Statistical analysis of effect of polyamide veil layer on quality and mechanical property of VaRTM CFRP

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Abstract

This study focused on VaRTM which is one of low-cost processing method, and intends to reveal the effect of to insert thermoplastic veil between carbon fiber on molding quality and strength property. Molding quality was assessed by measurement of the fiber volume fraction; the compression strength was determined by doing non-hole compression (NHC) and compression after impact (CAI) test. And design of experiments and analysis of variance were introduced to understand the effect of processing condition to the manufacturing quality and the compression strength properties. With the results obtained, it is confirmed that molding quality is affected by process condition. The CAI test clearly showed the increase in strength as a result of melt-infiltrated of the thermoplastic veil. NHC strength is significantly affected by molding quality. The effect of process condition change can be explained by introducing the design of experiments and the analysis of variance.

1 Introduction

In recent years, advanced composite materials have been widely used in aerospace structures since their specific strength and stiffness are higher than those of currently used aerospace metals. The application can therefore result in weight saving, hence an improved fuel consumption rate and composites also promise lower maintenance cost. However, the production cost of composites structures is higher than that of metal ones. Therefore, low-cost processing methods, such as Vacuum assisted Resin Transfer Molding (VaRTM), have attracted attention [1]. This molding process reduces manufacturing cost because it does not require expensive equipment and materials, such as an autoclave and prepregs. On the other hand, it does have some drawbacks, strength decrease, quality loss and dimensional instability as a result of the low pressure applied during the process. In the past, several studies have reported the effect of process parameters on the strength properties and molding quality [2], [3]. Focus attention on resin, in order to ensure moldability of VaRTM, low viscosity in low temperature during resin infusion is required. Generally, it is difficult to achieve a good balance between low viscosity of resin and compression after impact (CAI) strength required in aerospace application. In this study, we evaluate process that inserting thermoplastic veil as a way of improvement in CAI strength without effect on moldability of VARTM. To change the molding process based on design of experiments, effect of the molding process on molding quality and strength property is statistically evaluated. From these results, method of to determine the optimum molding process is proposed.

2 Experimental

2.1 Material and cure process

In VaRTM the carbon fiber preforms are impregnated with liquid resin using only vacuum (Fig.1). The CFRP laminates are composed of quasi-isotropic non crimp fabric (NCF) preforms that are stitched with thermoplastic fibers, and epoxy resin. The applied carbon fibers are IMS 60 (Toho Tenax) and the used epoxy resin is XNR/HNR 6809 (Nagase ChemteX Co., Ltd.).

All dry preform were treated with hot compaction before resin impregnation. Table 1 shows the focused processing conditions, and Table 2 shows the selected manufacturing conditions based on design of experiments. In the case of the heating at 80ºC, the laminates have been exposed to 80ºC for 2 hours. The ones heated at 180ºC were exposed the temperature for 15 minutes. In the case of included thermoplastic veils, these were inserted in between the carbon fiber NCFs before hot compaction process.

The resin temperature was kept at 40°C and vacuumed for degassing from 30 to 60 minutes before resin impregnation. During a resin impregnation process, perform temperature also was kept at 40°C. Then the resin was impregnated with the preform by vacuuming at -98kPa. After resin impregnation, the preform was cured at 80°C for 16 hours as first cure, and then temperature was risen up to 120°C and kept at 2 hours for as post cure. Dimensions of molded products are 330mm (width) × 330mm (length) × 3.9~6.0mm (thickness).

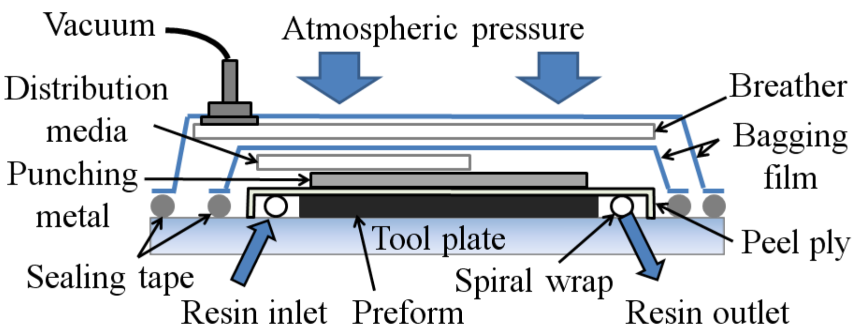


Fig 1. Schematic of VaRTM process

Factor＼Level	1	2	3
a: Thermoplastic veil [g/m^2]	N/A	7.5	19
b: Hot compaction temperature and time	80[°C], 2[h]	180[°C], 15[min]	
c: Hot compaction pressure [atm]	1(atmosphere)	+20	

Table 1. Focused factors and levels

No.	a: Thermoplastic veil[g/m^2]	Hot compaction b: Temperature and time	Hot compaction c: pressure[atm]
A	N/A	80[°C], 2[h]	1
B	N/A	180[°C], 15[min]	+20
C	N/A	80[°C], 2[h]	+20
D	N/A	180[°C], 15[min]	1
E	7.5	80[°C], 2[h]	+20
F	7.5	180[°C], 15[min]	1
G	19	80[°]C, 2[h]	1
H	19	180[°C], 15[min]	+20

Table 2. Process conditions selected by design of experiments

2.2 Measurement of molding quality and strength property

Molding quality was assessed by cross-sectional observation using optical microscope, and measurement of the fiber volume fraction (V_f) by JIS (Japanese Industry Standard) K 7075 which is combustion method (This method is compatible with ASTM D 3171.). The specimen dimensions of these tests are 8mm (width) × 8mm (length). The compression strength was determined by doing Non-hole compression (NHC) (ASTM D 6641) and CAI (ASTM D 7136 and 7137) tests. The specimen dimensions of NHC test are 12mm (width) × 140 (length) and CAI test are 100mm (width) × 150mm (length). For the CAI test, a drop-weight impact machine was used to damage the specimens (Fig.2); the impact energy was 6.7 J/mm. Extent of the applied damage was evaluated using non-destructive inspection (NDI) technique which is ultrasonic testing. Compression machine used in NHC and CAI tests was the Instron 4482 and 5589; crosshead speed was 1.00 mm/min. The number of specimen is n=4, n=6, and n=3 for V_f, NHC, and CAI test, respectively.

Fig 2. Drop-weight impact machine

2.3 Analysis of variance

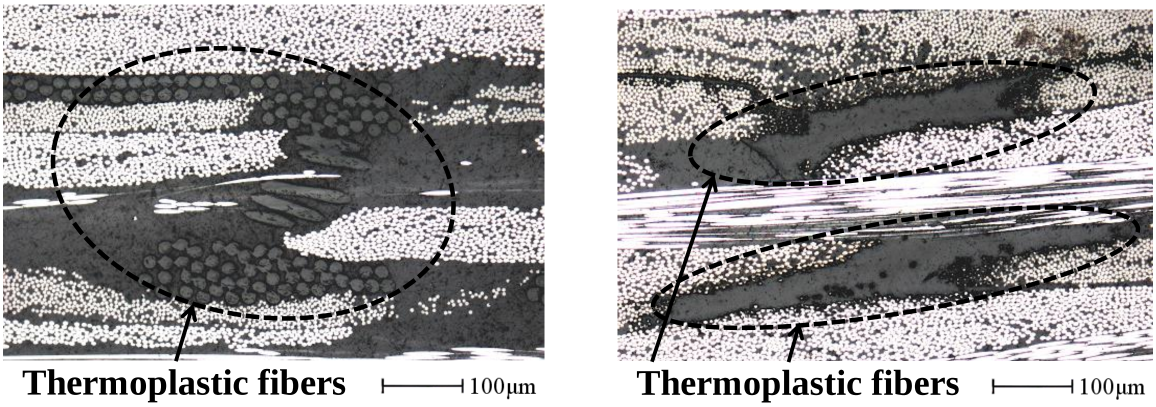
It is intended that significance of effect of focused factor, varied manufacturing process, on molding quality and strength property are determined; the results of each test were tested by F-test which is one of the statistical hypothesis testing. F-test can be obtained F-value and p-value. F-value is determined greatness of effect of focused factor. P-value is determined significance of effect of focused factor. As a result of F-test, if p-value is less than 0.05, the null hypothesis is rejected. This study focused on effect of the thermoplastic veil and hot compaction conditions. The detailed description of analysis of variance is in [4]

3 Results and discussion

3.1 Molding quality

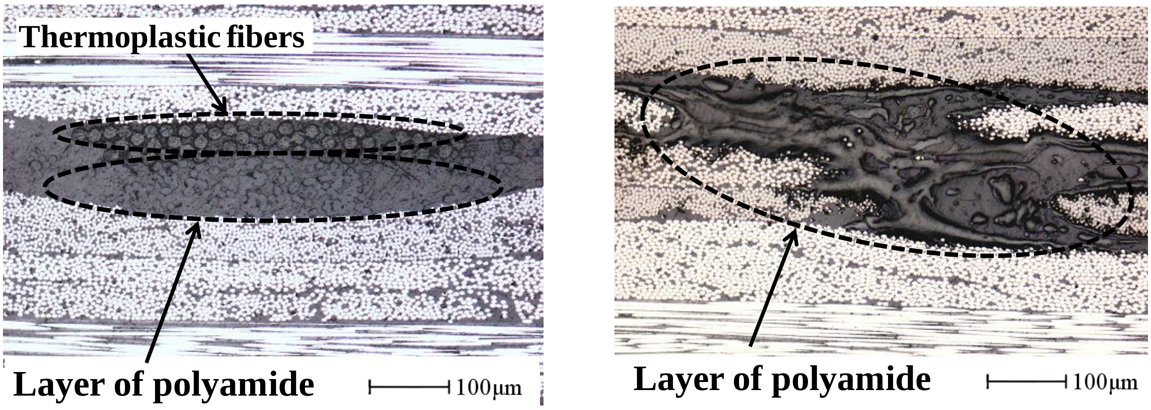
3.1.1 Cross-sectional observation

Typical results of cross-sectional observation results are shown in Fig 3 and 4. Fig 3 (a) and (b) shows process condition A and B, Fig 4 (a) and (b) shows process condition G and H.



(a) Process condition A (N/A, 80°C, 1atm) (b) Process condition B (N/A, 180°C, +20atm)

Fig 3. Cross-sectional observation results of Laminate without inserted the thermoplastic veil



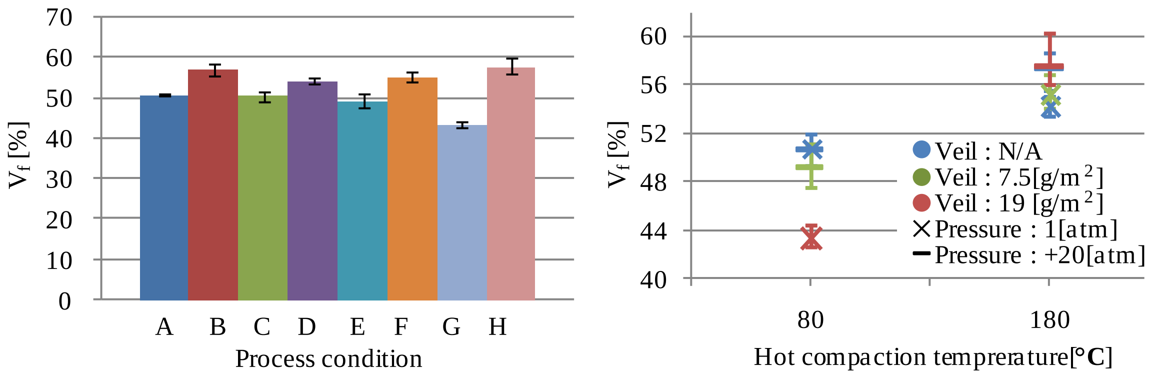
(a) Process condition G (19g/m^2, 80°C, 1atm) (b) Process condition H (19g/m^2, 80°C, +20atm)

Fig 4. Cross-sectional observation results of Laminate with inserted the thermoplastic veil (19g/m^2)

In Fig3, it was confirmed that thermoplastic fiber of NCF preform is melted by heating at 180°C but it remain as form of fiber by heating at 80°C. Fig 4 shows that thermoplastic veil is also melt and impregnate into the layer by heating at 180°C but it remain as form of veil by heating at 80°C. Glass-transition temperature (T_g) of thermoplastic veil is about 50°C, melting point of it is 176°C, and melting point of thermoplastic fiber is 110~120°C. Therefore, in the case of heating at 180°C, fiber of the NCF preform was kept densely by the thermoplastic veil and the thermoplastic fiber adhered to the NCF preforms. Although temperature of the post cure was higher than melting point of the thermoplastic fiber, thermoplastic fiber did not adhere. Epoxy resin completely loses fluidity during the first cure; thermoplastic fiber melts but cure without impregnation.

3.1.2 Measurement of V_f

Fig.5 shows the result of measurements of V_f. It is found that high V_f is achieved by hot compaction with high temperature. On the other hand the laminates including thermoplastic veil and heated at 80°C, the thermoplastic veil caused the reduction in V_f. From the cross-sectional observation, it was confirmed that thermoplastic veil and the thermoplastic fiber melt by heating at 180°C. This caused the NCF preforms are kept densely and high V_f is achieved. In the case of heating at 80°C, the thermoplastic veil did not melt and remained as form of veil between the NCF preforms. Therefore, it is obvious that this cause low V_f.

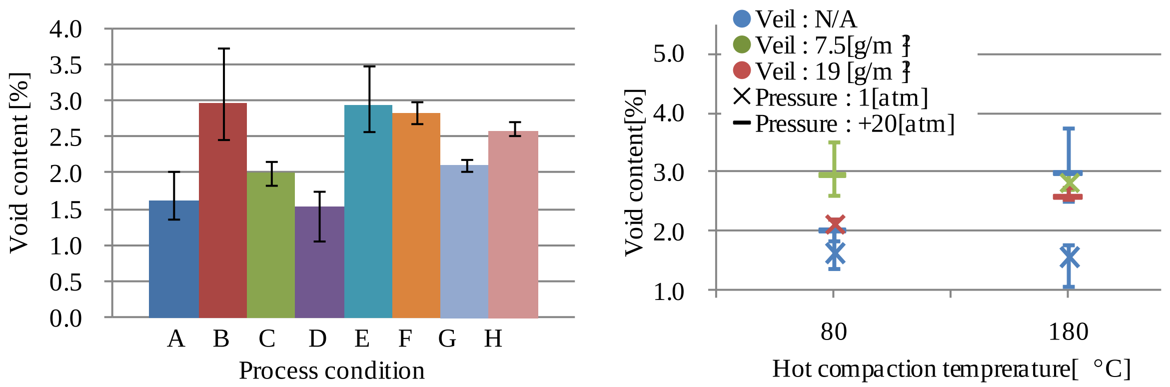


(a) Comparison of measured V_f (b) Relationship between V_f and temperature

Fig 5. Results of measurement of V_f

3.1.3 Measurement of void content

Void content of each process conditions are shown in Fig 6. It was confirmed that void content is increased by inserting the thermoplastic veil and pressing during hot compaction. The thermoplastic veil could possibly have decreased elimination route of void during resin impregnation. In addition, it is considered that high pressure process makes closer gap between carbon fibers and it traps smaller bubbles compared to not press case. However, void content was indirectly measures by using combustion method. In this method, void content is calculated by subtracting V_f and resin contents from total volume of composite; its include the content of thermoplastic veil inclusion. Thus, for detail discussion for void content, more precise measurement such as acid solution process should be performed.



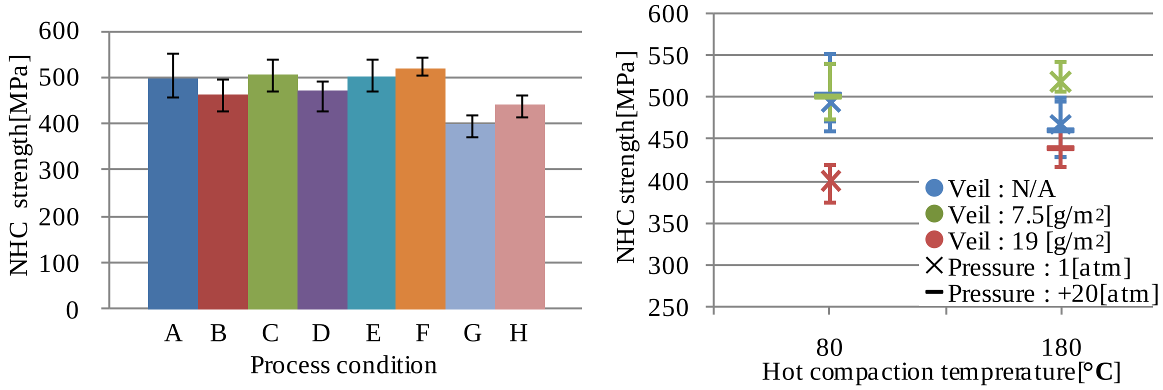
(a) Comparison of measured void content (b) Relationship between void content and temperature

Fig 6. Results of void content

3.2 Strength property

3.2.1 NHC strength

Fig 7 shows results of NHC strength. In Fig 7, it can be observed that NHC strength is increased with the addition of the thermoplastic veil whose weight per unit area is 7.5 [g/m^2] in both process condition E and F. On the other hand, NHC strength is decreased with the addition of the thermoplastic veil whose weight per unit area is 19 [g/m^2] in both process condition G and H. However, these results were not concluded that the conditions of hot compaction correlate with NHC strength. From this result, it is suggested that melting of the thermoplastic veil and the thermoplastic fiber have no direct effect on NHC strength.



(a) Comparison of NHC strength (b) Relationship between NHC strength and temperature

Fig 7. Results of NHC strength

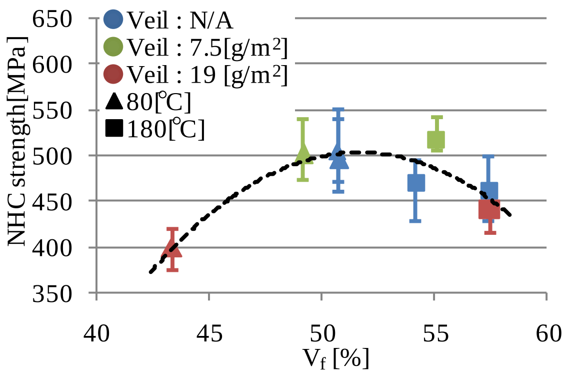
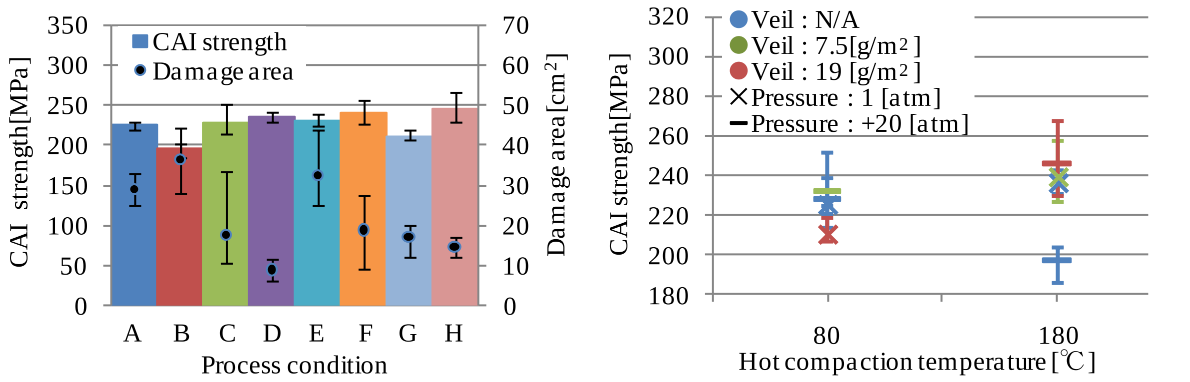


Fig 8. Relationship between V_f and NHC strength

Fig 8 shows relationship between NHC strength and V_f. It was confirmed that NHC strength has strong relationship with V_f. This result suggests the possibility that the highest NHC strength is obtained by V_f which is about 52%. Our previous study showed statistically that VaRTM CFRP has optimum value of V_f in NHC strength [5]. The trend shown in Fig 8 supports the findings of our previous study. Consequently, Data from NHC tests lead to the conclusion that NHC strength can be controlled by molding quality. However, it remains to be explained the fundamental reason that the reduction in NHC strength caused the increase in V_f. Future work is needed to determine the reason.

3.2.2 CAI strength

Fig 9 shows the obtained CAI strength and damage area. It was confirmed that the laminate heated at 80°C without the thermoplastic veil is not affected by pressure. On the other hand, the laminates including the thermoplastic veil and heated at 180°C is increased CAI strength. In the case of the laminate including low area density thermoplastic veil, the effect of the thermoplastic veil is smaller than high area density thermoplastic veil. Focus attention on the laminates heated at 180°C without the thermoplastic veil, pressing preform caused a reduction in CAI strength. The laminate is low fraction of resin by high V_f. Therefore, the increase in damage area caused the reduction in CAI strength.



(a) Comparison of CAI strength (b) Relationship between CAI strength and temperature

Fig 9. Results of CAI strength

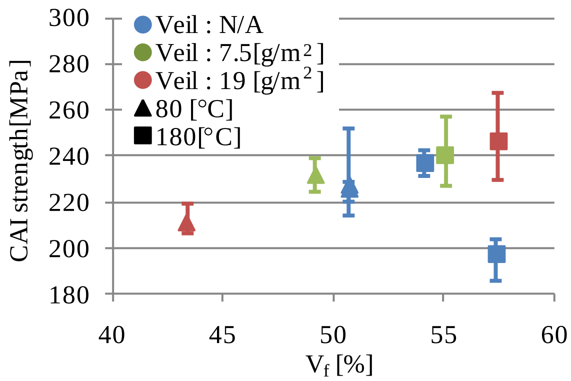


Fig 10. Relationship between V_f and CAI strength

Fig 10 shows relationship between CAI strength and V_f. As V_f increases, CAI strength also increases. However, large difference occurred in the area which is V_f over 55%. In the area, CAI strength is increased by the melted thermoplastic veil. Interlaminar toughness and crack extension resistance is increased by the melted thermoplastic veil. This caused the reduction in damage area inside laminate and the increased in CAI strength of the laminate which is high V_f.

3.3 Analysis of variance

In order to evaluate statistically an effect of varied process condition on molding quality and strength property, F-test performed. The results of F-test are shown in Table3.

Factor	V_f (n=4)		Void (n=4)	
	F	p	F	p
a :Thermoplastic veil	**5.405**	<0.05	**12.768**	<0.05
b : Temperature	**113.970**	<0.05	**5.082**	<0.05
c : Pressure	**16.412**	<0.05	**18.383**	<0.05

(a) Molding quality

Factor	NHC Strength (n=6)		CAI Strength (n=3)	
	F	p	F	p
a: Thermoplastic veil	**30.781**	<0.05	1.074	0.361
b : Temperature	0.130	0.720	0.599	0.449
c : Pressure	0.557	0.459	0.117	0.736

(b) Strength property

Table 3. Results of F-test

Focus attention on V_f and void content, F-value of all focused factors was more than 1.0 and p-value was less than 0.05. This results show that all focused factors have an influence. In particular, it was showed that hot compaction temperature was the main influencing factor of V_f. Consequently, these results explained that the molding quality can be controlled by inserting the thermoplastic veil and selecting the appropriate hot compaction conditions.
Focus attention on NHC strength, F-value of the thermoplastic veil was more than 1.0 and p-value was less than 0.05; the null hypothesis was rejected, i.e., presence or absence of the thermoplastic veil is the influence factor on NHC strength. However, NHC strength correlated with molding quality in Fig 8. It was shown statistically that NHC strength is affected by the thermoplastic veil, but it is also possible that NHC strength is not directly affected by the thermoplastic veil. In case of CAI strength, the null hypothesis of all focused factors was not rejected. However, it was confirmed the increase in CAI strength by fusion of thermoplastic veil. It is necessary to evaluate the effect of melting of the thermoplastic veil by F-test considered interaction.
From these results, it was revealed that strength property cannot be controlled directly by process conditions but can be controlled indirectly by the adjustment of molding quality which can be determined with process condition by using design of experiments.

4 Conclusions

The effect of the process conditions on the molding quality and the strength property has been made clear by determining the fiber volume fraction and performing NHC and CAI strength tests.
Required molding quality is achieved by control of process condition. The CAI test clearly shows the increase in strength as a result of the fusion of thermoplastic veil.
To analyze the effect of process condition on molding quality and strength property, design of experiments and analysis of variance are introduced. Consequently, it was concluded that the effect of process conditions on CFRP can be quantitatively evaluated. It was made it possible to assess the optimum process conditions.

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