

# Process-Structure-Property Relationships and Multi-Response Optimization of Z-Direction MEX-Printed Polyphenylene Sulfide

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## Research Article

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# **Process-Structure-Property Relationships and Multi-Response Optimization of Z-Direction MEX-Printed Polyphenylene Sulfide**

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## ABSTRACT

Material extrusion (MEX) of polyphenylene sulfide (PPS) is strongly governed by interlayer consolidation, particularly under Z-direction loading. However, whether minimizing the overall apparent porosity also maximizes Z-direction mechanical performance remains unclear. This study investigates the effects of nozzle temperature (315–345 °C), layer thickness (0.1–0.3 mm), and print speed (20–60 mm/s) on Young's modulus, tensile strength, elongation at break, and density-derived apparent porosity using a face-centred central composite design and response surface methodology. Dynamic mechanical analysis and scanning electron microscopy were used to support the process–structure–property interpretation. The tensile responses were strongly affected by the investigated processing variables, whereas print speed had no significant effect on apparent porosity ( $p = 0.517$ ). Importantly, the best measured mechanical response occurred at 345 °C, 0.1 mm, and 20 mm/s, whereas the minimum apparent porosity occurred at 330 °C, 0.2 mm, and 40 mm/s. This divergence demonstrates that minimum bulk porosity does not necessarily correspond to maximum Z-direction performance. The SEM images revealed that better mechanical performance correlates with lesser interlayer spaces as well as higher local fusion. The multi-response optimization method estimated an optimum value at 341.67 °C, 0.1 mm, and 20 mm/s. It could be concluded that assessment of the structural integrity of 3D prints in PPS by means of MEX technology is defined by the defects' shape.

Authors Keywords: Additive manufacturing (AM); material extrusion (MEX); PPS; 3D printing; Mechanical properties; Tensile strength; Apparent porosity; RSM

## Highlights

- Printing parameters governed Z-direction tensile behavior of PPS.
- Print speed was not significant for apparent porosity in the studied range.
- Maximum tensile performance did not coincide with minimum apparent porosity.
- SEM linked improved Z-direction strength to reduced interlayer gaps.
- RSM predicted an optimum at 341.67 °C, 0.10 mm and 20 mm/s.

# 1 INTRODUCTION

High-performance polymers (HPPs) are of significant interest across various industries due to their excellent strength-to-weight ratio and ability to endure extreme conditions. These properties offer several advantages including reduced weight, lower cost and the potential to serve as alternatives to metals in demanding applications [1]. HPPs are known for their ability to retain beneficial properties even under harsh conditions, such as exposure to corrosive environments, elevated temperature and high pressures. They encompass both amorphous types, such as polyetherimide (PEI), polysulfone (PSU), polyaryletherketone (PAEK) and polybenzimidazole (PBI), and semicrystalline types such as polyphenylene sulfide (PPS) and polyetheretherketone (PEEK), which are widely used in engineering applications [2]. These polymers are characterized by exceptional mechanical strength, thermal stability and chemical resistance, making them promising alternatives to metallic components. Due to their superior performance and cost-effectiveness, HPPs are increasingly applied in industries such as automotive, aerospace, agriculture, construction and textiles [3,4].

High-performance thermoplastics are increasingly processed using additive manufacturing (AM) techniques. AM is based on the conversion of a three-dimensional computer-aided design model into a physical part by successive material deposition. Several AM processes are currently available, including powder-bed fusion, material jetting and material extrusion (MEX). Among these techniques, MEX, also referred to as fused filament fabrication (FFF) or fused deposition modelling (FDM), has received particular attention for thermoplastic processing because of its relatively simple operation, material efficiency and ability to manufacture complex geometries. During MEX, a polymer filament is fed into a heated nozzle, melted and deposited layer by layer according to the tool path. The final properties of the printed component therefore depend not only on the intrinsic material properties but also on the thermal history and the quality of bonding between adjacent deposited roads and successive layers [5,6].

A large number of studies have demonstrated that MEX printing parameters can strongly affect the mechanical behavior of printed polymers. Chacón et al. [7] showed for PLA that layer thickness and build orientation significantly influence tensile and flexural performance, with the effect of layer thickness depending on the manufacturing orientation. Nabavi-kivi et al reported that printing speed modifies the fracture behavior of ABS specimens, confirming that deposition rate influences the quality of the printed structure [8]. More recently,

Maloney et al. [9] investigated semi-crystalline PEKK and found that print speed, layer thickness, nozzle temperature and build-plate temperature all contribute to the flexural response. These studies confirm the importance of process parameters, but their conclusions cannot be transferred directly to PPS because the viscosity, crystallization behavior and thermal processing window of high-performance semi crystalline polymers are strongly material dependent. They also show that individual parameter trends should not be interpreted without considering printing orientation and interactions between process variables.

For PPS specifically, several studies have provided important information on the relationship between printing conditions and mechanical behavior. Fitzharris et al. [10] investigated interlayer bonding of MEX-printed PPS and demonstrated the importance of processing conditions for improving bonding between deposited layers. El Magri et al. [11] subsequently studied the influence of printing temperature on the structural and mechanical performance of printed PPS and showed that temperature affects both tensile behavior and crystallinity. Also, Geng et al. examined high-performance PPS printing and confirmed that extrusion conditions and filament alignment influence tensile, bending and impact properties [12]. Retolaza et al. [13] quantified the anisotropic elastic behavior of printed PPS, while a subsequent study investigated the effects of infill percentage, printing direction and pattern and showed that the build direction plays an important role in the final mechanical response [14]. Taken together, these works clearly demonstrate that the mechanical performance of printed PPS is controlled by both the process conditions and the deposited structure. However, most of these studies focused on mechanical properties, orientation, raster configuration or individual processing effects, and the relationship between the statistical process response, porosity and the local fracture morphology remains less clearly established.

The Z direction (weakness direction) is particularly important when evaluating this relationship because the applied tensile load is mainly transferred through the interfaces between successive deposited layers. Consequently, Z-direction specimens are more sensitive to insufficient interlayer diffusion, incomplete fusion and internal defects than specimens loaded mainly along the deposited roads. This condition is therefore useful for studying the mechanisms that limit the mechanical performance of MEX parts. Studies on PPS and other high-performance thermoplastics have shown that higher nozzle temperature can promote interfacial bonding by reducing melt viscosity and increasing polymer mobility, whereas large layer thickness and high printing speed can limit the time and contact conditions required for adequate layer fusion [10,12,15]. Nevertheless, the effect of these parameters on porosity does

not necessarily follow the same trend as their effect on mechanical properties. In particular, a low total void fraction does not automatically indicate strong interlayer bonding, because the position and morphology of the defects and the effective bonded area can also govern crack initiation and propagation.

Experimental design and response surface methodology (RSM) have been increasingly applied to MEX in order to evaluate several processing parameters simultaneously and reduce the number of required experiments. El Magri et al. [11] applied RSM to printed PPS using nozzle temperature, printing speed and layer thickness and demonstrated that these parameters can be optimized with respect to mechanical properties and crystallinity. This study established the usefulness of statistical optimization for PPS processing. However, optimization of the process parameters alone does not fully explain why a given condition provides better Z-direction performance. A process condition that maximizes tensile properties may not correspond to the condition that minimizes porosity, and statistical models require complementary structural characterization to explain this difference. Chacón et al. [7] demonstrated that layer thickness significantly affects the tensile and flexural strengths of PLA samples, with variation depending on manufacturing orientation (flat, on-edge or upright). The number of contours also exerts a notable influence on mechanical properties [16,17]. Retolaza et al [14] systematically investigated the influence of infill percentage, printing direction and pattern type, showing that parts fabricated in the Z-axis orientation exhibit inferior mechanical properties compared to those printed in flat (XY or YX) or edge (XZ) orientations. Additionally, Soni et al. [18] found that the mechanical properties of bio-composites are influenced by weave angles and that specimens with weave angles of  $0^\circ$  and  $90^\circ$  exhibit lower tensile strength. These findings collectively highlight the importance of parameter selection in predicting mechanical behavior and ensuring a reliable transition from prototype to production. Moreover, studies on continuous carbon-fiber/PPS composites have related consolidation conditions, void formation and interlaminar behavior [15,19,20], but the presence of continuous reinforcement changes the material flow and interfacial mechanisms and these conclusions cannot be directly transferred to neat PPS.

Based on the available literature, there is therefore a need for a more complete analysis that relates the MEX processing parameters of neat PPS to Z-direction mechanical behavior, porosity and interlayer morphology within the same experimental design. In particular, it remains important to determine whether the parameters that improve tensile properties also reduce porosity and to identify the interactions between nozzle temperature, layer thickness and

printing speed. Establishing this relationship is important for the use of PPS in mechanically demanding printed components because the Z-direction generally represents the weakest loading direction and can limit the reliability of the final part. A better understanding of these relationships can also reduce trial-and-error parameter selection and provide a more rational process window for high-performance PPS printing.

The present study therefore investigates the effects of nozzle temperature, layer thickness and printing speed on the mechanical properties and porosity of PPS parts manufactured by MEX in the Z direction. A face-centred central composite design was used to evaluate the individual and interaction effects of the three parameters on Young's modulus, tensile strength, elongation at break and density-derived apparent porosity. Dynamic mechanical analysis (DMA) and scanning electron microscopy (SEM) were additionally used to compare contrasting printing conditions and to relate the macroscopic mechanical response to viscoelastic behavior and fracture morphology. Finally, RSM was applied to identify a multi-response process condition that maximizes the mechanical properties while minimizing apparent porosity. In this way, the study goes beyond the determination of an optimum parameter combination and focuses on the relationship between processing conditions, interlayer quality, porosity and Z-direction mechanical performance.

## 2 EXPERIMENTAL

### 2.1 Material and printing system

Commercial ThermaX PPS filament (3DXTECH, USA) with a nominal diameter of 1.75 mm and nominal density of  $1.28 \text{ g.cm}^{-3}$  was used. Specimens were produced on an INTAMSYS FUNMAT HT Enhanced V3 printer equipped for high-temperature thermoplastics. The machine provides a heated build platform, temperature-controlled chamber and high-temperature all-metal hot end. Slicing was performed using INTAM-Suite v3.4.0.

The investigated parameter ranges were selected to cover stable PPS printing conditions while varying the thermal and geometric conditions expected to control interlayer formation. Nozzle temperature was varied from 315 to 345 °C, layer thickness from 0.1 to 0.3 mm and printing speed from 20 to 60  $\text{mm.s}^{-1}$ . Bed temperature, chamber temperature, raster geometry, infill and extrusion settings were held constant as listed in Table 1.

Key material properties are summarized in Table 2.

*Table 1: MEX printing parameters used for PPS.*

| Parameters            | Values     | Units              |
|-----------------------|------------|--------------------|
| Nozzle temperature    | 315 to 345 | °C                 |
| Bed temperature       | 130        | °C                 |
| Chamber temperature   | 50         | °C                 |
| Print speed           | 20 to 60   | $\text{mm.s}^{-1}$ |
| Layer thickness       | 0.1 to 0.3 | mm                 |
| Infill pattern        | Lines      |                    |
| Infill line direction | 0          | (°)                |
| Wall lines count      | 2          |                    |
| Infill density        | 100        | %                  |
| Extruder diameter     | 0.4        | mm                 |
| Extrusion multiplier  | 100        | %                  |

*Table 2: Key thermal, mechanical, and structural properties of the PPS material used.*

| ThermaX PPS | Values | Units |
|-------------|--------|-------|
|-------------|--------|-------|

|                           |      |                   |
|---------------------------|------|-------------------|
| Tensile Modulus           | 2650 | MPa               |
| Tensile Strength          | 50   | MPa               |
| Tensile Elongation, Break | 18   | %                 |
| Density                   | 1.28 | g/cm <sup>3</sup> |
| Deflection temperature    | 90   | °C                |

## 2.2 Design of experiments

A face-centred central composite design (CCD) was employed to evaluate the effects of three MEX printing parameters, namely nozzle temperature, layer thickness and print speed, on four measured responses: Young's modulus, tensile strength, elongation at break and density-derived apparent porosity. Each factor was investigated at three levels. The selected levels were 315, 330 and 345 °C for nozzle temperature, 0.1, 0.2 and 0.3 mm for layer thickness and 20, 40 and 60 mm.s<sup>-1</sup> for print speed. The resulting design consisted of 15 experimental combinations, which allowed the linear, quadratic and two-way interaction effects to be evaluated within the investigated process window.

The parameters selected for this study are key variables in the material extrusion process. Nozzle temperature regulates the viscosity of the molten polymer and significantly influences diffusion and interlayer adhesion [1]. Print speed determines the residence time and thermal history during deposition, thereby affecting the quality of filament fusion and the formation of porosity [21]. The thickness of the layers influences the contact area between consecutive layers and the heat transfer conditions that occur during solidification. This, in turn, impacts the mechanical properties and porosity of the printed components [22].

The experimental data were fitted using a second-order polynomial response surface model. In the statistical analysis, nozzle temperature was defined as factor A, layer thickness as factor B and print speed as factor C. The model included the linear effects, quadratic effects and two-way interactions between the three factors. This approach was used to determine the contribution of each printing parameter and to identify the combinations that provide the best balance between mechanical performance and apparent porosity.

The CCD facilitates the collection of data required to develop response surface methodology (RSM) models. RSM is a statistical technique used to model the relationships

between independent factors and dependent responses and to identify optimal process conditions [23]. The experimental data were fitted using a second-order polynomial model as expressed in equation (1) [24].

$$Y = \beta_0 + \sum_{i=1}^N \beta_i X_i + \sum_{i=1}^N \beta_{ii} X_i^2 + \sum_{i<j}^N \beta_{ij} X_i X_j + \varepsilon \quad (1)$$

where  $\beta_0$  parameter is the model constant,  $\beta_i$  is the linear coefficient,  $\beta_{ii}$  is the quadratic coefficient,  $\beta_{ij}$  represents the interaction terms,  $\varepsilon$  is the random error component and  $Y$  represents the predicted response.

Statistical analyses were performed using Minitab version 18. The significance level was fixed at  $\alpha = 0.05$ , and terms with  $p < 0.05$  were considered statistically significant. Based on the parameter ranges defined in Table 3, the software generated the 15 experimental combinations listed in Table 4. Minitab was used to calculate the regression coefficients,  $p$ -values and coefficients of determination ( $R^2$ ) for each response model. Multi-response optimization was subsequently performed by maximizing Young's modulus, tensile strength and elongation at break while minimizing apparent porosity.

Table 3: Tested levels for experimental Design.

| Factor | Name               | Code | Unit | Level | Value |     |     |
|--------|--------------------|------|------|-------|-------|-----|-----|
| 1      | Nozzle temperature | A    | °C   | 3     | 315   | 330 | 345 |
| 2      | Layer thickness    | B    | mm   | 3     | 0.1   | 0.2 | 0.3 |
| 3      | Print speed        | C    | mm/s | 3     | 20    | 40  | 60  |

Table 4: Summary table of experimental tests.

| Run | Nozzle temperature | Layer thickness | Print speed |
|-----|--------------------|-----------------|-------------|
| 1   | 330                | 0.2             | 40          |
| 2   | 330                | 0.2             | 20          |
| 3   | 330                | 0.1             | 40          |
| 4   | 345                | 0.1             | 60          |
| 5   | 345                | 0.3             | 20          |

|    |     |     |    |
|----|-----|-----|----|
| 6  | 345 | 0.1 | 20 |
| 7  | 345 | 0.2 | 40 |
| 8  | 315 | 0.3 | 60 |
| 9  | 315 | 0.1 | 60 |
| 10 | 330 | 0.3 | 40 |
| 11 | 315 | 0.2 | 40 |
| 12 | 315 | 0.1 | 20 |
| 13 | 345 | 0.3 | 60 |
| 14 | 315 | 0.3 | 20 |
| 15 | 330 | 0.2 | 60 |

## 2.3 Characterization

### 2.3.1 Density evaluation and Tensile test

Density measurement was performed to assess porosity using the Archimedes method in accordance with ASTM D792. Cubic specimens with dimensions of  $1 \times 1 \times 1 \text{ cm}^3$  were used for this purpose (Fig. 1a). Two independently printed cubic specimens were measured for each experimental condition ( $n = 2$ ), and the results are reported as mean  $\pm$  standard deviation. During testing, the weight of each specimen was recorded first in atmospheric air and then in distilled water at  $25^\circ\text{C}$ . Density was calculated using equation (2), where  $m_{\text{air}}$  and  $m_{\text{fluid}}$  denote the specimen weights in air and in water respectively. The density of distilled water was set at  $0.9979 \text{ g/cm}^3$ . Porosity was subsequently calculated using equation (3).

$$\text{Density } (\rho) = \frac{m_{\text{air}}}{m_{\text{air}} - m_{\text{fluid}}} \times \rho_{\text{Water}} \quad (\text{g/cm}^3) \quad (2)$$

$$\text{Porosity } (P) = 100 - \frac{\rho_{\text{measured}} \times 100}{\rho_{\text{theoretical}}} \quad (\%) \quad (3)$$

Tensile properties were evaluated using a Criterion C45.105 electromechanical universal testing machine (MTS, USA) equipped with a 10 kN load cell and a contact extensometer for precise deformation measurement. Specimens conforming to ASTM D638 Type IV (Fig. 1b) were tested at a crosshead displacement speed of  $0.08 \text{ mm/s}$  until fracture. Six tensile specimens were tested for each experimental condition ( $n = 6$ ), corresponding to a

total of 90 tensile specimens. Young's modulus, tensile strength and elongation at break are reported as mean  $\pm$  standard deviation. Young's modulus was calculated from the resulting stress-strain data using equation (5).

$$E = \frac{\sigma \text{ (MPa)}}{\varepsilon} \quad (5)$$

The printing orientation used for all specimens is illustrated in Fig. 2.

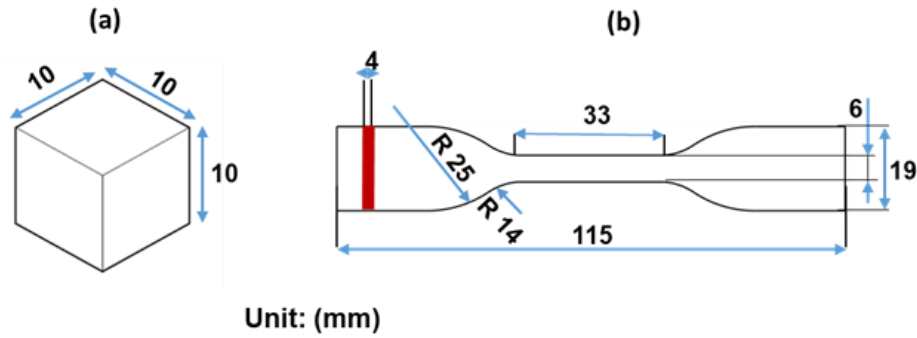


Fig. 1: (a) Sample size requirements for ASTM D792 density testing, (b) ASTM D638 Type IV tensile test specimen configuration

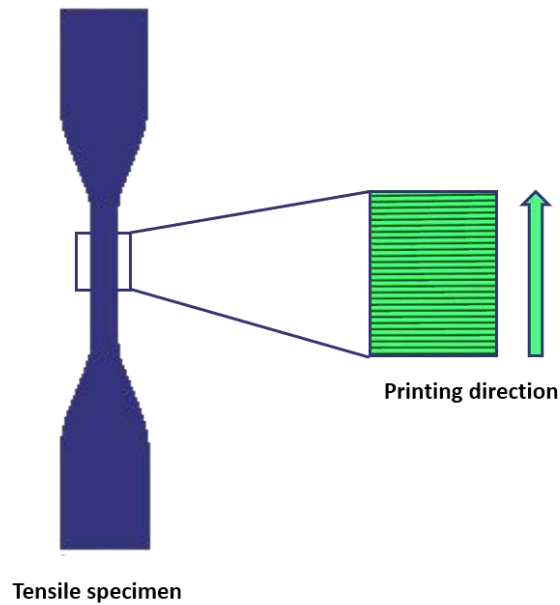


Fig. 2: Orientation and deposited layer direction

### 2.3.2 Dynamic mechanical analysis (DMA)

Dynamic mechanical analysis (DMA) was performed to characterize the viscoelastic behavior of printed PPS specimens. The glass transition temperature was evaluated using three

approaches, based on the onset of reduction in the storage modulus ( $E'$ ), the peak of the loss modulus ( $E''$ ) and the peak of  $\tan(\delta)$ . All specimens were tested using a TA Instruments DMA 850 in dual cantilever bending mode. Rectangular specimens measuring 60 mm in length, 12 mm in width and 3.2 mm in thickness were used (Fig. 3), in accordance with ASTM D5418. Specimens were heated from room temperature to 200 °C at a rate of 10 °C/min. The oscillation frequency was set to 1 Hz and the amplitude to 0.02%. Two specimens were tested for each experimental condition ( $n = 2$ ).

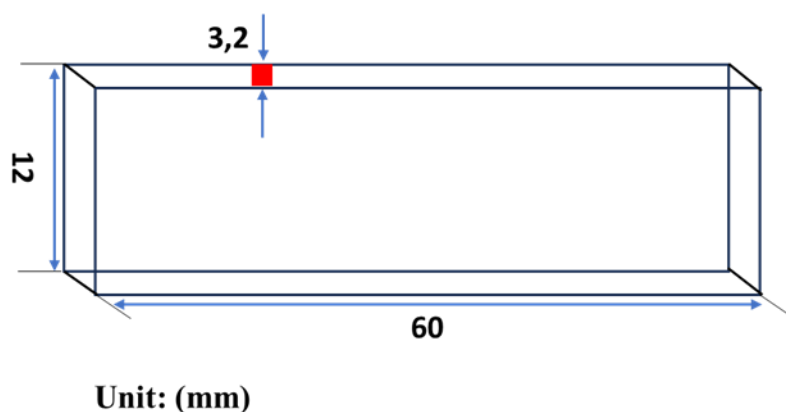


*Fig. 3: DMA sample dimensions according to ASTM D5418*

### 2.3.3 Morphological evaluation

Scanning electron microscopy (SEM) was used to analyze the fracture surfaces of tested specimens, enabling detailed assessment of internal microstructural features. Observations were carried out using a JEOL JSL IT500HR instrument (JEOL Ltd., Akishima, Tokyo, Japan) at an accelerating voltage appropriate for polymer imaging. Prior to imaging, a carbon coating was deposited on each specimen using a JEOL JFC 1300 automatic metallizer. This approach enabled detailed visualization of the printed microstructure and facilitated correlation between microstructural features and measured mechanical responses.

## 3 RESULTS AND DISCUSSION

### 3.1 Analysis of tensile strength and porosity characterization

The mechanical properties and porosity of MEX-printed PPS specimens were evaluated across 15 experimental conditions defined by the CCD. A total of 90 specimens were tested with 6 replicates per experimental run. The measured responses including Young's modulus

(E), tensile strength ( $\sigma$ ), elongation at break ( $\epsilon$ ) and porosity (P) exhibited notable variability across the investigated parameter space, ranging from 1988 to 2350 MPa, 25.9 to 46.3 MPa, 1.42 to 2.52% and 4.18 to 7.57% respectively. The stress-strain curves and porosity distribution for all runs are presented in Fig. 4 and the corresponding average results are summarized in Table 5.

Examination of Table 5 reveals that the highest mechanical properties were obtained under printing conditions of 345 °C, 20 mm/s and 0.1 mm, whereas the lowest mechanical performance was recorded at 315 °C, 60 mm/s and 0.3 mm. A similar trend was reported by El Magri et al. [11] for PPS material, where improved mechanical properties were achieved at higher nozzle temperatures combined with lower printing speeds and reduced layer thicknesses. These results can be interpreted in light of the individual roles of each parameter. Printing with a thin layer of 0.1 mm increases the total number of deposited layers and produces a more compact internal structure [25,26]. This compactness reduces interlayer porosity and improves load transfer between successive layers. In contrast, greater layer thickness compromises interlayer adhesion by reducing the contact pressure and thermal bonding at layer interfaces. It should be noted that Khabia and K. Jain. [27] reported an exception for specimens printed in the edge orientation, where the applied load acts parallel to the layers and variations in layer thickness do not significantly affect tensile strength. This observation underlines the importance of printing orientation as a co-determining factor in mechanical performance.

Regarding nozzle temperature, higher extrusion temperatures enhance fusion between adjacent filaments and promote polymer chain diffusion at the interfaces through reptation mechanisms, thereby reducing the formation of voids and internal defects. Shuto et al. [15] proved this correlation experimentally for carbon fiber/PPS composites, indicating that a decline in the average voids fraction takes place at the higher nozzle temperatures along with the decrease of resin viscosity which leads to better impregnation quality. These results are consistent with the current findings, where samples printed at 345 °C had lower porosity and higher mechanical performance when compared to samples printed at 315 °C.

As the printing speed increases, the thermal energy available for bonding between layers decreases causing voids, and interior and exterior morphological defects [28]. this microstructural quality deterioration results in lower mechanical properties. However, such a decrease in printing speed increases the time of manufacturing and thus in industrial conditions a compromise between quality and productivity needs to be found [29].

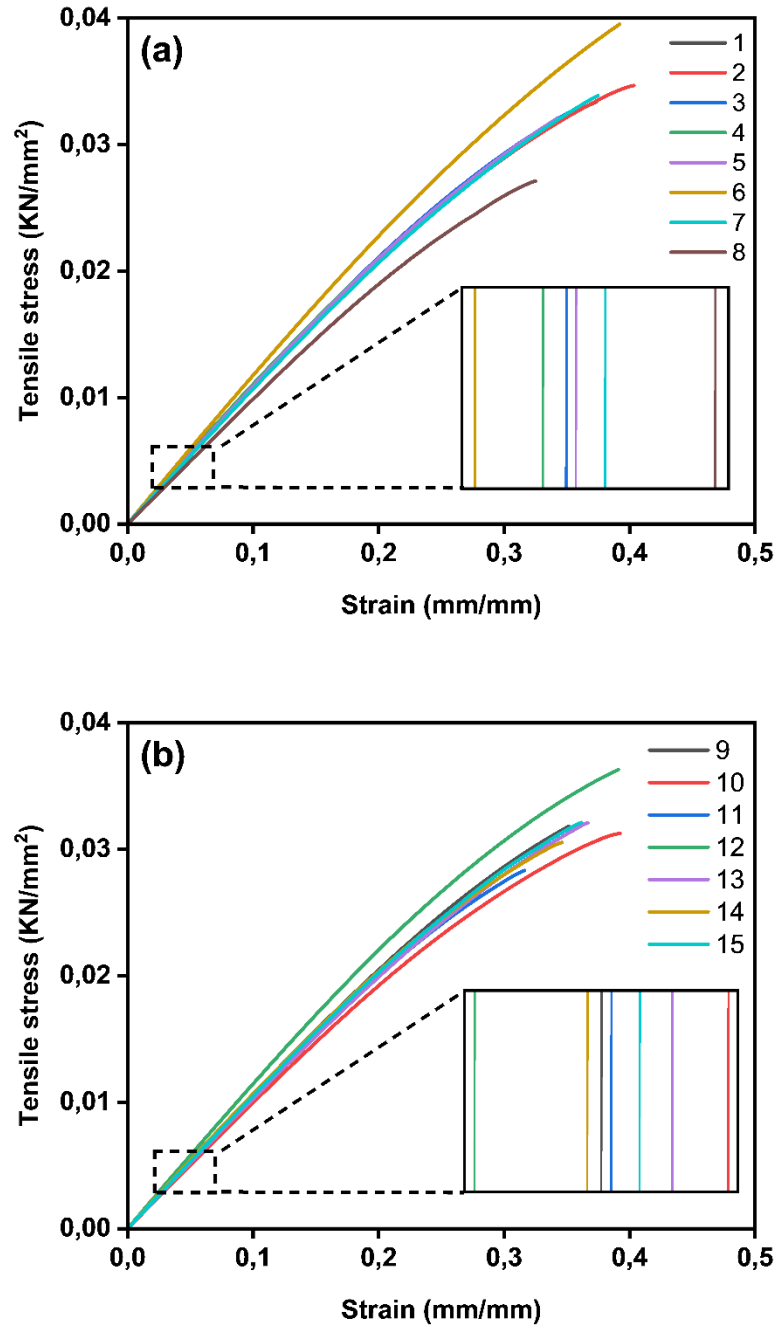


Fig. 4: Stress-strain curves of printed PPS under different printing conditions: (a)Runs 1 to 8, (b)Runs 8 to 15.

Table 5: Design of Experiments (DOE) and average results for tensile properties and porosity of PPS specimens.

| Run | Input parameters    |                 |                   | Measured parameters |           |            |           |
|-----|---------------------|-----------------|-------------------|---------------------|-----------|------------|-----------|
|     | Nozzle              | Print           | Layer             | E                   | $\sigma$  | $\epsilon$ | P         |
|     | temperature<br>(°C) | speed<br>(mm/s) | thickness<br>(mm) | (MPa)               | (MPa)     | (%)        | (%)       |
| 1   | 330                 | 40              | 0.2               | 2120±56             | 31.4±3.16 | 1.8±0.26   | 4.18±0.24 |
| 2   | 330                 | 20              | 0.2               | 2220±59             | 35.8±1.76 | 2.08±0.24  | 4.67±0.78 |
| 3   | 330                 | 40              | 0.1               | 2213±77             | 31.1±1.99 | 1.69±0.11  | 4.77±0.62 |
| 4   | 345                 | 60              | 0.1               | 2252±51             | 29.4±3.65 | 1.69±0.21  | 5.26±0.22 |
| 5   | 345                 | 20              | 0.3               | 2175±65             | 31.2±1.56 | 1.76±0.11  | 7.57±0.92 |
| 6   | 345                 | 20              | 0.1               | 2337±54             | 41.3±2.01 | 2.29±0.26  | 4.94±0.25 |
| 7   | 345                 | 40              | 0.2               | 2144±85             | 32.8±3.47 | 1.84±0.31  | 4.56±0.14 |
| 8   | 315                 | 60              | 0.3               | 1988±46             | 25.9±1.43 | 1.61±0.12  | 6.28±0.62 |
| 9   | 315                 | 60              | 0.1               | 2143±61             | 28.7±2.41 | 1.59±0.51  | 4.87±0.44 |
| 10  | 330                 | 40              | 0.3               | 2112±51             | 29.4±1.58 | 1.79±0.18  | 5.33±0.64 |
| 11  | 315                 | 40              | 0.2               | 2131±63             | 28.5±3.16 | 1.58±0.16  | 4.73±0.31 |
| 12  | 315                 | 20              | 0.1               | 2303±70             | 34.8±3.07 | 1.81±0.18  | 4.33±0.31 |
| 13  | 345                 | 60              | 0.3               | 2076±39             | 31.2±1.32 | 1.80±0.15  | 7.05±0.95 |
| 14  | 315                 | 20              | 0.3               | 2151±44             | 26.8±3.17 | 1.42±0.18  | 5.64±0.16 |
| 15  | 330                 | 60              | 0.2               | 2114±52             | 32.0±3.78 | 1.88±0.23  | 4.44±0.20 |

### 3.2 Analysis of variance and response surface methodology for model fitting

The influence of nozzle temperature, printing speed and layer thickness on the four measured responses was assessed through analysis of variance (ANOVA) and response surface methodology (RSM). The main effects plots presented in Fig. 5 illustrate the mean response values across the different levels of each printing parameter. Table 6 summarizes the ANOVA results including the  $p$ -values for each source of variation. Table 7 presents the developed regression model equations together with the associated coefficient of determination ( $R^2$ ) and  $p$ -values for each response.

Statistical analysis confirmed that all three parameters, namely layer thickness, printing speed and nozzle temperature, exert a statistically significant influence on Young's modulus ( $p < 0.05$ ). The response model demonstrated an excellent fit to the experimental data with  $R^2 = 95.45\%$  (Table 7). As shown in Fig. 5a, Young's modulus increases with nozzle temperature,

which is consistent with the enhanced interlayer fusion achieved at elevated printing temperatures [30]. Conversely, layer thickness and print speed exhibit a negative effect on this response, with thin layers and low speeds yielding higher modulus values. This improvement is attributed to greater dimensional precision and more homogeneous bonding between successive layers, which minimizes the formation of cavities and delamination at layer interfaces [31]. Reducing layer thickness also increases the total number of deposited layers, which can improve structural integrity and raise the load at which the breaking point is reached [32,33]. The combination of 345 °C, 0.1 mm and 20 mm.s<sup>-1</sup> were identified as the condition yielding the maximum Young's modulus, while 315 °C, 0.3 mm and 60 mm.s<sup>-1</sup> produced the minimum.

The model for tensile strength achieved  $R^2 = 94.08\%$  with a model  $p$ -value of 0.000, confirming a statistically significant and well-fitted relationship. All three printing parameters were found to exert a highly significant influence on tensile strength ( $p = 0.000$ ). As shown in Fig. 5b, nozzle temperature exhibits a nonlinear increasing trend while layer thickness shows a decreasing effect on tensile strength. Maximum tensile strength was obtained at the highest temperature (345 °C) and minimum layer thickness (0.1 mm). Reducing layer thickness minimizes interlayer spacing and significantly improves mechanical performance [34], which is consistent with the broader literature identifying layer thickness as a key parameter governing mechanical strength in FFF [35]. Print speed was found to have a decreasing effect on tensile strength with a plateau at intermediate values, consistent with the findings of Nabavi-Kivi et al. [8] who demonstrated that tensile strength is optimized at moderate speeds while higher speeds lead to progressive reduction. The optimal combination for tensile strength was identified as 345 °C, 0.1 mm and 20 mm.s<sup>-1</sup> [36].

As shown in Fig. 5c, apparent porosity was primarily governed by nozzle temperature and layer thickness, both of which had statistically significant effects on this response (Table 6). Layer thickness showed the strongest effect, with the highest apparent porosity values obtained at 0.3 mm. The temperature response was nonlinear, while print speed had no statistically significant effect on apparent porosity within the investigated range ( $p = 0.517$ ). The minimum experimental mean apparent porosity was obtained for Run 1 at 330 °C, 0.2 mm and 40 mm.s<sup>-1</sup>, whereas the multi-response mechanical optimum occurred at a different combination. This separation between the mechanical response and apparent porosity confirms that total density-derived porosity alone is not sufficient to describe the quality of the Z-direction interfaces.

All three studied factors had a statistically significant effect on elongation at break ( $p < 0.05$ ), and the model achieved  $R^2 = 91.34\%$ , indicating a good fit to the experimental data. Nozzle temperature had the strongest linear effect, while layer thickness ( $p = 0.008$ ) and print speed ( $p = 0.005$ ) were also significant. The effect of nozzle temperature followed a nonlinear trend, with elongation increasing toward the upper part of the investigated temperature range. The observed reduction at the highest conditions may be related to changes in the thermal history and interlayer morphology; however, thermal degradation or crystallinity changes cannot be confirmed from the present data because these mechanisms were not independently measured. Regarding layer thickness, the response decreased at the highest thickness, while low print speed produced the highest elongation values. These trends show that ductility in the Z direction depends on the combined quality of the deposited interfaces rather than on a single printing parameter.

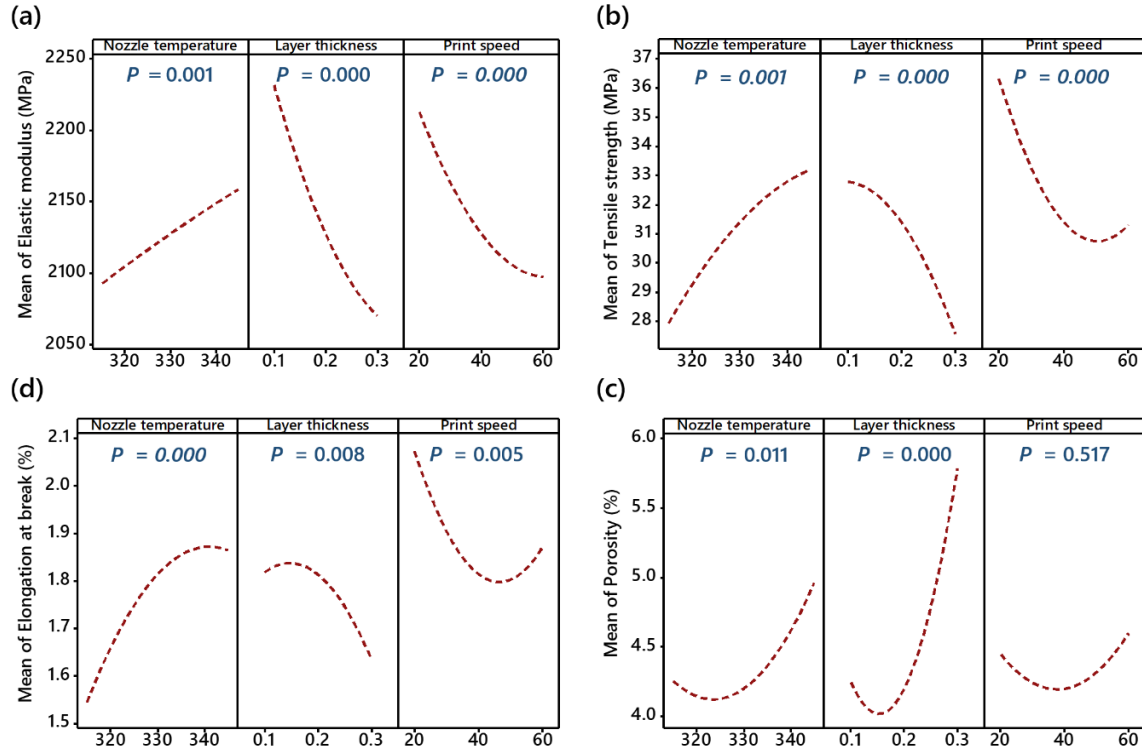


Fig. 5: Analysis of the main effects on Young's modulus (a), tensile strength (b), elongation at break (c) and porosity (d).

Table 6: Analysis of variance for the effect of process parameters on the mechanical properties and apparent porosity.

| Source                                         | Abbreviation   | <i>p</i> - Value |                   |                   |          |
|------------------------------------------------|----------------|------------------|-------------------|-------------------|----------|
|                                                |                | E<br>(MPa)       | $\sigma$<br>(MPa) | $\epsilon$<br>(%) | P<br>(%) |
| <b>Linear</b>                                  |                | 0.000            | 0.000             | 0.000             | 0.000    |
| Nozzle temperature (°C)                        | A              | 0.001            | 0.000             | 0.000             | 0.011    |
| Layer thickness (mm)                           | B              | 0.000            | 0.000             | 0.008             | 0.000    |
| Print speed (mm/s)                             | C              | 0.000            | 0.000             | 0.005             | 0.517    |
| <b>Square</b>                                  |                | 0.015            | 0.097             | 0.040             | 0.000    |
| Nozzle temperature $\times$ Nozzle temperature | A <sup>2</sup> | 0.905            | 0.352             | 0.066             | 0.088    |
| Layer thickness $\times$ Layer thickness       | B <sup>2</sup> | 0.135            | 0.179             | 0.134             | 0.004    |
| Print speed $\times$ Print speed               | C <sup>2</sup> | 0.085            | 0.017             | 0.013             | 0.166    |
| <b>2-Way Interaction</b>                       |                | 0.645            | 0.000             | 0.001             | 0.223    |
| Nozzle temperature $\times$ Print speed        | A $\times$ C   | 0.287            | 0.010             | 0.012             | 0.207    |
| Nozzle temperature $\times$ Layer thickness    | A $\times$ B   | 0.939            | 0.712             | 0.282             | 0.123    |
| Layer thickness $\times$ Print speed           | B $\times$ C   | 0.517            | 0.000             | 0.000             | 0.480    |

Table 7: Regression models and associated R<sup>2</sup> and p-values for each response.

| Response                | RSM model                                                                                                                                               | R <sup>2</sup><br>(%) | <i>p</i> - Value |
|-------------------------|---------------------------------------------------------------------------------------------------------------------------------------------------------|-----------------------|------------------|
| Elastic modulus (MPa)   | $E = 1499 + 6.0 A - 2005 B - 19.43 C - 0.0079 A^2 + 2347 B^2 + 0.0689 C^2 + 0.44 A \times B + 0.0317 A \times C + 2.84 C \times B$                      | 95.45                 | 0.000            |
| Tensile strength (MPa)  | $\sigma = -480 + 2.85 A + 20 B + 0.798 C - 0.00367 A^2 - 122.4 B^2 + 0.00603 C^2 - 0.180 A \times B - 0.00520 A \times C + 1.551 C \times B$            | 94.08                 | 0.000            |
| Elongation at break (%) | $\epsilon = -59.1 + 0.349 A + 7.18 B + 0.0520 C - 0.000486 A^2 - 8.65 B^2 + 0.000398 C^2 - 0.0236 A \times B - 0.000317 A \times C + 0.0788 C \times B$ | 91.34                 | 0.000            |
| Porosity (%)            | $P = 199 - 1.183 A - 70.2 B + 0.137 C + 0.001819 A^2 + 81.7 B^2 + 0.000807 C^2 + 0.1426 A \times B - 0.000570 A \times C - 0.0465 C \times B$           | 92.96                 | 0.000            |

### 3.3 Pareto chart

The Pareto chart provides a visual assessment of the relative importance of each factor and interaction term with respect to the measured responses. In this study it was used to identify

the standardized effects of linear (A, B, C), quadratic ( $A^2$ ,  $B^2$ ,  $C^2$ ) and two-way interaction ( $A \times B$ ,  $A \times C$ ,  $B \times C$ ) terms relative to the critical value of 2.228 (Fig. 6). The vertical reference line at 2.228 corresponds to the critical Student's  $t$ -value at  $\alpha = 0.05$  with 10 residual degrees of freedom. Standardized effects exceeding this value in absolute magnitude were considered statistically significant. Any effect whose standardized value exceeds this threshold is considered statistically significant. The printing variables are nozzle temperature (A), layer thickness (B) and print speed (C).

As shown in Fig. 6a, the three main factors A, B and C exhibit statistically significant effects on Young's modulus, as all corresponding  $p$ -values fall below the 0.05 significance threshold (Table 6). This confirms that each parameter independently exerts a meaningful influence on this response within the investigated range. For tensile strength (Fig. 6b), a significant quadratic effect of print speed ( $C^2$ ) was identified with  $p = 0.017$ . Two significant two-way interactions were also detected, namely  $B \times C$  ( $p < 0.001$ ) and  $A \times C$  ( $p = 0.01$ ), indicating that the combined effect of these parameters cannot be neglected when optimizing tensile performance. A similar pattern was observed for elongation at break (Fig. 6c), although the ranking of factor influence differs. The  $B \times C$  interaction was found to be highly significant ( $p < 0.001$ ), and both the  $A \times C$  interaction ( $p = 0.012$ ) and the quadratic effect of print speed ( $C^2$ ,  $p = 0.013$ ) were also significant. These results indicate that elongation at break is sensitive not only to individual parameters but also to their combined effects. For porosity (Fig. 6d), two linear effects were identified as statistically significant. Layer thickness (B) was the most influential factor ( $p = 0.000$ ) followed by nozzle temperature (A,  $p = 0.011$ ). The quadratic effect of layer thickness ( $B^2$ ) ranked second among significant terms ( $p = 0.004$ ), reflecting the nonlinear relationship between layer thickness and void formation previously observed in the main effect's analysis. Print speed had no significant effect on porosity under the conditions investigated, which is consistent with the ANOVA results reported in Table 6.

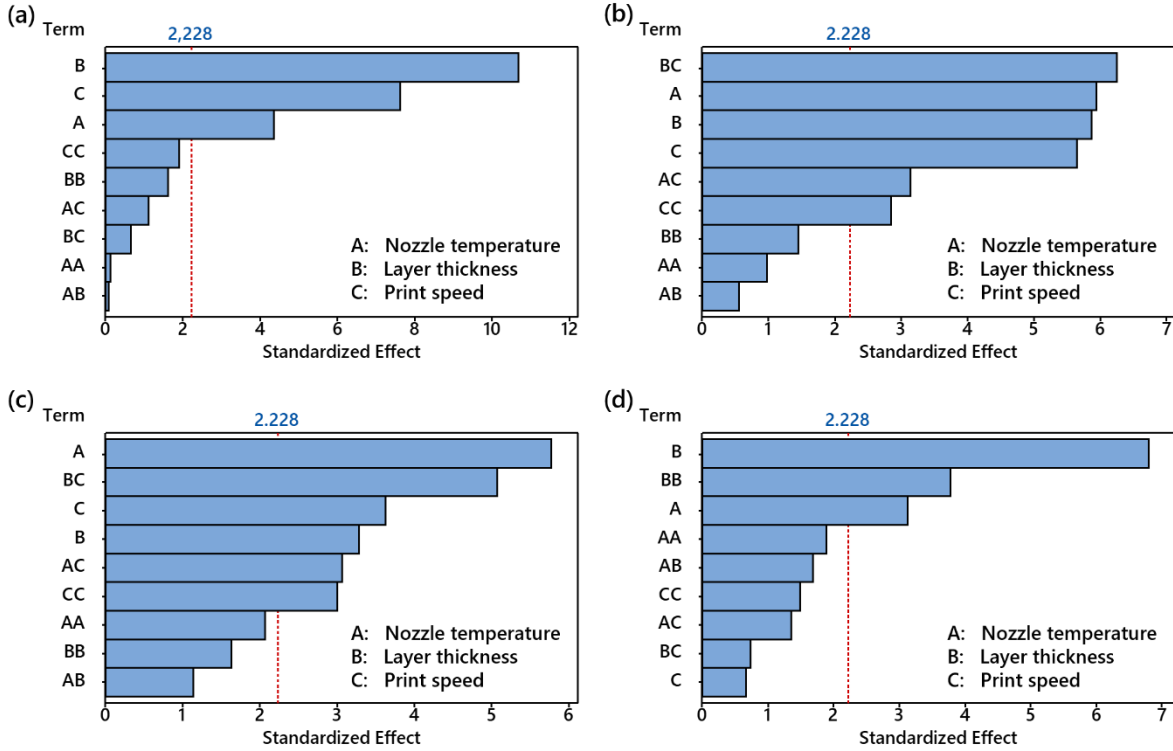


Fig. 6: Pareto diagram of standardized effects on (a) Young's modulus, (b) tensile strength, (c) elongation at break and (d) porosity.

### 3.4 Dynamic Mechanical Analysis

DMA was performed to characterize the viscoelastic behavior of printed PPS specimens and to assess the influence of manufacturing parameters on dynamic mechanical properties. Two experimental runs were selected for comparison based on their contrasting printing conditions. Run 6 (345 °C, 0.1 mm, 20 mm.s<sup>-1</sup>) represents the condition associated with the best mechanical performance, while Run 8 (315 °C, 0.3 mm, 60 mm.s<sup>-1</sup>) represents the condition yielding the poorest performance. Fig. 7 presents the evolution of storage modulus ( $E'$ ) and loss modulus ( $E''$ ) as a function of temperature from 40 to 180 °C.

In the glassy region, run 6 exhibited a significantly higher storage modulus ( $E' \approx 1873$  MPa) than Run 8 ( $E' \approx 1437$  MPa), indicating greater elastic stiffness and load-bearing capacity. This result is consistent with the tensile test data and confirms that  $E'$  serves as a reliable indicator of macroscopic stiffness in FFF-printed parts. The superior stiffness of Run 6 is attributed to the combined effect of high nozzle temperature, low print speed and thin layer thickness, which collectively promote interlayer diffusion and polymer chain entanglement through reptation mechanisms. These conditions enhance interfacial cohesion and yield lower porosity (4.94% for Run 6 versus 6.28% for Run 8). In contrast, the processing conditions of

Run 8 provide insufficient thermal energy and diffusion time, which limits interlayer bonding and results in a less consolidated microstructure.

In the viscoelastic transition region, the loss modulus ( $E''$ ) was higher and more pronounced for Run 6 ( $E'' \approx 125$  MPa) than for Run 8 ( $E'' \approx 110$  MPa), indicating more uniform energy dissipation and a more cohesive polymer network. The glass transition temperature ( $T_g$ ) was identical for both configurations at approximately 111 °C, which confirms that the intrinsic segmental mobility of the PPS chains is not affected by the printing parameters investigated. This invariance in  $T_g$  confirms that the observed differences in mechanical behavior do not originate from thermodynamic changes in the material but arise exclusively from differences in microstructural quality, including interfacial cohesion, porosity and entanglement density. Similar findings have been reported for other high-performance polymers processed by FFF, where process-induced microstructural variations altered dynamic mechanical properties without shifting the glass transition temperature [37,38].

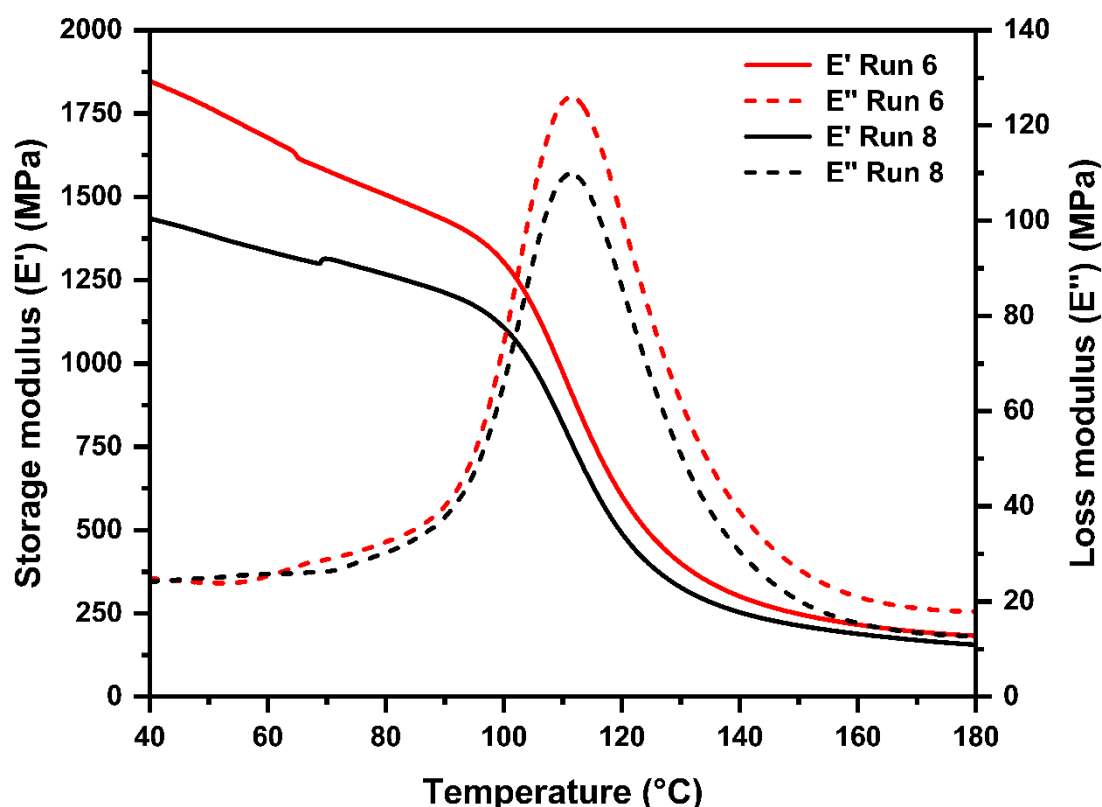


Fig. 7: DMA curves of PPS samples comparing Run 6 (345 °C, 0.1 mm, 20 mm/s) and Run 8 (315 °C, 0.3 mm, 60 mm/s). printed condition

### 3.5 Surface morphology analysis and optimization chart

SEM observations were conducted in conjunction with the mechanical and porosity results to provide a comprehensive understanding of the structure-property relationships in MEX-printed PPS. Fig. 8 presents the fracture surface micrographs of two specimens printed under contrasting conditions.

Fig. 8a represents the fracture area of an item produced while subjected to a temperature of 345 °C, under a pressure of 0.1 mm and 20 mm.s<sup>-1</sup>. The surface is characterized by a fairly dense layered structure, due to the lower gaps in between layers caused by the better mechanical characteristics observed for this case (Young's modulus:  $E = 2330$  MPa, strength:  $\sigma = 40.3$  MPa, strain:  $\epsilon = 2.20\%$ , power:  $P = 4.94\%$ ). The small number of hollows in the article indicates the sufficient interlayer bonding which is a result of high temperatures in the nozzle and slow speed of the printing. According to El Magri et al. [38], strong bonding between the layers allows for good stress transfer in the material under tension showing the high value of the storage modulus and the improved energy absorption during dynamic stress. These observations are further supported by the work of Hamoud et al. [39] who reported that printing speeds around 30 mm/s enhance interlayer adhesion and reduce defect density, thereby improving overall mechanical performance.

Fig. 8b shows the fracture surface of a specimen printed at 315 °C, 0.3 mm and 60 mm.s<sup>-1</sup>. Significant porosity and poor layer alignment are evident on this surface, indicating that the combination of low temperature, thick layers and high speed was insufficient to achieve adequate interlayer bonding. These defects are primarily attributed to partial melting of the PPS material at 315 °C, which limits polymer diffusion at layer interfaces and prevents complete void elimination [40,41]. The high print speed of 60 mm.s<sup>-1</sup> further disrupts nozzle movement consistency, resulting in irregular layer deposition and increased pore formation. Although geometric refinement through reduced layer thickness is generally expected to improve surface resolution, this benefit is not fully realized when bonding quality is compromised. As demonstrated by Nguyen et al. [42], mechanical performance in FFF is primarily governed by interlayer bonding quality rather than geometric resolution alone, and porosity together with weak interfacial fusion remains the dominant factor limiting load transfer between deposited filaments.

The optimization diagram (Fig. 9) identifies the optimal printing parameters for Z-direction MEX of PPS by simultaneously maximizing Young's modulus, tensile strength and elongation at break while minimizing porosity. The optimal conditions identified by the RSM optimizer are a nozzle temperature of 341.67 °C, a layer thickness of 0.1 mm and a printing

speed of  $20 \text{ mm.s}^{-1}$ . These conditions yield predicted properties of  $E = 2339 \text{ MPa}$ ,  $\sigma = 43.76 \text{ MPa}$ ,  $\varepsilon = 2.40\%$  and  $P = 4.89\%$ . The SEM micrograph of a specimen printed under the optimized conditions (Fig. 10) reveals a rough fracture surface with localized delamination, which is characteristic of diffusion-limited interlayer bonding in FFF-processed PPS. Crack propagation along layer interfaces confirms the anisotropic fracture behavior inherent to Z-direction printing, while the limited plastic deformation indicates a predominantly brittle fracture mode. Overall the optimized parameters improve the global balance of mechanical properties and reduce defect severity relative to the worst-case conditions, though interlayer diffusion remains the primary governing factor of fracture behavior in Z-direction printed PPS.

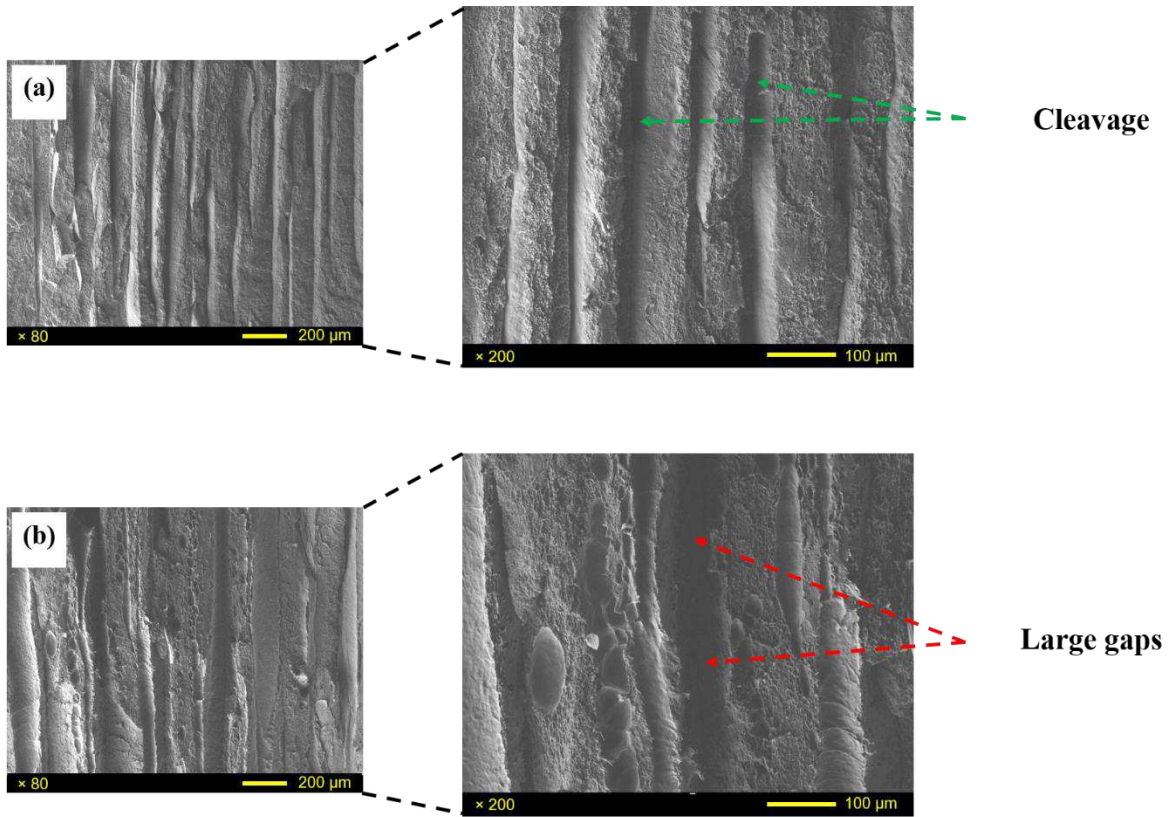


Fig. 8: Scanning electron microscopy micrographs of the fractured surfaces of PPS samples printed under -a (345 °C, 0.1 mm, 20 mm/s) and -b (315 °C, 0.3 mm, 60 mm/s) conditions.

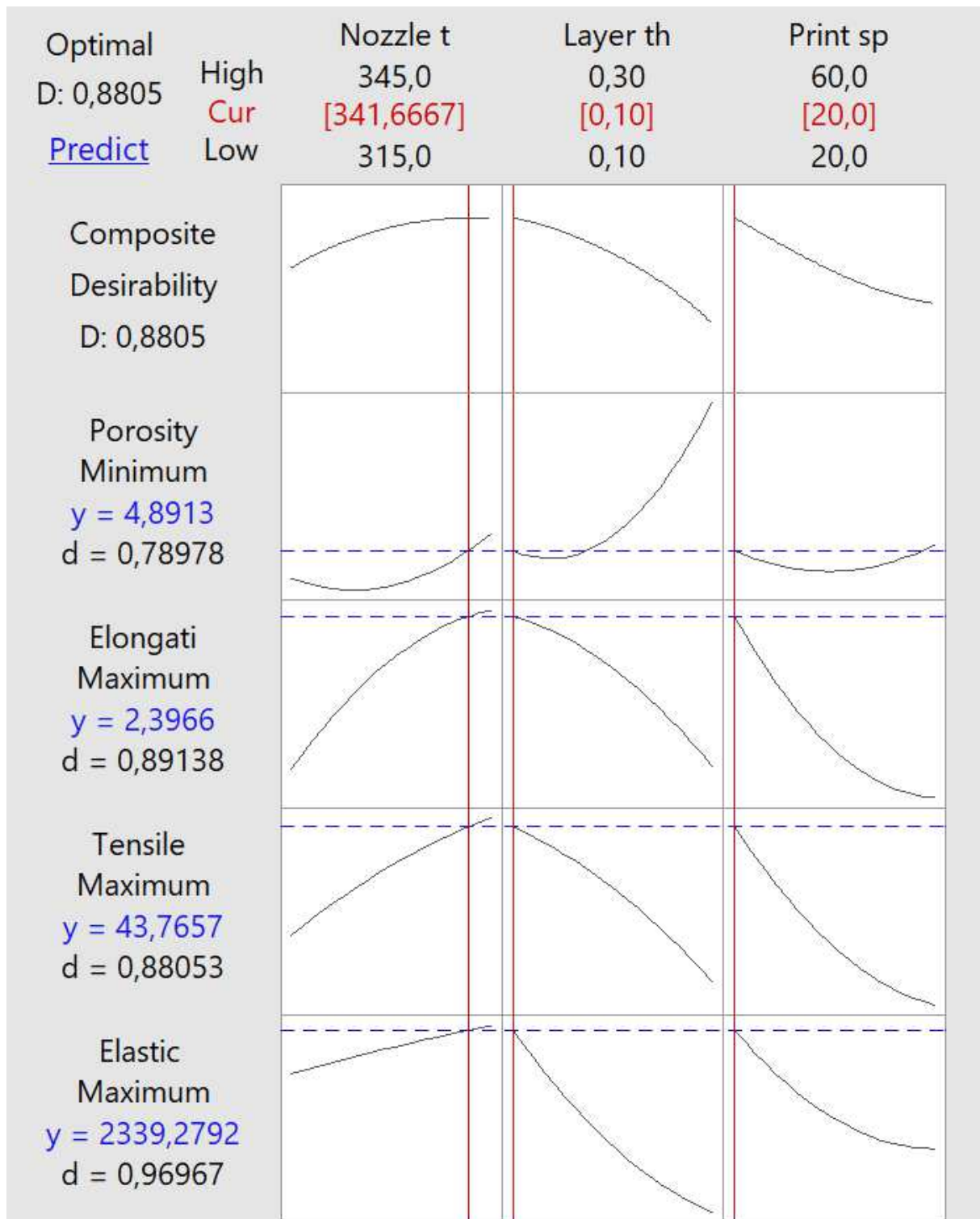


Fig. 9: Optimal levels of printing parameters obtained from the response optimizer module.

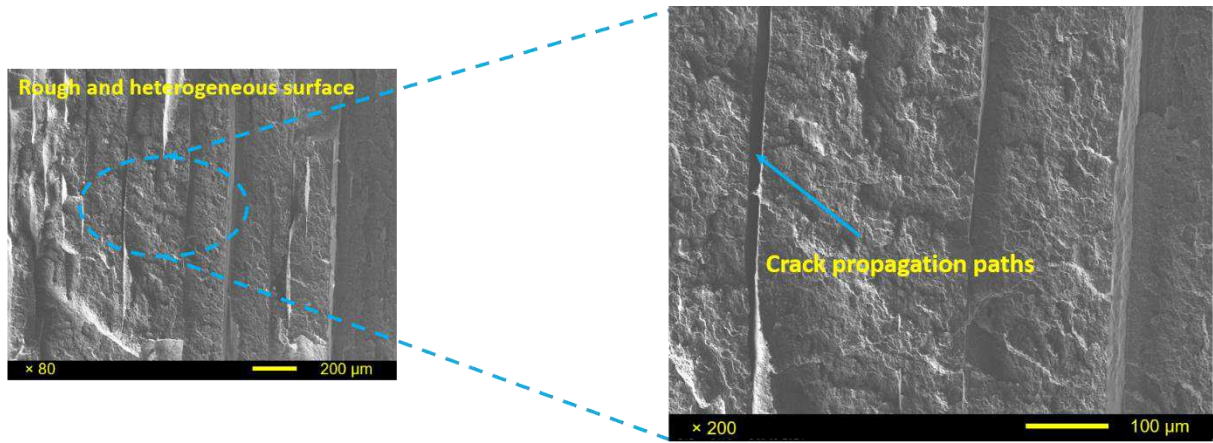


*Fig. 10: SEM micrographs of fracture surfaces of RSM-optimized 3D-printed PPS.*

## 4 CONCLUSION

This study investigated the influence of nozzle temperature, layer thickness and printing speed on the mechanical properties and porosity of MEX-printed PPS in the Z-direction. A central composite design combined with RSM was applied to evaluate individual parameter effects and their interactions. The following conclusions were drawn from the experimental and statistical analyses.

For mechanical characterization, the specimens created at a temperature of 345 °C, a layer thickness of 0.1mm and a deposition speed of 20 mm/s showed the best performance and lowest porosity. A very high nozzle temperature promotes the diffusion of polymer chains and adhesion between layers, while thin layers and slow deposition speed allow for denser and more homogeneous microstructure. Upon examining the SEM images, it was confirmed that the best specimens have very low void content as well as a high interlayer connection, whereas the worst specimens are characterized by a high porosity and poorly aligned layers. ANOVA confirmed that these three parameters have a significant effect on Young's modulus, tensile strength and elongation at break. In the data set analyzed, no statistically significant effect of print speed on porosity was found, with nozzle temperature and layer height being the major factors affecting the formation of cavities. There were significant two-way interactions between print speed and other parameters when it comes to tensile strength and elongation at break. DMA investigations revealed that the best printing parameters led to the enhancement of the storage and loss modulus, while the glass transition temperature was essentially stable at around 111 °C for all the studied systems, which supports the assumption of the importance of parameters, which affect the microstructure rather than the polymer behavior itself. RSM optimization allowed to determine the predicted multi-response optimum at the nozzle temperature of 341.67 °C, which corresponds to 0.1 mm layer thickness and printing speed of 20 mm/s, which resulted in the following predicted parameters:  $E = 2339$  MPa,  $\sigma = 43.76$  MPa,  $\varepsilon = 2.40\%$ , and  $P = 4.89\%$ . This approach allows to develop a statistical model, which demonstrates the applicability of the proposed methods in selection of the MEX processing parameters for Z-directional PPS and provides evidence that mechanical performance of the produced items must be analyzed together with the inter-layer morphology. Independent confirmation testing at the exact optimizer condition is required before the predicted optimum can be described as experimentally validated.

Future work should focus on experimental validation of the predicted optimum and on deeper characterization of the printed microstructure using techniques such as DSC and X-ray micro-computed tomography. In addition, a larger experimental database could be developed to support machine-learning models capable of predicting mechanical properties, porosity and interlayer quality from MEX processing parameters. Such approaches could contribute to more intelligent and reliable optimization of high-performance PPS components

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**Declarations:** Conflict of interest: The authors declare no competing interests.

**Ethical approval:** Not applicable. Funding

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## **Author Contributions Statement:**

Z.B., A.E.M., and H.R.V. conceived and designed the study.

Z.B. and A.E.M. carried out the experiments and data acquisition.

Z.B., A.E.M., and H.R.V. performed data analysis and interpretation.

Z.B., A.E.M., B.H., and A.E.H. developed the methodology and validation procedures.

Z.B. and A.E.M. wrote the original draft of the manuscript.

A.E.M., H.R.V., B.H., and A.E.H. reviewed and edited the manuscript.

A.E.M., B.H., and A.E.H. supervised the research and provided critical revisions.

All authors have read and approved the final manuscript.

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