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Full Factorial Design for Formulation and Optimisation of Polyvinyl Alcohol with Starch Composite Using Conventional Injection Moulding

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ABSTRACT

The combination of polyvinyl alcohol (PVOH) and starch composites leads to the production of biodegradable polymer composites. This polymer compound has good qualities for being used as a biodegradable material, which makes it easier to stop the buildup of synthetic substances made from petroleum. This study is intended to analyse the processability characteristics of PVOH-starch composites using actual injection moulding. The processability study of PVOH-starch found that the compound could be injection moulded. The name tag product (NTP) was used as a product outcome during the injection moulding. The verification of the optimisation was conducted by means of statistical analysis employing full-factorial design methodologies. In short, the statistical results have indicated that optimal processing techniques can contribute to the production of NTP with little volumetric shrinkage (as observed in short-shot scenarios) while maintaining acceptable levels of variability. Finally, the optimum injection moulding process parameters were found at 200°C and a pressure of 90 bar. This is because the NTP was produced at the lowest variation of the NTP's total weight in this set of parameters.

Keywords: Composite, full factorial design, injection moulding, polyvinyl alcohol, starch

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INTRODUCTION

The composite of polyvinyl alcohol (PVOH) loaded starch was developed at the beginning of the 1980s as an alternative biodegradable polymer technology to the existing conventional polymers, such as polyethene,

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polypropylene and other petroleum-based polymers (Ray et al., 2021; Wang et al., 2023). The utilisation of starch as a matrix for the advancement of biodegradable polymers has gained attention due to its complete biodegradability and cost-effectiveness in production (Chopra et al., 2013). Numerous researchers have extensively explored starch in the field of edible films and coatings to utilise it as a protective agent for food products (Flores et al., 2007).

The present research indicates that incorporating starch with synthetic polymers has enhanced the resulting material's biodegradability. Starch exhibits polarity, rendering it incompatible with non-polar synthetic polymers like polyethene when combined. According to the findings of Jagannath et al. (2006), the mechanical characteristics of low-density polyethene filled with starch were observed to deteriorate. Hence, to address this issue, incorporating an intrinsic polar polymer into the blending process will provide compatibility between the components. It is probable that a composite material consisting of both PVOH and starch, which are both polar polymers, will exhibit superior mechanical capabilities and barrier behaviour (Siddaramaiah et al., 2004). In the context of polymer films, incorporating glycerol as a plasticiser mitigates brittleness by disrupting the hydrogen bonding interactions between lipid and hydrocolloid molecules (Pan et al., 2022).

Currently, limited efforts have been made to develop processable injection-moulded starch composites using either native or modified starch. This study evaluated the processing parameters for the composites to be processed via conventional injection moulding. The comprehensive examination and determination of thermal and flow characteristics are crucial in optimising the processing parameters in order to facilitate the broad utilisation of these compounds across diverse industries, particularly in conventional polymer processing methods like injection moulding, extrusion, blown film, and thermoforming techniques (Ramesh, 2016). In addition, an investigation was conducted on the processing behaviour of PVOH/Starch using the injection moulding technique often employed in various industrial applications. This study's findings also have significant industrial implications. The optimised injection moulding parameters for PVOH/starch composites provide a cost-effective and environmentally friendly alternative for manufacturing biodegradable products, such as packaging and consumer goods.

Typically, if a new material is introduced to the injection moulding process, trial and error are required to determine the optimal processing parameters. Most of the time, personnel will resolve this issue based on their experience or with the aid of a processing troubleshooting guide (Gülçür et al., 2023). All variables except one are held constant in this task, and elements are tolerated through trial and error. This situation may have required hours or days to determine the optimisation parameter. More variables will bring more problems, squander time, and sometimes not detect the troublesome variables (Krantz et al., 2023). When this occurs, the operator must continually adjust the system to produce quality parts without resolving the underlying issue (Trotta et al., 2021). This will result in a loss of competitiveness in the long term. The optimal parameter is determined using a full factorial design to encourage further development and enhancement of biodegradable starch-based polymer compounds for injection moulding (Moo-Tun et al., 2020). Compared to previous studies, which primarily focused on the mechanical properties of starch-based composites, this research uniquely integrates a full factorial design methodology to statistically optimise processing parameters for conventional injection moulding applications.

The optimised parameters demonstrated in this study align with recent advancements in biopolymer research but provide a novel focus on statistical optimisation. This sets the study apart from prior works relying on trial-and-error methods for parameter determination. As highlighted in Jung et al. (2023), statistical optimisation plays a pivotal role in improving process efficiency, which is directly addressed in this study (Jung et al., 2023). The study examined two process parameters using a 2-level full factorial design approach. The two process parameters involved in the injection moulding process are temperature and pressure. The variables of interest in the experiment were the weights of the samples. A complete factorial experiment includes all possible level combinations for each factor. According to Antony (2023), the total number of experiments for investigating factors at the 2-level was 2. In the early phases of experimentation, the full factorial design is particularly useful, especially when the number of process parameters or design parameters (or factors) is less than or equal to four (Antony, 2023). One of the assumptions for 2-level factors is that the response is approximately linear over the range of the selected factor setting.

MATERIALS AND METHODS

Injection Moulding

Polyvinyl alcohol-starch (PVOH/Starch) blends were successfully injected through the "JSW N100 BII" injection moulding machine, complete with a reciprocating unit. Testing specimen mound shape was used for the injection moulding process. On the other hand, name tag product shape (NTP) was used to optimise the product injection moulding processing parameter. The NTP was drawn using commercial computer-aided design (CAD) software. Figure 1 shows the details of the NTP drawn by CAD based on the mould, as shown in Figure 2. This process was done using the injection moulding machine Demag EL-EXISE 60/370, as shown in Figure 3.

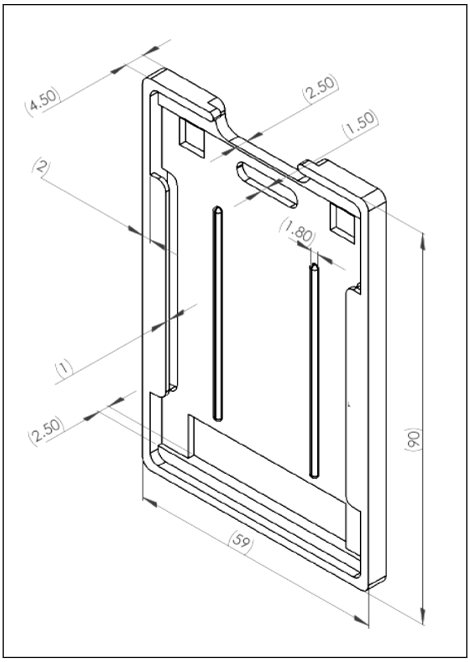
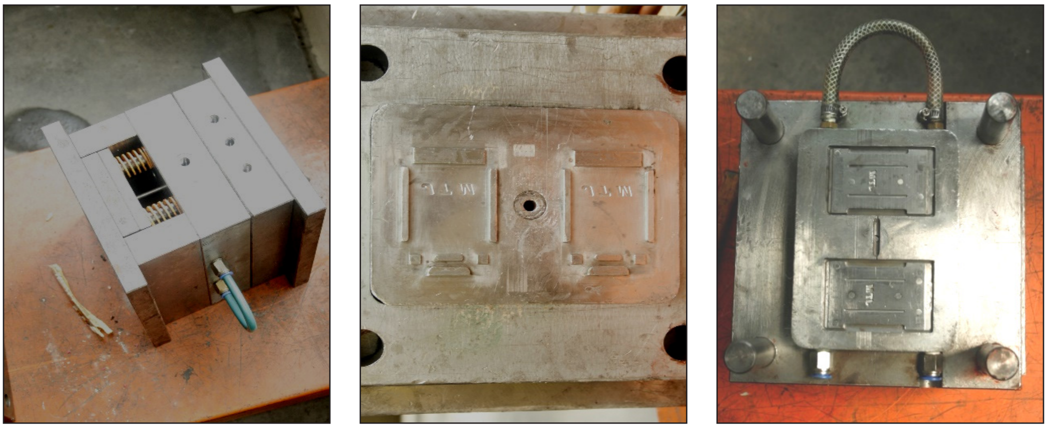


Figure 1. Drawing and measurement of NTP (unit in mm)



(a) (b) (c)

Figure 2. (a) NTP mould set; (b) Female mould of NTP; and (c) Male mould of NTP

Figure 3. Injection moulding machine Demag EL-EXISE 60/370

Full Factorial Design

The online investigations were carried out to identify the optimal processing parameters, namely the injection temperature and pressure. The 2-level full factorial design method was employed to investigate two process parameters. The variable of interest was the weight of the sample. Based on a 2-level full factorial design, 2κ trials must be conducted to provide a comprehensive statistical analysis. κ represents the quantity of process parameters (Antony, 2023). Thus, in this scenario involving two process factors, four experiments were conducted to assess the impact of the process parameters on the weight of the samples. The arrangement of the experimental full factorial design layouts for PVOH/Starch is presented in Table 1.

The collected data was imported into the statistical software tool Minitab 15 for analysis to determine the impact of each process parameter on the weight of the sample. The main goal was to determine the most favourable process condition to maintain a consistent sample weight. Following the completion of the analysis of the outputs, an experiment validation was conducted to verify the reproducibility of the results. Ultimately, the specific processing parameters were documented for subsequent utilisation.

Table 1
Full factorial design of the experimental layout of PVOH/starch injection moulding process parameters

Trial	**A**	**B**	**AB**	**Sample Weight (g)**		
1	-1	-1	1	W1	W2	W3
2	-1	1	-1	W1	W2	W3
3	1	-1	-1	W1	W2	W3
4	1	1	1	W1	W2	W3

Note. A= Injection temperature (°C); B= Injection Pressure (Bar)

Differential Scanning Calorimetry (DSC) Analysis

Thermal analysis of the PPVA/TS blends was carried out using Differential Scanning Calorimetry (DSC-7 Perkin-Elmer) to determine the melting temperature (Tm) of the PVOH/starch blend. The samples were placed in sealed 10 mg aluminium pans under constant nitrogen flow. DSC was performed by heating a 5–12 mg blend sample from 30°C to 260°C. The heating rate used was at 10°C/min.

RESULTS AND DISCUSSION

The injection moulding processing parameters were optimised using the Demag EL-EXISE 60/370 machinery. The selection of the mould shape for the name tag product (NTP) was made as the final result for evaluating the processing parameters in the injection moulding process—the analysis aimed to determine the optimal injection moulding parameters for manufacturing NTP. The trial session was conducted to identify the range of settings for each parameter before determining the appropriate and optimised variable tuning using the full factorial design method.

Differential Scanning Calorimetry (DSC) Analysis

The particular thermal scanning range was selected to include the melting point of PVOH at 230°C but lower than 250°C to avoid dehydration of hydroxyl groups (Bercea, 2024).

In general, the melting stage can be detected through the presence of the endothermic peak in DSC thermograms (Sangthongdee et al., 2022). The onset temperature, end-point temperature, and melting temperature (T_m) are presented in Table 2. Incorporating starch into the PVOH has led to a decrease in the onset and end-point temperature as well as melting temperature (Tm) shown in Table 2. The melting properties of PVOH/starch blends are significantly affected by crystal formation within the blends, which depends on the starch composition in the matrix. The reduction in PVOH content significantly affects the amount and size of crystallites, weakening the interaction between PVOH and starch and consequently reducing the crystalline region (Othman & Azahari, 2011). The results indicated a melting range between 180°C and 240°C, which aligns with the experimental temperature settings. This data justifies the chosen temperature range and explains the formation of flashing and burn marks at higher temperatures (above 220°C).

Table 2
Onset and end-point melting temperature and melting temperature (T_m)

Sample	**Onset (°C)**	**End-point (°C)**	**T_m (°C)**
PVOH	183.41	200.00	196.03
SD	4.33	3.04	2.18
PVOH/starch	173.89	189.97	186.37
SD	1.25	1.78	2.28

*SD= Standard Deviation

Injection Moulding Start-up Setting

Two parameters were chosen for the optimisation process. Initially, the setting was established in the presence of pre-injected tensile bars. The NTP injection during the initialisation phase exhibited inconsistencies with respect to the samples' weight. Adjusting the setting was a viable solution to achieve the desired tolerance level. During the trial session, a list of parameters was established to determine the most optimal set for the subsequent validation experiment.

The two parameters subject to optimisation were injection temperature and injection pressure. Other parameters, including the mould temperature, holding time (comprising both cooling and packing time), injection speed, and packing pressure, were fixed at 45°C, 20 s, 60 mm/s, and 70 bar values, respectively. The experimental conditions employed during the trial injection moulding of NTP were recorded and organised in Table 3. The outcomes resulting from each alteration were carefully monitored and documented.

In the injection moulding of PVOH/starch composites, ensuring sufficient injection pressure is crucial to prevent short shots and situations where the mould cavity is inadequately filled, resulting in faulty components (Shogren, 1995). Pressures below 90 bar have been observed to induce such problems, highlighting the necessity of adequate

Table 3
Setting range injection temperature and injection pressure during trial run

Sample	Injection Temperature (°C)	Injection pressure (bar)	Remarks
PVOH/Starch	180	90	Short shot
	200	90	Fully moulded
	220	90	Fully moulded
	240	90	Fully moulded, but have burn marks
	180	110	Short shot
	200	110	Fully moulded
	220	110	Fully moulded
	240	110	Flashing and have burn marks
	180	130	Short shot
	200	130	Flashing
	220	130	Flashing
	240	130	Overpacking and having burn marks

injection pressure. Insufficient pressure prevents the molten polymer from fully occupying the mould cavity, leading to inadequate filling and diminished product (Liou et al., 2023).

Full Factorial Analysis and Actual Injection Moulding Verification

The study employed a full-factorial design methodology to examine the effectiveness of various processing parameters in producing NTP. The investigation of processing parameters by injection moulding was conducted using the design of experiment (DOE) methodology. The experimental design for the injection moulding validation experiment is presented in Table 4, which displays the process parameters and their corresponding levels. The chosen parameter ranges were further analysed using the full factorial design procedure.

Each set of combinations was repeated four times to calculate the NTP weight's standard deviation (SD). The standard variation was used to measure the level of variability in the process parameters. The remaining parameters, such as the mould's temperature, the duration of holding (which includes cooling and packing time), the speed of injection, and the pressure of packing, were set at 45°C, 20 seconds, 60 millimetres per second, and 70 bars, respectively.

Table 4
The level of process parameters utilised in the verification experiment

Sample	Process Factor	Low Level	High Level
PVOH/Starch	Injection temperature (°C)	200	210
	Injection pressure (bar)	90	110

Analysis of Process Factors: Effects on Total Weight of PVOH/Starch

This analysis aimed to investigate the influence that process factors had on the total weight of the sample in order to determine the ideal combination of process factors that would result in the highest possible sample weight. The analysis was performed by providing the PVOH/Starch compound, with the parameters established in line with the specifications in Table 4. The entire weight of NTP was the response to this analysis, tabulated in Table 5.

According to the data presented in Table 5, the maximum aggregate weight of NTP was observed at a temperature of 200°C and a pressure of 110 bar. The findings demonstrated that the diverse combinations of parameters led to significant variations in the total weight of NTP, suggesting interactions among the process components. In summary, a comprehensive statistical analysis can be employed to derive the optimal set of process parameters. The results were imported into Minitab 15 for analysis.

Figures 4 and 5 depict the normal probability plot and Pareto chart of the standardised effects based on the computed data in Minitab 15. Figure 4 depicts the reaction, represented by a red dot, which exhibits a similar pattern to the normal distribution condition, illustrated by a blue line. The scenario suggests that the reaction fell within the established parameters and can be subjected to additional examination using a Pareto chart, as depicted in Figure 5. The Pareto chart displayed the process factor along with its standardised effect. According to the information provided, when the standardised effect exceeds the threshold indicated by the red line in Figure 4, the specific process factor or the interaction of process factors has considerably influenced the total weight filled by PVOH/Starch in the NTP.

According to the Pareto Chart, it can be observed that both factor A (injection temperature) and component B (injection pressure) significantly impact the total weight of the NTP. The injection temperature factor influenced the overall weight, as the greater temperature facilitated the free movement of the polymer complex, enabling it to effectively occupy any available space (Moo-Tun et al., 2020; Ray et al., 2021). Therefore, the issue of premature solidification of material prior to complete cavity filling might be effectively mitigated (Ghazy et al., 2016). Furthermore, the use of increased injection pressure has resulted in the compression of the molten polymer compound, enabling a greater quantity

Table 5
Total weight of NTP filled by PVOH/Starch

Trial	Injection Temperature (˚C)	Injection Pressure (bar)	Weight (g)				Mean
			1	2	3	4	
1	200	90	26.51	26.43	26.07	26.22	26.31
2	200	110	27.33	26.88	26.53	26.58	26.83
3	210	90	25.33	24.17	25.11	24.01	24.66
4	210	110	25.02	24.93	25.40	25.11	25.12

of molten PVOH/Starch compound to be injected into the mould (Liu et al., 2021). This is indicative of the larger overall weight of the final product. The investigation of the interaction of process factors was conducted comprehensively by generating a normal interaction plot, as depicted in Figure 6. In terms of the high total weight response, the interaction plot results indicated that the injection temperature and pressure should consistently be set at 200°C and 110 bar, respectively.

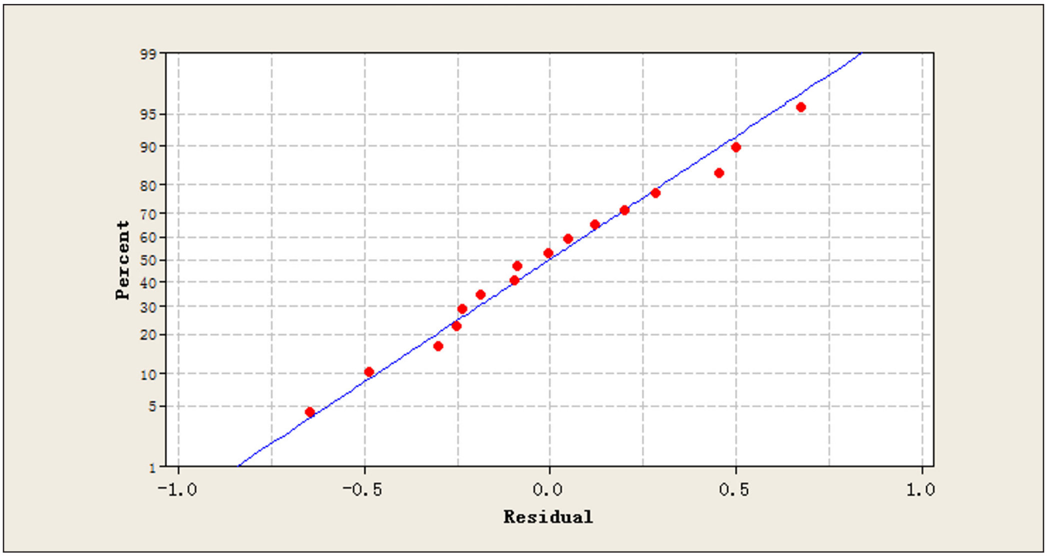


Figure 4. Normal probability plot of NTP (response is weight)

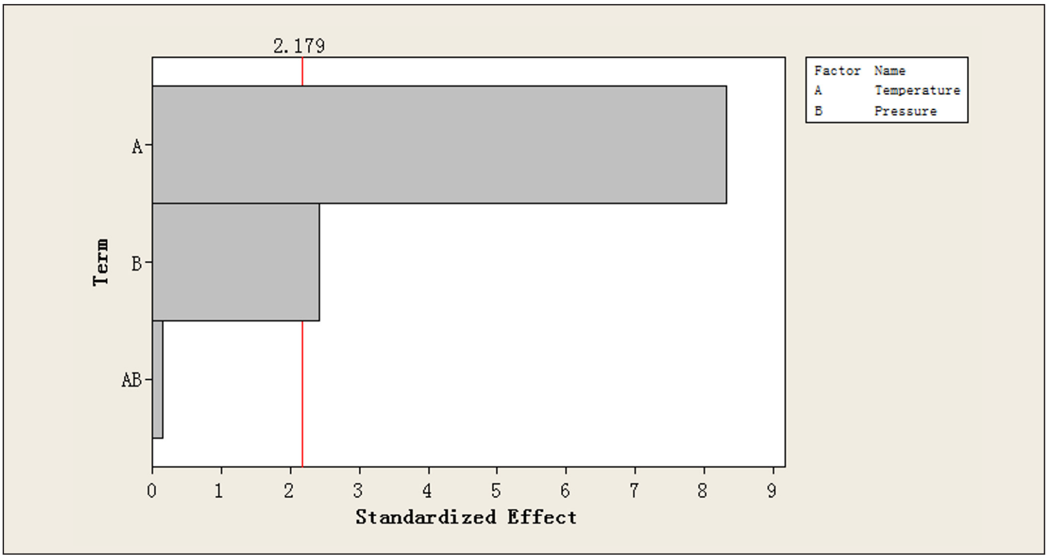


Figure 5. Pareto chart plot of NTP (response is weight, alpha= 0.05)

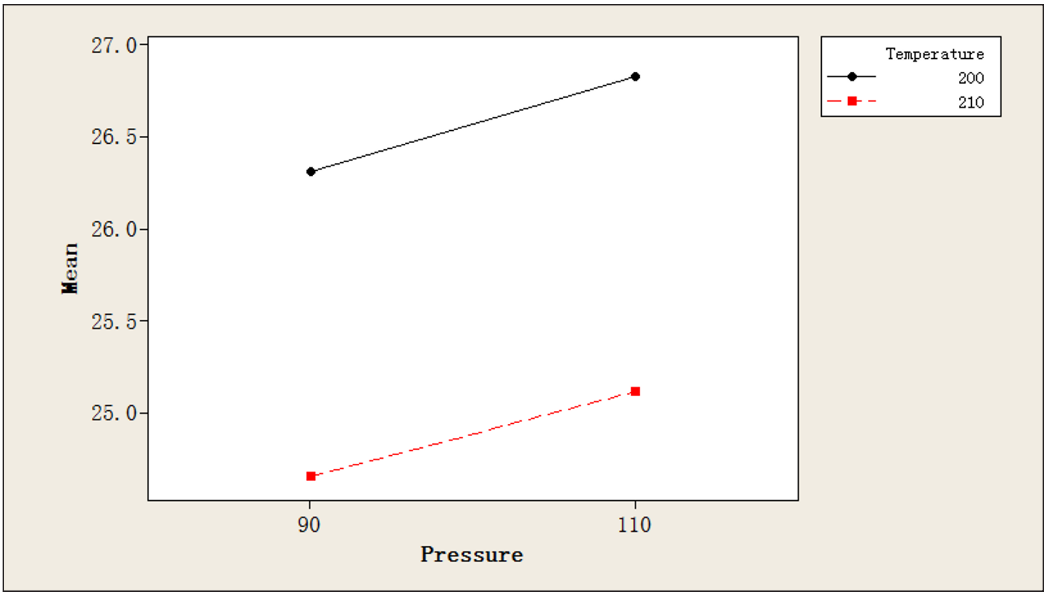


Figure 6. Interaction plot for a total weight of NTP (data means)

Analysis of Process Factors: Effects on the Variability of Total Weight of PVOH/Starch

This analysis aims to identify the process element affecting the variability of the NTP's overall weight when filled with PVOH/Starch. The table marked as Table 6 contains standard deviations (SD) information. Standard deviation (SD) is a crucial factor in this analysis and is transformed into logarithmic form to bring the standard deviations closer to a normal distribution (Antony, 2023). A high standard deviation of the individual process parameters results in a significant variability in the quantity of NTP generated. As a result, there was a lack of uniformity in the manufacturing of NTP, potentially resulting in deviations from the specified product requirements. The Pareto chart depicted in Figure 7 indicates that the injection temperature (A) and pressure (B) do not exert a substantial impact on the variability of NTP's total weight. Furthermore, there has been an interaction between these two process elements (AB).

Figure 8 illustrates the fluctuation in the weight of the total NTP (Nucleotide Triphosphate) at an injection temperature of 200°C and an injection pressure of 90 bar. These findings suggest that the largest total weight of NTP occurred at a temperature of 200°C and a pressure of 110 bar, resulting in a greater variance compared to the conditions set at 200°C and 90 bar. These findings indicate that the injection temperature and pressure do not have a substantial impact on the fluctuation in the production of NTP. Despite possessing the greatest overall weight, it exhibited the second-highest degree of variability at a temperature of 200°C and a pressure of 110 bar. Currently, the most favourable

Table 6
SD of NTP filled by PVOH/Starch

Trial	Injection Temperature (°C)	Injection Pressure (bar)	Weight (g)				Mean	SD
			1	2	3	4		
1	200	90	26.51	26.43	26.07	26.22	26.31	0.200062
2	200	110	27.33	26.88	26.53	26.58	26.83	0.367423
3	210	90	25.33	24.17	25.11	24.01	24.66	0.661791
4	210	110	25.02	24.93	25.40	25.11	25.12	0.203715

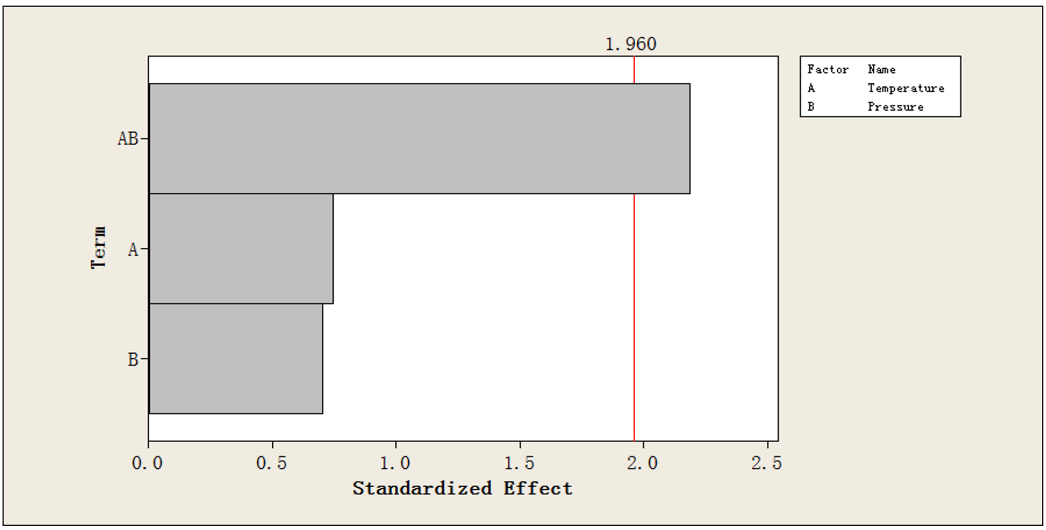


Figure 7. Pareto chart of NTP-filled PVOH/Starch (response is ln of weight SD, alpha= 0.05)

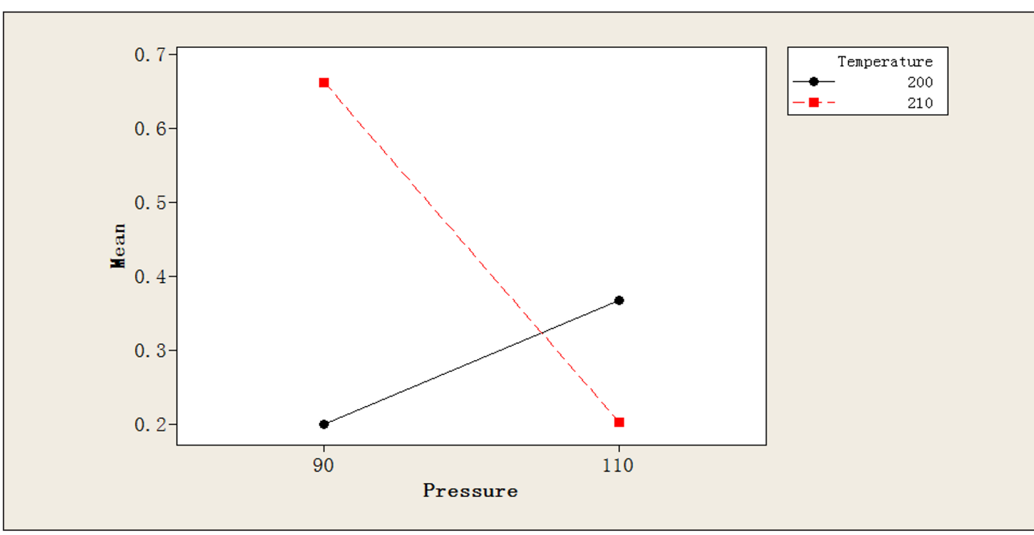


Figure 8. Interaction plot for a total weight of NTP (data means)

condition for generating NTP is observed at a temperature of 200°C and a pressure of 90 bar. This condition significantly impacts the quality of the resulting product. Additionally, by reducing variance, it is possible to mitigate the inconsistency that may arise during the production of NTP (Moo-Tun et al., 2020).

A validation experiment was undertaken to ensure the reproducibility of the process parameter acquired for the PVOH/starch blend. The experiment validated the feasibility of doing the analyses in a real-world conventional injection moulding process. The findings are documented in Table 7. The statistical study confirmed that the findings obtained from optimising processing parameters for producing NTP filled with PVOH/Starch are reproducible. These findings also validate the reproducibility of the optimal parameters in a real-world injection moulding process. These results underscore the practicality of employing biodegradable composites in industries aiming to reduce reliance on petroleum-based materials.

Table 7
NTP's lower variation validation experiment

Sample	Injection Temperature (°C)	Injection Pressure (bar)	Weight (g)				Mean	SD
			1	2	3	4		
PVOH/Starch	200	90	26.37	26.38	26.61	26.12	26.37	0.200167

CONCLUSION

A combination of polyvinyl alcohol (PVOH) and starch has been effectively developed and established. The development of PVOH/Starch blends for injection moulded applications was evaluated. The reproducibility of optimal parameters at 200°C and 90 bar reinforces their industrial applicability for producing consistent, high-quality biodegradable products. This work provides a foundation for scaling up PVOH/starch composites in environmentally sustainable applications. Under these conditions, the NTP exhibited higher precision because of reduced variation. Additionally, by reducing variance, it is possible to mitigate the inconsistency that may arise during the manufacture of NTP. The selected set of parameters was subsequently validated to assess its reproducibility, which exhibited a comparable trend to that observed in standard injection moulding practices in the industry.

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