CHAPTER 3

RESULT

3.1 Results of part 1

3.1.1 Tensile-shear bond strength

The tensile-shear bond strength values (in MPa), obtained in part 1, are displayed in table 2 and fig. 13. Two-way ANOVA revealed that the tensile-shear bond strength between the post and composite core-build up material was significantly influenced by the type of post, type of plasma treatment, and their interaction ($p<0.01$) (table 3). Tukey's test revealed significant differences between the control group and the other plasma treatment groups. Besides, a significant difference in the tensile-shear bond strength was detected between the O_2 and the other plasma treatment groups. For the FRC posts, 1-way ANOVA revealed that all of the plasma treatments significantly influenced the tensile-shear bond strength when compared with the control group ($p<0.05$). For the DT posts, only the Ar, N_2 and He+N_2 plasma treatments significantly influenced the tensile-shear bond strength when compared with the control group ($p<0.05$).

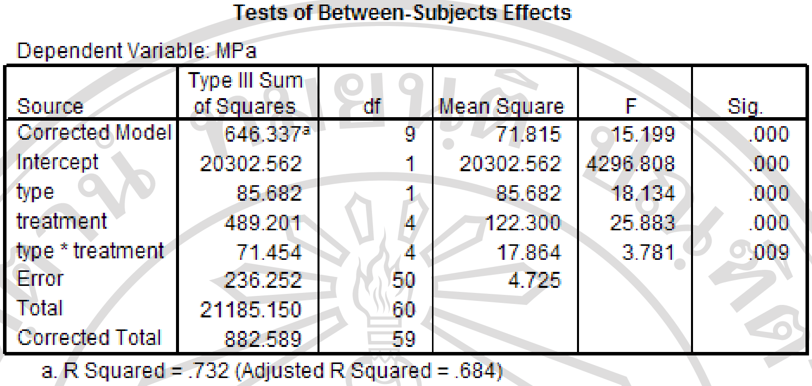
Table 2 Means (MPa) ± s.d. of tensile-shear bond strength for all groups.

Type of post	Treatment				
	Control	O_2	Ar	N_2	He+N_2
FRC	13.85±0.6^a	19.33±2.9^b	20.32±2.6^b	21.73±3.2^b	22.80±2.6^b
DT	13.90±1.3^c	13.67±1.8^c	19.75±2.0^d	17.60±2.4^d	21.08±1.2^d

Different in alphabet denotes significant differences at $p<0.05$

Amongst all the plasma treatment groups, the He + N_2 plasma treatment yield the highest tensile-shear bond strength for both types of posts. Consequently, this plasma was used in part 2.

Table 3 Two-way ANOVA revealed p-value<0.01 for the type of the post (type), the type of plasma treatment (treatment), and their interactions (type*treatment)



Tests of Between-Subjects Effects

Dependent Variable: MPa

Source	Type III Sum of Squares	df	Mean Square	F	Sig.
Corrected Model	646.337[a]	9	71.815	15.199	.000
Intercept	20302.562	1	20302.562	4296.808	.000
type	85.682	1	85.682	18.134	.000
treatment	489.201	4	122.300	25.883	.000
type * treatment	71.454	4	17.864	3.781	.009
Error	236.252	50	4.725		
Total	21185.150	60			
Corrected Total	882.589	59			

a. R Squared = .732 (Adjusted R Squared = .684)

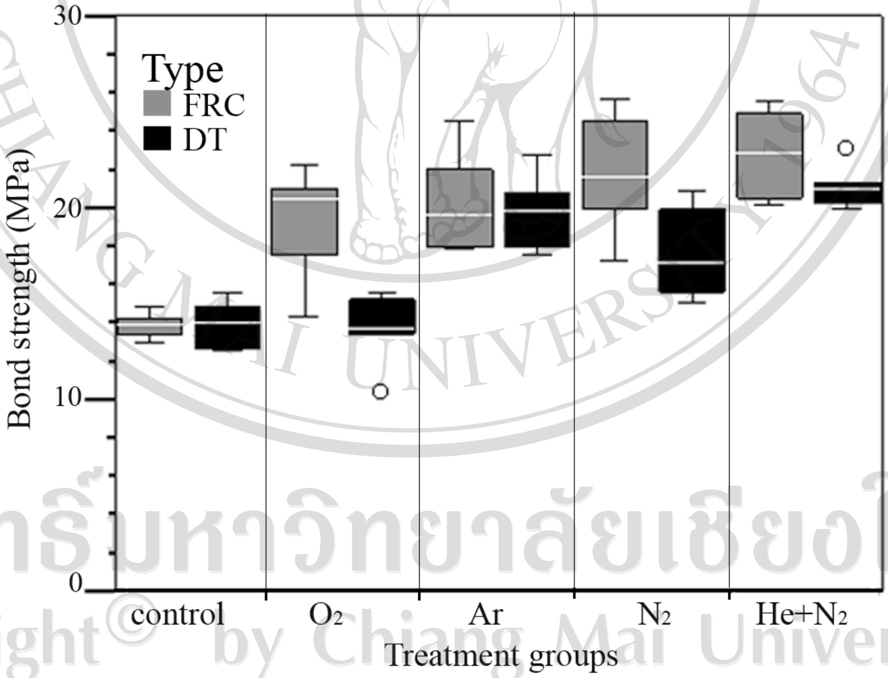


Figure 13 Tensile-shear bond strengths (in MPa) for all groups displayed in a box and whisker plot.

3.1.2 Surface roughness

Table 4 shows the surface roughness results of the FRC and DT posts in all treatment groups. Two-way ANOVA revealed that the average surface roughness on the surface of the post was significantly influenced by the type of post and type of plasma treatment, but not by their interaction. Among all the treatment groups, Tukey's test revealed significant differences in surface roughness between the control and the Ar treatment groups and between the O_2 and Ar treatment groups. For the FRC posts, 1-way ANOVA revealed no significant differences in surface roughness between any of the groups. However, for the DT posts, there was a significant difference between the O_2 and the Ar treatment groups.

Table 4 Means (μm) ± s.d. of surface roughness calculated for all the treatment groups.

Type of post	Treatment				
	Control	O_2	Ar	N_2	He+N_2
FRC	0.83 ± 0.06^{a}	1.04 ± 0.10^{a}	1.23 ± 0.16^{a}	1.17 ± 0.12^{a}	0.96 ± 0.06^{a}
DT	0.73 ± 0.30^{b}	0.70 ± 0.26^{b}	1.14 ± 0.15^{c}	$0.93 \pm 0.15^{b,c}$	$0.85 \pm 0.32^{b,c}$

Different alphabet denotes significant differences at $p<0.05$

3.1.3 Light microscopic analysis

Light microscopic images of the debonded specimens after the pull-out test demonstrated that mixed adhesive/cohesive failure at the composite core build-up material/post occurred in both types of posts (fig. 14).

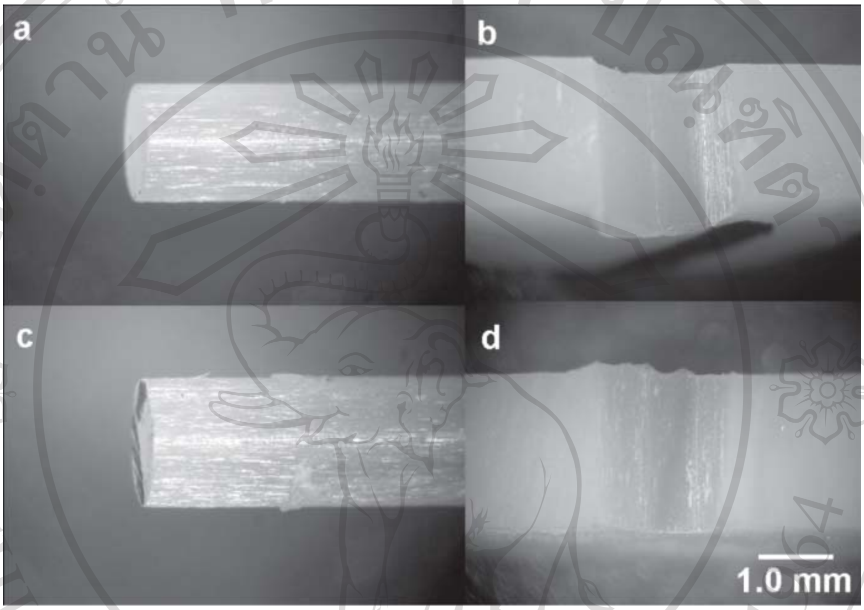


Figure 14 Light microscopic image of the debonded surfaces of FRC and DT posts: (a, b) debonded surfaces of FRC Postec: (c, d) debonded surface of DT Light-Post.

3.2 Results of part 2

3.2.1 Step 1 Gas pressure

The tensile-shear bond strength values (MPa), obtained in this step, were displayed in table 5 and fig. 15a, b. For both FRC and DT post, 1-way ANOVA revealed that the tensile-shear bond strength between the post and composite core build-up material was not significantly influenced by the gas pressure ($p<0.05$).

Table 5 Means (MPa) ± s.d. of tensile-shear bond strength calculated for all gas pressure groups for FRC and DT post.

Type of post Gas pressure (Pa)	FRC	DT
2.7	21.55±3.41	20.97±2.70
6.7	19.97±2.86	19.60±3.60
13.3	21.00±3.23	20.73±4.64
26.7	22.80±2.25	20.95±1.36
40.0	21.47±3.19	20.27±1.79

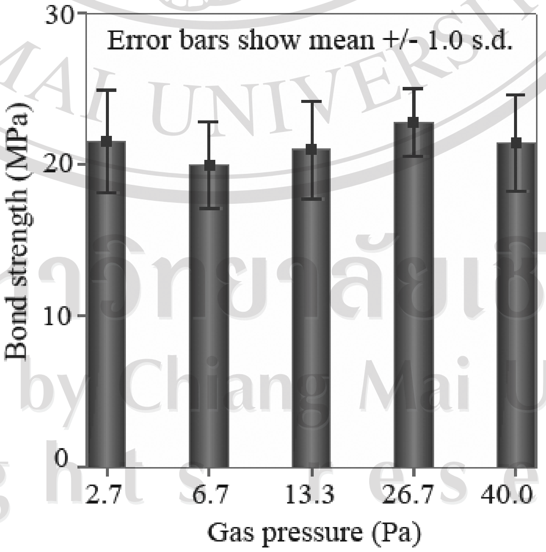


Figure 15a Tensile-shear bond strengths (MPa) for all gas pressure groups for FRC post.

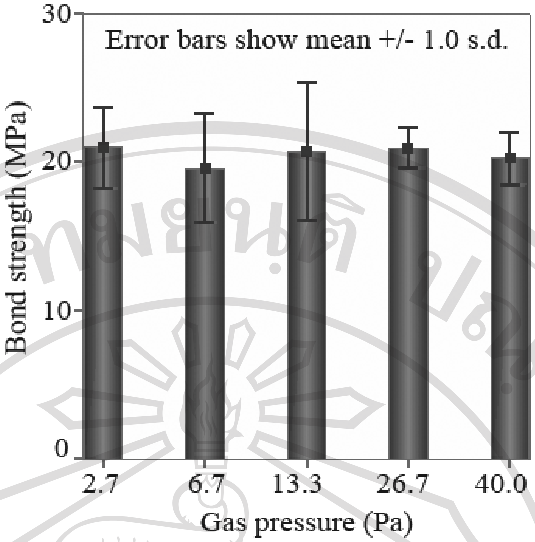


Figure 15b Tensile-shear bond strengths (MPa) for all gas pressure groups for DT post.

Although there was no significant difference in tensile-shear bond strength between each gas pressure group, the gas pressure of 26.7 Pa was selected because of their highest tensile-shear bond strength between the FRCPs and the composite core build-up material and more reliable results when compared with the other groups.

3.2.2 Step 2 Discharge power

The tensile-shear bond strength values (MPa), obtained in this step, were displayed in table 6 and fig. 16a, b. For both types of posts, 1-way ANOVA revealed that the tensile-shear bond strength between the post and composite core build-up material was not significantly influenced by discharge power ($p<0.05$).

Table 6 Means (MPa) ± s.d. of tensile-shear bond strength calculated for all discharge power groups.

Type of post / Discharge power (W)	FRC	DT
25	22.67±3.40	19.77±3.29
50	22.80±2.25	21.08±1.16
75	25.02±4.52	19.22±2.10

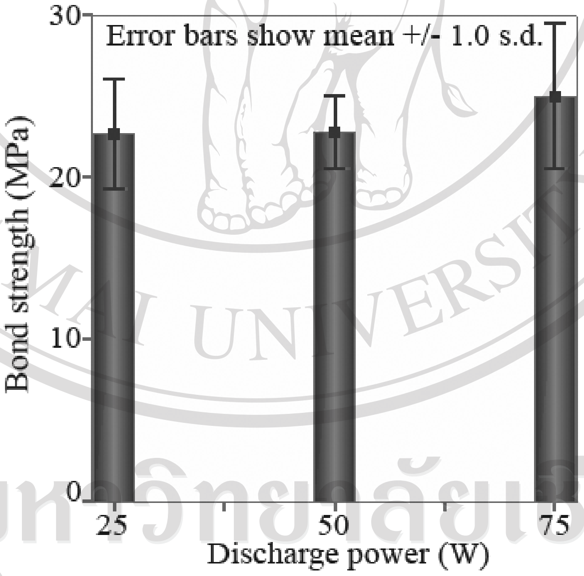


Figure 16a Tensile-shear bond strengths (MPa) for all discharge power groups for FRC post.

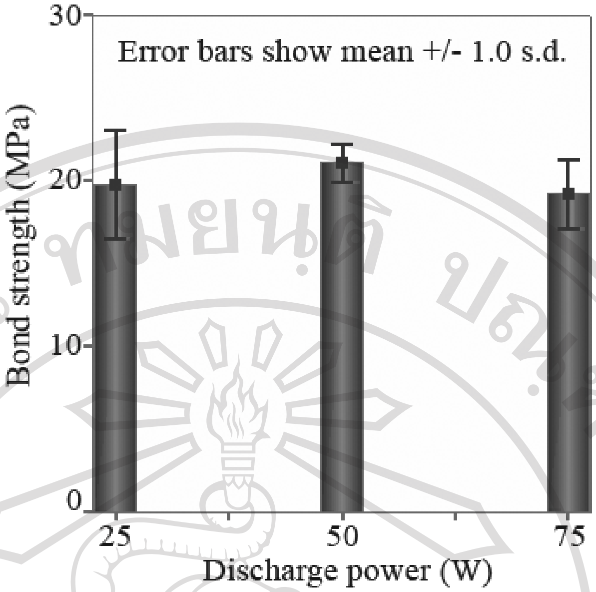


Figure 16b Tensile-shear bond strengths (MPa) for all discharge power groups for DT post.

The discharge power at 75 W and 50 W were selected for the FRC posts and the DT posts respectively because of their highest tensile-shear bond strength and the level of ion energy was suitable for the chemical structure of each type of post that will be discussed later.

3.2.3 Step 3 Plasma treatment time

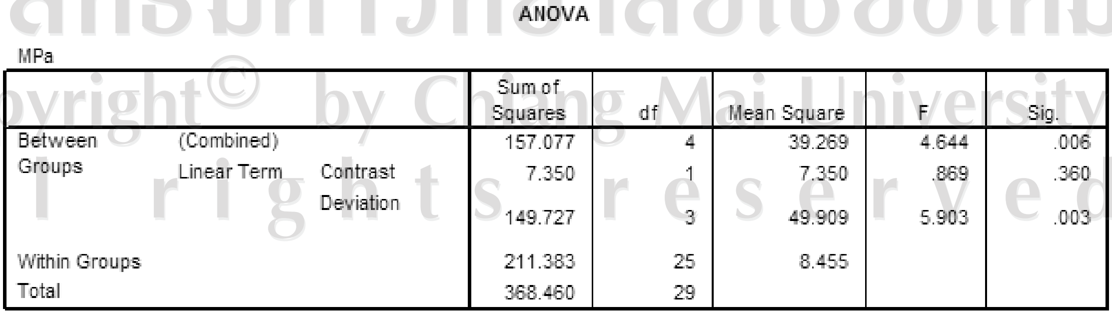
Table 7 and fig. 17a, b showed the tensile-shear bond strength of the FRC and DT posts in different plasma treatment time. For both types of posts, 1-way ANOVA revealed that the tensile-shear bond strength was significantly influenced by plasma treatment time ($p<0.05$) (table 8 for FRC post, table 9 for DT post). For the FRC posts, Tukey's test revealed significant differences between the treatment time of 10 minutes and the other groups except for the treatment time of 15 minutes. For the DT posts, Tukey's test revealed significant differences between the treatment time of 15 minutes and the other groups except for the treatment time of 30 minutes.

Table 7 Means (MPa) ± s.d. of tensile-shear bond strength calculated for all the treatment time groups.

Type of post Treatment time (minute)	FRC	DT
3	23.48±3.08^{a}	21.42±3.51^{1}
5	23.10±3.45^{a}	26.60±2.09^{2}
10	29.37±3.36^{b}	28.35±2.60^{2}
15	25.53±1.40^{a,b}	32.68±1.37^{3}
30	24.02±2.88^{a}	31.80±2.47^{2,3}

Different in alphabet and number denote significant differences at $p<0.05$

Table 8 One-way ANOVA revealed p-value<0.01 for FRC post in plasma treatment time groups.

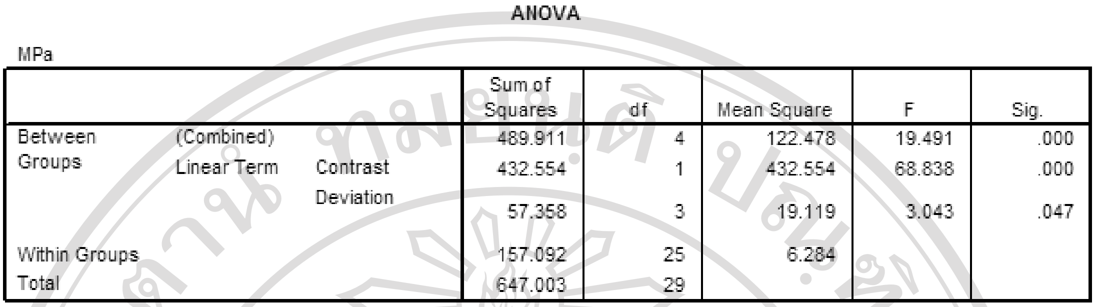


ANOVA

MPa

			Sum of Squares	df	Mean Square	F	Sig.
Between Groups	(Combined)		157.077	4	39.269	4.644	.006
	Linear Term	Contrast	7.350	1	7.350	.869	.360
		Deviation	149.727	3	49.909	5.903	.003
Within Groups			211.383	25	8.455		
Total			368.460	29			

Table 9 One-way ANOVA revealed p-value<0.001 for DT post in plasma treatment time groups.



ANOVA

MPa

			Sum of Squares	df	Mean Square	F	Sig.
Between Groups	(Combined)		489.911	4	122.478	19.491	.000
	Linear Term	Contrast	432.554	1	432.554	68.838	.000
		Deviation	57.358	3	19.119	3.043	.047
Within Groups			157.092	25	6.284		
Total			647.003	29			

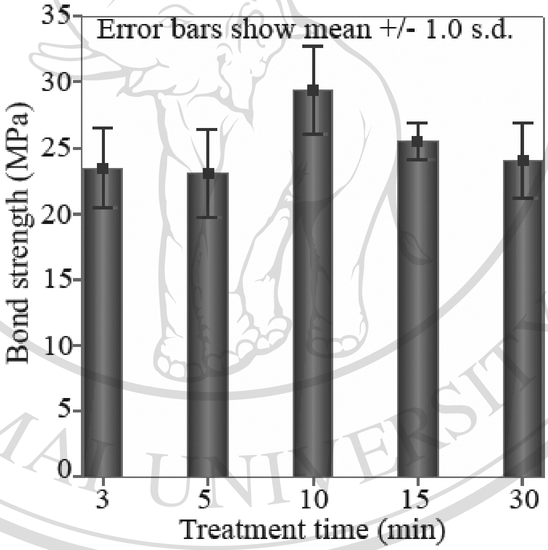


Figure 17a Tensile shear bond strength (MPa) for all the treatment time groups for the FRC posts.

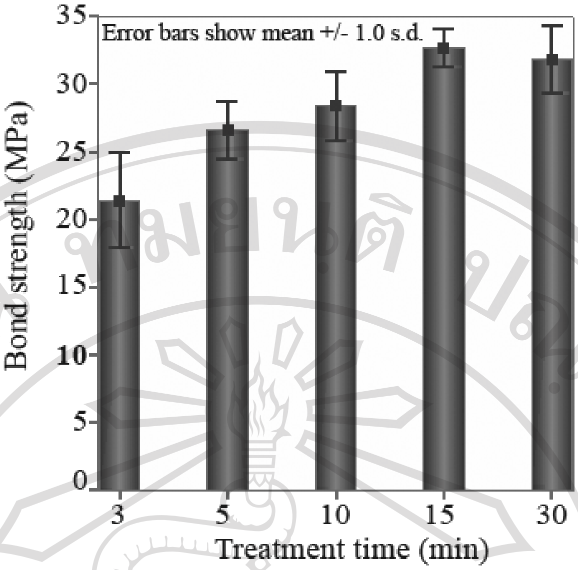


Figure 17b Tensile shear bond strengths (MPa) for all the treatment time groups for the DT posts.

Based on the results obtained in this study, it could be concluded that the most suitable parameters for He+N_2 plasma treatment, including the gas pressure, the discharge power, and the treatment time were 26.7 Pa, 75 W, 10 minutes and 26.7 Pa, 50 W, 15 minutes for the FRC posts and the DT posts respectively.

3.3 Results of Fourier transform infrared spectroscopy (FTIR) for chemical analysis

Fig. 18a, b show the FTIR spectra of the untreated and treated FRC and DT posts respectively. Fig. 19a, b revealed the EDX spectra for FRC and DT post. For the FRC posts, there is no obvious difference between the spectra of the untreated post and the plasma-treated posts. The possible reason is that nitrogen is one of the chemical components of the FRC post (UDMA and TGDMA components) and the new expected functional group on the plasma-treated post may also contain terminal nitrogen, thereby the FTIR spectra of them were not different. However, EDX has shown the increasing of nitrogen element from 1.88 to 6.60 wt% after the post was treated by plasma. With the small increasing in nitrogen, the differences of FTIR spectra between both of them may not be detected. However, FTIR spectra have shown that both of them contained C-N bond (amines component) at the peak around 1000-1350 cm^{-1} (a in fig. 18a). For the DT posts, the chemical component of the polymer matrix is epoxy resin that is absolutely absence of nitrogen, so the spectra of plasma treated post showed the new peak of nitrogen bond. Peaks around 1000-1350 cm^{-1}, 1550 cm^{-1}, and 3100-3500 cm^{-1} (a, b, and c in fig. 18b) can be assigned as C-N, N=O (nitro component), and N-H (primary and secondary amines and amides, stretch type) respectively. In addition, EDX of plasma treated-DT post also revealed an increasing of nitrogen (from 0 to 3.76 wt%). Based on these results, the induced functional group on the FRC and the DT posts that performed by He+N_2 plasma treatment can be assumed as nitrogen functional group.

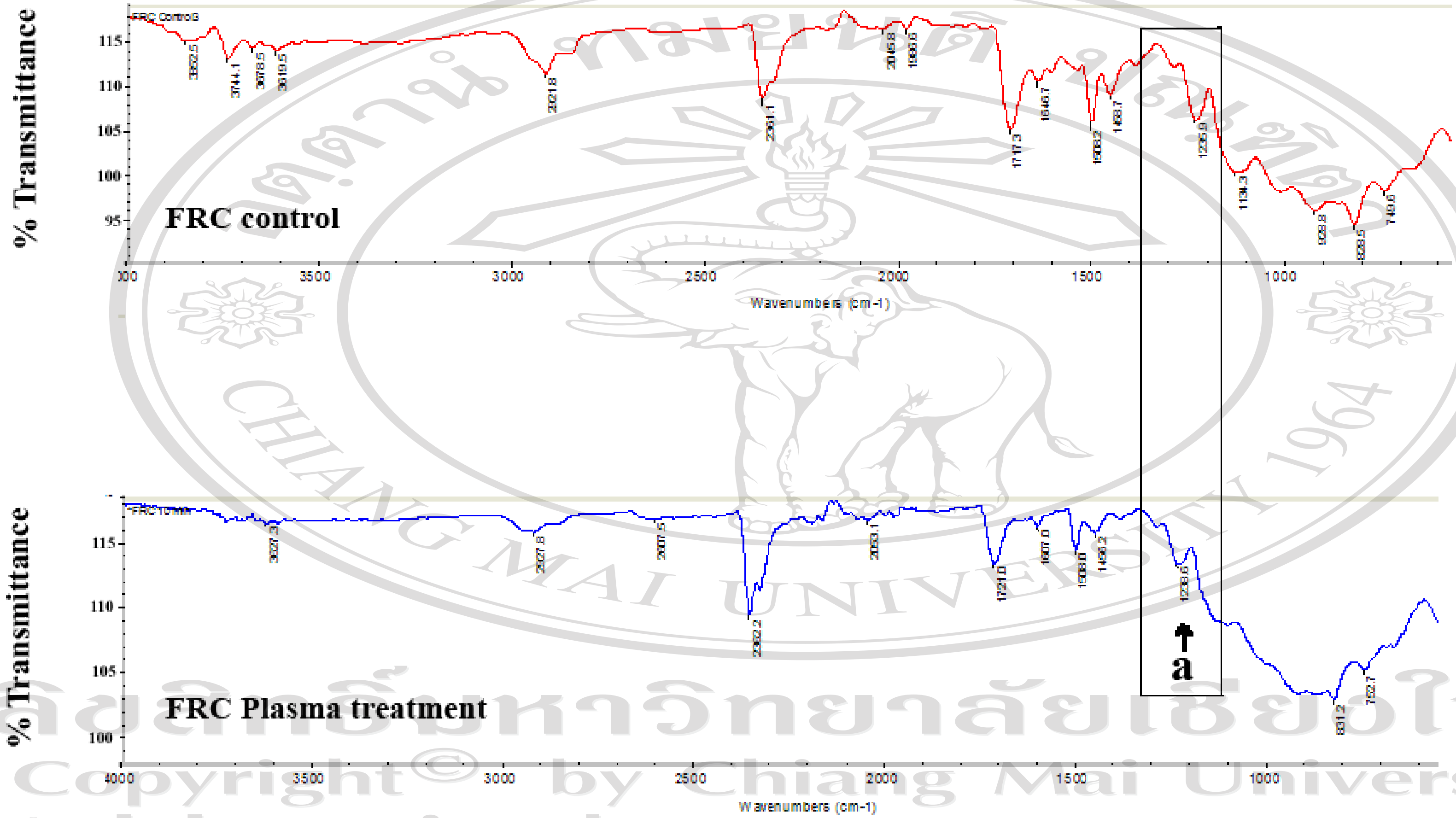


Figure 18a FTIR spectra of the untreated post (above) and the plasma-treated post (below) of the FRC post. No significant different in spectra was found, peak around 1000-1350 cm^{-1} (a) can be assigned as C-N bond.

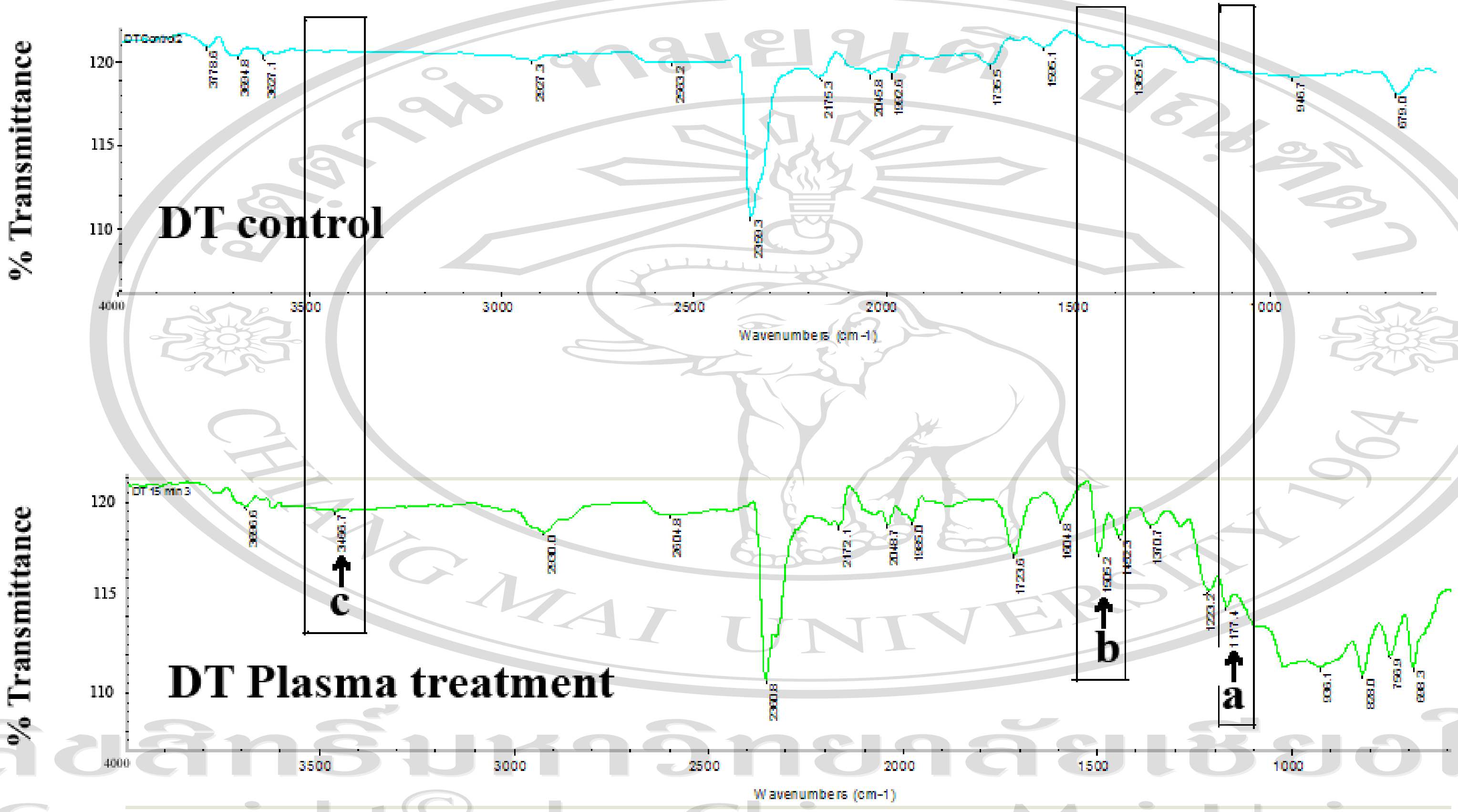
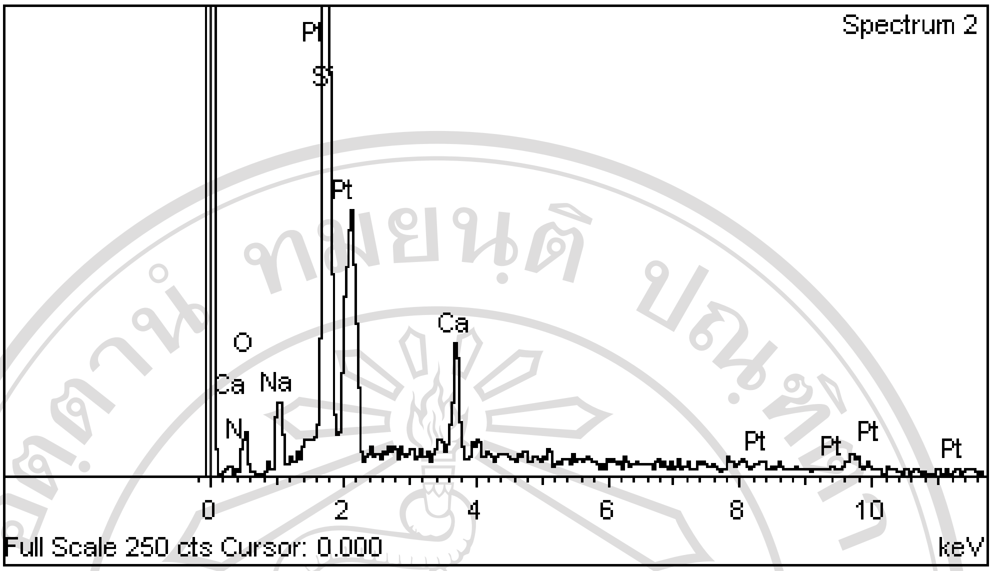
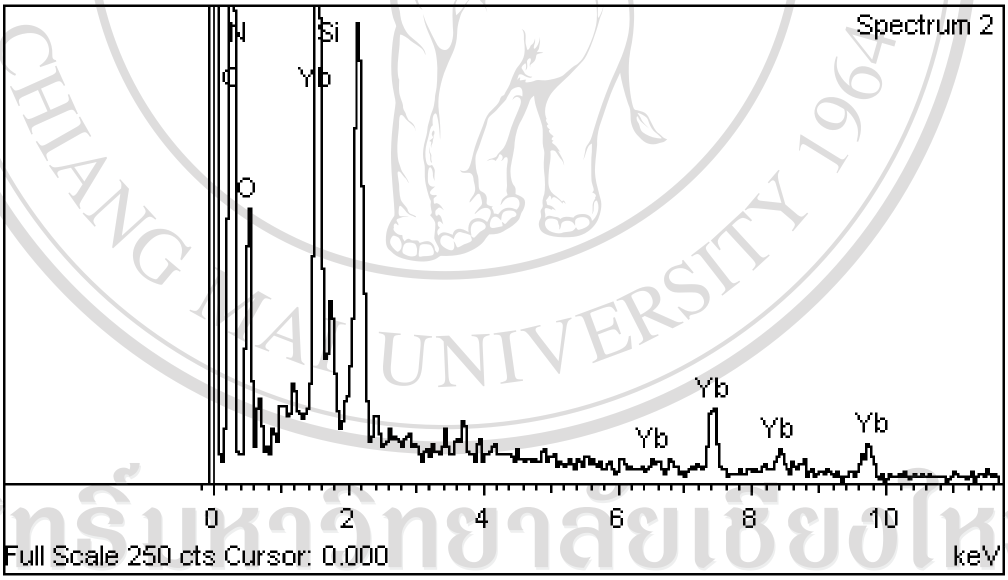


Figure 18b FTIR spectra of the untreated post (above) and the plasma treated post (below) of the DT posts. The new peaks appeared around 1000-1350 cm^{-1} (a), 1550 cm^{-1} (b), and 3100-3500 cm^{-1} (c) can be assigned as C-N, N=O, and N-H bonds respectively.

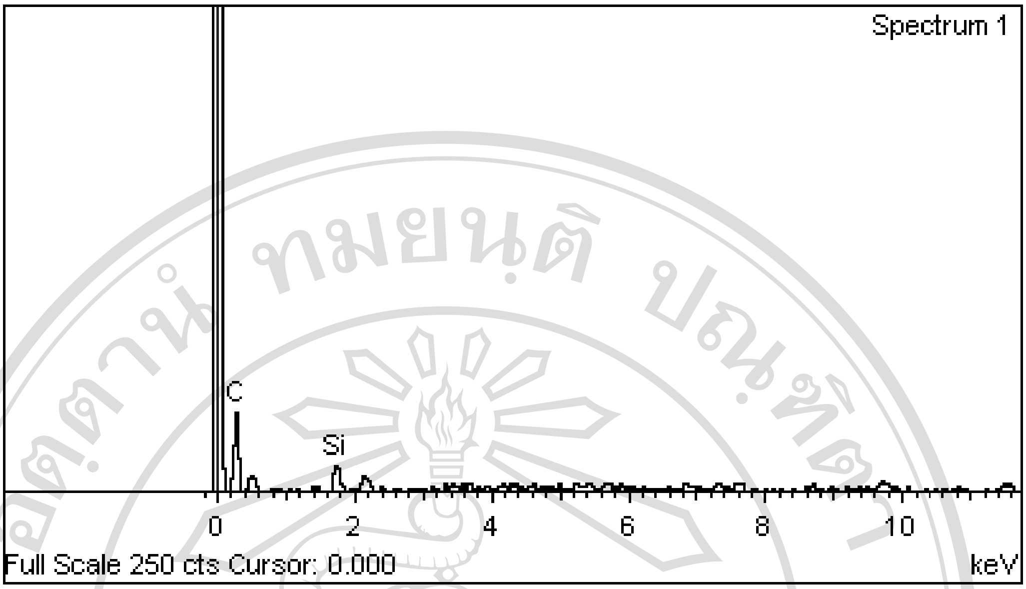


EDX spectra of untreated FRC post

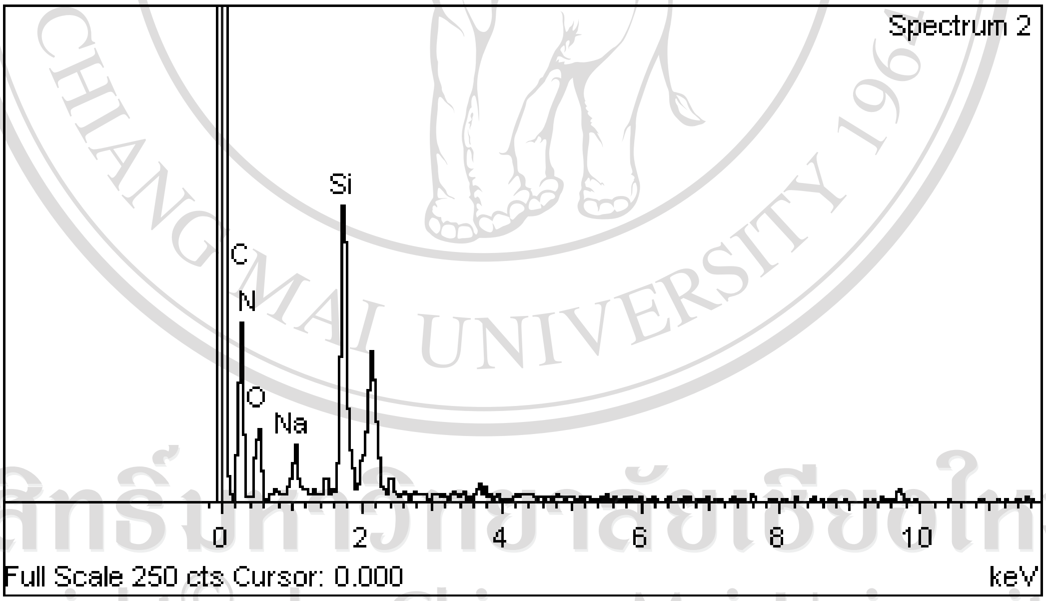


EDX spectra of plasma treated FRC post

Figure 19a EDX spectra of the untreated and plasma-treated FRC posts.



EDX spectra of untreated DT post



EDX spectra of plasma treated DT post

Figure 19b EDX spectra of the untreated and plasma-treated DT posts.

3.4 Results of part 3

3.4.1 Results of division 1: Effect of plasma treatment time and hydrothermal storage condition

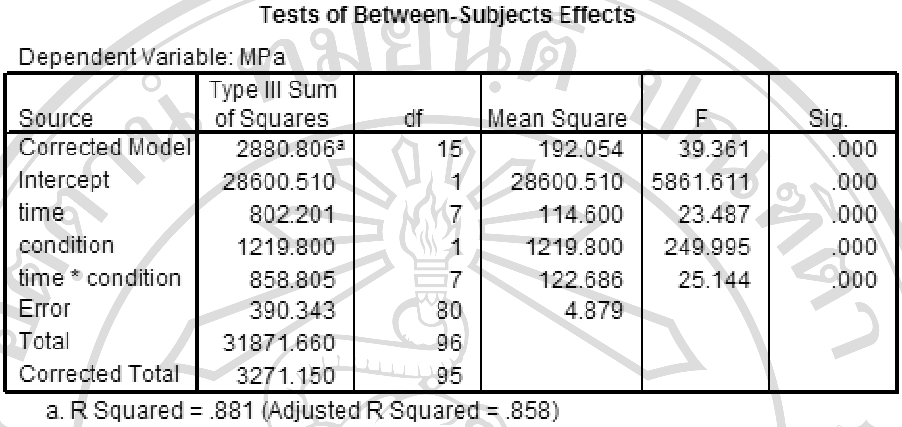
The tensile-shear bond strength values (MPa) of all plasma treatment time groups in different storage conditions (room temperature and 37 °C deionized water for 7 days) for FRC posts were displayed in table 10 and fig. 20. Two-way ANOVA revealed that the tensile-shear bond strength was significantly influenced by the treatment time, the storage condition, and their interaction ($p<0.05$) (table 11). Tukey's test revealed significant different in tensile-shear bond strength between the two conditions at the treatment time 3, 5, 10, 15, and 30 minutes ($p<0.05$). In water storage condition at 37 ^{o}C, there were no significant differences in tensile-shear bond strength among all the treatment time groups.

Table 10 Means (MPa) ± s.d. of the tensile-shear bond strength calculated for all treatment time groups in both storage conditions for the FRC posts.

Storage condition Treatment time	Room temp. (25 ^{o}C)	In water 37 ^{o}C
Control	13.85 ± 0.63^{a}	14.03 ± 3.33^{a}
Plasma 30 sec	13.62 ± 1.90^{a}	13.17 ± 1.97^{a}
Plasma 1 min	13.63 ± 1.65^{a}	13.85 ± 1.60^{a}
Plasma 3 min	23.48 ± 3.08^{b}	14.95 ± 1.56^{a}
Plasma 5 min	23.10 ± 3.35^{b}	14.75 ± 2.08^{a}
Plasma 10 min	29.37 ± 3.36^{c}	13.58 ± 1.40^{a}
Plasma 15 min	$25.53 \pm 1.40^{b,c}$	11.75 ± 8.83^{a}
Plasma 30 min	24.02 ± 2.90^{b}	13.48 ± 1.43^{a}

Different alphabet denotes significant differences at $p<0.05$

Table 11 Two-way ANOVA revealed p-value<0.001 for plasma treatment time (time), storage condition (condition), and their interaction (time*condition) for FRC posts.



Tests of Between-Subjects Effects

Dependent Variable: MPa

Source	Type III Sum of Squares	df	Mean Square	F	Sig.
Corrected Model	2880.806ᵃ	15	192.054	39.361	.000
Intercept	28600.510	1	28600.510	5861.611	.000
time	802.201	7	114.600	23.487	.000
condition	1219.800	1	1219.800	249.995	.000
time * condition	858.805	7	122.686	25.144	.000
Error	390.343	80	4.879		
Total	31871.660	96			
Corrected Total	3271.150	95			

a. R Squared = .881 (Adjusted R Squared = .858)

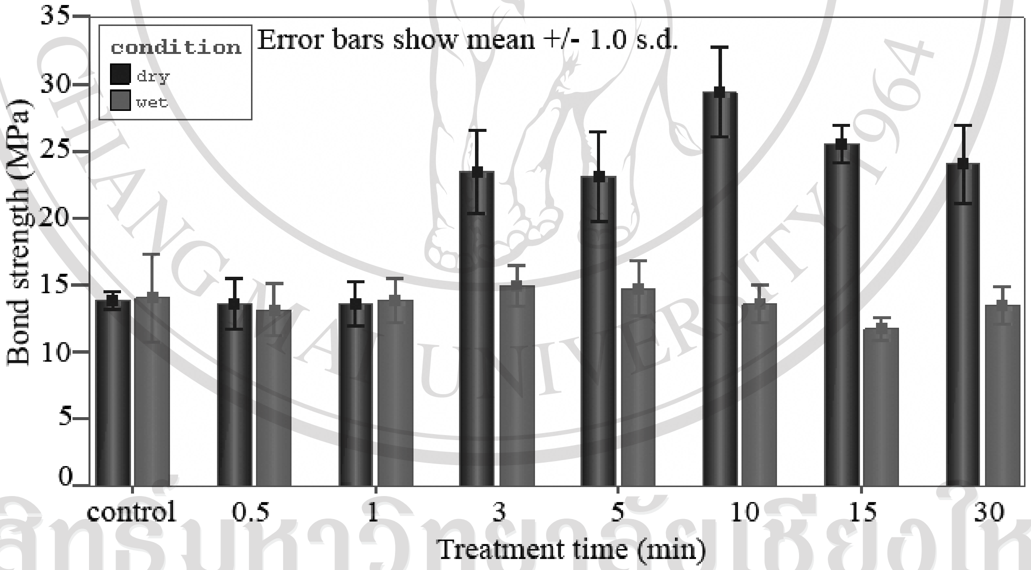


Figure 20 Tensile-shear bond strengths (MPa) for all treatment time groups in both storage conditions for the FRC posts.

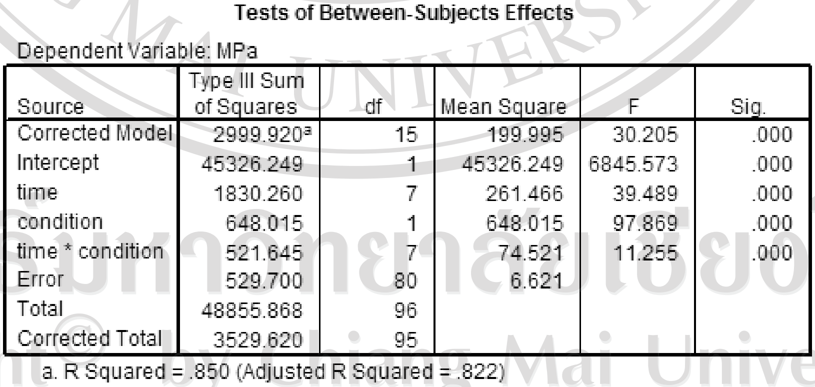
Table 12 and fig. 21 displayed the tensile-shear bond strength values (MPa) of all plasma treatment time groups for DT post. Two-way ANOVA revealed that the tensile-shear bond strength was significantly influenced by the treatment time, the storage condition, and their interaction (p<0.05) (table 13). Tukey's test revealed significant different in tensile-shear bond strength among the two storage conditions at the treatment time 5, 10, 15, and 30 minutes (p<0.05). There were significant differences in tensile-shear bond strength between the control group and the treatment time groups at 30 seconds, 1, 3, 10, and 15 minutes which storage in 37 °C deionized water.

Table 12 Means (MPa) ± s.d. of the tensile-shear bond strength calculated for all treatment time groups in both storage conditions for the DT posts.

Storage condition Treatment time	Room temp. (25 ^{o}C)	In water 37 ^{o}C
Control	13.90±1.26^{a}	12.01±1.54^{a}
Plasma 30 sec	19.93±1.23^{b}	19.06±3.34^{b}
Plasma 1 min	19.93±2.10^{b}	19.62±3.37^{b,e}
Plasma 3 min	21.42±3.51^{b}	19.83±3.33^{b,e}
Plasma 5 min	26.60±2.09^{c}	16.90±3.52^{a,b}
Plasma 10 min	28.35±2.60^{c,d}	21.78±2.03^{b,e}
Plasma 15 min	32.68±1.37^{d}	24.25±1.00^{b,e}
Plasma 30 min	31.80±2.47^{c,d}	17.05±2.7^{a,b}

Different alphabet denotes significant differences at $p<0.05$

Table 13 Two-way ANOVA revealed p-value<0.001 for plasma treatment time (time), storage condition (condition), and their interaction (time*condition) for DT post.



Tests of Between-Subjects Effects

Dependent Variable: MPa

Source	Type III Sum of Squares	df	Mean Square	F	Sig.
Corrected Model	2999.920^{a}	15	199.995	30.205	.000
Intercept	45326.249	1	45326.249	6845.573	.000
time	1830.260	7	261.466	39.489	.000
condition	648.015	1	648.015	97.869	.000
time * condition	521.645	7	74.521	11.255	.000
Error	529.700	80	6.621		
Total	48855.868	96			
Corrected Total	3529.620	95			

a. R Squared = .850 (Adjusted R Squared = .822)

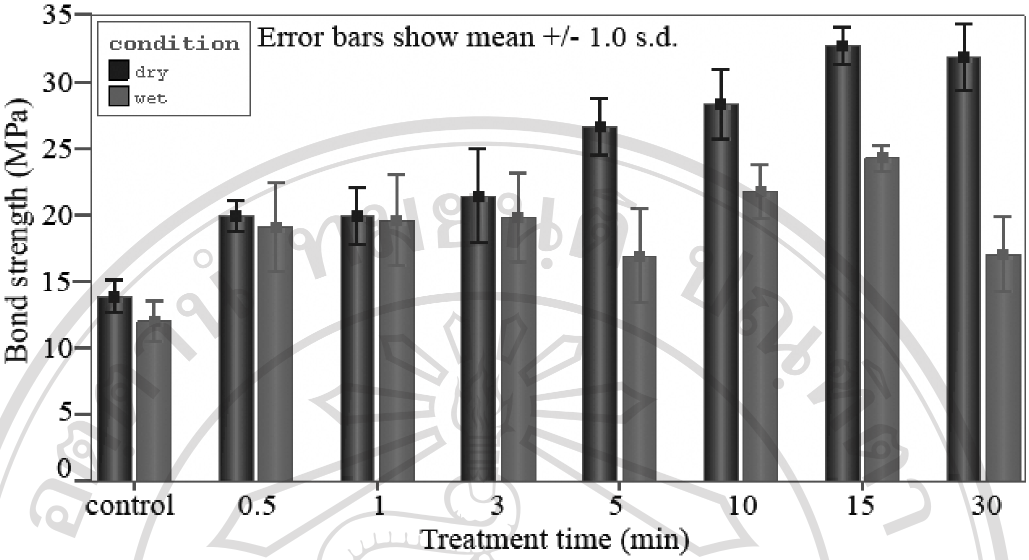


Figure 21 Tensile-shear bond strengths (MPa) for all treatment time groups in both storage conditions for the DT posts.

3.4.2 Results of division 2: Main factor of hydrothermal condition

In this division, the results were divided into 2 sections.

3.4.2.1 Result of section 1: Thermal effect.

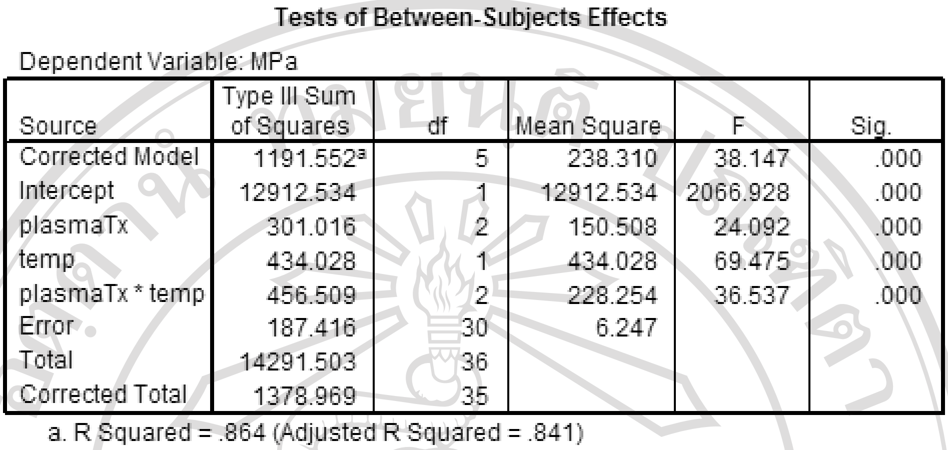
The tensile-shear bond strength values (MPa) for the control and treatment groups of FRC post which storage in 37 °C dry storage condition were displayed in table 14 and fig. 22. Two-way ANOVA revealed that the tensile-shear bond strength was significantly influenced by the plasma treatment, the storage temperature, and their interaction ($p<0.05$) (table 15). Tukey's test revealed no significant different between control group and plasma treatment time 10 and 30 minutes in dry storage condition. However, there were significant differences between plasma treatment time 10 and 30 minutes that storage in room temperature and that storage in 37 ^{o}C dry condition.

Table 14 Means (MPa) ± s.d. of the tensile-shear bond strength calculated for the control and all treatment groups for the FRC posts in section 1.

Plasma treatment / Storage condition	non	10 min	30 min
Room temp (25 ^{o}C)	13.85±0.63^{a}	29.37±3.36^{b}	24.02±2.88^{c}
In chamber 37 ^{o}C	16.58±2.24^{a}	15.17±2.76^{a}	14.65±2.21^{a}
In water 37 ^{o}C	14.03±3.33^{a}	13.58±1.40^{a}	13.48±1.43^{a}

Different alphabet denotes significant differences at $p<0.05$

Table 15 Two-way ANOVA revealed p-value<0.001 for temperature (temp), plasma treatment (plasma Tx), and their interaction (plasmaTx* temp) for the FRC posts.



Tests of Between-Subjects Effects

Dependent Variable: MPa

Source	Type III Sum of Squares	df	Mean Square	F	Sig.
Corrected Model	1191.552[a]	5	238.310	38.147	.000
Intercept	12912.534	1	12912.534	2066.928	.000
plasmaTx	301.016	2	150.508	24.092	.000
temp	434.028	1	434.028	69.475	.000
plasmaTx * temp	456.509	2	228.254	36.537	.000
Error	187.416	30	6.247		
Total	14291.503	36			
Corrected Total	1378.969	35			

a. R Squared = .864 (Adjusted R Squared = .841)

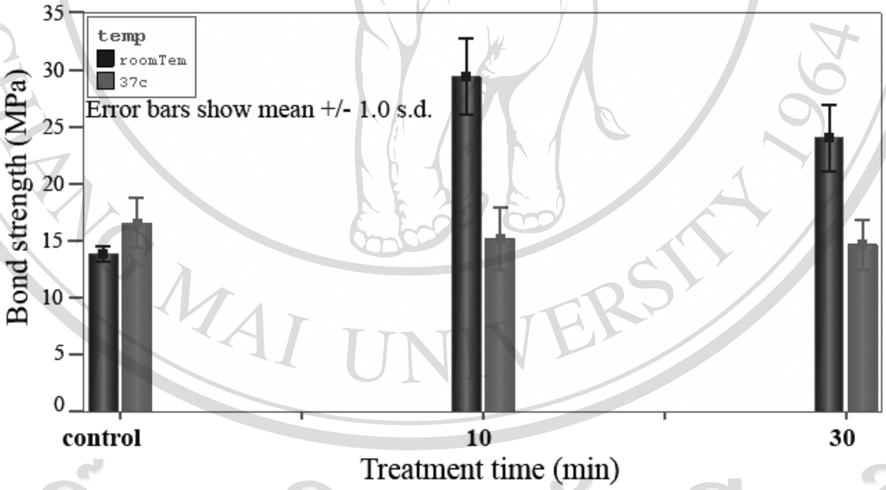


Figure 22 Tensile-shear bond strengths (MPa) for the control and all treatment groups for the FRC posts in section 1.

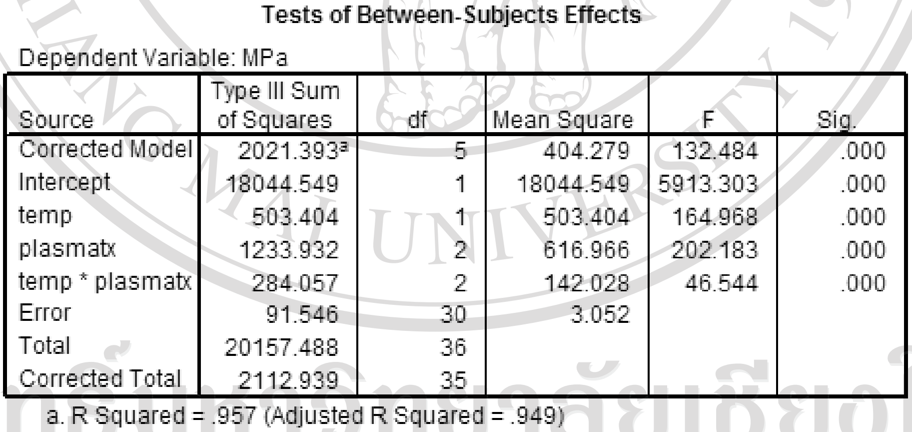
Table 16 and fig. 23 displayed the tensile-shear bond strength values (MPa) for the control and all treatment groups for DT post in section 1. Two-way ANOVA revealed that the tensile-shear bond strength was significantly influenced by the plasma treatment, the storage temperature, and their interaction (p<0.05) (Table 17). Tukey’s test revealed significant different between the plasma treatment time 15 and 30 minutes that storage in room temperature and that storage in 37 °C dry condition.

Table 16 Means (MPa) ± s.d. of the tensile-shear bond strength calculated for the control and all treatment groups of the DT posts in section 1.

Plasma treatment Storage condition	non	15 min	30 min
Room temp (25 °C)	13.90 ± 1.26^{ad}	32.68 ± 1.37^{b}	31.80 ± 2.47^{b}
In chamber 37 °C	14.57 ± 2.41^{ad}	21.42 ± 1.89^{c}	20.17 ± 0.95^{c}
In water 37 °C	12.01 ± 1.54^{a}	24.25 ± 1.00^{c}	17.05 ± 2.79^{d}

Different alphabet denotes significant differences at p<0.05

Table 17 Two-way ANOVA revealed p-value<0.001 for temperature (temp), plasma treatment (plasmatx), and their interaction (temp*plasmatx) for the DT posts.



Tests of Between-Subjects Effects

Dependent Variable: MPa

Source	Type III Sum of Squares	df	Mean Square	F	Sig.
Corrected Model	2021.393[a]	5	404.279	132.484	.000
Intercept	18044.549	1	18044.549	5913.303	.000
temp	503.404	1	503.404	164.968	.000
plasmatx	1233.932	2	616.966	202.183	.000
temp * plasmatx	284.057	2	142.028	46.544	.000
Error	91.546	30	3.052		
Total	20157.488	36			
Corrected Total	2112.939	35			

a. R Squared = .957 (Adjusted R Squared = .949)

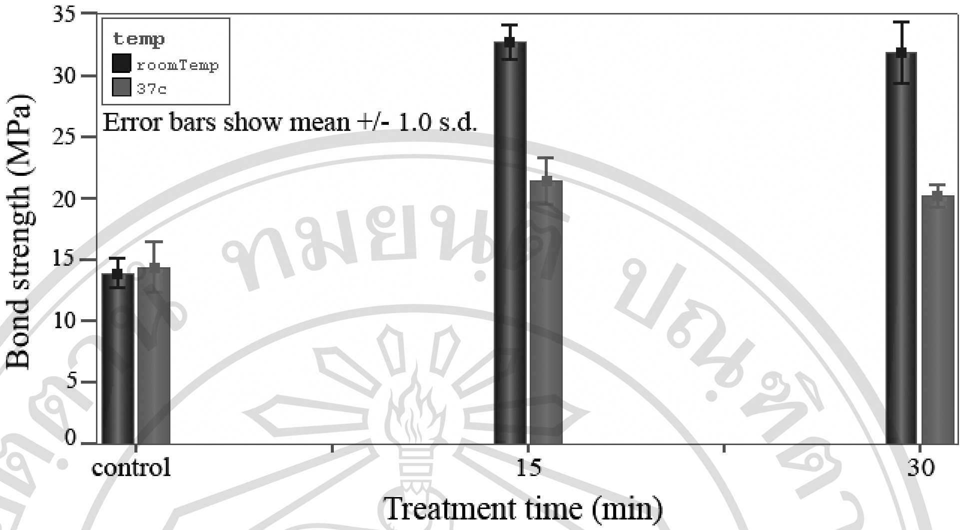


Figure 23 Tensile-shear bond strengths (MPa) for the control and all treatment groups for the DT posts in section 1.

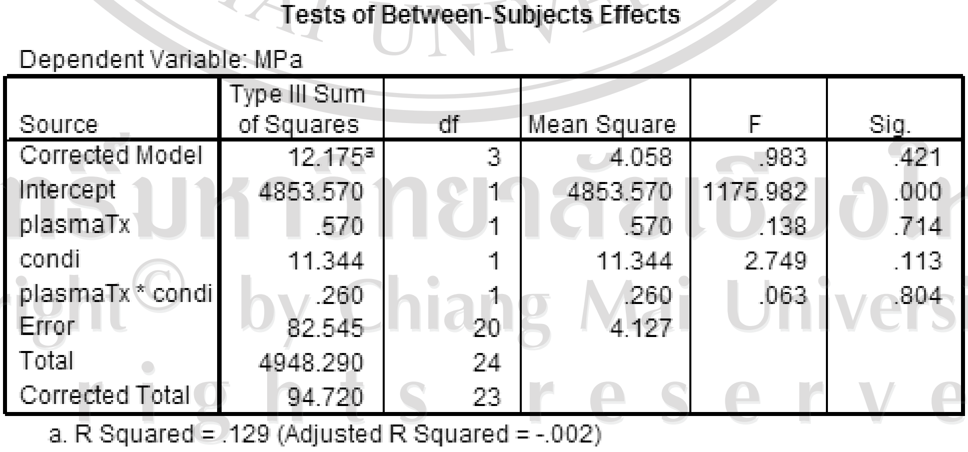
3.4.2.2 Result of section 2: Hydration effect.

The tensile-shear bond strength values (MPa) for all plasma treatment groups in different storage condition (dry and wet storage conditions) for FRC post were displayed in table 18 and fig. 24. Two-way ANOVA revealed that the tensile-shear bond strength was not significantly influenced by the plasma treatment, the storage condition, and their interaction ($p<0.05$) (table 19).

Table 18 Means (MPa) ± s.d. of the tensile-shear bond strength for plasma treatment time 10 and 30 minutes in 37 °C dry and 37 °C wet storage conditions for the FRC posts in section 2.

Plasma treatment Storage condition	10 min	30 min
Dry (37 ^{o}C)	15.17 ±2.76	14.65± 2.21
Wet (37 ^{o}C)	13.58 ±1.40	13.48 ±1.43

Table 19 Two-way ANOVA reveal p-value>0.05 for plasma treatment (plasma Tx), storage condition (condi), and their interaction (plasmaTx*condi) for the FRC posts in section 2.



Tests of Between-Subjects Effects

Dependent Variable: MPa

Source	Type III Sum of Squares	df	Mean Square	F	Sig.
Corrected Model	12.175[a]	3	4.058	.983	.421
Intercept	4853.570	1	4853.570	1175.982	.000
plasmaTx	.570	1	.570	.138	.714
condi	11.344	1	11.344	2.749	.113
plasmaTx * condi	.260	1	.260	.063	.804
Error	82.545	20	4.127		
Total	4948.290	24			
Corrected Total	94.720	23			

a. R Squared = .129 (Adjusted R Squared = -.002)

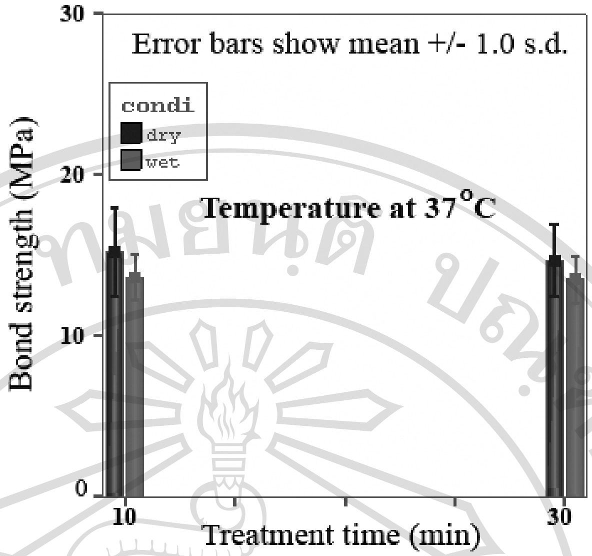


Figure 24 Tensile-shear bond strengths (MPa) for plasma treatment time 10 and 30 minutes in 37 °C dry and 37 °C wet storage conditions for the FRC posts in section 2.

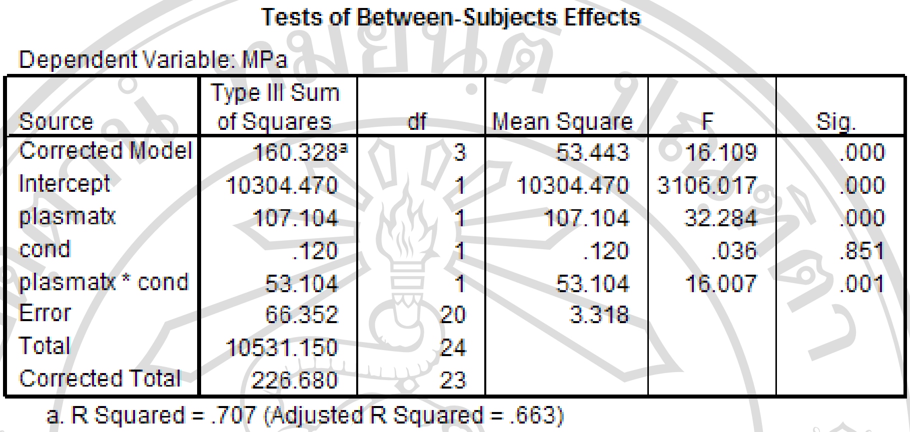
Table 20 and fig. 25 revealed the tensile-shear bond strength values (MPa) for all plasma treatment groups in different storage condition for DT post in section 2. Two-way ANOVA revealed that the tensile-shear bond strength was not significantly influenced by the storage condition ($p<0.05$), but it was significantly influenced by the plasma treatment and their interactions ($p<0.05$) (table 21). Tamhane's test revealed no significant different between the plasma treatment time 15 and 30 minutes that storage in 37 ^{o}C dry condition and that storage in 37 ^{o}C wet condition.

Table 20 Means (MPa) ± s.d. of the tensile-shear bond strength for plasma treatment time 15 and 30 minutes in 37 ^{o}C dry and 37 ^{o}C wet storage conditions for the DT posts in section 2.

Plasma treatment Storage condition	15 min	30 min
Dry (37 ^{o}C)	$21.42 \pm 1.90^{a,b}$	20.17 ± 0.95^{a}
Wet (37 ^{o}C)	24.25 ± 1.00^{b}	17.05 ± 2.79^{a}

Different alphabet denotes significant differences at $p<0.05$

Table 21 Two-way ANOVA reveal p-value>0.05 for storage condition (cond) but p-value<0.05 for plasma treatment (plasmatx) and their interaction (plasmatx*cond) for the DT posts in section 2.



Tests of Between-Subjects Effects

Dependent Variable: MPa

Source	Type III Sum of Squares	df	Mean Square	F	Sig.
Corrected Model	160.328[a]	3	53.443	16.109	.000
Intercept	10304.470	1	10304.470	3106.017	.000
plasmatx	107.104	1	107.104	32.284	.000
cond	.120	1	.120	.036	.851
plasmatx * cond	53.104	1	53.104	16.007	.001
Error	66.352	20	3.318		
Total	10531.150	24			
Corrected Total	226.680	23			

a. R Squared = .707 (Adjusted R Squared = .663)

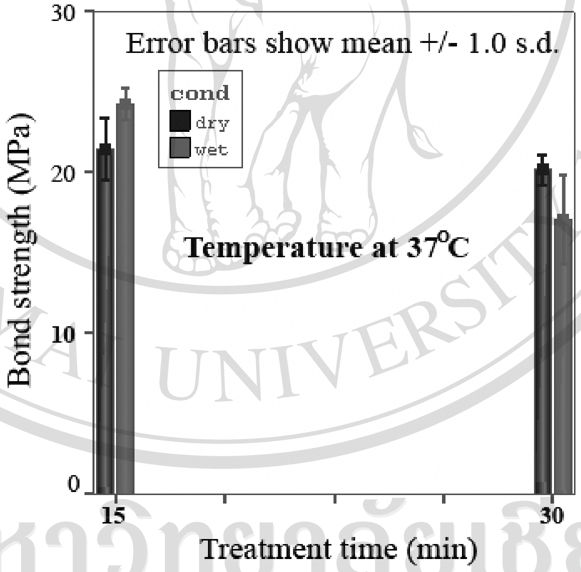


Figure 25 Tensile-shear bond strengths (MPa) for plasma treatment time 15 and 30 minutes in 37 °C dry and 37 °C wet storage conditions for the DT posts in section 2.