

Group 3: sorted from highest to lowest (SBS) (see Tables: 12 and 13.).

Adhesive Agent/Type of crown	SBS-N	SBS-MPa	Adhesive agent/Type of crown	SBS-N	SBS-MPa
R-X U/E-NT	65.3	7.3	R-X U/Z-NT	81	9
R-X U/E-NT	55.1	6.1	R-X U/Z-NT	68.2	7.6
R-X U/E-NT	52.2	5.8	R-X U/Z-NT	65.9	7.3
R-X U/E-NT	51.4	5.7	R-X U/Z-NT	58.5	6.5
R-X U/E-NT	49.4	5.5	R-X U/Z-NT	52.5	5.8
R-X U/E-NT	45.8	5.1	R-X U/Z-NT	40.8	4.5
R-X U/E-NT	45.5	5.		38.8	4.3
R-X U/E-NT	40.3	4.		34.6	3.8
R-X U/E-NT	26.6	3.		27.2	3.0
R-X U/E-NT	22.2	2.		0	0
Average/Mean	45.5	5.1	Average/Mean	51.9	5.8
Median	47.6	5.3	Median	52.5	5.8



Table: 12.

Table: 13.

Group 4: Results of the Non-Thermocycled eMax/Transbond XT adhesive and Porcelain-veneered zirconia crown/ Transbond XT adhesive combinations (see Table: 14).

#	Adhesive Agent	Type of crown	T./NT.	SBS N	SBS MPa	ARI score	PFI Score
61	Tb XT	E	NT	65.4	7.3	3	0
62	Tb XT	E	NT	172.2	19.1	0	0
63	Tb XT	E	NT	63.3	7.0	0	0
64	Tb XT	E	NT	65.7	7.3	0	0
65	Tb XT	E	NT	43.7	4.9	3	0
66	Tb XT	E	NT	68.8	7.6	3	0
67	Tb XT	E	NT	43.4	4.8	3	0
68	Tb XT	E	NT	103.9	11.5	0	0
69	Tb XT	E	NT	50.6	5.6	1	0
70	Tb XT	E	NT	50.2	5.6	0	0
71	Tb XT	Z	NT	57.3	6.4	0	0
72	Tb XT	Z	NT	52.2	5.8	2	0
73	Tb XT	Z	NT	49.6	5.5	3	0
74	Tb XT	Z	NT	52.2	5.8	3	0
75	Tb XT	Z	NT	62.5	6.9	1	0
76	Tb XT	Z	NT	54.2	6.0	0	0
77	Tb XT	Z	NT	41.2	4.6	3	0
78	Tb XT	Z	NT	72.1	8.0	3	0
79	Tb XT	Z	NT	71.9	8.0	0	0
80	Tb XT	Z	NT	59.8	6.6	3	0

Table: 14.

Group 4: sorted from highest to lowest (SBS) (see Table 15 and 16.).

Adhesive agent/Type of crown	SBS-N	SBS-MPa	Adhesive agent/Type of Crown	SBS-N	SBS-MPa
Tb XT/E-NT	172.2	19.1	Tb XT/Z-NT	72.1	8.0
Tb XT/E-NT	103.9	11.5	Tb XT/Z-NT	71.9	8.0
Tb XT/E-NT	68.8	7.6	Tb XT/Z-NT	62.5	6.9
Tb XT/E-NT	65.7	7.3	Tb XT/Z-NT	59.8	6.6
Tb XT/E-NT	65.4	7.3	Tb XT/Z-NT	57.3	6.4
Tb XT/E-NT	63.3	7.0	Tb XT/Z-NT	54.2	6.0
Tb XT/E-NT	50.6	5.6	Tb XT/Z-NT	52.2	5.8
Tb XT/E-NT	50.2	5.6	Tb XT/Z-NT	52.2	5.8
Tb XT/E-NT	43.7	4.9	Tb XT/Z-NT	49.6	5.5
Tb XT/E-NT	43.4	4.8	Tb XT/Z-NT	41.2	4.6
Average/Mean	72.7	8.1	Average/Mean	57.3	6.4
Median	64.4	7.2	Median	55.75	6.2

Table: 15.

Table: 16.

4.2. Statistical Analysis of shear bond strengths in Newtons (N) and Mega Pascals (MPa)

The following layout for IPS eMax (E) and Zirconia (Z) crowns were used.

Group A-

Thermocycled (T); IPS eMax (E) and Zirconia (Z) crowns bonded with RelyX™ Unicem 2 (R-X U)

Group B-

Thermocycled (T); IPS eMax (E) and Zirconia (Z) crowns bonded with Transbond™ XT (Tb XT)

Group C-

Non-Thermocycled (NT); IPS eMax (E) and Zirconia (Z) crowns bonded with RelyX™ Unicem (R-X U)

Group D-

Non-Thermocycled (NT); eMax (E) and Zirconia (Z) crowns bonded with Transbond™ XT (Tb XT)

This is also known as a factorial layout and the aim was to have ten replicates for each crown/adhesive treatment combination, eight combinations in total. It was analysed as a one-way analysis of variance, ANOVA.

The data in Newtons (N) which was obtained from the shear bond strength test performed with the Instron testing machine was converted into Mega Pascals using the following equation:

$$\textit{Shear bond strength (MPa)} = \textit{Shearing force (Newtons)} / \textit{Bracket base surface area (mm}^2\textit{)}$$

The orthodontic bracket used had a surface area of 9mm², as confirmed by the manufacturer.

A/C T/NT	Sample size	Average N	Median N	S.D	Average MPa	Median MPa	S.D
R-X U/ E-T	10	44.46	44.55	26.07	4.94	4.95	2.89
R-X U/ Z-T	10	29.14	20.70	19.36	3.24	2.30	2.14
TbXT/ E-T	10	46.17	43.20	22.17	5.13	4.80	2.47
TbXT/ Z-T	10	45.81	50.40	18.72	5.13	5.60	2.08
R-X U/ E-NT	10	45.54	47.70	12.94	5.09	5.30	1.43
R-X U/ Z-NT	10	51.80	52.20	17.88	5.76	5.80	2
TbXT/ E-NT	10	72.63	64.35	39.12	8.07	7.15	4.33
TbXT/ Z-NT	10	57.24	55.80	9.69	6.36	6.20	1.07

Data

Table:17:

analysed

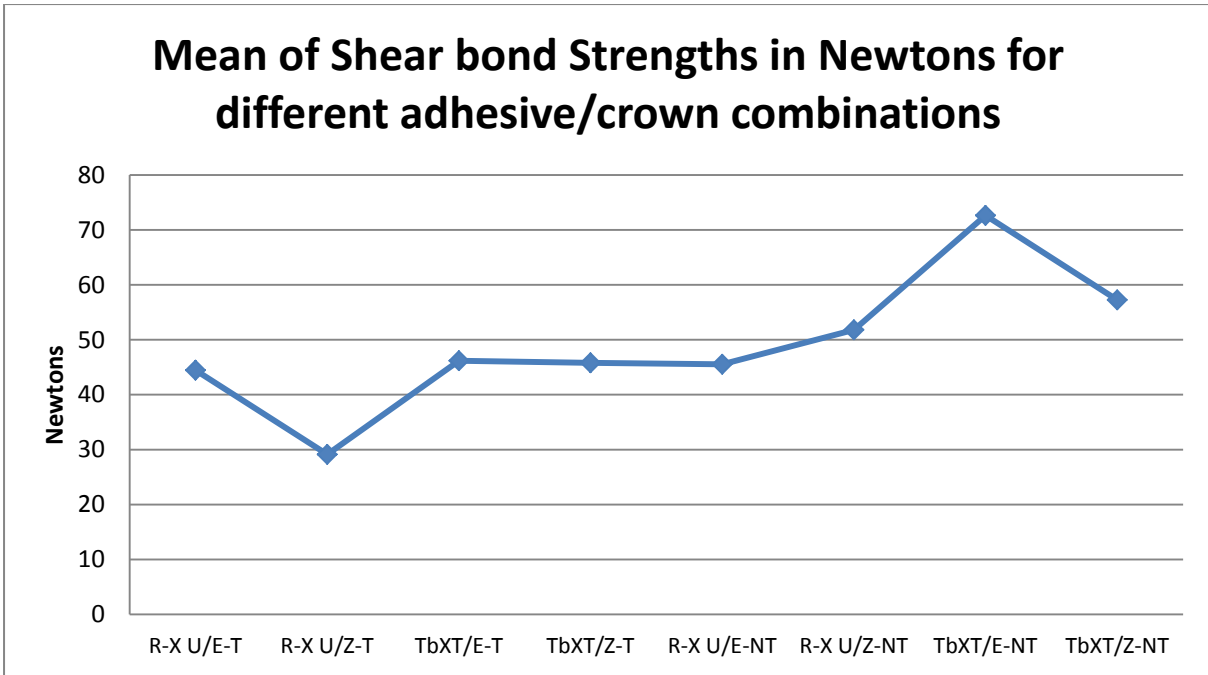


Figure 10: Line graph: Mean shear bond strengths in Newtons for the 8 combinations.

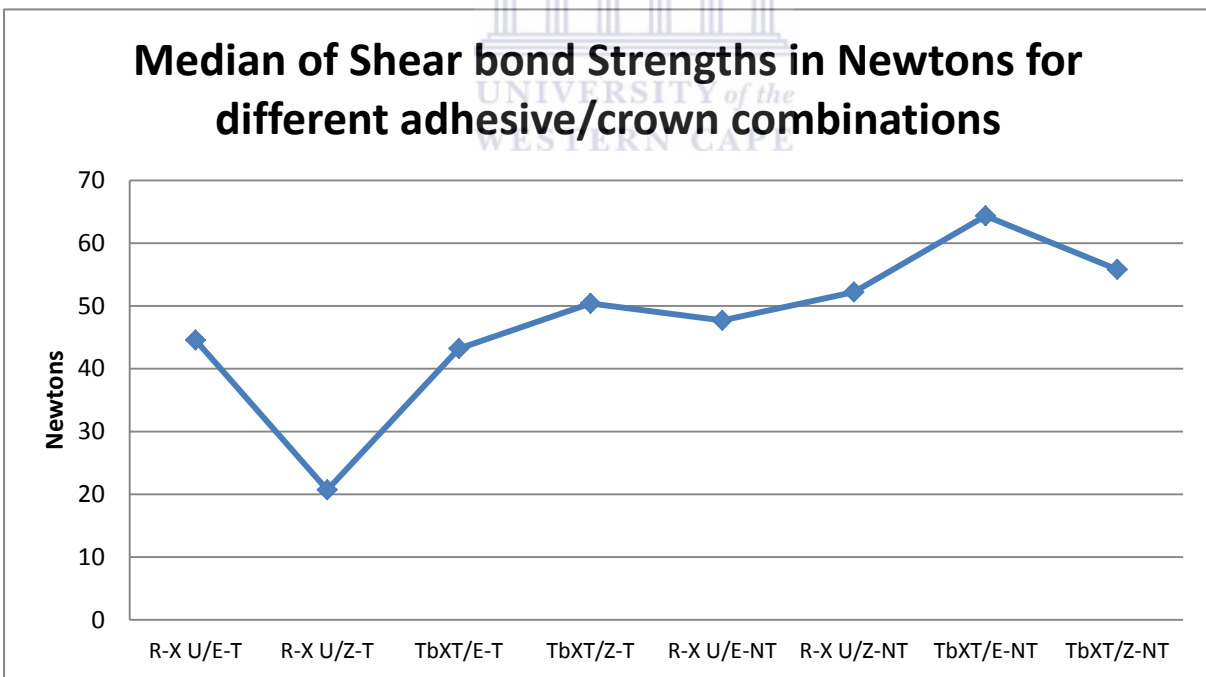


Figure 11: Line graph showing the Median shear bond strengths in Newtons for each adhesive/crown combination.

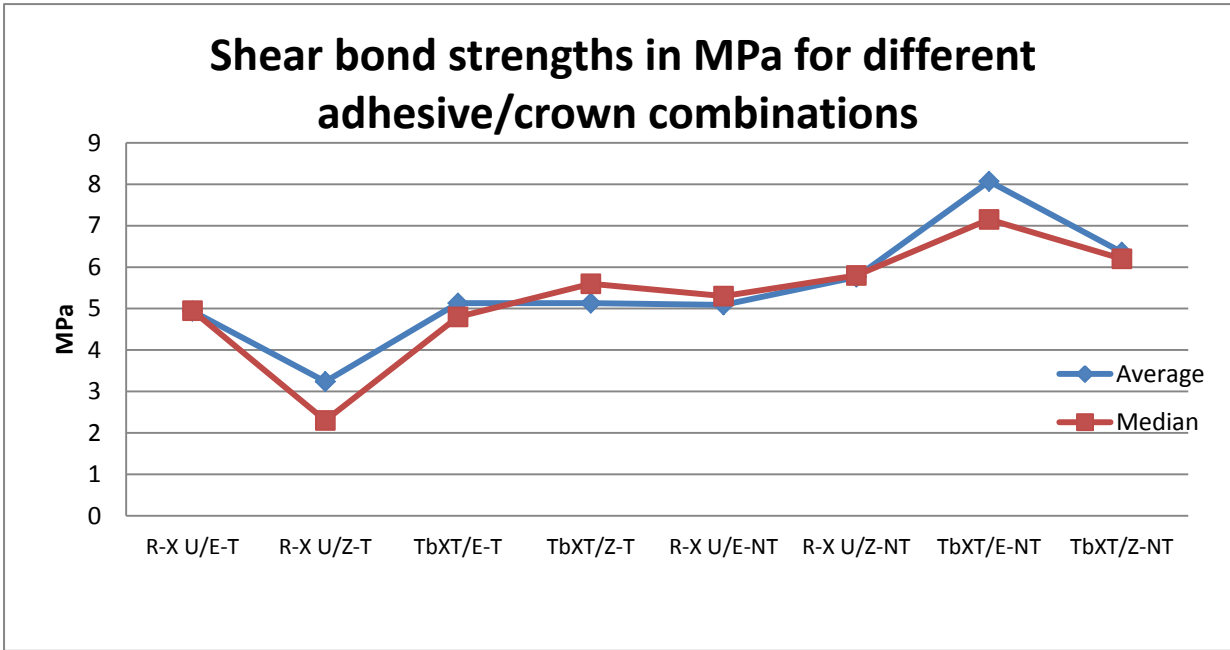


Figure: 12: Line graph showing the shear bond strengths in Mega Pascals (MPa) for each adhesive/crown combination.

The red line represents the median.

The blue line represents the average/mean.



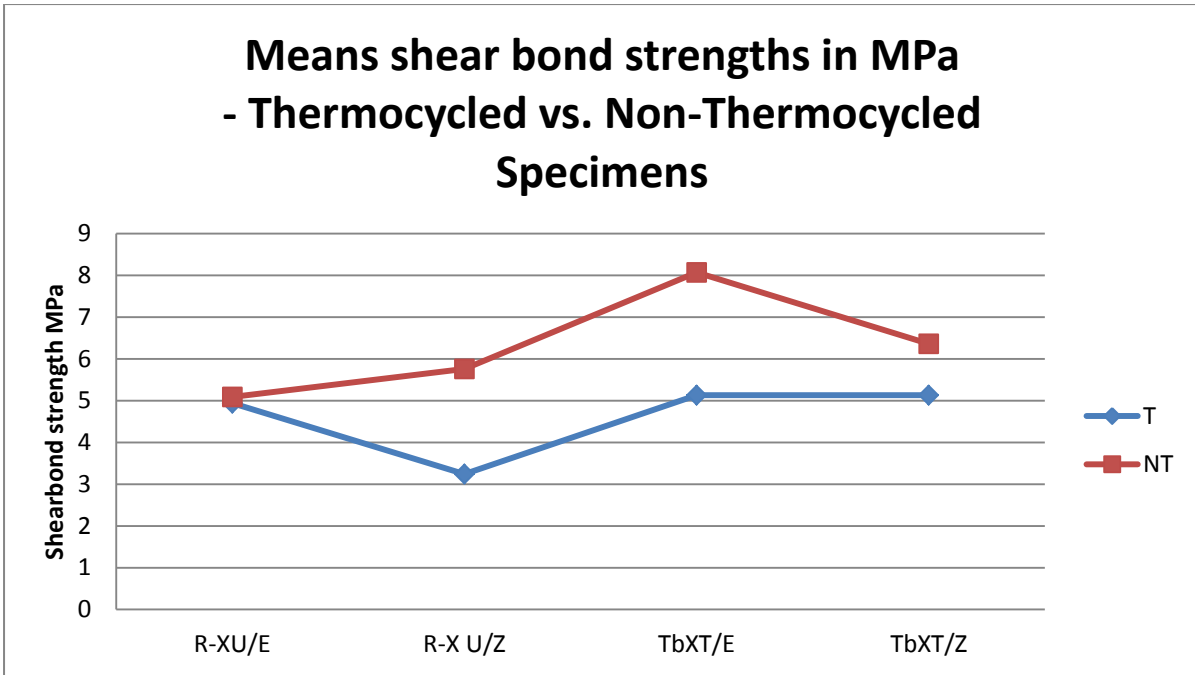


Figure: 13: Line graph showing the Mean shear bond strengths in MPa for the adhesive/crown combinations.

The blue line shows the Thermocycled specimens.

The red line shows the Non –Thermocycled specimens.

For a graphical display of the SBS determinations for each of the eight adhesive/crown combinations a Stem-and-Leaf was constructed (see Tables: 18-21.)

The bold digits in the **Leaves**-columns represents the individual observations (SBS in Newtons).

E-max- E

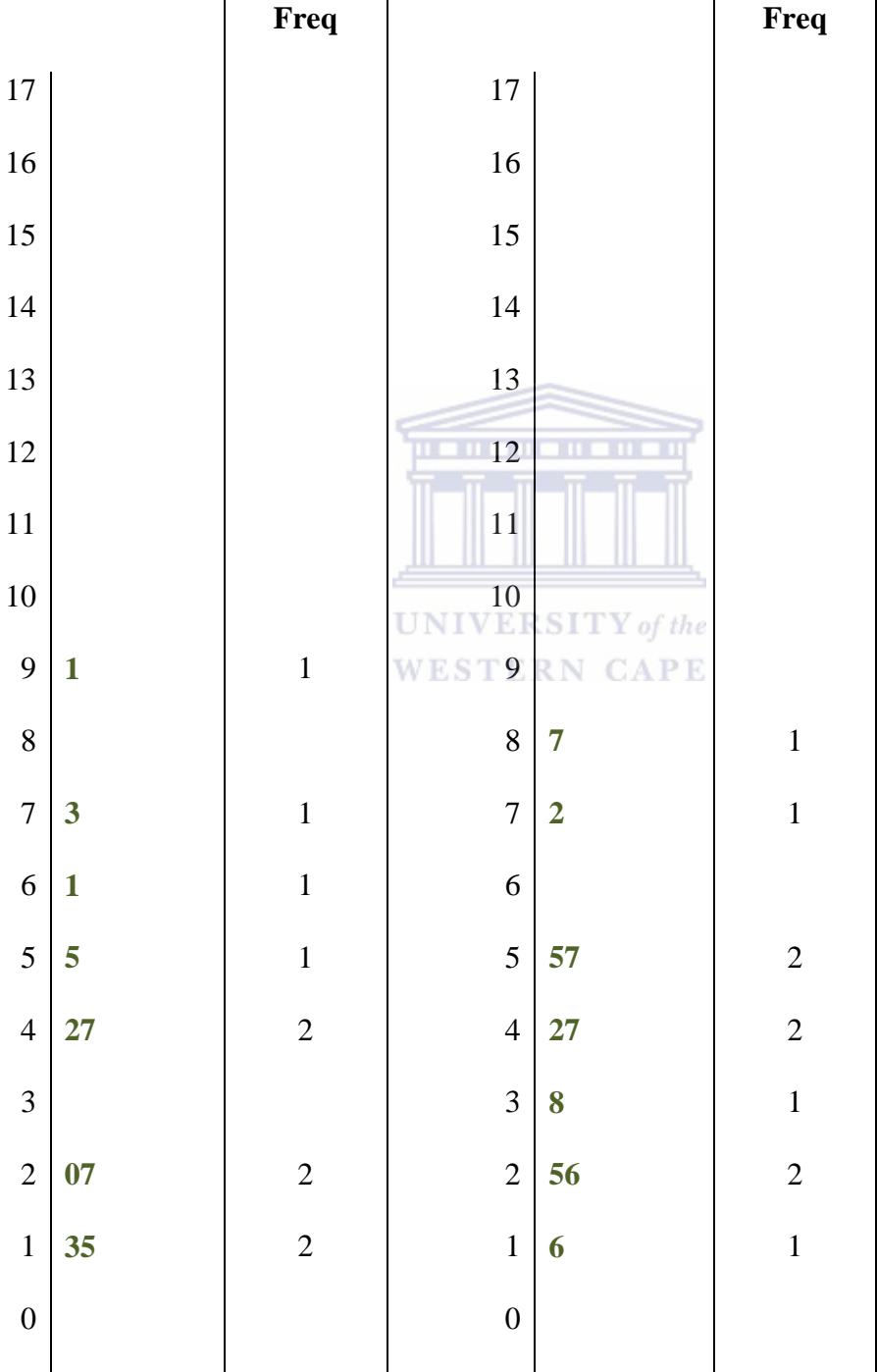
E-max – E

Thermo	Rely X
Cycled	Unicem

Thermo	Transbond
cycled	XT

Stem- Leaves

Stem- Leaves



Distribution very flat	Distribution very flat
------------------------	------------------------

*Table: 18. Stem-and-Leaf Diagram for two of the eight adhesive/crown combinations (**Freq** \equiv Frequency).*



E-max - E	
Non-	
Thermo	Rely X
Cycled	Unicem

Stem- Leaves

E-max - E	
Non-	
Thermo	Transbond
cycled	XT

Stem- Leaves

	Freq		Freq
17		17	2
16		16	
15		15	
14		14	
13		13	
12		12	
11		11	
10		10	4
9		9	
8		8	
7		7	
6	5	6	3569
5	125	5	01
4	0669	4	34
3		3	
2	27	2	
1		1	
0		0	



Dispersion narrow	Contains two outliers 172 & 104
-------------------	---------------------------------

Table 19. Stem-and-Leaf Diagram for two of the eight adhesive/crown combinations (**Freq** \equiv Frequency).



Zirconia – Z

Zirconia - Z

Thermo	Rely X
Cycled	Unicem

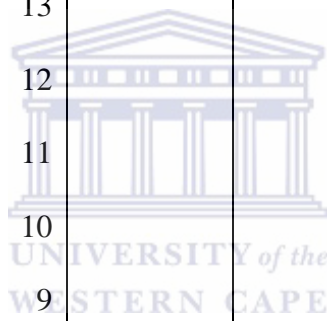
Thermo	Transbond
cycled	XT

Stem- Leaves

Stem- Leaves

		Freq
17		
16		
15		
14		
13		
12		
11		
10		
9		
8		
7		
6	0	1
5	3	1
4	0	1
3		
2	3	1
1	238	3
0	003	3

		Freq
17		
16		
15		
14		
13		
12		
11		
10		
9		
8		
7	1	1
6	16	2
5	16	2
4	9	1
3	3	1
2	78	2
1	6	1
0		



Distribution; skewed towards the larger values	Dispersion wide
---	-----------------

*Table: 20: Stem-and-Leaf Diagram for two of the eight treatment combinations (**Freq** \equiv Frequency).*



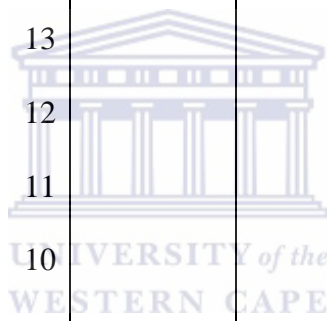
Zirconia – Z	
Non-	
Thermo	Rely X
Cycled	Unicem

Stem- Leaves

Zirconia - Z	
Non-	
Thermo	Transbond
cycled	XT


Stem- Leaves

		Freq		Freq
17			17	
16			16	
15			15	
14			14	
13			13	
12			12	
11			11	
10			10	
9			9	
8	1	1	8	
7			7	22
6	68	2	6	03
5	39	2	5	02247
4	1	1	4	1
3	59	2	3	
2	7	1	2	
1			1	
0	0	1	0	



Dispersion wide	Dispersion narrow
-----------------	-------------------

Table: 21: Stem-and-Leaf Diagram for two of the eight adhesive/crown combinations (**Freq** \equiv Frequency).



Group	SBS N	Group	SBS Mpa
R-X U-Z-T	20.70	R-X U-Z-T	2.30
Tb XT-E-T	43.20	Tb XT-E-T	4.80
R-X U-E-T	44.55	R-X U-E-T	4.95
R-X U-E-NT	47.70	R-X U-E-NT	5.30
Tb XT-Z- T	50.40	Tb XT-Z-T	5.60
R-X U-Z-NT	52.20	R-X U-Z-NT	5.80
Tb XT-Z-NT	55.80	Tb XT-Z-NT	6.20
Tb XT-E-NT	64.35	Tb XT-E-NT	7.15

Table: 22.: The SBS in Newtons and MPa **Medians** of the eight adhesive/crown combinations sorted from small to large.

Group	SBS N	Group	SBS Mpa
R-X U-Z-T	29.14	R-X U-Z-T	3.24
R-X U-E-T	44.46	R-X U-E-T	4.94
R-X U-E- NT	45.54	R-X U-E-NT	5.06
Tb XT-Z-T	45.81	Tb XT-Z-T	5.09
Tb XT-E-T	46.17	Tb XT-E-T	5.13
R-X U-Z- NT	51.80	R-X U-Z-NT	5.76
Tb XT-Z- NT	57.24	Tb XT-Z-NT	6.36
Tb XT-E- NT	72.63	Tb XT-E-NT	8.07

Table: 23.: The SBS in Newtons and MPa Means of the eight adhesive/crown combinations sorted from small to large.

The order for the two measures are exactly the same because the two units are linearly related

In the two Tables above (*see Tables: 22 and 23.*) there is a strong correspondence between the two rankings (Medians and the Means). The smallest shear bond (SBS) values remain the same as well

as the last three at the higher end of the spectrum. Towards the middle section of the rankings R-X U-E-T, R-X U-E-NT, Tb XT-Z-T and Tb XT-E-T are only slightly rearranged.

Group		R-X	Tb	R-X	R-X	Tb	R-X	Tb	Tb
		U-Z-T	XT-E-T	U-E-T	U-E-NT	XT-Z-T	U-Z-NT	XT-Z-NT	XT-E-NT
Median	SBS N	20.70	43.20	44.55	47.70	50.40	52.20	55.80	64.35
Mean	SBS N	29.14	46.17	44.46	45.54	45.81	51.80	57.24	72.63



Table: 24: The Medians and Means of the SBS in Newtons of the eight different adhesive/crown combinations.

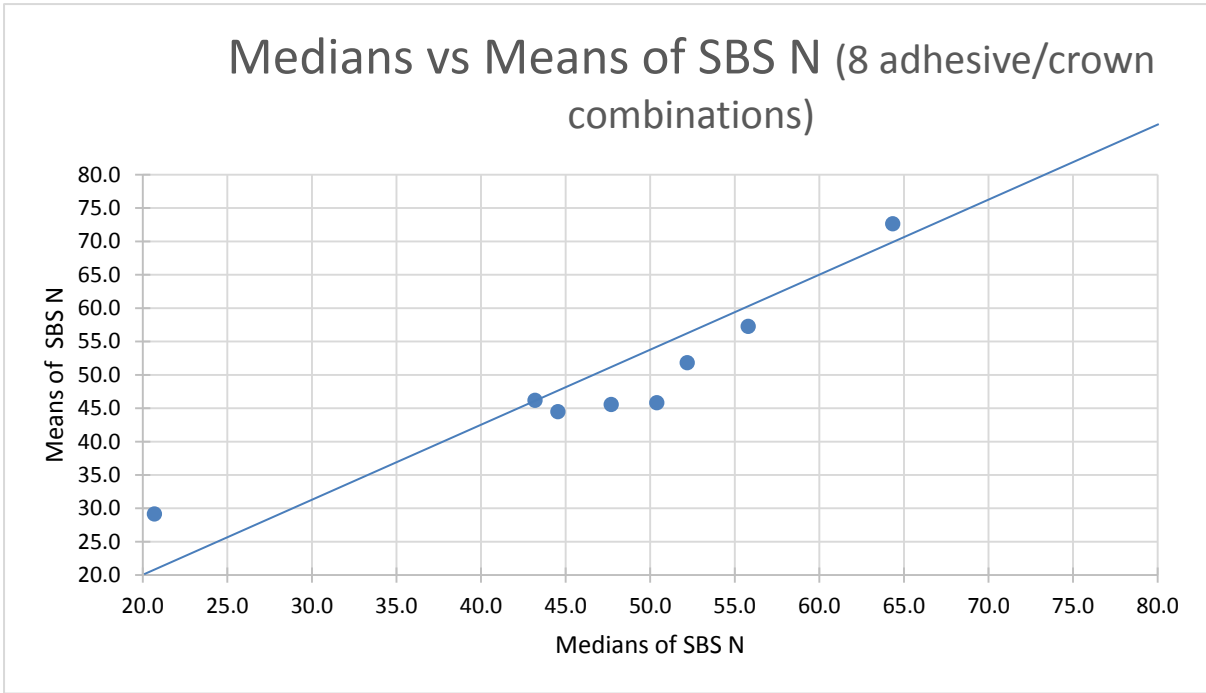


Figure: 14: Scatter plot of the Medians vs Means of the SBS in Newtons of the eight adhesive/crown combinations.



In the above graph of the eight adhesive/crown combinations Medians vs Means of SBS N are displayed and the symmetry of the statistical distribution of SBS-N is confirmed. The smallest SBS N is much less than the second smallest observation.

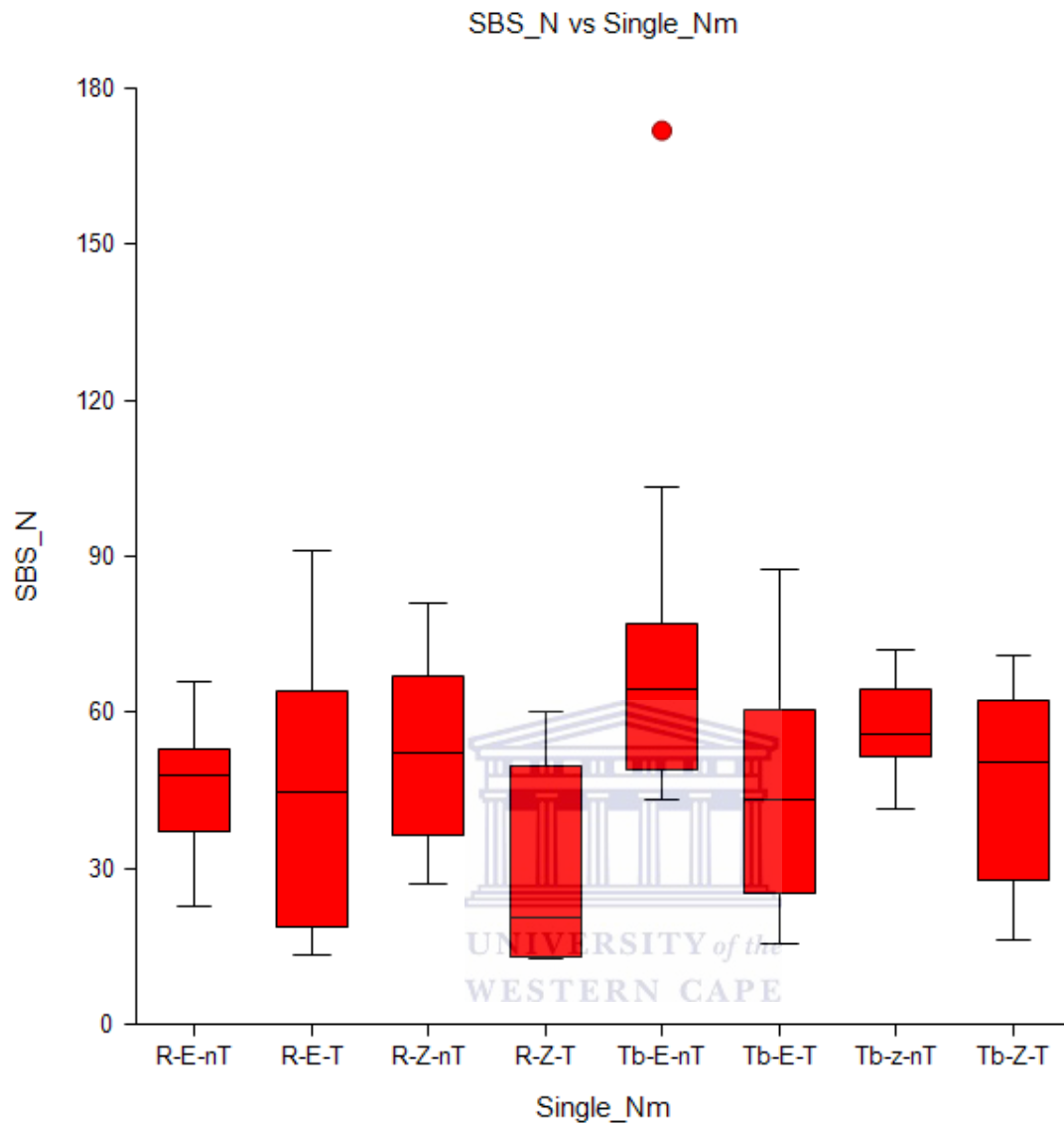


Figure: 15: Side by side Box-and-Whisker Plots of the SBS (N) values for the eight adhesive/crown combinations (Single combination Names).

Each box represents the interquartile area (50% of the readings for each combination).

The red line in each box represents the medians.

The red dot is representative of an extreme value obtained.

The wide and overlapping dispersion (interquartile ranges, the red boxes) of the adhesive/crown combinations will consequently lessen the probability of significant differences between the eight adhesive/crown combinations. The excessive outlier in the treatment combination Tb XT-E-NT would not affect the analysis because non-parametric methods were used.

From the Kruskal-Wallis test with respect to the Medians, the following Table (*see Table: 25.*) (for all the pairwise comparisons) can be constructed ($p < 0.05$).

				R-E-	R-Z-	Tb-Z-	Tb-E-	
Group	R-Z-T	Tb-E-T	R-E-T	NT	Tb-Z-T	NT	NT	
Median	20.7	43.2	44.55	47.7	50.4	52.2	55.8	64.35

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Table: 25. Medians in Newtons of the eight adhesive/crown combinations.

From this Table we could learn that SBS N for the treatment R-X U-Z-T which has a median of 20.7 N is different from Tb XT-E-T, R-X U-E-T, R-X U-E-NT, Tb XT-Z-T, R-X U-Z-NT, Tb XT-Z-NT and Tb XT-E-NT with corresponding medians 43.2, 44.55, 47.7, 50.4, 52.2, 55.8 and 64.35. Using the Bonferroni Test for Medians, it implies that those adhesive/crown combinations linked by the dark horizontal line do not differ significantly.

Relaxing the significance level (p-value) somewhat one could arrive at the following Table (*see Table 26.*).

				R-E-		R-Z-	Tb-Z-	Tb-E-
Group	R-Z-T	Tb-E-T	R-E-T	NT	Tb-Z-T	NT	NT	NT
Median	20.7	43.2	44.55	47.7	50.4	52.2	55.8	64.35

Table:26. Relaxing p-value

This figure is corresponding to the Scatter plot (*see Figure: 14.*). From this follows that Tb XT-E-NT (Transbond XT and e-Max and not Thermocycled) yielded the maximum SBS, but after being Thermocycled it dropped to the second lowest position of SBS N (a fall of six positions). Tb XT-Z-NT, R-X U-Z-NT and R-X U-E-NT dropped two positions, five positions and one position respectively.

R-X U-Z-NT, Tb XT-Z-NT, Tb XT-E-NT three of the not Thermocycled treatments are in the top positions, showing the adverse effects of Thermocycling. The treatment R-X U-Z-T (20.7) had the lowest SBS.

4.3. Shear bond strength comparisons

The results after debonding were sorted into maximum and minimum values and the means and medians were calculated (*see Tables: 6-16.*).

Table: 17. expresses the mean Shear bond strength (SBS) of the 8 adhesive/crown combinations in Newtons (N) and Mega Pascals (MPa) and their respective standard deviations (S.D).

Figures: 10 and 11. are line graphs showing the mean and median shear bond strengths (SBS) values in Newtons (N) of each adhesive/crown combination. Figure: 12. are superimposed line graphs comparing the mean and median shear bond strength (SBS) values of each adhesive/crown combination in Mega Pascals (MPa). Figure: 13. are superimposed line graphs comparing the mean shear bond strength (SBS) values in Mega Pascals (MPa) of the thermocycled and non-thermocycled adhesive/crown combinations.

For a graphical display of the shear bond strength (SBS) values in Newtons (N) for each of the 8 adhesive/crown combinations Stem-and-Leaf diagrams were constructed. Table: 18. displays a flat distribution of shear bond strength (SBS) values for both the R-X U/E-T and the Tb XT/E- T combinations. Table: 19. displays a narrow dispersion of shear bond strength (SBS) values for the R-X U/E-NT combination and the Tb XT/E-NT combination contained two outliers (104 N and 172 N).

Table: 20. displays a distribution of shear bond strength (SBS) values which is skewed towards the larger values for the R-X U/Z-T combination and a wide dispersion of shear bond strength (SBS) values for the TbXT/Z-T combination. Table: 21. displays a wide dispersion of shear bond strength (SBS) values for the R-X U/Z-NT combination and a narrow dispersion of shear bond strength (SBS) values for the TbXT/Z-NT combination.

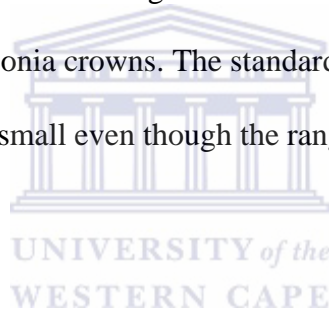
Table: 22. represents the medians of the shear bond strength (SBS) values of the 8 adhesive/crown combinations in Newtons (N) and Mega Pascals (MPa). Table: 23. represents the Means of the shear bond strength (SBS) values of the 8 adhesive/crown combinations in Newtons (N) and Mega Pascals (MPa). The order in these two tables is exactly the same because the two units are linearly related.

Figure: 14. shows a scatter plot of the Medians and Means of the shear bond strength (SBS) values in Newtons (N) of the 8 adhesive/crown combinations (*see Table: 24.*) and the symmetry of the statistical distribution of the shear bond strength (SBS) values in Newtons (N) is confirmed.

4.3.1. Rely X™ Unicem 2 self-adhesive resin

Comparative shear bond strengths of the 2 all ceramic non-thermocycled crowns

The results after debonding were compared. The mean shear bond strength for this adhesive bonded to the all ceramic non-thermocycled crowns ranged from a low of 5.1 MPa (45.5 Newtons) when brackets were bonded to the eMax crowns to a high of 5.8 MPa (51.9 Newtons) when brackets were bonded to the porcelain veneered zirconia crowns. The standard deviation in the shear bond strength values displayed in the 2 groups was small even though the range between the maximum and minimum values was large.



Rely X Unicem 2 self-adhesive resin/non-thermocycled eMax crown combination

The Rely-X Unicem 2 self-adhesive resin displayed the sixth highest shear bond strength value of the two adhesive resin cements when bonded to the non-thermocycled eMax crowns (Transbond™ XT adhesive resin cement displayed the 5 highest shear bond strength values). The shear bond strength values for this combination ranged from a minimum of 2.5 MPa (22.2 Newtons) to a maximum of 7.3 MPa (65.3 Newtons) with a mean value of 5.1 MPa (45.5 Newtons) (*see Table: 12.*). This combination displayed a standard deviation of 1.43 (*see Table: 17.*).

Rely-X Unicem 2 self-adhesive resin/non-thermocycled porcelain veneered zirconia crown combination

The Rely-X Unicem 2 self-adhesive resin displayed the highest shear bond strength value of the two adhesive resin cements when bonded to the non-thermocycled porcelain veneered zirconia crowns. The shear bond strength values for this combination ranged from a minimum of 0 MPa (the bracket debonded without registering a value on the shearing machine) to a maximum of 9 MPa (81 Newtons) with a mean value of 5.8 MPa (51.9 Newtons) (*see Table: 13.*). This combination displayed a standard deviation of 2 (*see Table: 17.*).

4.3.2. Transbond XT adhesive resin

Comparative shear bond strengths of the 2 all ceramic non-thermocycled crowns

The results after debonding were compared. The mean shear bond strength for this adhesive bonded to the all ceramic non-thermocycled crowns ranged from a low of 6.4 MPa (57.3 Newtons) when brackets were bonded to the porcelain veneered zirconia crowns to a high of 8.1 MPa (72.7 Newtons) when brackets were bonded to the eMax crowns.

Transbond XT adhesive resin/non-thermocycled eMax crown combination

The Transbond XT adhesive resin displayed the highest shear bond strength value of the two adhesive resin cements when bonded to the non-thermocycled eMax crowns. The shear bond strength values for this combination ranged from a low of 4.8 MPa (43.4 Newtons) to a maximum of 19.1 MPa (172.2 Newtons) with a mean value of 8.1 MPa (72.7 Newtons) (*see Table: 15.*). This combination displayed a standard deviation of 4.33 (*see Table: 17.*).

Transbond XT adhesive resin/non-thermocycled porcelain veneered zirconia crown combination

The Transbond XT adhesive resin displayed the second highest shear bond strength value of the two adhesive resin cements when bonded to the non-thermocycled porcelain veneered zirconia crowns. The shear bond strength values for this combination ranged from a low of 4.6 MPa (41.2 Newtons) to a maximum of 8 MPa (72.1 Newtons) with a mean value of 6.4 MPa (57.3 Newtons) (*see Table: 16.*). This combination displayed a standard deviation of 1.07 (*see Table: 17.*).

The side by side Box-and-Whisker plots of the shear bond strengths (*see Figure: 15.*) show wide and overlapping dispersions of the treatment combinations which consequently lessen the probability of significant differences between the treatment combinations. According to the Kruskal-Wallis test (*see Tables: 25 and 26.*) ($p < 0.05$), and the Bonferroni Test the non-thermocycled crown/adhesive resin combination do not differ significantly.

4.4. The Adhesive Remnant index (ARI) And Porcelain Fracture Index (PFI) results:

Analysis of the ARI and the PFI:

Group 1	Adhesive Remnant Index (ARI)					Porcelain Fracture Index (PFI)				
	0	1	2	3	Average	0	1	2	3	Average
R-X U/E-T	4	4	2	0	0.8	10	---	---	---	0
R-X U/Z-T	10	0	---	---	0	10	---	---	---	0
Group 2										
Tb XT/E-T	10	---	---	---	0	10	---	---	---	0
Tb XT/Z-T	10	---	---	---	0	9	---	---	1	0.3
Group 3										
R-X U/E-NT	1	2	2	5	2.1	10	---	---	---	0
R-X U/Z-NT	10	0	---	---	0	10	---	---	---	0
Group 4										
Tb XT/E-NT	5	1	0	4	1.3	10	---	---	---	0
Tb XT/Z-NT	3	1	1	5	1.8	10	---	---	---	0

Table: 27: Adhesive Remnant Index (ARI) and Porcelain Fracture Index (PFI) (sorted)-Groups

1,2,3,4.

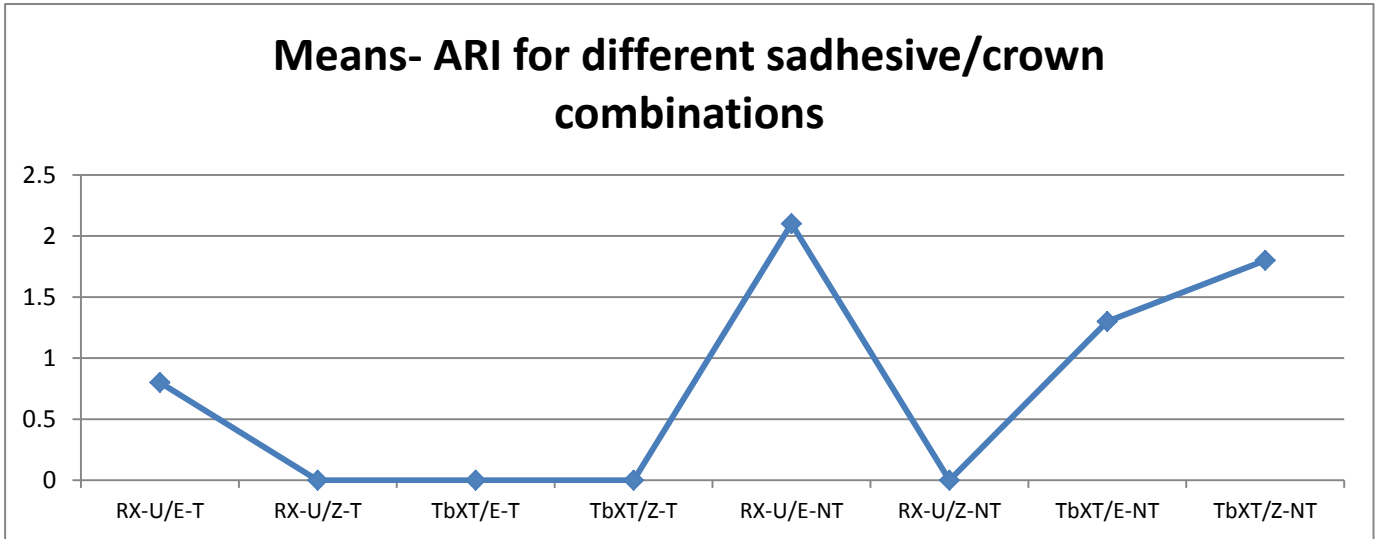


Figure: 16: Line graph showing the Means of the Adhesive Remnant Index (ARI) for the different adhesive/crown combinations.



For the next two measurements: ARI-score and PFI-score the values consists discrete integers. For the ARI-score the following summary table provides a list of similarities and differences.

Group	R-X U-	Tb XT-	Tb XT-	R-X U-	R-X U-	Tb XT-E-	Tb XT-Z-NT	R-X U-E-NT
	Z-T	E-T	Z-T	Z-NT	E-T	NT		
Mean	0	0	0	0	0.8	1.3	1.8	2.1

Table: 28. Mean ARI-scores for the eight adhesive/crown combinations.

For the last measurement, PFI only one observation was different from zero, therefore all eight medians were equal to zero.

4.4. Conclusions

Despite the small sample sizes and the overlapping dispersions, the study gives an indication of a trend, in the Shear Bond Strengths (SBS). The two units (Newtons and Mpa) differ only for a linear transformation of nine (9), therefore the statistical outcomes hold for both units. The detrimental influence of Thermocycling was observed in the measured shear bond strengths.





Chapter 5

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Discussion

Discussion

Introduction

Optimal bracket adhesion to the bonding surface of porcelain crowns is always of concern to orthodontists because the forces applied during treatment should not result in bond failure. Glazed porcelain is not an appropriate surface for resin penetration and orthodontic bonding due to the physical properties of glazed surfaces and the chemical properties of bonding resins (Smith *et al* 1988). Recommended surface treatment methods can be time consuming or even harmful to soft tissues. Hydrofluoric acid (HFA) etching is an effective surface treatment for porcelain-composite bonding (Kocadereli *et al* 2001). However, the risk of soft tissue burns and toxic effects of HFA requires extreme care during intraoral application, causing many orthodontists to be hesitant in its use (Zachrisson *et al* 1996, Lamour *et al* 2006).

In the present study, due to the potential toxicity of HFA, the ceramic surfaces were treated with 37% phosphoric acid and a silane coupling agent. Etching of porcelain surfaces with phosphoric acid alone does not provide adequate shear bond strength, capable of resisting the forces applied during orthodontic treatment (Guimaraes *et al* 2012). Anecdotal evidence suggests brackets bonded with silane coupling agents and phosphoric acid or hydrofluoric acid has sufficient bond strength for orthodontic treatment (Nebbe and Stein 1996, Schmage *et al* 2003, Ajlouni *et al* 2005, Lamour *et al* 2006, Abu Alhajja and Al-Wahadani 2007). Phosphoric acid does not etch porcelain, and it does not produce physical or topographical changes in the porcelain surface. Instead, phosphoric acid has the effect of neutralising the alkalinity of the adsorbed water layer, which is present on all porcelain restorations in the oral cavity. This enhances the chemical activity of the silane coupling agents which are subsequently applied (Wolf *et al* 1993, Samruajbenjakul and Kukiattrakoon 2009, Purnal *et al* 2013). Silane coupling agents have been reported to enhance bond strength to porcelain surfaces (Wood *et al* 1986, Kao 1988, Winchester and Orth 1991, Newman 1994, Bourke and Rock 1999,

Kocadereli *et al* 2001) . The silane reacts with the silica within the porcelain and the organic groups of the bonding resin, thus forming a bridge between the two materials (Newman 1994).

Commercially available porcelains are usually similar in chemical formula but have distinct differences in constituents in particle size, and crystalline structure. Therefore, different results are expected regarding bonding to different types of porcelain. In the present study, 40 IPS eMax and 40 porcelain-veneered zirconia crowns were fabricated off a single die and were divided into 4 groups containing 20 crowns each (10 IPS eMax crowns and 10 porcelain-veneered zirconia crowns). The IPS eMax crown and the porcelain-veneered zirconia crowns were chosen because currently they are the most commonly used crowns to restore teeth in the anterior region (Fradaeni 2012). A minimum of 10 specimens is recommended to perform shear bond strength testing (Fox *et al* 1994). However, a sample size greater than 10 specimens per group is recommended for bond strength testing of natural teeth where variations in tooth shape exist (Eliades and Brantley 2000). The maxillary anterior teeth are the teeth most frequently restored with porcelain restorations (Fradaeni 2012). Therefore, in this present study, the lateral incisor tooth form was selected to allow clinical simulation. Some studies (Nebbe and Stein 1996, Schmage *et al* 2003, Purnal *et al* 2013), have used porcelain tabs with flattened surfaces, while some have used porcelain discs (Guimaraes *et al* 2012) and others porcelain denture teeth (Lamour *et al* 2006).

The maximum bond strength which may be achieved to porcelain is not usually required for orthodontic purposes. The ideal bond should be sufficiently strong to endure a course of orthodontic treatment, yet be sufficiently weak at debond to permit restoration of the original porcelain surface.

There are a few scientifically-based recommendations in the literature for minimum orthodontic bracket shear bond strength. Reynolds (1975) recommended a tensile force of 60kg/cm² to 80kg/cm², while Newman (1994) stated that 14kg/cm² was the maximum that should be applied by an orthodontic appliance. Whitlock *et al* (1994) based upon the works of Reynolds (1975), also suggested that 6-8 MPa was adequate for orthodontic attachments and this was used in the present study. The Adhesive Remnant Index and the Porcelain Fracture Index was also examined to establish which regime produced adequate strength for orthodontic bracket attachment to all-ceramic crowns, with least porcelain surface damage following bracket removal.

The overall time required to place an appliance is an important factor in the cost of the treatment (Ajlouni *et al* 2005). Newer, self-adhesive cements have the potential to further simplify the bonding process, that is, by reducing the bonding of orthodontic brackets to a one-step procedure, and thereby reduce chair time and increase cost effectiveness, resulting in increased convenience and reduced costs for the patient (Hayakawa *et al* 1992). Reducing the steps during the bonding process will also reduce the risks of saliva contamination and the effects of humidity which could both have an adverse effect on the bond strength of the cement.

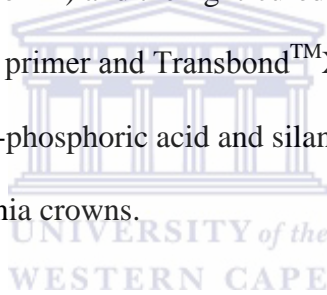
Although there are innumerable protocols for bonding orthodontic brackets to porcelain, there is still no scientific consensus about which of the techniques would be the ideal standard protocol for the purpose of overcoming the two points of contrast mentioned above (Herion *et al* 2010).

Increasing demands of adults for orthodontic treatment and the variation of the results in efficient methods of bonding to ceramics require more investigations. Hence, the purpose of the present study was to test and compare the shear bond strength and the resultant failure pattern of 2 types of resin cements (a self-adhesive, dual cured resin cement and a 2-step bonding, light cured resin cement) to etched and silane treated ceramic crowns.

Additionally, a further aim of this study was to substitute the etching using hydrofluoric acid which is noxious and potentially harmful. Instead, etching with 37% ortho-phosphoric acid and silane coupling application as a pre-treatment conditioning procedure of the ceramic crown surfaces before bonding was used.

Furthermore, examining the effect of thermocycling (ie. some the ceramic specimens were thermocycled to simulate the oral environment prior to bonding of the orthodontic bracket to the ceramic crown) on the shear bond strengths, which many studies have not included, was also documented.

The first objective of this study was to compare the shear bond strengths of the dual-cured, self-adhesive resin cement (RelyX™ Unicem 2) and the light-cured, 2 step bonding resin cement (Transbond™ XT light cure adhesive primer and Transbond™ XT adhesive resin cement) (3M, Unitek) to the pre-treated (35% ortho-phosphoric acid and silane coupling agent application) IPS eMmax and porcelain veneered zirconia crowns.



The results of the non-thermocycled groups (Group 3 and Group 4) show the highest mean shear bond strength (SBS) of 8.1 MPa (72.7 Newtons) was for the Transbond XT/eMax crown combination, second highest shear bond strength of 6.4 MPa (57.3 N) for the Transbond XT/porcelain veneered zirconia crown combination, third highest shear bond strength of 5.8 MPa (51.9 N) for the RelyX Unicem 2/ porcelain veneered zirconia crown combination and the lowest mean shear bond strength of 5.1 MPa (45.5 Newtons) was for the RelyX Unicem 2/eMax crown combination (*see Tables: 12 and 13. and Tables:15 and 16.*). Guimaraes *et al.*'s (2012) study on shear bond strength (SBS) of Transbond XT bonded to feldspathic porcelain discs conditioned with 37% phosphoric acid and silane application showed a mean SBS value of 7.32 MPa and concluded the shear bond strength (SBS) to be ideal for orthodontic bonding. Larmour *et al.*'s (2006) study on

shear bond strength (SBS) of Transbond™ XT bonded to porcelain denture teeth conditioned with 37% phosphoric acid and a silane coupling agent showed a mean shear bond strength (SBS) value of 7.9 MPa. However, it must be borne in mind that these studies have used feldspathic porcelain discs and porcelain denture teeth respectively, which may behave differently then when bonding to porcelain crowns. The Mean shear bond strengths (SBS) of the 4 non-thermocycled adhesive/crown combinations in the present study (ie. 8.1 MPa for Transbond™ XT/IPS eMax crown, 6.4 MPa for the Transbond™ XT/porcelain veneered zirconia crown, 5.8 MPa for the RelyX™ Unicem 2/porcelain veneered zirconia crown and 5.1 MPa for the RelyX™ Unicem 2/ IPS eMax crown combinations) are in agreement with the current literature and even though the Mean SBS of 5.8 MPa for the RelyX™ Unicem 2/ porcelain veneered crown and 5.1 MPa for the RelyX™ Unicem 2/ IPS eMax crown combination are lower than the ideal rupture force of 5.9 MPa (Guimaraes 2012), in this study, there is no statistically significant difference between the SBS of RelyX™ Unicem 2 dual-cured, self-adhesive resin cement and Transbond™ XT light-cured, 2-step adhesive resin cement (which is a commonly used orthodontic adhesive resin cement) to IPS eMax and porcelain veneered zirconia crowns, and should therefore still be clinically acceptable. Moreover, cohesive fractures may be seen on the ceramic surface, if the bond strength results between the ceramic and the composite resin are greater than 13 MPa (Thurmond *et al* 1994). In our present study, the bond strength values in all 4 groups did not exceed this value.

As this is the first shear bond strength study on IPS eMax and porcelain-veneered zirconia crowns conditioned with 35% phosphoric acid and a silane coupling agent in the literature, there are no values to compare the results with.

Shear bond strength values will be compared with results from bonding orthodontic brackets to ceramic crowns conditioned with Hydrofluoric acid (HFA) and a silane coupling agent. Jivanescu and Bratu (2014) compared RelyX™ Unicem self-adhesive resin to a light cured bonding system on porcelain-fused to metal crowns which were conditioned with 10% HFA, a primer and an adhesive. No statistically significant difference was found between the RelyX™ Unicem resin (SBS-5.18MPa) and the light cured bonding system. They concluded that both materials may be recommended for bonding orthodontic brackets to ceramic surfaces. In this study, the shear bond strength of the RelyX™ Unicem 2 dual-cured, self adhesive resin cement/ IPS eMax crown combination was 5.1 MPa and 5.8 MPa for the RelyX™ Unicem 2 dual-cured, self-adhesive resin cement/ porcelain veneered zirconia crown combination.

In Group 3 and Group 4, no statistically significant differences were found in the shear bond strengths of metal brackets bonded with the RelyX™ Unicem 2 dual-cured, self-adhesive resin cement and metal brackets bonded with the Transbond™ XT light-cured, 2-step bonding orthodontic adhesive cement to IPS eMax and porcelain-veneered zirconia crowns which were treated with 35% phosphoric acid and a silane coupling agent. This is in agreement with a study by Bilgic *et al* (2013) who had treated the porcelain surfaces with 9.6% HFA and a silane primer. This is also in agreement with a study by Elham *et al* (2007). However, Turk *et al* (2006) reported that lithium disilicate had a higher shear bond strength (SBS) than feldspathic porcelain restorations. Moreover, Abu Alhajja and Al-Wahadani (2007) observed significant differences between feldspathic and lithium disilicate ceramic restorations (IPS empress 2), with higher mean shear bond strength (SBS) reported in the feldspathic porcelain group. This may also be due to the structural differences between IPS empress 2 crown (earlier version of IPS eMax crown) and the IPS eMax crown. Ahluwalia *et al's* (2013) study which used a 9.6% HFA etch and silane primer found the IPS eMax crowns to have the greatest shear bond strength. The ceramo-metal and ceramo-zirconia crowns had comparable shear

bond strengths. This may be due to the differences in the processing methods and the molecular structure of the all-ceramic restorations.

5.1. Shear bond strengths (SBS) comparisons: non-thermocycled groups vs thermocycled groups (see Tables: 6-16.)

The third objective of this study was to compare the effects of thermocycling on the shear bond strengths of the tested groups. The results of the thermocycled groups (Group 1 and Group 2) show the Transbond™ XT/non-thermocycled eMax crown combination yielded the highest mean shear bond strength of 8.1 MPa (72.7 Newtons) but dropped to a mean shear bond strength of 5.1 MPa (46.1 Newtons) (36.4% drop in shear bond strength) when the crowns were thermocycled prior to bonding. The Transbond™ XT/non-thermocycled porcelain veneered zirconia crown combination yielded the second highest mean shear bond strength of 6.4 MPa (57.3 Newtons) and dropped to a mean shear bond strength of 5.1 MPa (45.8 Newtons) (19.3% drop in shear bond strength) when the crowns were thermocycled prior to bonding. The RelyX™ Unicem 2/non-thermocycled porcelain veneered zirconia crown combination yielded the third highest mean shear bond strength of 5.8 MPa (51.9 Newtons) but dropped significantly to a mean shear bond strength of 3.2 MPa (29.1 Newtons) (a significant 43.8% drop in shear bond strength) when the crowns were thermocycled prior to bonding (see Table: 23.). Lastly, the RelyX™ Unicem 2/non-thermocycled eMax crown combination yielded the fourth highest mean shear bond strength of 5.1MPa (45.5 Newtons) but dropped to a mean shear bond strength of 4.9 MPa (44.5 Newtons) (a drop in shear bond strength of only 3%) when the crowns were thermocycled prior to bonding. Relaxing the significance level (p-value) somewhat demonstrates the adverse effect of thermocycling on the shear bond strength of the adhesive/crown combinations (see Tables: 23-26.).

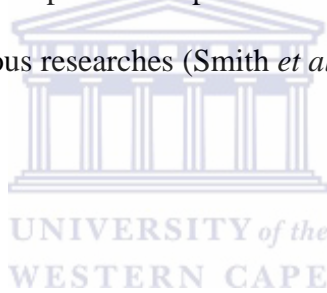
In this study, a statistically significant difference was found between the shear bond strengths of the non-thermocycled and thermocycled groups. As this is the first study on the influence of thermocycling prior to bonding on shear bond strength in the literature, there are no values to compare the results with. However, the adverse influence of thermocycling can be seen on the measured shear bond strength values.

It should be emphasized that the difference between in vitro versus in vivo bond strengths needs to be considered carefully, especially when bonding brackets to other restorative materials. Andreasen and Stieg (1988) indicated that the shear and tensile bond strengths of in vivo incisor and premolar enamel were significantly less than those of in vitro incisor and premolar enamel. They suggested that part of the in vivo increase in the rate of deterioration may be because of the mechanical and masticatory stresses placed on the bonds in the oral environment. They listed other factors, which may be of importance, including the moisture within the living tooth, flexing of the enamel during mastication, moisture contamination during bonding, as well as the thermal fluctuation in the oral cavity and the constant bathing of saliva. However, as this is a first in-vitro study on the influence of thermocycling prior to bonding of orthodontic brackets, there is no explanation in the literature as to why bonding of orthodontic brackets using adhesive resin cements to porcelain, which is an inert material, would be adversely affected by thermocycling prior to bonding. This may be an interesting topic for future research in order to gain a better understanding of the bonding orthodontic brackets to porcelain crowns.

Andreasen and Stieg (1988) calculated that there was a decrease of approximately 17% to 22% in tensile strengths and 48% to 52% in shear strengths in vivo when compared with the in vitro bond strengths. They suggested that if this percent of in vivo decline is evident when bonding to porcelain surfaces, stronger bond strengths would be required for efficient bonding of orthodontic brackets in the actual patient. In this study, even though the shear bond strengths of the adhesive/crown combinations were reduced statistically significantly when the porcelain crowns were thermocycled

prior to bonding, according to the literature (Andreason and Stieg 1988), may be something that is expected. In this study, a decrease of 36.4% in shear bond strengths for the Transbond™ XT/ thermocycled eMax crown combination, a decrease of 19.3% in shear bond strengths for the Transbond™ XT/ thermocycled porcelain veneered zirconia crown combination, a decrease of 43.8% in shear bond strengths for the RelyX™ Unicem 2/ thermocycled porcelain veneered zirconia crown combination, and a decrease of 3% in shear bond strengths for the RelyX™ Unicem 2/ thermocycled eMax crown combination, are significant, but according to the literature (Andreason and Stieg 1988), may still be clinically acceptable.

The number of thermal cycle is another point of dispute between different researchers. It has been 100, 150, 200 and 500 times in previous researches (Smith *et al* 1988, Newman 1994). We applied the biggest number in our study.



5.2. Adhesive Remnant Index comparisons (non-thermocycled crowns- group 3 and group 4)

The second objective of this study was to compare the resultant failure pattern of the tested groups.

Description of each category of the adhesive remnant index (*see Table: 1.*)

RelyX™ Unicem 2 adhesive/non-thermocycled eMax crown combination (*see Table: 27.*)

- 5 specimens debonded at the adhesive/bracket interface (ARI 3)
- 2 specimens had more than 50% of the adhesive on the ceramic surface (ARI 2)
- 2 specimens had less than 50% of the adhesive on the ceramic surface (ARI 1)
- 1 specimen had all the adhesive removed with the bracket (ARI 0)

- mean score of 2.1

RelyX™ Unicem 2 adhesive/non-thermocycled porcelain veneered zirconia crown combination (see Table: 27.)

- All 10 specimens had all the adhesive removed with the bracket (ARI 0)
- mean score of 0

Transbond™ XT adhesive/non-thermocycled eMax crown combination (see Table: 27.)

- 4 specimens debonded at the adhesive/bracket interface (ARI 3)
- 1 specimen had less than 50% of the adhesive on the ceramic surface (ARI 1)
- 5 specimens had all the adhesive removed with the bracket (ARI 0)
- mean score of 1.3



Transbond™ XT adhesive/non-thermocycled porcelain veneered zirconia crown combination (see Table:27.)

- 5 specimens debonded at the adhesive/bracket interface (ARI 3)
- 1 specimen had more than 50% of the adhesive on the ceramic surface (ARI 2)
- 1 specimen had less than 50% of the adhesive on the ceramic surface (ARI 1)
- 3 specimens had all the adhesive removed with the bracket (ARI 0)
- mean score of 1.8

The ARI results for the non-thermocycled crown/adhesive combinations display a mean score of 2.1 for the RelyX™ Unicem 2/non-thermocycled eMax crown combination, a mean of 0 for the RelyX™ Unicem 2/non-thermocycled porcelain veneered zirconia crown combination, a mean of 1.3 for the Transbond™ XT/ non-thermocycled eMax crown combination and a mean of 1.8 for the Transbond™ XT/non-thermocycled porcelain veneered zirconia crown combination (*see Table: 27. and Figure: 16.*). Study of the mean ARI values for the non-thermocycled crown/adhesive combinations shows that brackets bonded with RelyX™ Unicem 2/non-thermocycled porcelain veneered zirconia crowns failed entirely at the ceramic/adhesive interface and for all the other non-thermocycled ceramic/adhesive combinations most of the failures of the bond (70%) occurred at the bracket/adhesive interface and cohesive fractures within the composite resin. No cohesive fractures of the porcelain crowns were noted. The present findings indicate that there was no significant difference in the debonding patterns of the four non-thermocycled ceramic/adhesive combinations. This finding is different to the study by Bishara *et al* (2000) who tested RelyX™ Unicem with Transbond™ XT and their findings indicated that the brackets bonded with RelyX™ Unicem failed at the enamel/adhesive interface, whereas brackets bonded using Transbond™ XT typically, failed at the bracket/adhesive interface.

5.3. Adhesive Remnant Index (ARI): comparison of non-thermocycled and thermocycled groups

In this present study a similar trend to the shear bond strength was noted when ARI scores were examined. The non-thermocycled all ceramic crown/adhesive combinations showed mean ARI values of between 1.3 and 2.1 indicating cohesive fractures within the composite resin and efficient bonding of the adhesive material to the porcelain. However, the thermocycled all ceramic crown/adhesive treatment combinations showed mean ARI values of between 0 and 0.8 indicating a

bond failure between adhesive and porcelain and highlighting the adverse influence of thermocycling on bond strength of the adhesive resin cement (*see Table: 27. and Figure: 16.*). Bracket failure at each of the two interfaces has its own advantages and disadvantages. As an example, bracket failure at the bracket/adhesive interface is advantageous because it leaves the porcelain surface intact; however, considerable chair time is needed to remove the residual adhesive with the added possibility of damaging the porcelain during the cleaning process (Bishara *et al* 2000). On the other hand, when brackets fail at the porcelain/adhesive interface, less residual adhesive remains, therefore making the cleaning of the porcelain surface so much easier (Bishara *et al* 1998).

5.4. Porcelain Fracture Index (PFI)

The fourth objective of this study was to compare the surface integrity of the IPS eMax and porcelain veneered zirconia crowns after debonding for each of the groups tested using the Porcelain Fracture Index (PFI). Description of each category of the porcelain fracture index (*see Table 2.*).

All specimens were scored 0 but one specimen from the TransbondTM XT adhesive/thermocycled porcelain veneered zirconia crown combination was scored 3 due to delamination of the veneered porcelain from the underlying zirconia core. This may possibly be due to the poor bonding of the veneered porcelain to the underlying zirconia core. In the present study, optical microscope examination revealed no damage to the porcelain surfaces of 98.75% of the all ceramic crowns after debonding (*see Table: 28.*). A previous study (Thurmond *et al* 1994) showed that if the bond strength between the porcelain and the adhesive is greater than 13 MPa, the porcelain is fractured. In this study all 4 groups obtained shear bond strength values less than 13 MPa.

5.5. Ethical Consideration

This is a full laboratory study and no human tissue was used.

5.6. Conflict of interest

No conflict of interest was declared.

5.7. Limitations

This study has some limitations that may preclude the extrapolation of the results: a small sample size was used; the use of one type of orthodontic bracket and it is an in vitro study, which tested only resistance to shear forces, under constant load. Thermocycling studies also have limits.

Thermocycling in water poorly represents the dynamic environment of the oral cavity (Mair and Padipatvuthikul 2009). There is also important variability in the methods used to evaluate bond strength within the orthodontic literature, partially due to the lack of standardization protocols. As a result, it is difficult to draw any meaningful conclusion when comparing studies.

5.8. Future Research

Future research avenues can be orientated towards alternative debonding methods. Debonding should be explored using manual debonding, electrothermal debonding devices and lasers (Tocchio *et al* 1993, Azzeh and Feldon 2003, Bishara and Trulove 1990). Studies comparing machine debonding and manual debonding can be interesting.

Chapter 6

Conclusion

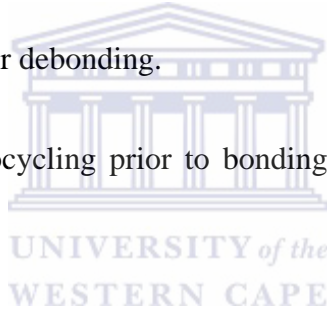


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Conclusion

Within the limitations of this study, it can be concluded that:

1. There was no significant difference in the shear bond strengths of metal orthodontic brackets bonded with RelyXTM Unicem self-adhesive resin cement and metal orthodontic brackets bonded with TransbondTM XT adhesive cement to non-thermocycled IPS eMax and porcelain-veneered zirconia crowns which were conditioned with 35 % phosphoric acid and a silane coupling agent.
2. Conditioning the porcelain surface with 35% phosphoric acid and a silane coupling agent would be safer to use than Hydrofluoric acid and should make it less risky for clinicians to clean the adhesive on the porcelain surface after debonding.
3. The negative influence of thermocycling prior to bonding can be seen on shear bond strength values.
4. Most of the bond failures for the non-thermocycled crown/adhesive combinations occurred at the bracket/adhesive interface and cohesive fractures within the composite resin and most of the bond failures for the thermocycled crown/adhesive combinations occurred at the adhesive/porcelain interface. No cohesive fractures of the porcelain crowns were noted.





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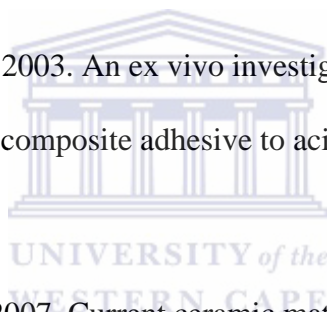
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