, 2011) as a result. It is worth noting that the majority of these studies did not measure the water content directly, often only hypothesising that observed changes in properties were due to increased water content. Additionally, the studies that did measure the water content only considered two cases of "dry" and "wet" samples, which were saturated under accelerated conditions (namely being submerged at 90°C for 80+ days). This process introduces more potential causes of any differences observed (such as physical ageing or higher temperatures) and only looks at the extremes of the possible water contents, meaning it was not possible to observe a trend in the results. Another key point, which has not been investigated, is whether after exposure to these adverse conditions, the properties of the parts can be recovered through simple methods such as drying, or whether they represent a permanent degradation of the polymer.

The research presented here investigates the effect of **a single** steam sterilisation **cycle** on the mechanical properties of LS polyamide 12 parts, separately investigating the effects of autoclaving (temperature and moisture) and temperature alone on the properties. The reversibility of any changes in mechanical properties was investigated as an indicator of the root causes, and a methodology to simply measure the water content at the time of tensile testing is presented.

2. Materials and Methods

2.1. Materials

Test specimens were printed on an EOS Formiga P100 Laser Sintering machine using a polyamide 12 powder (PA2200). In total, 45 test specimens were built in a 1×5×9 (XYZ) stack, in the same orientation (XY – with the longest dimension in the x-direction, parallel to the front of the machine), in the centre of a dedicated build. **This ensured comparability between the samples; however it should be noted that parts built in other orientations are likely to have different properties.** All parts were built with a 50/50 mix of virgin (unused) and used powder, a combination widely used in industry and recommended by the manufacturer. The default **"performance" parameters for 50/50 PA2200 were used (Pfefferkorn and Weilhammer, 2017)**, these being laser power 21 W, scan spacing 0.25 mm, scan speed 2500 mm/s, **layer height 100 µm** and a bed temperature of 170°C. Excess powder was removed from the parts in post-processing by bead blasting and compressed air.

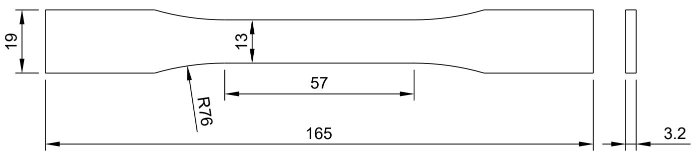


Figure 1: **Dimensions of a Type I tensile specimen from ASTM D638. All dimensions in mm.**

	As Built	Heat and Steam	Heat Only
No Drying	A	B	C
Air-dried	D	E	F
Oven-dried	G	H	J

Table 1: Combinations of conditioning (As Built, Heat and Steam, and Heat Only) and drying (No Drying, Air-dried, and Oven-dried) for each sample set.

2.2. *Mechanical Testing*

In order to determine the mechanical properties of the LS samples, tensile testing was used. **All testing was carried out in accordance with ASTM D638 (ASTM, 2014), with a type I geometry used (as shown in Figure 1)**. A Tinius Olsen 5K with Laser Extensometer was used, with 5 × specimens tested per sample set **at a rate of 5 mm/min**. The Young's Modulus (E), Ultimate Tensile Strength (σ_{uts}), and Elongation at Break (ε_{max}) were subsequently determined to characterise the mechanical properties.

2.3. *Specimen Preparation*

Some of the tensile test specimens were kept "as built", while the remainder received a combination of either heating with steam, or heating only (henceforth referred to as the conditioning step), followed by a drying step prior to testing. These were chosen to determine the effect of autoclaving (Heat and Steam) and of heat only, with three drying methods chosen to identify the causes of any changes observed. The combinations of these sample sets are summarised in Table 1, where each label represents a set of 5 × specimens.

Details for each of these conditioning and drying steps are shown in Sections 2.3.1 and 2.3.2.

2.3.1. *Conditioning*

To separately investigate the combined effects of autoclaving and of temperature alone, three different conditioning methods were used:

- As Built – no conditioning (control group).
- Heat and Steam – samples were subjected to steam at 121°C for 20 minutes. **Specimens were placed in autoclave pouches for steam sterilisation, allowing them to remain sterile after removal from the autoclave.**
- Heat Only – samples were heated to 121°C for 20 minutes (no steam).

The conditioning was carried out immediately after post-processing of the parts, with the autoclaving and heating taking place simultaneously to ensure comparability.

2.3.2. *Drying*

In order to test the reversibility of any changes, three methods of drying the specimens were investigated:

- No Drying – samples were tested immediately **after conditioning,** without any drying.
- Air-dried – samples were left uncovered in a non-dessicated environment for 7 days.
- Oven-dried – samples were held at 50°C for 7 days.

The "As Built – No Drying" specimens were used as a control group to compare all of the other combinations of conditioning and drying to, as this represented the standard properties after printing. The air-drying and oven-drying were fixed at 7 days to minimise any ageing effect. This was also expected to be sufficiently long to ensure the specimens held at 50°C reached their dried mass (see Section 2.4). As both these methods of drying were of equal length, tensile testing of all the **"Oven-dried" and "Air-dried"** samples could be carried out at the same time.

2.4. *Water Content*

In order to determine the water content at any given time t (w_t) during the sample conditioning and drying, two different methods were used. When the dried mass of the specimens (m_{dried}) could be measured, the value of w_t could be calculated directly (as detailed in Section 2.4.1). Where m_{dried} could not be measured directly (whenever there was no oven-drying step before tensile testing), the initial water content of the build (w_{int}) was determined and used to calculate the value of m_{dried}, which could then be used to find w_t (as detailed in Section 2.4.2).

To increase the accuracy of the measured water content value at the time of tensile testing (w_{test}), both the direct and indirect methods were used to calculate the pre- and post-test w_t. The mean of these was then used as the final value shown.

A summary of the entire conditioning and drying process is shown in Section 2.4.3, detailing all of the required measurements to calculate w_t using the direct and indirect methods.

2.4.1. *Directly from the Dried Mass*

Where oven-drying of the specimens was possible, the value of m_{dried} was easily obtained by weighing the dried parts. Using Equation 1, the value of w_t was then be calculated for time t, where m_t was the mass of the sample at time t (ASTM, 2010; ISO, 2008).

$$w_t = \frac{m_t - m_{\text{dried}}}{m_{\text{dried}}} \times 100 \qquad (1)$$

As this process removed all moisture from the specimens, and had the potential to affect the mechanical properties (through the exposure to elevated temperatures), it could not be used before tensile testing. However, since the effect on the broken (post-test)

Figure 2: View of a fractured tensile test specimen.

specimens was of no concern, further oven-drying was carried out on these to measure m_{dried} for the broken specimens.

Note that it was not possible to use the value of m_{dried} for the post-test specimens to calculate the pre-test water content. This was due to the tendency of polyamide 12 to fracture into more than 2 pieces during testing (see Figure 2); and as these small fragments could be easily lost, a comparison was not practical.

2.4.2. Indirectly from the Initial Water Content

For the times where the direct method could not be used, the value of m_{dried} was calculated, rather than measured (denoted as $m_{\text{dried}}{}^*$), by using the initial water content of the build (w_{int}). Since sample sets G, H and J were oven-dried as whole specimens as part of the conditioning process, the value of m_{dried} (and their initial mass, m_{int}) were used to calculate w_{int} using Equation 1. This initial water content (w_{int}) was assumed to be the same for all sample sets as all the specimens were manufactured in the same build.

With this value of w_{int}, the dried mass for any whole (pre-test) specimen could be determined by measuring their initial mass (m_{int}) and by rearranging Equation 1 for m_{dried} (as shown in Equation 2).

$$m_{\text{dried}}{}^* = m_{\text{int}} \left(\frac{100}{100 + w_{\text{int}}} \right) \tag{2}$$

This value of $m_{\text{dried}}{}^*$ was then used in place of m_{dried} in Equation 1 to determine w_t.

2.4.3. Protocol Summary

As previously mentioned, the values of water content at the time of tensile testing w_{test} were an average of the pre- and post-test water content. The measurements required to calculate these for each sample set are shown in Table 2.

Working backwards from these required measurements, and including the additional oven-drying steps necessitated by the direct method, an overview of the entire experimental procedure is shown in Figure 3.

3. Results

3.1. Mechanical Properties

The results of the tensile testing are shown in Figures 4 and 5, both as the raw data and the measured properties respectively. In Figure 4, it can be seen that all the stress-strain curves are broadly similar to one another, with the exception of the "Heat and Steam – No Drying" and "Heat and Steam – Air-dried"; which can be seen to be considerably different to the others, while following a similar profile.

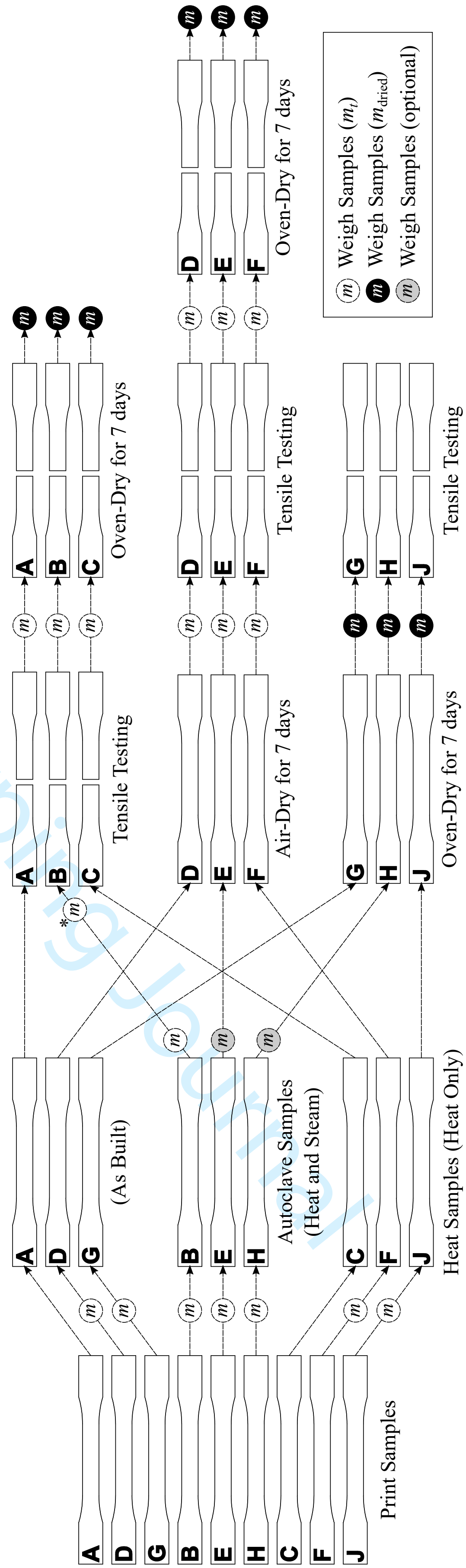


Figure 3: Overview of the protocol for conditioning and taking mass measurements. The additional pre-test measurement $^*m$ was only taken as there was an 18 hour delay between autoclaving and testing.

Sample	Values of w_t to average for w_{test}	
	Pre-Test	Post-Test
A	As printed (w_{int})	($m_{A,\text{pt}}$, $m_{A,\text{dried}}$)
B	After autoclaving (from $m_{B,\text{ac}}$ $m_{B,\text{int}}$, w_{int})	($m_{B,\text{pt}}$, $m_{B,\text{dried}}$)
C	As printed (w_{int})	($m_{C,\text{pt}}$, $m_{C,\text{dried}}$)
D	Air-dried (from $m_{D,\text{ad}}$, $m_{D,\text{int}}$, w_{int})	($m_{D,\text{pt}}$, $m_{D,\text{dried}}$)
E	Air-dried (from $m_{E,\text{ad}}$, $m_{E,\text{int}}$, w_{int})	($m_{E,\text{pt}}$, $m_{E,\text{dried}}$)
F	Air-dried (from $m_{F,\text{ad}}$, $m_{F,\text{int}}$, w_{int})	($m_{F,\text{pt}}$, $m_{F,\text{dried}}$)
G	Assumed 0	–
H	Assumed 0	–
J	Assumed 0	–

Table 2: Values of w_t to average to obtain w_{test}. Shown are the conditions to calculate, and the values required (shown in brackets) for use with the either the direct or indirect method. The mass values (m), are initial (m_{int}), after autoclaving (m_{ac}), after air-drying (m_{ad}), after testing (m_{pt}), and after oven drying (m_{dried}).

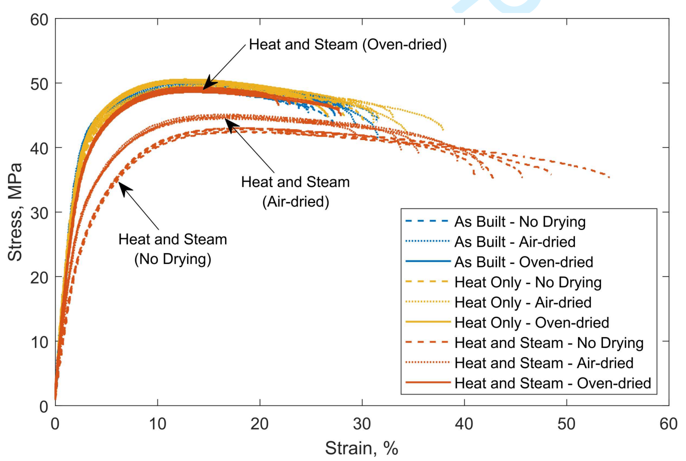
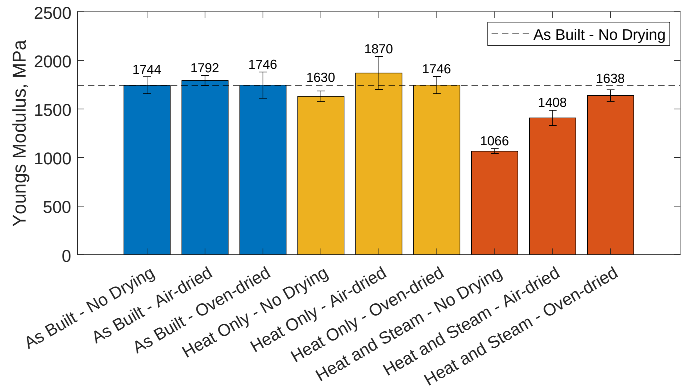


Figure 4: Stress-Strain data from tensile testing, with the results of all 45 test specimens shown. The majority of the curves can be seen to be similar, with "Heat and Steam – No Drying" and "Heat and Steam – Air-dried" samples appearing to be significantly different.

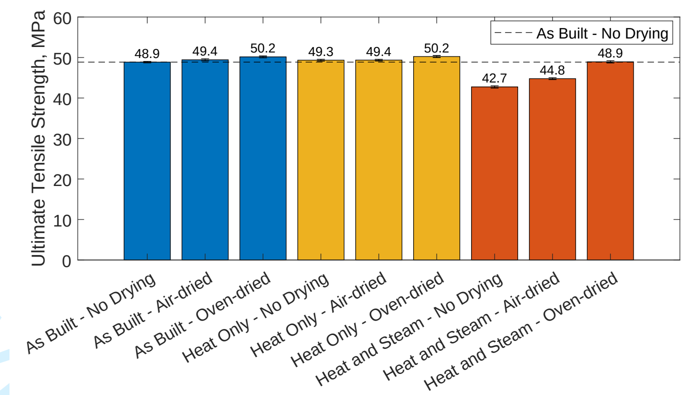
In Figure 5, it is worth noting that all of the samples initially resembled the "As Built – No Drying", with any subsequent changes due to the conditioning and drying. This initial value is therefore shown across all samples for comparison.

3.2. *Water Content*

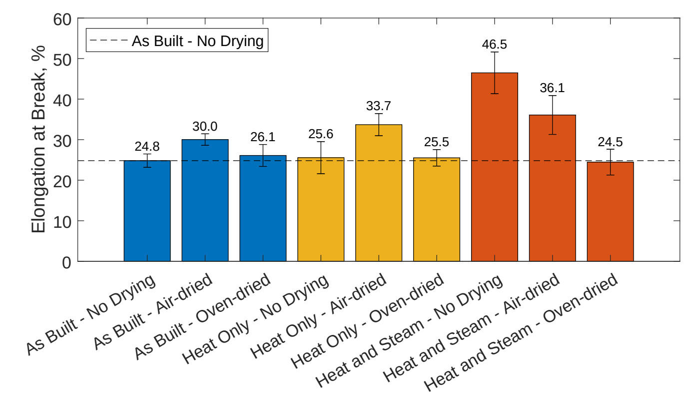
The values of w_{test} for the samples are shown in Table 3, where the pre- and post-test vales are shown alongside the mean. **The negative values shown indicate that the mass increased after oven-drying, suggesting the water content increased during drying. However, the mass change for this was approximately 0.002 g, which could be attributed to a zeroing error, to a drift in the machine calibration or to human error when handling the samples. Since these values were small relative to the changes observed for the "Heat and Steam" samples, this was**



(a) Elastic Modulus (E).



(b) Ultimate tensile strength (σ_{uts}).



(c) Elongation at break ($\varepsilon_{\max}$).

Figure 5: Tensile properties for all combinations of conditioning and drying, values are shown as the mean ± standard deviation. Here it can be seen that the "Heat Only" samples do not show any significant difference to the "As Built" samples, whereas the "Heat and Steam" samples show a marked decrease in modulus, decrease in ultimate tensile strength and increase in elongation at break. These differences are shown to be reversed by the drying steps, with all of the oven-dried samples re-gaining their original mechanical properties.

Sample Description		Water Content / %		
		Pre-Test	Post-Test	Average
As Built	A – No Drying	0.13	-0.02	0.05
	D – Air-dried	0.14	0.07	0.11
	G – Oven-dried	0	–	0
Heat Only	C – No Drying	0.13	-0.03	0.05
	F – Air-dried	0.07	0.06	0.07
	J – Oven-dried	0	–	0
Heat and Steam	B – No Drying	1.01	0.68	0.84
	E – Air-dried	0.60	0.48	0.54
	H – Oven-dried	0	–	0

Table 3: Calculated water content during testing, see Table 2 for methodology. All values are ±0.01.

deemed acceptable and the values can be assumed to represent a 0% water content.

The properties shown in Figure 5 are again shown in Figure 6 with respect to w_{test}. A linear fit has been added, with the calculated R^2 value shown as a measure of the goodness of fit. The value of w_{int} was found to be 0.13±0.00%, and the value of w_{ac} was found to be 1.49±0.02%.

4. Discussion

From the data shown in Figures 4 and 5, it can be clearly seen that there was a change in the mechanical properties after autoclaving (Heat and Steam). For these samples, E and σ_{uts} were initially lower than the as built values, whereas $\varepsilon_{\max}$ was significantly higher. These trends match those found in the literature for σ_{uts} and E, and agrees with the trend in $\varepsilon_{\max}$ where an increase in water content was not achieved under accelerated conditions. The samples which were exposed to heat only also showed the expected behaviour, with no significant change in the mechanical properties after exposure to the higher temperatures.

The reason for examining the different drying processes, was to determine the underlying cause of the differences observed. In the scenario where water content was the cause of any differences, logic dictates that after drying, the properties would revert to their original values. However, if these differences were caused by other factors (such as high temperatures), then this reversibility would not be expected. The results shown in Figures 4 and 5 clearly show that the "Heat and Steam" samples regain their original mechanical properties after oven-drying and partially regain them after air-drying. When combined with the lack of a difference in the "Heat Only", this suggests that the causes of the differences were due to the water content of the parts.

This theory was further supported by the measured water content (shown in Figure 6), where all the mechanical properties **show a negative trend** with the measured w_{test}; in this, the air-dried samples show a higher w_{test} than the oven-dried, whereas all the other samples have a similar water content. The samples initially exposed to the oven heating at 121°C, do not display a large divergence from the as built samples (Figure 5), again suggesting that the water content, rather than the temperature was the cause of the differences. Although a linear fit is shown in these results, similar experiments on injection moulded polyamide 6 samples (Jia and Kagan, 2001), suggest that these trends may not in fact be linear. Further work could focus on measuring the effects of water content over a wider range of values to obtain a more accurate measure of the effects on the mechanical properties.

Throughout this research, the sterility of the parts after the initial steam sterilisation was not maintained. For practical applications, this would need to be investigated further to ensure that the parts remained sterile after conditioning. For oven-drying, this could be achieved by using autoclave bags / pouches during steam sterilisation (as in this experiment) and subsequently drying both the part and bag inside an oven. Alternatively for air-drying, this could be carried out in a class II biological safety cabinet, enabling the part to be uncovered while increasing the airflow over the part. However, it is worth noting that the time required to fully dry the parts is likely to be dependent on the geometry, meaning it is not possible to determine a standard drying time. In these instances, repeated weighing of the part should be carried out, with the 0% water content achieved when the mass values plateau.

5. Conclusion

It has been shown that steam sterilisation (exposure to heat and steam in an autoclave) does affect the mechanical properties of LS PA2200 parts. Lower values of E and σ_{uts} were found after sterilisation, along with higher values of $\varepsilon_{\max}$. These changes were found to directly relate to the water content, with the original properties re-obtained after drying of the samples. From these results, a further post-processing step of oven-drying is suggested after autoclaving for **single-use** applications where the properties of the printed parts are critical.

6. Further Work

Before application in a critical setting, the effect the drying processes have on the sterility of the printed parts would have to be tested. In terms of the mechanical properties, the effect of orientation in the build could be investigated, as well as whether the crystallinity of the parts is affected by either the conditioning or drying processes.

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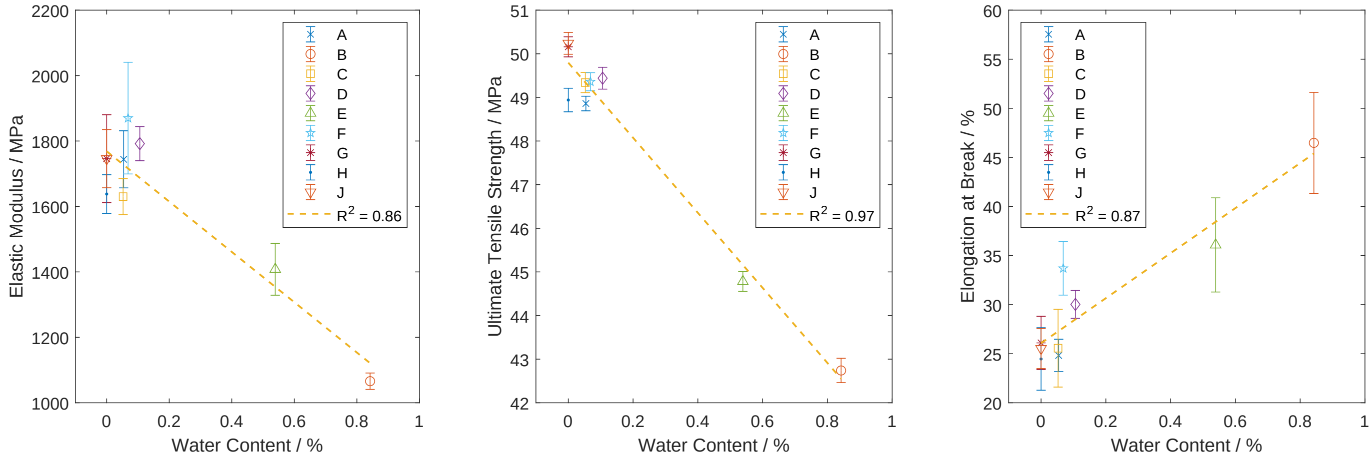

(a) Elastic Modulus (E). (b) Ultimate Tensile Strength (σ_{uts}). (c) Elongation at Break (ε_{max}).

Figure 6: Effect of water content (w_{test}) on mechanical properties. Linear fits have been added, with the R^2 values **shown, and all of the properties displaying a negative trend with increasing w_{test}.**

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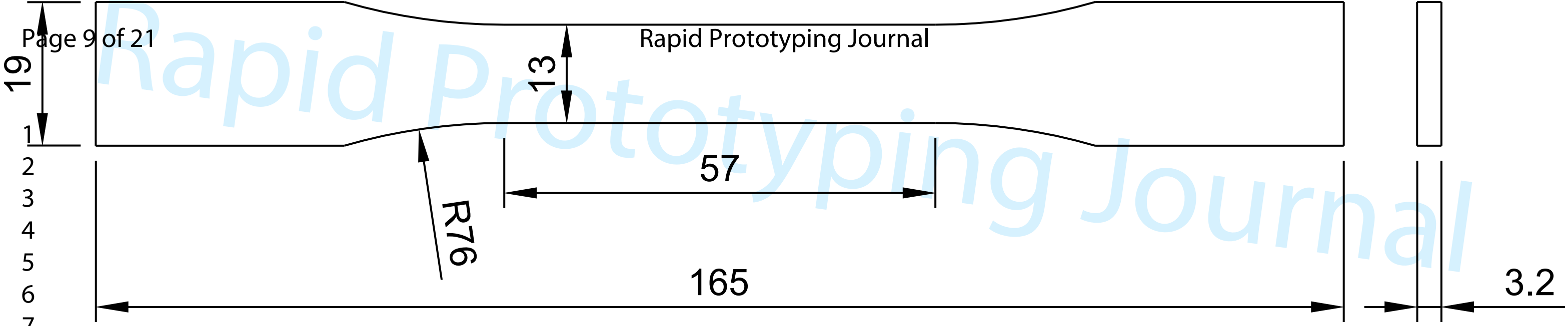
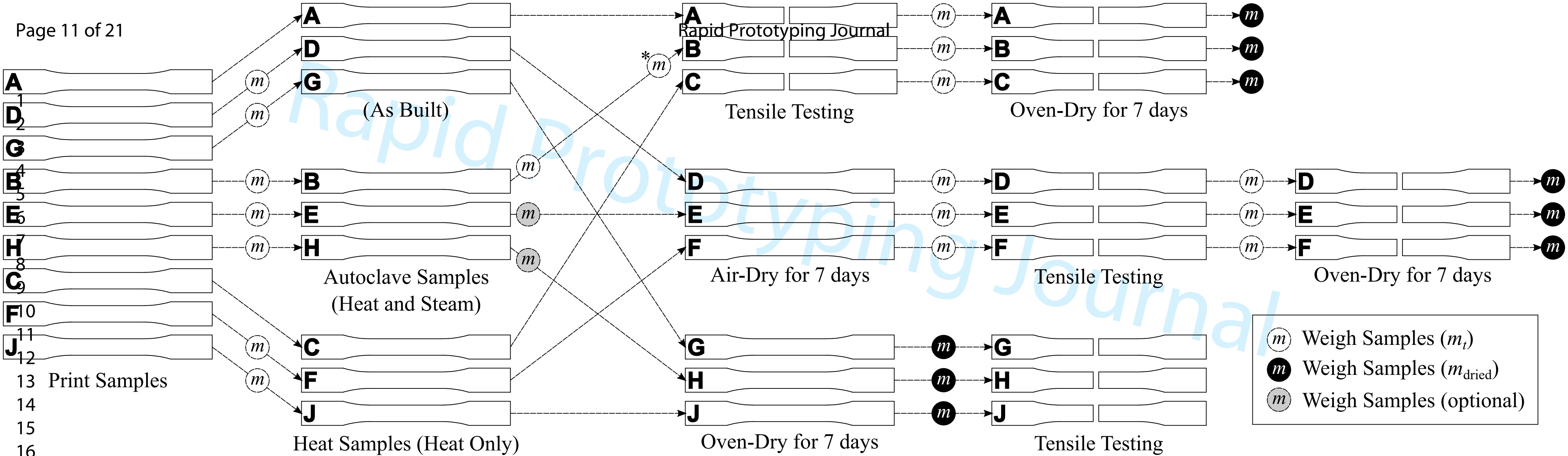

19
13
57
R76
165
3.2

Figure 2: View of a fractured tensile test specimen.

1378x246mm (72 x 72 DPI)


Print Samples
(As Built)
Autoclave Samples
(Heat and Steam)
Heat Samples (Heat Only)
Tensile Testing
Oven-Dry for 7 days
Air-Dry for 7 days
Tensile Testing
Oven-Dry for 7 days
Oven-Dry for 7 days
Tensile Testing
Weigh Samples (m_t)
Weigh Samples (m_{dried})
Weigh Samples (optional)

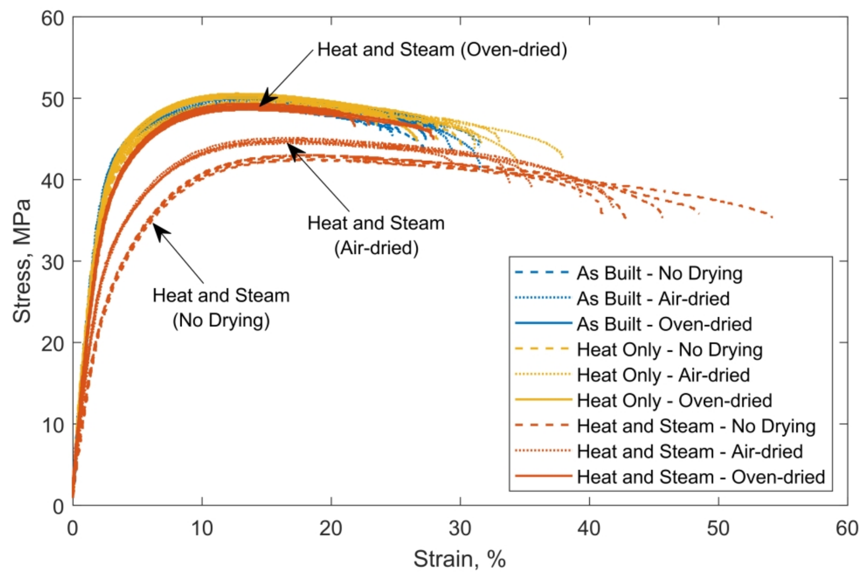


Figure 4: Stress-Strain data from tensile testing, with the results of all 45 test specimens shown. The majority of the curves can be seen to be similar, with "Heat and Steam - No Drying" and "Heat and Steam - Air-dried" samples appearing to be significantly different.

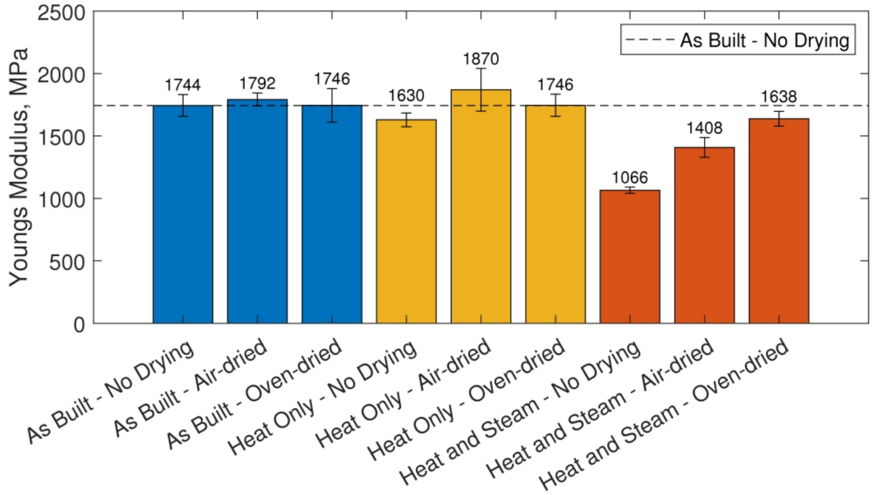


Figure 5 (a): Tensile properties for all combinations of conditioning and drying, values are shown as the mean ± standard deviation. Here it can be seen that the "Heat Only" samples do not show any significant difference to the "As Built" samples, whereas the "Heat and Steam" samples show a marked decrease in modulus, decrease in ultimate tensile strength and increase in elongation at break. These differences are shown to be reversed by the drying steps, with all of the oven-dried samples re-gaining their original mechanical properties. (a) Elastic modulus (E).

211x111mm (600 x 600 DPI)

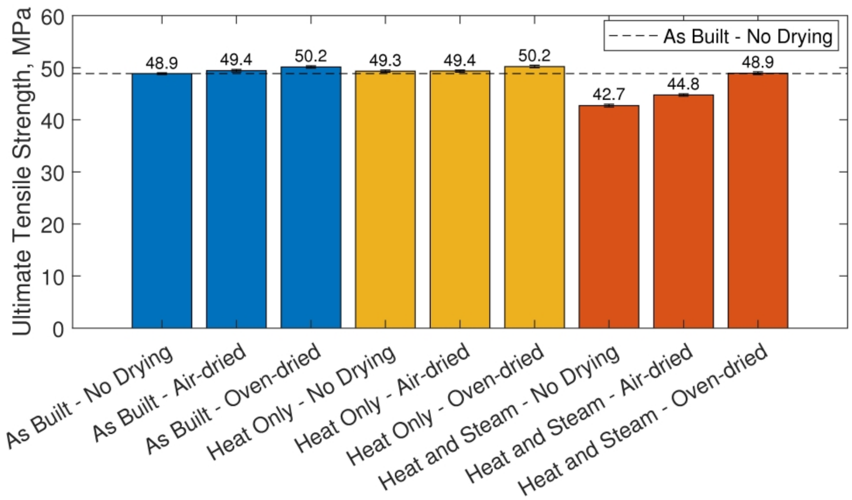


Figure 5 (b): Tensile properties for all combinations of conditioning and drying, values are shown as the mean ± standard deviation. Here it can be seen that the "Heat Only" samples do not show any significant difference to the "As Built" samples, whereas the "Heat and Steam" samples show a marked decrease in modulus, decrease in ultimate tensile strength and increase in elongation at break. These differences are shown to be reversed by the drying steps, with all of the oven-dried samples re-gaining their original mechanical properties. (b) Ultimate tensile strength (σ_{uts}).

211x111mm (600 x 600 DPI)

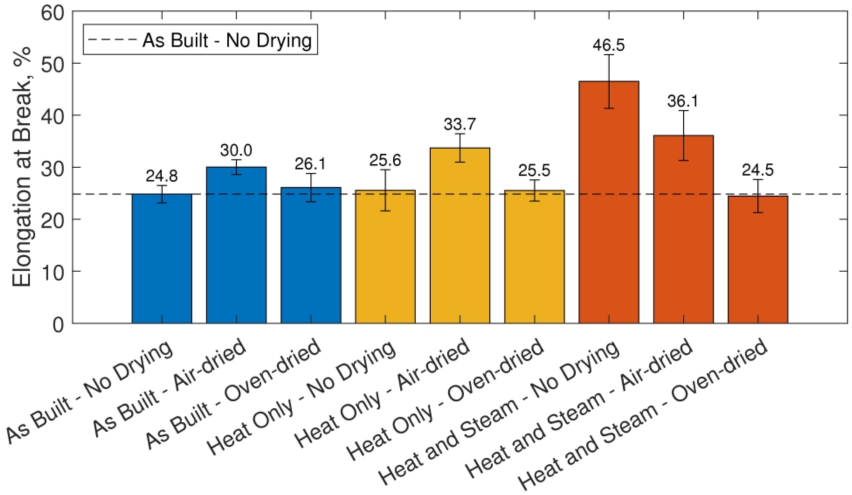


Figure 5 (c): Tensile properties for all combinations of conditioning and drying, values are shown as the mean ± standard deviation. Here it can be seen that the "Heat Only" samples do not show any significant difference to the "As Built" samples, whereas the "Heat and Steam" samples show a marked decrease in modulus, decrease in ultimate tensile strength and increase in elongation at break. These differences are shown to be reversed by the drying steps, with all of the oven-dried samples re-gaining their original mechanical properties. (c) Elongation at break (ε_{max}).

211x111mm (600 x 600 DPI)

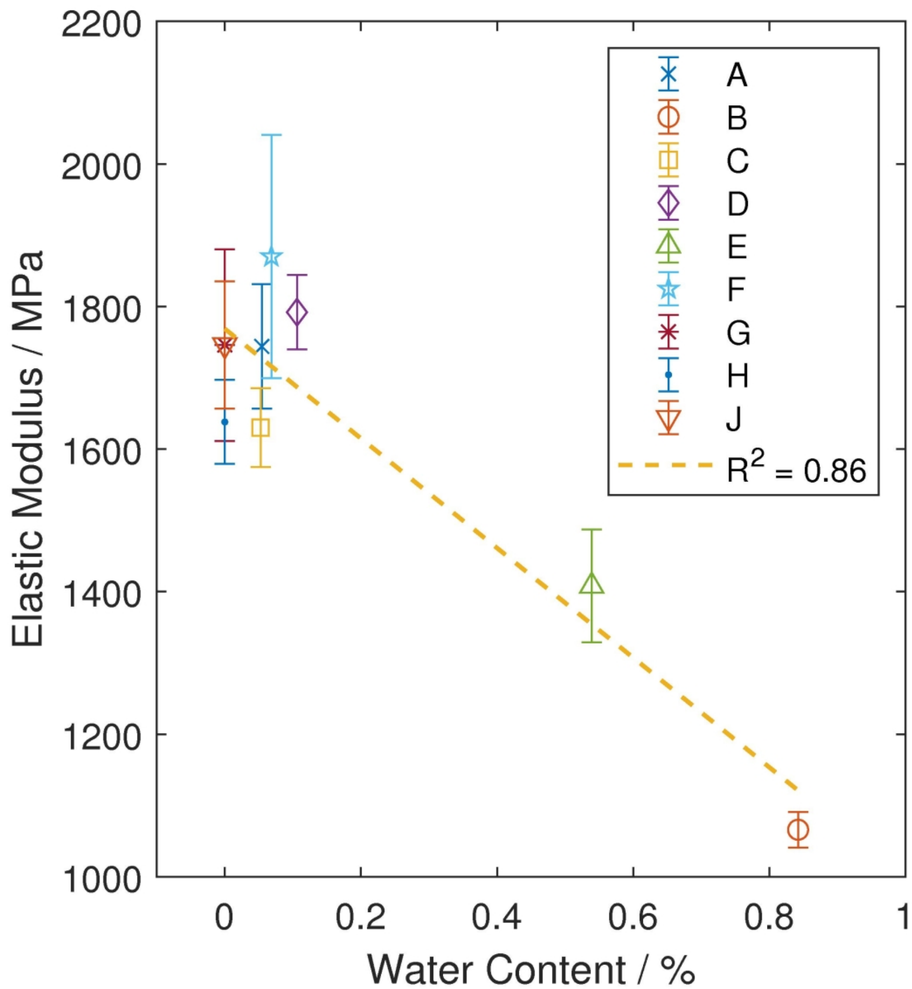


Figure 6 (a): Effect of water content (w_{test}) on mechanical properties. Linear fits have been added, with the R^2 values shown, and all of the properties displaying a negative trend with increasing w_{test}.

105x111mm (600 x 600 DPI)

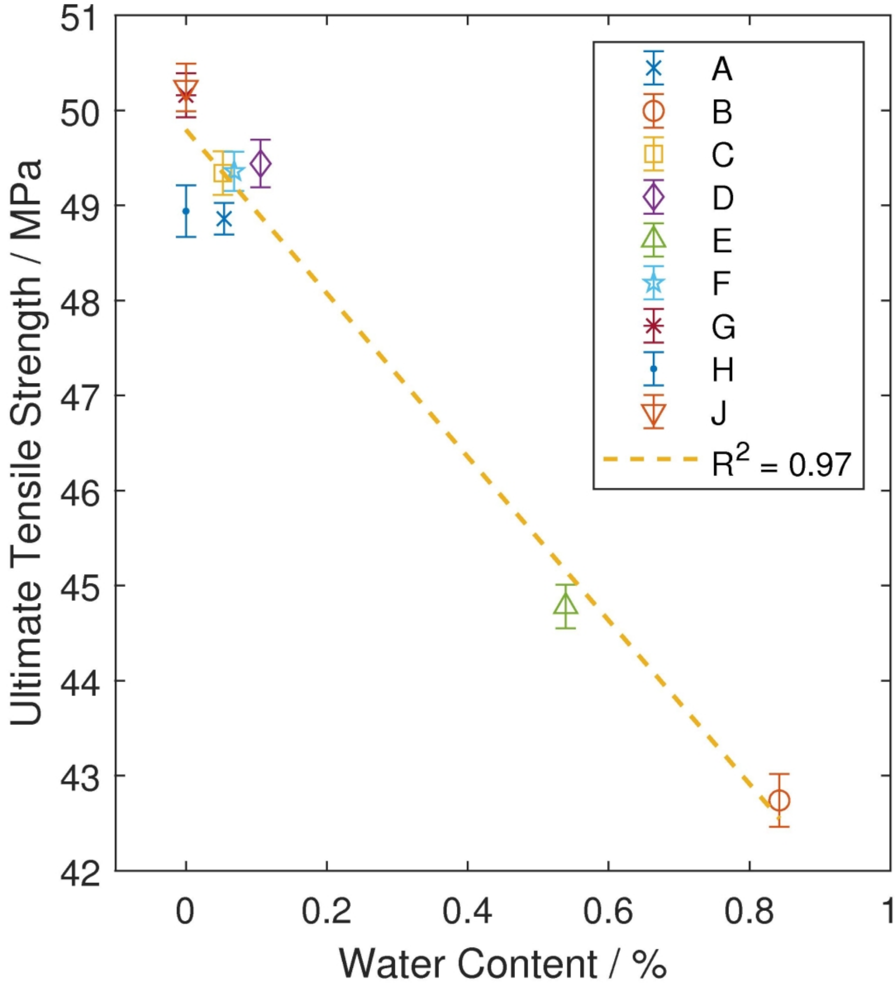


Figure 6 (b): Effect of water content (w_{test}) on mechanical properties. Linear fits have been added, with the R^2 values shown, and all of the properties displaying a negative trend with increasing w_{test}.

105x111mm (600 x 600 DPI)

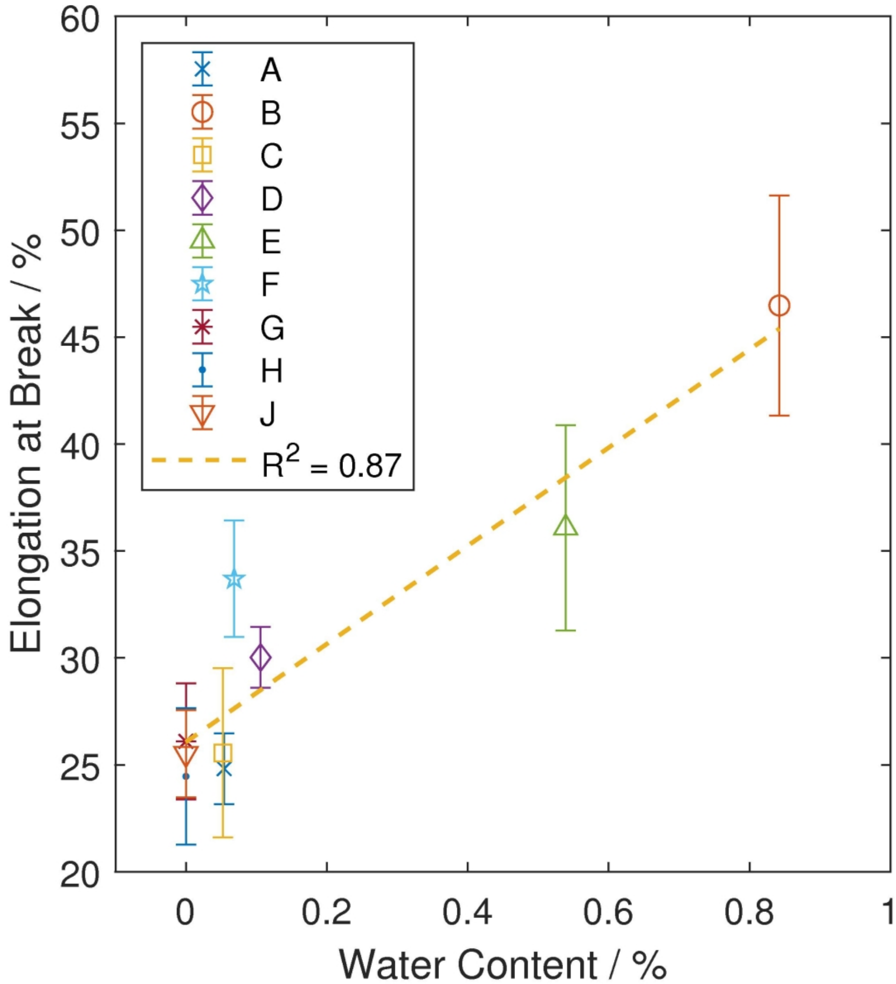


Figure 6 (c): Effect of water content (w_{test}) on mechanical properties. Linear fits have been added, with the R^2 values shown, and all of the properties displaying a negative trend with increasing w_{test}.

105x111mm (600 x 600 DPI)

	As Built	Heat and Steam	Heat Only
No Drying	A	B	C
Air-dried	D	E	F
Oven-dried	G	H	J

Sample	Values of w_t to average for w_{test}	
	Pre-Test	Post-Test
A	As printed (w_{int})	($m_{A,pt}$, $m_{A,dried}$)
B	After autoclaving (from $m_{B,ac}$ $m_{B,int}$, w_{int})	($m_{B,pt}$, $m_{B,dried}$)
C	As printed (w_{int})	($m_{C,pt}$, $m_{C,dried}$)
D	Air-dried (from $m_{D,ad}$, $m_{D,int}$, w_{int})	($m_{D,pt}$, $m_{D,dried}$)
E	Air-dried (from $m_{E,ad}$, $m_{E,int}$, w_{int})	($m_{E,pt}$, $m_{E,dried}$)
F	Air-dried (from $m_{F,ad}$, $m_{F,int}$, w_{int})	($m_{F,pt}$, $m_{F,dried}$)
G	Assumed 0	–
H	Assumed 0	–
J	Assumed 0	–

Sample Description		Water Content / %		
		Pre-Test	Post-Test	Average
As Built	A – No Drying	0.13	-0.02	0.05
	D – Air-dried	0.14	0.07	0.11
	G – Oven-dried	0	–	0
Heat Only	C – No Drying	0.13	-0.03	0.05
	F – Air-dried	0.07	0.06	0.07
	J – Oven-dried	0	–	0
Heat and Steam	B – No Drying	1.01	0.68	0.84
	E – Air-dried	0.60	0.48	0.54
	H – Oven-dried	0	–	0