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PROCESSING PARAMETERS OPTIMIZATION OF VACUUM-BAGGING PREFORMING IN OVEN CURE FOR INTER-LAMINAR SHEAR STRENGTH (ILSS) IN GLASS/EPOXY COMPOSITE LAMINATE

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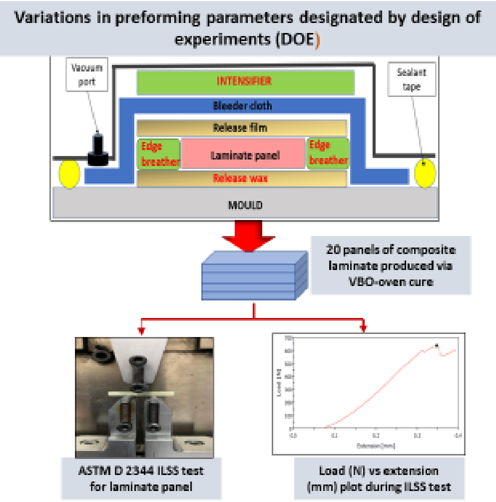


Abstract

Autoclave had been restricted to immense production expenses and overabundant residual stress. These disadvantages had prompted development on the alternative of out-of-autoclave (OoA) processing. The optimization on vacuum-bagging-only (VBO) pre-forming process in producing high inter-laminar shear strength (ILSS) of composite laminate was proposed. The effects of individual and combined pre-forming parameters of VBO-oven cure processing for the conventional low-cost glass/epoxy composite material towards ILSS of cured laminates were quantified. 20 composite panels were manufactured following the designated parameter combinations based on central composite design in fractional factorial for response surface model. Three factors of vacuum debulk duration, edge breather number of sides and intensifier weight were investigated. For validation, two laminates without additional processing parameters were produced via oven (baseline) and autoclave. ILSS test was conducted based on ASTM D 2344. The interaction between combined parameters was analyzed using analysis of variance (ANOVA). Lowest ILSS was found in laminates where intensifier was absent, while highest ILSS was measured with edge breather, debulk and intensifier at different levels. An optimum combination of 30 minutes debulk, each sides of edge breather and 1kg intensifier were validated to produce laminate with highest ILSS of 38.96 MPa, which was 23.53% higher than baseline laminate.

Keywords: inter-laminar shear strength, VBO-oven cure, out-of-autoclave, optimization, composite laminates

1.0 INTRODUCTION

Laminated composite materials have attracted numerous interests from the airline industry, which pursued components with high ratio of strength to weight and minimal environmental impact in order to improve the performance capacity of the vehicle [1]. Generally, prepregs were used to produce such composite structures, where the fabrication processes were mainly categorized into two stages of pre-forming and curing [2]. During pre-forming process, plies of B-stage prepreg were carefully prepared at room temperature before stacked into vacuum bagging mould. Vacuum pressure was then introduced to eliminate voids in the laminate, forcing excess air and resin from the mould. Then, the cure process took place where heat and pressure were applied simultaneously to consolidate the materials [3]. A massive pressure application was typically used to eliminate entrapped air and voids, and further consolidate the individual plies and fibers in a uniform manner. There were a wide variety of preforming processing techniques available inclusive of liquid moulding, filament winding, vacuum bagging (VBO), etc., while the curing was continued in autoclave or an out-of-autoclave (OoA) cure such as microwave, oven, Quickstep, room temperature and so forth [4-6]. Vacuum bagging with autoclave curing was the most common and effective manufacturing technique in producing composite laminate with minimal void content for extreme performance applications especially in aerospace industry [7, 8].

Nevertheless, autoclave curing yielded the drawbacks of approximately 30 to 50% longer time consumption, excessive residual stress (leading to composite core crush, skin pillowing and dimpling), and 30 to 50% of higher costs (capital, production and running) [5, 9-11]. Hence, lower cost of OoA curing process especially vacuum-bagging-only with oven curing (VBO-oven cure), offered an attractive and closest alternative from autoclave, removing the high-pressure application and steep cost in autoclave [12-14]. Despite the lower cost benefiting from the VBO-oven cure method, the composite parts hardly achieved the mechanical properties requirement as those of autoclave counterparts due to the insufficient compaction pressure, leading to elevated void content [15,16]. In turn, the void content was found to have a great effect on the composite inter-laminar shear strength (ILSS) which affect the performance of structural component critically. Hence, the investigation in the processing parameters was crucial since void were mostly generated during composite manufacturing process. Regardless those studies considering on the curing techniques [17-19], several researchers focused on the pre-forming methods enhancement in VBO, which involves layup (debulking) [20] and bagging configurations (e.g. mould release type, intensifier and edge breather) [21,22].

Kratz and Hubert [23] indicated that the key challenge associated with VBO was to develop techniques enhance the permeability of the prepreg, enabling effective removal of trapped air and volatile substances prior to resin solidification.

It was observed that the air permeability exhibited a reverse correlation with the magnitude of the compaction pressure, wherein the characteristic decreased as the pressure was raised. Davies et al. [24] and Hubert and Pousartip [21] suggested that debulking should be performed on each separate prepreg arrangement for a duration of 20 minutes at ambient temperature, utilizing a vacuum pressure of 100kPa. This will ensure effective removal of voids between the layers of prepreg. Liu and Hubert [25] investigated the effect of heat treatment combined with debulk on carbon/epoxy laminates and found that heated debulk reduced the manufacturing cycle time by 26-71%. The debulk was done for 120 minutes at 48 ºC, resulted in only slightly higher ILSS of 75.98 MPa and void content of 2.44%, as compared to 72.36 MPa and 2.88% of its 16 hours room temperature debulk counterparts, respectively. By employing a rubber seal as the intensifier and positioning it under the steel plate, a uniform dispersion of compaction pressure was achieved during the curing process [26]. Conversely, dry glass fiber utilization as breather or bleeder at laminate edges within the bagging was claimed to be one of the successful technique employed in VBO-oven cure. Edge breather ensured improved air permeability along the laminate's edges, facilitating the path for trapped air to escape [27].

Caubergh s and Hubert [28] justified that air-permeability was optimum and sufficient despite when at least one laminate edge was connected to edge breathing system, contributing to reduced void distribution and improved thickness uniformity. The edge breather was used to alleviate in-plane air and porosity extraction in transverse direction, however longer time consumption was needed for a wider and thicker composite laminate, where air flow path was more convoluted [29, 30]. Hence, an immaculate edge breathing mechanism was necessary since improper setup would directly interfere in laminate's ply-slippage and resulted in a larger undesirable compressive hoop stress within laminate's fiber [31]. In a review conducted by Judd and Wright, the majority of studies focused on the influence of voids on the ILSS characteristics of composite structures. They reported that an approximately 1% rise in void content led to a decrease of around 7% in ILSS of laminate structure [32]. Additional studies also noted that the outcomes of investigations concerning the reduction in ILSS and tensile strength were associated with an increase in void content [33-35]. Hou et al. [36] stated that ILSS characteristics were crucial for enhancing the aerodynamic performance of aircraft control-lift surfaces like elevators and rudders during their operational lifespan. Attaining a sufficient ILSS was a vital demand for aileron ribs, given that the sidewalls of these structures endure considerable flexural pressures and aerodynamic loads during their operational life [37].

Findings from previous researches emphasized that the pre-forming processes had indeed affected the final properties of the cured composite, in which the qualities could be further improved if the VBO processing techniques were optimized. The influence of the parameters was investigated, and the contribution of individual and combination of the processing routes was quantified. An experimental study based on central composite design in fractional factorial method for response surface model was proposed to investigate the contribution of each and combined vacuum-bagging only (VBO) pre-forming parameters towards the low-cost, conventional material composite laminate ILSS, leading to 20 different processing routes. For validation purpose, two laminates without additional processing parameters were produced via oven (baseline) and autoclave. ILSS test based on ASTM D 2344/D 2344M-00 was carried out to determine the ILSS of laminate. The interaction between the combined parameters was subsequently analyzed using an analysis of variance (ANOVA) tool. Based on the results, an optimum method of processing parameters in VBO-oven curing technique for highest ILSS of composite laminates were evidently established.

2.0 METHODOLOGY

2.1 Materials

The prepreg used was an epoxy reinforced plain weave glass fiber Cycom 7668/7881-1 material for conventional autoclave cure. Epoxy resin content was 36±2% and had 177°C flame. The prepreg was manufactured via hot-melt technique and demonstrated strong adhesion and pliability for a minimum of 15 days at 24°C. After curing, it showcased remarkable resistance to thermal aging and displayed exceptional tensile and compression characteristics throughout its operational duration. This prepreg type was usually cured using autoclave where the laminate and sandwich structural composite product was proposed for the external components of aircraft structure.

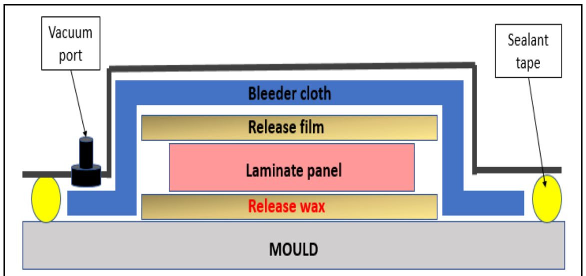
2.2 Processing Routes Of Glass Fiber / Epoxy Laminates

Prepregs were cut into dimension in a controlled humidity and temperature conditions at 50% and 24°C. Nine plies of prepreg were prepared into square panels with the dimension of 300mm × 300mm, producing the nominal thickness of about 2 mm. Laminates were manufactured using the VBO method, following various configurations of bagging arrangements, in accordance with the processing routes described in the following. Three factors of vacuum debulk duration, edge breather number of sides and intensifier weight were investigated at three different levels via central composite design in fractional factorial method for response surface model. 20 different composite panels were produced following the designated parameter combinations as illustrated in Table 1. For validation purpose, two laminates without additional processing parameters were produced via oven (baseline) and autoclave, respectively.

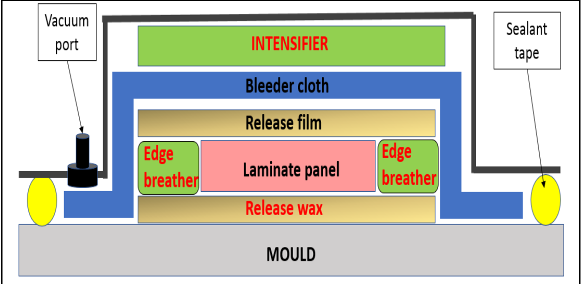
Table 1 Processing parameter's factors and levels.

Symbol	**Control Factor**	**Level 1**	**Level 2**	**Level 3**
A	Debulk duration (minutes)	*0 min*	*30 min*	*60 min*
B	Edge breather (no. of sides)	*1 side*	*2 sides*	*All sides*
C	Intensifier weight (kg)	*0 kg*	*1 kg*	*2 kg*

Figure 1 shows the differences in vacuum bagging employed in the study. In the baseline-oven and autoclave cure bagging of Figure 1(a), the plies were stacked over a flat aluminum mould with the application of TR-104 high temperature mould wax in between laminate plies and metallic tool. Other consumables were position above the prepreg in the subsequent sequence: non-perforated release film of RF260 by Tygavac Advanced Materials, bleeder cloth of Airweave N10 (Airtech Advanced Materials) and vacuum-bag of WL7400 (Airtech Advanced Materials), enclosing the assembly with the sealant tape of GS2131/2 (General Sealants Inc), employed for the purpose of sealing the perimeters of the vacuum bagging. A single channel of vacuum port (Airtech Advanced Materials) was utilized to remove the air from the sealed bagging.



(a) Baseline oven and autoclave configuration



(b) Bagging configurations with additional parameters

Figure 1 The different configurations of vacuum bagging

The bagging set up with additional processing parameters included intensifier or caul plate to boost the contact pressure that may contribute to lower porosity and void content of cured laminates. A 300mm x 300mm of g stainless steel plate of different weight was used as an intensifier and was placed on the laminates, on top of the bleeder. A 1 cm thick x 2.5 cm width of Tygavac Y-0094 dry glass fiber was employed as the edge breather material, positioned adjacent to the laminate layers to facilitate the removal of in-plane voids. This breather fabric served as an in-plane bleeder, effectively aiding the removal of air and excess resin from the laminate through the in-plane direction.

Vacuum debulking was executed during the layup process employing JK-VP-3C single stage vacuum pump of Shanghai Jingke Scientific Instrument with ultimate vacuum of 5 Pa, with an Airtech-Airflow 65R hose and VV 401 Airtech vacuum port was connected to the vacuum bagging. An approximate vacuum pressure of 3.86 kPa (equivalent to 29 mmHg) was employed for a duration of 20 minutes at room temperature for every 3-ply prepreg placement. This process was repeated until a total of 20 prepreg plies had been stacked.

For the oven-cured laminates, the curing cycle was performed in an oven with one dwell section for 120 minutes at 180 ± 12°C. This cure cycle entails heating and cooling rates of 2°C/minute. A primary vacuum pump was connected to the vacuum bag, providing a vacuum pressure of 1 kPa to evacuate air from the beginning of oven curing until the removal of laminate panel, when the temperature was cooled to 60 °C. Whereas, a pressure compaction increment from 103 kPa to 310 kPa was supplied to the autoclave-cured laminate for two hours during autoclave cure, as per recommendation by Cycom 7668/7881-1 prepreg manufacturer.

2.3 ILSS Test

ILSS test was done according to ASTM standard of D 2344/ D 2344 M–00. A total of 10 samples per panel were prepared with dimensions of 25 mm x 8 mm. Test specimens underwent quasi-static loading at a consistent speed of 1 mm/min using an Instron universal testing machine. A constant span length of 18 mm for each ILSS specimen was required by ASTM D 2344/D 2344 M-00 standard to fulfill the calculation requirement for ILSS value. The average ILSS value from 10 samples were then recorded as the ILSS value.

3.0 RESULTS AND DISCUSSION

3.1 ILSS analysis

Results of ILSS were tabulated in Table 2. It was observed that ILSS of the panels with processing parameters were at the range of 32.63 MPa to 38.96 MPa. On the contrary, the lowest ILSS of 31.54 MPa was still obtained without any processing parameters of baseline laminate, while autoclave yielded a moderate ILSS value of 36.44 MPa. ILSS was quantified at the lowest with values of 32.63 MPa, 33.33 MPa and 33.67 MPa when intensifier was absent in all these settings. Conversely, highest ILSS was obtained with the values of 38.69 MPa, 38.43

MPa and 37.89 MPa via the combinations of edge breather, debulk and intensifier at assorted levels. These higher ILSS values signified stronger interlaminar bonding and better resistance to shear forces within the composite material.

Table 2 ILSS value of laminate panels

Trial	Factor			ILSS (MPa)
	A* *(Debulk)	***B (Edge Breather)***	***C (Intensifier)***	
1	1	1	1	33.33
2	1	3	1	33.67
3	2	2	1	34.31
4	3	1	1	32.63
5	3	3	1	33.58
6	1	2	2	36.02
7	2	1	2	35.12
8	2	2	2	37.76
9	2	2	2	37.01
10	2	2	2	36.37
11	2	2	2	35.17
12	2	2	2	36.46
13	2	2	2	36.25
14	2	3	2	38.96
15	3	2	2	38.43
16	1	1	3	35.37
17	1	3	3	37.33
18	2	2	3	37.54
19	3	1	3	37.89
20	3	3	3	36.67
Autoclave				36.44
Baseline (oven)				31.54

Generally, intensifier was used to intensify the compaction and consolidation during composite cure, minimizing the void content, and subsequently improved the mechanical qualities. In this research work, it was found that intensifier application was a mandatory in oven cured composite to achieve product with highest ILSS value. The absence of intensifier was presumed to contribute in weaker interlaminar bonding and reduced resistance to shear forces, resulting in lower ILSS values. Utilizing the additional pressure via intensifier strengthened fiber-matrix bonding by facilitating resin to fill the spaces between fibers, ensuing in a more densely packed, homogenous and uniform composite. Since ILSS was validated as resin-dominated property, intensifier helped in improving the compaction during cure which aided in promoting resin flow, interfacial adhesion and fiber wetting, contributing to sturdier interlaminar bonding and increased ILSS.

Conversely, the influence of different levels of factors debulk and edge breather towards ILSS was exhibited to be dispersed in all tested laminates. Nevertheless, edge breather was fully utilized in laminate 14 with highest ILSS, contrasted with the minimum edge breather employment for laminate 4 of lowest ILSS. However, debulking was utilized at highest level for laminate 4, but lowest for laminate 14. Generally, edge breather enhanced permeability and promoted excessive resin removal in in-plane direction, which aided in higher fiber volume fraction of produced panels. These ideal fiber volume fraction of laminates led to improved ILSS value due to augmented fiber-to-fiber contact and improved load transfer [38]. In contrast, inappropriate levels, frequency and conditions of debulking was unfavorable to ILSS as a result of void increment of oven-cured composite. The laminate would be partially cured without an appropriate and uniform resin flow, resulting in poor fiber wetting, incomplete resin filling with dry spots, resin rich area and deficient ILSS value.

Indeed, the combination of processing parameters in this validation work namely edge breather, debulk and intensifier, applied at their appropriate levels, contributed in a crucial role in improving the ILSS of composite panel, leading to better mechanical properties and overall performance of composite product. Nonetheless, the specific relationship between the processing parameters and ILSS where optimum level of processing parameters combinations was critical due to the non-linear relationship between ILSS quality and factor parameters. Accordingly, ANOVA analysis for ILSS in was fundamental to examine the interaction between ILSS and those processing parameters with their levels.

In order to evaluate the substantial impact of individual and collective input factors on the resulting response, an ANOVA analysis was performed as shown in Table 3. The value of α or significance level of 0.1 was selected. Hence, model P-value of 0.023 indicated that there was strong evidence to reject the null hypothesis and the model fits the data adequately. Factors C and CC presented P-values of 0.001 and 0.085, indicating highest and significant contributions towards ILSS.

Table 3 ANOVA for ILSS

Source	Sum of Squares	Df	F-value	P-value
Model	48.071	9	3.88	0.023
A	1.211	1	0.88	0.37
B	3.446	1	2.5	0.145
C	29.86	1	21.68	0.001
AA	0.007	1	0.01	0.944
BB	0.153	1	0.11	0.746
CC	5.019	1	3.64	0.085
AB	0.826	1	0.6	0.457
AC	0.878	1	0.64	0.443
BC	0.038	1	0.03	0.872
Std. Dev.		Mean		PRESS
1.1736		35.99		103.939
R-Squared		Adj R-Squared		BIC
0.7773		0.577		82.25

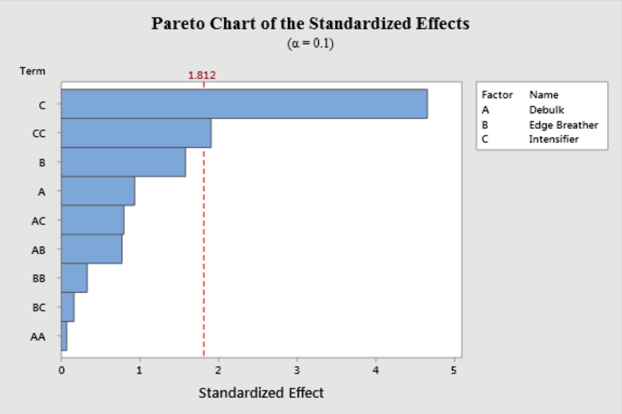


Figure 2 Pareto chart of standardized effects on ILSS

The Pareto chart of Figure 2 visualized the most important contributing factors that delivered the utmost impact towards output response. It was discovered that factors C (intensifier) and CC (intensifier-intensifier) were statistically significant towards ILSS at 0.10.1 α level in the model term, crossing the reference line at 1.812. Attainably, the percentage contribution of factors was calculated from Pareto plot with factor C and CC had the most significant effect of approximately 41.47% and 17%, respectively. The main effect plot demonstrated the differences between factor's level mean, while interaction effect plots scrutinized relationship between factors towards output response, which were illustrated in Figure 3 and 4, respectively. From Figure 3, it was found that the main effect existed in all three factors since each line were plotted in steep slope manners. Factor debulk level 1, edge breather level 1 and intensifier level 1were associated with lowest ILSS, where intensifier had the strongest effect on this quality reduction of approximately 10.45% from none to level 3 intensifier. Whereas, debulk level 2, edge breather level 2 and intensifier level 3 were associated with highest ILSS, where intensifier level 3 contributed to the maximum impact in generating highest ILSS in producing composite laminate. In accordance with the reference line of ILSS, processing parameters of no debulk, one-hour debulk, one-sided edge breather and no intensifier were unfavorable since ILSS were below the average mean. Nevertheless, as compared to the baseline panel in Table 2, the laminate produced without addition processing parameters was discovered to be poorer with lowest ILSS value.

Based on main effect plot of debulk factor in Figure 3, ILSS was increased by approximately 3.69% from none to 30 minutes debulk, then reduced by approximately 1.92% from 30 minutes to one-hour debulk. Meanwhile, from edge breather plot, it was shown that ILSS was increased by approximately 5.03% when two laminate sides were attached with edge breathers as compared to single-sided edge breather. Consequently, ILSS was dropped by approximately 1.23% with the implementation of edge breather on each laminate side. These proved that debulk and edge breather processing parameters should be optimal in producing composite panel with best ILSS value. Conversely for intensifier, ILSS was largely increased by approximately 9.55% from no intensifier to 1 kg intensifier application. As the intensifier weight was then augmented to 2 kg, ILSS was further improved by approximately 0.18%. These attested that the additional dead weight was successful to boost the contact pressure and ensure adequate contact between bagging film and sealant tape to enhance the fiber wetting throughout curing, since ILSS value was critically dominated by resin quality [39].

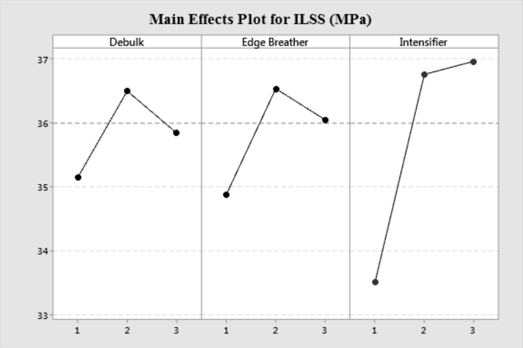


Figure 3 The main effects plot of ILSS

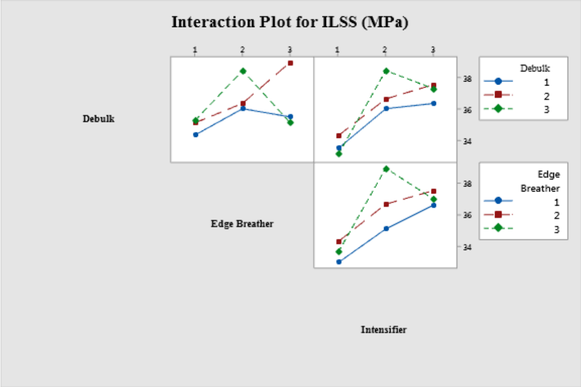


Figure 4 The interaction plot for ILSS

Figure 4 shows the interaction plot for ILSS, which illustrating several significant interactions between two combined factors due to the non-parallel lines plotted. From the first interaction graph of debulk versus edge breather, it was found that the relationship between debulk factor and void content depended on the level of edge breather. When there was no debulk (level 1) employed, single-sided edge breather (level 1) was associated with lowest ILSS quality of approximately 34.5 MPa. However, if 30 minutes debulk (level 2) was utilized, all-sided edge breather (level 3) was associated with the highest ILSS quality of approximately 38.5 MPa.

From the second graph of debulk versus intensifier, it was also demonstrated that the relationship of debulk factor and ILSS depended on intensifier level. Poorest ILSS quality of approximately 33.5 MPa was produced when one-hour debulk (level 3) associated with the absent of intensifier (level 1). Nevertheless, if 1kg of intensifier (level 2) was utilized, best ILSS quality of approximately 38.3 MPa was achieved with longest debulk time for one hour. Likewise, the relationship of edge breather and void content was dependable on intensifier level, according to the interaction plot of edge breather versus intensifier. Lowest ILSS of approximately 33 MPa was yielded with the combination of one-sided edge breather (level 1) along with no intensifier utilization. Nonetheless, when all sides of laminates were placed with edge breather (level 3), then 1kg intensifier (level 2) was associated with the highest ILSS of approximately 38.5 MPa.

It was discovered that debulking and intensifier factors enhanced the resin flow within laminate in inter-plane direction, while edge breather acted as the medium perimeter to ensure enhancement in in-plane direction. The optimal combination of debulk, intensifier and edge breather at distinct condition and frequencies were validated to further intensify the fiber wetting by the resin during layup and curing. As a result, each laminate panel with additional processing parameters produced in this study possessed higher and better ILSS quality than the baseline laminate. In a conclusion, ILSS was found to be optimum with factors combination of debulking, edge breather and intensifier were employed for 30 minutes (level 2), on each side (level 3) and weighted at 1kg (level 2).

4.0 CONCLUSION

Experimental study had been conducted to quantify the contribution of individual and combination of VBO pre-forming parameter variants towards ILSS of the composite laminates. 20 composite panels were produced following the designated parameter combinations based on central composite design in fractional factorial method for response surface model. Three factors of vacuum debulk duration, edge breather number of sides and intensifier weight were investigated. For validation purpose, two laminates without additional processing parameters were produced via oven (baseline) and autoclave, respectively. Lowest ILSS was found in the laminates where intensifier was absent in the processing setup, while highest ILSS was measured with the mandatory combination of edge breather, debulk and intensifier at different levels. In contrast with the panels without any additional processing parameters, lowest ILSS of 31.54 MPa was obtained for baseline laminate (laminate cured in oven without any additional processing parameters), while moderate ILSS value of 36.44 MPa was cured through autoclave processing route. The combination of 30 minutes debulk, each sides of edge breather and 1kg intensifier were validated to yield ILSS property of composite laminate optimally.

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