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Three point bending test of recycled Nickel-Titanium alloy wires

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The purpose of this study was to investigate the change of 3 point bending properties of various nickel titanium wires after recycling. Four types of nickel-titanium (Align: martensitic type, NiTi, Optimalloy, Sentalloy: austenitic type) wires were divided to three groups: as-received condition (T0: control group), treated in artificial saliva for four weeks (T1) and autoclaved after being treated in artificial saliva (T2). Detrimental changes were observed for the selected mechanical properties in three point bending test. Loading force at 3mm deflection, unloading force at 3mm deflection, stress hysteresis, loading force at 1mm deflection, unloading force at 1 mm deflection and stress hysteresis at 1mm deflection were calculated.

The findings suggest that :

1. Align demonstrated statistically significant increase in loading force ($p<0.05$) and unloading force ($p<0.01$) at 3mm deflection after recycling(T2), but NiTi, Optimalloy and Sentalloy showed no statistically difference after recycling.
2. Align demonstrated statistically significant decrease in hysteresis($p<0.01$) after recycling(T2) but NiTi, Optimalloy and Sentalloy showed no statistically significant difference after recycling.
3. All wires showed no statistically significant difference in loading force at 1mm deflection after recycling(T2).
4. Align demonstrated statistically significant decrease in unloading force in 1mm deflection ($p<0.05$) after recycling(T2) but NiTi, Optimalloy and Sentalloy showed no statistically difference after recycling.
5. Loading force and unloading force of T1 showed no significant change compared with those of T0, but loading force and unloading force of T2 showed significant changes compared with those of T0($p<0.05$, $p<0.01$ respectively).
6. Align demonstrated a tendency to lose some of this pseudoelsticity in T1 and pseudoplasticity and pseudoelasticity in T2.

Key words : nickel titanium wire, recycling, 3 point bending properties, hysteresis

Although nickel-titanium wires are attractive as orthodontic arch wires due to their excellent elasticity, unique properties of high springback and low stiffness[1], their high cost has led to their reuse. Buckthal et al.[2] noted that 52% of clinicians recycle them. Recycling involves sterilization and repeated exposure of the wire to physical stresses and chemical elements in the oral environment for several weeks or months. The effect of double exposure to the oral environment and sterilization may induce change of mechanical properties and surface condition.

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Several studies have demonstrated physical changes caused by sterilization and clinical use of orthodontic arch wires. Buckthal[3] said cold disinfectant induced, if any, change in the mechanical properties of nickel-titanium wires. Mayhew[4] showed that neither the heat sterilization nor multiple cycling procedures had a deleterious effect on the elastic moduli, surface topography, or tensile properties of Nitinol or Titanal. But Kapilla[5,6], on the contrary, showed recycling produced significant changes in both the three point bending characteristics and increased pitting of both Nitinol and NiTi wires. Sarkar[7] demonstrated that nickel titanium wire had a tendency to corrode when subjected to either oral fluid or chloride solutions. Schwaninger[8] found no changes in the load deflection characteristics of Nitinol following long term immersion in a 1% NaCl solution.

Lee and Chang[9] showed that the recycling had no significant effect on the tensile properties, surface topography and frictional properties of Nickel-titanium alloy wires lately. However, orthodontic wires do not usually get pressure on tensile, but tension, compression and bending in clinical situation.

Thus the purpose of this investigation was to evaluate the change of three point bending properties of nickel-titanium wires, and to evaluate the efficiency of reuse of nickel-titanium wires.

MATERIALS AND METHOD

Four brands of nickel-titanium wire (Align, A company, U.S.A ; NiTi, Ormco, U.S.A. ; Sentalloy, Tomy, Japan ; Optimalloy, Jinsung Ind. Co., Korea) was tested. 0.016 x 0.022 rectangular wires were selected. 15 wires of each product were prepared. Five wires of each served as controls (T0). The experimental sample consisted of five wires of each type were subjected to artificial saliva (Oralube, VA medical Center, Houston, Texas : Table 1) , maintaining in an incubator at 37 C for 3 weeks (T1) and 5 wires of each subjected to same artificial saliva and incubation treatment as T1, and then sterilized by autoclave at 121C (250 F) and 15 to 20 psi for 20 minutes(T2) according to ADA method.

Table 1. The composition of Oralube (VA Medical Center, Houston, Texas)

Methyl Paraben	2.00000 gm
Na CMC (Sodium CarboxyMethyl Cellulose)	10.00000 gm
Potassium Chloride	0.62500 gm
Magnesium Chloride Hexahydrate	0.05875 gm
Calcium Chloride Dihydrate	0.16625 gm
Potassium Phosphate (Dibasic, Anhydous)	0.80375 gm
Potassium Phosphate (Monobasic)	0.32625 gm
Sorbitol Solution, 70%, USP/FCC	42.7500 gm
FD & C Red Dye #40 (2% sterilized)	0.2500 ml
Distilled Water	1.0000 L

Test methods

Three point bending test was done with an Instron (Model 4466, Instron Corp., Cantom, Mass., USA) with a 5kg load cell. Each specimen was 50mm in straight area of preformed arch wires. 3 point bending fixture was in Liquid bath cell (S4190A, Instron Corp., Cantom, Mass.) that kept the temperature 371C during the test. Temperature controlled recirculator provided the capability to recirculate artificial saliva (Oraluve : VA medical Center, Houston, Texas) between external bath and a sealed heater unit. Two grips which permit sliding were positioned at each end of the wire with a distance of 30mm remaining between the jaws. 4mm diameter anvil pushed 3mm at the middle of the wire and replaced to original position. The machine was operated in the up and down mode with a crosshead speed of 5mm per min. Six characteristics of each sample were quantified from the Instron readouts using Series IX software: loading force (g/mm^2) , unloading force (g/mm^2), stress hysteresis (g/mm^2), loading force at 1mm deflection (g/mm^2) and unloading force at 1mm deflection (g/mm^2), stress hysteresis (g/mm^2) at 1mm deflection.

All means and standard deviations were calculated for the control and test values (for groups T1 and T2). Student t-test was used for all statistical analyses.

RESULTS

NiTi, Optimalloy and Sentalloy demonstrated no sta

Table 2. Summery of three point bending properties at 3mm deflection before and after recycling

	Align		*NiTi*		*Optimalloy*		*Sentalloy*	
	Mean	*SD*	*Mean*	*SD*	*Mean*	*SD*	*Mean*	*SD*
Loading force (g /mm^2)								
T0	2077.27	78.07	1640.91	104.64	1690.91	165.14	1213.64	12.45
T1	2145.46	67.42	1577.27	20.33	1818.18	55.67	1231.82	33.71
T2	2409.10	227.28	1590.91	16.07	1818.18	64.28	1236.36	20.33
Unloading force (g /mm^2)								
T0	1440.90	139.73	1022.73	60.13	1018.18	130.95	668.19	33.71
T1	1600.00	113.73	1027.27	24.89	1136.364	32.14	663.64	19.02
T2	1900.00	204.29	1018.18	24.90	1113.64	39.36	681.82	16.01
Stress Hysteresis (g /mm^2)								
T0	636.37	66.26	618.18	63.07	672.73	59.27	545.46	16.07
T1	545.45	55.67	550.00	10.16	681.82	32.14	568.18	16.08
T2	509.09	38.03	572.73	19.02	704.55	45.45	554.55	12.45

Table 3. Summery of three point bending properties at 1mm deflection before and after recycling

	Align		*NiTi*		*Optimalloy*		*Sentalloy*	
	Mean	*SD*	*Mean*	*SD*	*Mean*	*SD*	*Mean*	*SD*
Loading force (g /mm^2)								
T0	1640.90	38.03	1568.18	90.91	1418.18	38.03	1145.46	12.45
T1	1654.56	43.72	1536.36	34.47	1454.55	27.84	1154.55	24.90
T2	1690.91	98.54	1572.73	24.90	1445.46	20.33	1163.64	24.90
Unloading force (g /mm^2)								
T0	1295.46	106.60	859.09	82.57	804.55	120.91	336.36	10.16
T1	1409.09	80.35	827.27	12.45	904.55	40.64	336.36	19.02
T2	1454.55	55.67	872.73	25.91	868.19	51.82	354.54	12.45
Stress Hysteresis (g /mm^2)								
T0	345.46	80.99	718.18	12.45	613.64	83.51	809.09	12.44
T1	245.46	40.66	709.09	19.02	550.00	40.64	818.18	16.07
T2	236.37	49.79	700.00	19.02	577.28	54.95	809.09	12.45

tistically significant differences in three point bending properties : loading force (g/mm^2), unloading force (g/mm^2), stress hysteresis (g/mm^2), loading force at 1mm deflection (g/mm^2) and unloading force at 1mm deflection (g/mm^2), but Align showed statistically significant difference in loading force (g/mm^2), unloading force (g/mm^2), stress hysteresis (g/mm^2) and unloading force at 1mm deflection (g/mm^2) between groups. (Table II, III, IV and V)

Table 4. The effect of heat sterilization on three bending properties at 3mm deflection of various nickel titanium tested statistically by t-test

		Align		*NiTi*		*Optimalloy*		*Sentalloy*	
		P value	*sig*	*P value*	*sig*	*P value*	*sig*	*P value*	*sig*
Loading force	T0 vs T1	0.178	NS	0.219	NS	0.141	NS	0.291	NS
	T0 vs T2	0.015	*	0.322	NS	0.147	NS	0.066	NS
	T1 vs T2	0.038	*	0.273	NS	1.000	NS	0.803	NS
Unloading force	T0 vs T1	0.083	NS	0.880	NS	0.086	NS	0.724	NS
	T0 vs T2	0.003	**	0.880	NS	0.157	NS	0.273	NS
	T1 vs T2	0.021	*	0.580	NS	0.347	NS	0.141	NS
Stress Hysteresis	T0 vs T1	0.047	*	0.044	NS	0.771	NS	0.056	NS
	T0 vs T2	0.006	**	0.161	NS	0.369	NS	0..347	NS
	T1 vs T2	0.262	NS	0.046	NS	0.388	NS	0.172	NS

(* : p<0.05, ** : p<0.01)

Table 5. The effect of heat sterilization on three bending properties at 1mm deflection of various nickel titanium tested statistically by t-test

		Align		*NiTi*		*Optimalloy*		*Sentalloy*	
		P value	*sig*	*P value*	*sig*	*P value*	*sig*	*P value*	*sig*
Loading force	T0 vs T1	0.623	NS	0.485	NS	0.123	NS	0.486	NS
	T0 vs T2	0.325	NS	0.917	NS	0.195	NS	0.182	NS
	T1 vs T2	0.472	NS	0.092	NS	0.572	NS	0.580	NS
Unloading force	T0 vs T1	0.093	NS	0.419	NS	0.118	NS	1.000	NS
	T0 vs T2	0.019	*	0.734	NS	0.311	NS	0.057	NS
	T1 vs T2	0.329	NS	0.077	NS	0.252	NS	0.111	NS
Stress Hysteresis	T0 vs T1	0.039	*	1.000	NS	0.164	NS	0.347	NS
	T0 vs T2	0.033	*	0.471	NS	0.439	NS	1.000	NS
	T1 vs T2	0.760	NS	0.535	NS	0.392	NS	0.347	NS

(* : p<0.05, ** : p<0.01)

DISCUSSION

When a wire is deflected to place it in a bracket on a malaligned tooth, it is subjected to loading. The inherent tendency of the wire on loading is to try to return to its original shape or to unload, provided that its elastic limit is not exceeded during loading. This unloading of the wire represents its springback and provides the force required to cause biologic tissue response, which tends to move the tooth into alignment. The loading portion of the graphs obtained from the three point bending test in the present study simulates the activation of the wire, whereas the unloading portion of the graph provides some information of the forces associated with the wire as it undergoes deactivation. Therefore the unloading forces associated with the wire provided some indication of its potential clinical behavior[5].

The mechanical properties of various nickel-titanium wires differ according to their composition and the manufacturing processes. In the control groups, Align had the greatest loading force, unloading force at 3mm deflection and loading and unloading force at 1mm deflection, followed by NITi and Optimalloy which showed similar level of forces and Sentalloy showed lowest values (Fig. 1). Loading force at a deflection of 3.0mm for Align was 2077.27, Optimalloy 1690.91, NiTi 1640.91 and Sentalloy 1213.64. Unloading force at a deflection of 3.0mm for Align was 1440.90, NiTi 1022.73, Optimally 1018.18 and Sentalloy 668.19. However all the wires demonstrated pseudoplasticity as represented by small change in loads when the wires were displaced from 1.0mm to 3.0mm during loading and psedoelasticity as represented by similarly small change in forces from 2.0mm to 1.0mm deflection during unloading. Pseudoeplastic and pseudoelastic properties are unique to the nickel-titanium ally wires and imply that the forces associated with the wire remain fairly constant during loading and unloading respectively over a relatively large range of deflections. Clinically the small change in unloading forces signifies that the wire generates a nearly constant amount of force over some distance during deactivation.

They also showed relatively similar values in stress hysteresis at 3mm deflection compared with loading and unloading forces: Align 636.37, NiTi 618.18, Optimalloy 672.73 and Sentalloy 545.46. Therefore, though the graph of wires showed similar shapes, Sentalloy showed lowest position in the graph (Fig. 1).

Orthodontic wires are almost bent within the range of 3mm in clinical situation, and this is why the level of loading and unloading forces were mostly measured at 3mm deflection in three point bending test in orthodontic fields. Mean while, tooth movement in labiolingual direction and incisal-gingival direction of orthodontic treatment is usually done within 1mm. Therefore, level of loading and unloading force and stress hysteresis at 1mm deflection were also measured in this experiment.

NiTi, Optimally and Sentalloy showed no significant

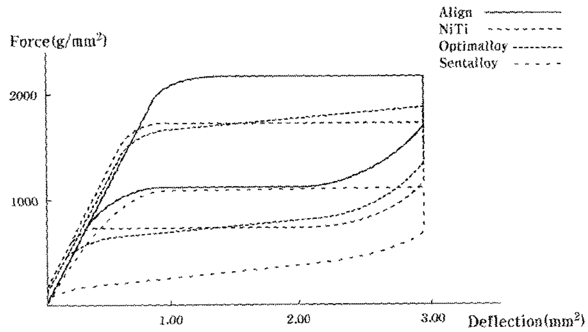

Fig. 1. Three point bending curves of control groups

change in all the three point bending properties after recycling, but Align was significantly altered by recycling (T0 vs T2 : Table IV and V). Loading force was increased significantly ($p<0.05$), unloading force was also increased significantly ($p<0.05$), and stress hysteresis was reduced significantly ($p<0.01$). Loading force at 1mm deflection showed no significant change, but unloading force at 1mm deflection and stress hysteresis at 1mm deflection showed significant change ($p<0.05$). Loading force and unloading force of T1 showed no significant change compared with those of T0, but loading force and unloading force of T2 showed significant changes compared with those of T1($p<0.05$, $p<0.01$ respectively). Therefore, autoclave process influenced the three point bending properties of Align more than treatment in artificial saliva.

Not only the values but also the shape of the curve of three point bending test were changed significantly (Fig 2). Pseudoplastic and pseudoelastic properties of unused Align(T0) is represented by relatively small decrease in forces when the wires is loaded from 1.4mm to 3.0mm and unloaded from 2.8mm to 1.0mm. However Align demonstrated a tendency to lose some of this pseudoelsticity in T1 and pseudoplasticity and pseudoelasticity in T2. Align showed significant changes in three bending properties, which concurs with Kapilla's[5,6] experiment in which he observed three bending properties after placing two types of nickel titanium wire in vivo for 8 weeks, and then through clinical recycling and dry heat sterilization. He ascertained that there was a small amount of change, but he

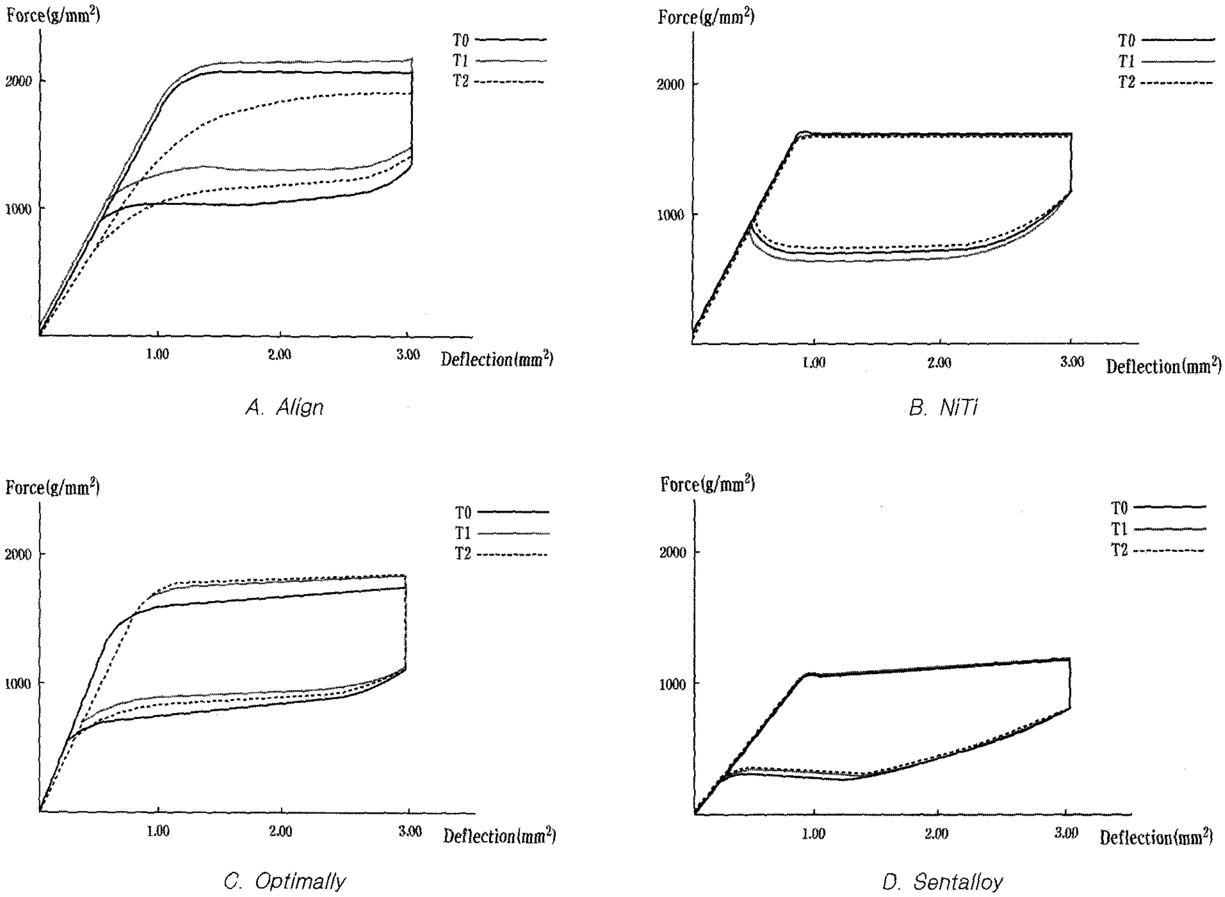

Fig 2. The change of three point bending curves in various nickel-titanium wires after recycling

left the clinical significance of his results open to question.

In the studies of Honma[10] and Okamoto[11]'s in which they examined the thermal expansion curves of nickel-titanium wires, they showed no significant change in the curve when the nickel-titanium wires were put through hot and cold cyclic treatment at under 350C. Though they showed that significant changes occurred in the thermal expansion curve when the wires were heated over 500°C.

In this study, an autoclave was used for sterilization at 121C for 15-20 minutes. Only Align altered in this range of heat. NiTi, Optimalloy and Sentalloy are Austenitic Ni-Ti wires that has lower transition temperature range, so they are stabilized in austenitic phase and do phase transformation in mouth temperature. However, Align is martensitic Ni-Ti wire that is stabilized in martensitic phase[12]. Martensitic Ni-Ti wire is not suitable in orthodontic treatment usually, and some procedures in manufacture are added to be useful in orthodontic treatment: for example, adding cobalt or work hardening. Therefore, we can conclude austenitic nickel-titanium wires were not significantly affected in three point bending properties by recycling, but martensitic nickel-titanium wires were changed by recycling.

In our previous report, recycled nickel-titanium wires showed no significant change on the tensile properties, surface topography and frictional properties[9]. Furthermore recycled austenitic nickel-titanium wires showed no significant change in three point bending properties.

CONCLUSION

1. Align(martensitic NiTi) demonstrated statistically significant increase in loading force ($p<0.05$) and unloading force ($p<0.01$) at 3mm deflection after recycling, but NiTi, Optimalloy and Sentalloy(austenitic NiTi) showed no statistically difference after recycling.
2. Align demonstrated statistically significant decrease in hysteresis($p<0.01$) after recycling but NiTi, Optimalloy and Sentalloy showed no statistically difference after recycling.
3. All wires showed no statistically significant difference in loading force at 1mm deflection after recycling.
4. Align demonstrated statistically significant decrease in unloading force in 1mm deflection ($p<0.05$) after recycling but NiTi, Optimalloy and Sentalloy showed no statistically difference after recycling.
5. Loading force and unloading force of T1 showed no significant changes compared with those of T0, but loading force and unloading force of T2 showed significant changes compared with those of T1($p<0.05$, $p<0.01$ respectively). Therefore, autoclave process influenced the three point bending properties of Align more than treatment in artificial saliva
6. Align demonstrated a tendency to lose some of this pseudoelsticity in T1 and pseudoplasticity and pseudoelasticity in T2, but NiTi, Optimally and Sentally showed no significant change in pseudoplasticity and pseudoelasticity.
7. Consequently recycled austenitic nickel-titanium wires showed no significant change in three bending properties.

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국문초록

재생한 니켈 티타늄 호선의 3점 굴곡물성실험

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본 연구의 목적은 재생된 니켈-티타늄 호선의 3점 굴곡물성의 변화를 조사하여, 임상적으로 니켈-티타늄 호선을

재생하여 사용하는 것이 타당한지를 알아보는 것이다. 4종의 니켈-티타늄 호선(Align, NiTi, Optimallo, Sentalloy)을 처리전(T0: 대조군)과 인공타액에 4주간 처리한 군(T1), 그리고 인공타액 처리 후 가압증기 멸균소독한 군(T2)으로 구분하여 3점 굴곡실험을 하였다. 3mm 변형시의 loading force, unloading force, hysteresis와 1mm 변형시의 loading, unloading force, stress hysteresis의 변화를 관찰하여 다음과 같은 결과를 얻었다.

1. Align은 재생(T2)후에 3mm 변형시의 loading force, unloding force가 통계적으로 유의성있게 증가하였다(loading force: p,0.05, unloading force:p<0.01). NiTi, Optimalloy, Sentalloy에서는 통계적으로 유의성있는 차이가 보이지 않았다.
2. Align은 재생(T2)후에 hysteresis가 통계적으로 유의성있게 감소하였다(p<0.01). NiTi, Optimalloy, Sentalloy에서는 통계적으로 유의성있는 차이가 보이지 않았다.
3. 모든 호선에서 1mm 변형시의 loading force는 재생(T2)후에 통계적으로 유의성있는 차이를 보이지 않았다.
4. Align은 재생(T2)후에 1mm 변형시의 unloading force가 통계적으로 유의성있게 증가하였다(p<0.05). NiTi, Optimalloy, Sentalloy에서는 통계적으로 유의성있는 차이가 보이지 않았다.
5. Align의 loading force와 unloading force는 T1과 T0는 통계적으로 유의한 차이가 없었으며, T2와 T0사이에 유의성 있는 차이가 있었다(각각 p<0.05, p<0.01).
6. Align은 T1에서 pseudoelasticity가 감소하고, T2에서는 pseudoplasticity와 pseudoelasticity가 감소하였다.

주요 단어 : 니켈-티타늄 호선, 재생, 3점 굴곡물성, 히스테레시스