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Evaluation and Comparison of Effect of Different Surface Treatments and Varying Alloy Percentage on the Elemental Composition of Ni Cr Base Metal Alloy

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Authors' contributions

This work was carried out in collaboration between all authors. Authors DG and RPN designed the study, performed the statistical analysis, wrote the protocol and wrote the first draft of the manuscript and managed literature searches. Authors DG, RPN and RP managed the analyses of the study and literature searches. All authors read and approved the final manuscript.

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ABSTRACT

Aim: To evaluate and compare the Effect of Different Surface Treatments and Varying Alloy Percentage on The Elemental Composition of Ni Cr Base Metal Alloy.

Study Design: The elemental composition of Ni Cr alloy with different surface treatments (oxidation, sandblasting, silane coupling agent) with varying alloy percentage (100% new, 50% new and 50% old, 100% old once cast) of all specimens were tabulated and subjected to statistical analysis.

Place and Duration of Study: Department of Prosthodontics and Crown and Bridge, V.K Institute Of Dental Sciences, KLE University, Belgaum, Karnataka, India and Indian Institute Of Technology, Mumbai India.

Methodology: Forty five disc shaped Ni Cr alloy specimens were made and subjected to different surface treatments which include oxidation, sandblasting and silane coupling agent application.

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Elemental composition was evaluated with Energy Dispersive X-Ray Analysor System (EDX) and surface topography was analyzed using Scanning Electron Microscope (SEM) at 1500 x magnification.

Results: The elemental composition was significantly different in the casting groups and treatment groups ($p < 0.05$). The highest mean weight percentage Ni value was recorded for Group A with oxidation treatment. Aluminum-oxide sandblasting of the alloy surface reduced the mean weight percentage for Cr.

Conclusion: Recasting metal alloys may adversely affect surface quality of the Ni Cr alloy.

Keywords: Ni Cr (nickel chromium) alloy; EDX (Energy Dispersive X-Ray Analysor System); SEM (Scanning Electron Microscope); Mo (Molybdenum); Cu (Copper); Ag (Silver); Zn (Zinc).

1. INTRODUCTION

High noble and base metal alloys have been in use to fabricate cast dental prosthesis. High noble alloys were used because of their resistance to corrosion and biocompatibility. Due to economic reasons and ease of availability, base metal alloys came into wide use. Ni-Cr alloys possess good mechanical properties, such as high hardness, low density, and high tensile strength [1].

Dental laboratories reuse the casted metal for recasting of cast restorations. Literature guidelines for recasting Ni-Cr alloy vary from not adding any new metal, or to some new metal, or 50% new metal with previously casted alloys. The manufacturer’s information on Ni-Cr casting alloys also states that casted metals can be reused to fabricate clinically acceptable castings, provided at least 50% new metal is used. Certain important secondary elements, present in small percentages in the original alloy compositions may be lost during recasting through volatilization or oxidation [2].

Chromium provides tarnish resistance and stainless properties to these alloys. When chromium content exceeds 30%, the alloy becomes more difficult to cast as it forms a brittle phase, known as the sigma (б) phase. Hence, cast base metal dental alloys should not contain more than 28% or 29% chromium. Cobalt increases strength, hardness and elastic modulus. Nickel may increase the ductility. The presence of 3% to 6% molybdenum contributes to the strength of the alloys. Aluminum increases the ultimate tensile and yield strengths of the alloy. Silicon and manganese are added to increase the fluidity and castability of these alloys.

Zinc acts as an oxygen scavenger during melting to minimize the oxidation of other elements in the alloy. Almost all elements in these alloys, such as chromium, silicon, molybdenum, and nickel, react with carbon to form carbids, which change the properties of the alloys [3,4].

Different surface treatments like sandblasting and degassing may affect the elemental composition and their surface structure. Silane coupling agents which are commonly used to enhance the bond strength between metal and ceramic may also affect the surface of these alloys. The studies on recasting base metal alloys are restricted to examining the physical characteristics of these alloys. There is very less literature on the change in the elemental composition of the recasted alloys. Hence this study was undertaken to evaluate the effect of different surface treatments and varying alloy percentage on the elemental composition of base metal alloys.

2. MATERIALS AND METHODS

2.1 Sample Preparation

Forty five (n = 45) disc shaped specimens measuring about (15*2 mm) were fabricated using Ni-Cr base metal alloy (Girobond CBS, Germany). These specimens were further divided into 3 Groups (Groups A, B and C) consisting of fifteen specimens in each group. (n=15) (Fig. 1)

In the first group (Group A) the specimens were cast from 100% new alloy. The second group (Group B) consisted of specimens that were cast from 50 wt% new alloys with 50 wt% recast (once) alloys and the third group (Group C) specimens were cast from 100% recast (once) alloys. The metal used for recasting was obtained from left over buttons and sprues of the alloys that had been previously casted [3].

The specimens were fabricated using a conventional lost-wax technique. Wax patterns were fabricated using inlay casting wax (Bego, Bremen, Germany), invested using a phosphate bond investment (Bellawest, Bego, Bremen, Germany) and subjected to casting procedure. The rings were submitted to thermal cycling according to the manufacturers' recommendations, and a casting temperature of 1330°C was followed for all the castings. After the sprues had been cut off, the specimens were airborne-particle abraded using aluminum oxide (Alox, Bego, Bremen, Germany) in order to be cleaned from remaining remnants of investment material. Then specimens were polished and finished by stone burs and rubber polishing wheels (BEGO, Bremen, Germany) (Fig. 2).

The resultant specimens were subjected to different surface treatments which include oxidation, sandblasting and silane coupling agent application (Fig. 3).

2.2 Surface Treatments

The resulting specimens were randomly divided into three subgroups (Subgroup 1, 2 and 3) of five samples each (n=5).

Sub Group 1- The specimens were subjected to oxidation at 970°C for 10 min in the Ceramic furnace (Vita Zahnfabrik, Germany).

Sub Group 2- The specimens were subjected to sand blasting treatment with 110 μmm Al2O3 in a sandblasting unit.

Sub Group 3- Specimens were treated with Silane coupling agent (Monobond N) according to manufacturer instructions. After sandblasting and cleaning the specimens in an ultrasonic unit for about 1 minute, Monobond N was applied with brush on the surface of the specimen.

Fig. 1. 45 samples divided in three groups (A,B,C).

Fig. 2. Armamentarium used in the study

Fig. 3. Samples divided into three Subgroups (1,2,3)

Fig. 4. Energy dispersive X-ray analysor system

2.3 Elemental Analysis

The elemental compositions of each specimen in weight percentage was calculated with the EDX (Fig. 4). It is used for chemical characterization of a sample and is based on the fundamental principle that each element has a unique atomic structure allowing unique set of peaks on its X-ray emission spectrum. A high-energy beam of charged particles such as electrons or protons or a beam of X-rays, is focused into the sample being studied. The incident beam may excite an electron in an inner shell, ejecting it from the shell while creating an electron hole and an electron from an outer, higher-energy shell then fills the hole, and the difference in energy between the higher-energy shell and the lower energy shell may be released in the form of an X-ray. The number and energy of the X-rays emitted from a specimen were measured by an energy-dispersive spectrometer.

For qualitative analysis of the surface topography of the specimens, one specimen from each group randomly was subjected to SEM at 1500 x magnification. The specimens were rinsed with distilled water, dried, and fixed onto an aluminum cylinder (13 mm in diameter and 10 mm in height). The specimens were sputter-coated with gold-palladium alloy and evaluated by SEM (Figs. 5-13).

SEM images Group A

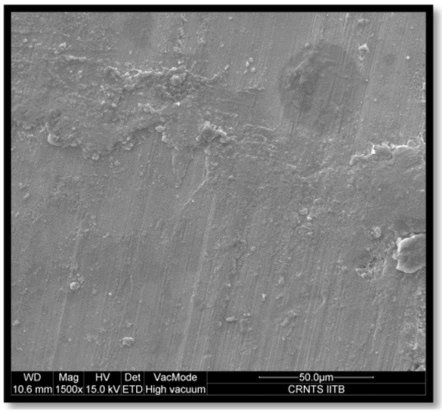

Fig. 5. SEM image at 1500 X Subgroup 1 (Oxidation)

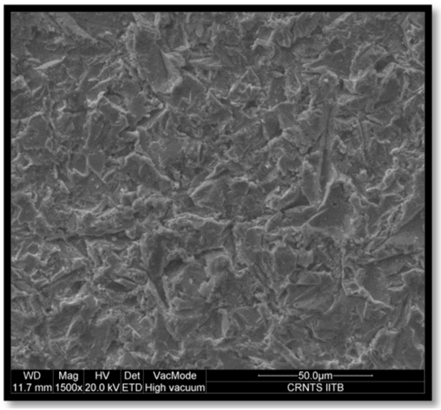

Fig. 6. SEM image at 1500 X subgroup 2. (Sandblasting)

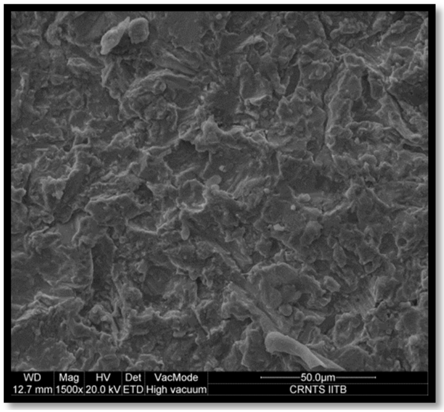

Fig. 7. SEM image at 1500 X Subgroup 3 (Silane coupling agent)

SEM images of Group B

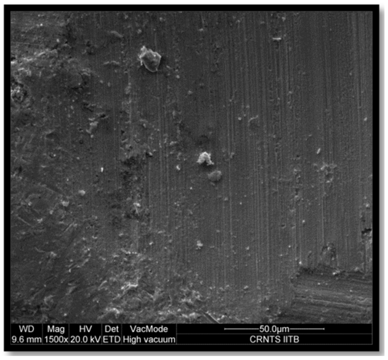

**Fig. 8. SEM image at 1500 X
Subgroup 1 (Oxidation)**

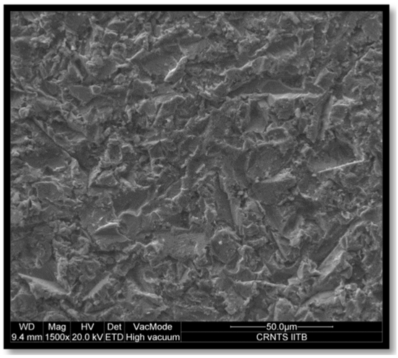

**Fig. 9. SEM image at 1500 X
Subgroup 2. (Sandblasting)**

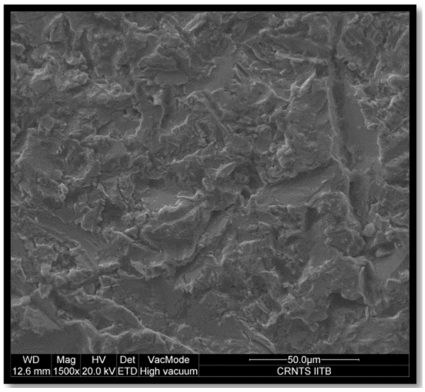

**Fig. 10. SEM image at 1500 X
Subgroup 3 (Silane coupling agent)**

SEM images of Group C

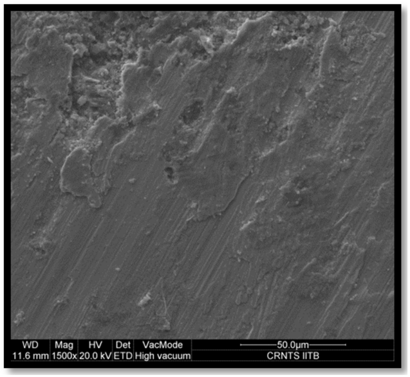

**Fig. 11. SEM image at 1500 X
Subgroup 1 (Oxidation)**

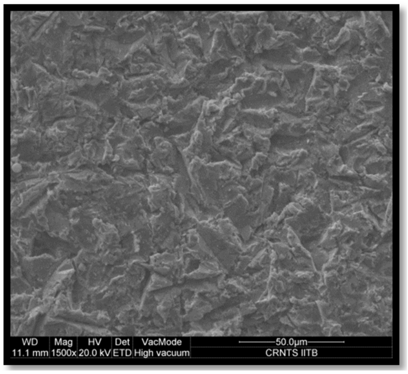

**Fig. 12. SEM image at 1500 X
Subgroup 2 (Sandblasting)**

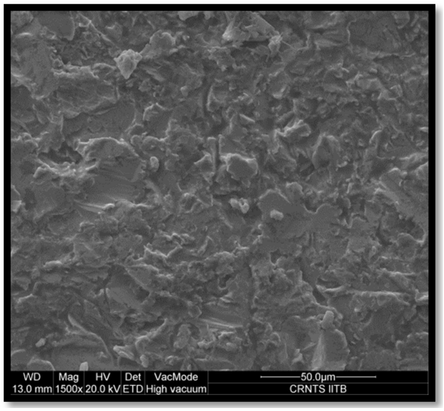

Fig. 13. SEM image at 1500 X
Subgroup 3 (Silane coupling agent)

Elemental percentage for each specimen was calculated in terms of its weight% and tabulated and the resultant data was subjected to statistical analysis to draw conclusions from the experimental data.

3. RESULTS

Means and standard deviations were calculated for each group, and the results were analyzed with two-way ANOVA and Tukey HSD tests. Statistical differences were analyzed and tabulated. Of the three groups the percentage of Nickel was maximum with 100% new alloy and oxidation treatment (Group A with Subgroup 1) whereas least with the 100% old alloy (Group C) suggesting decrease in concentration of Ni in recast alloy (F 2.8785 and P 0.0363) (Tables 2 and 5). The percentage of Cr was maximum in 100% new alloy with oxidation (Group A with Subgroup 1) and was least with 100% old alloy (Group C) with sandblasting suggesting decrease in Cr percentage in recast alloy with sandblasting treatment (F 1.2398 and P 0.3115) (Tables 3 and 6). Other specimens did not show any significant differences in elemental composition.

FLOWCHART OF METHODOLOGY

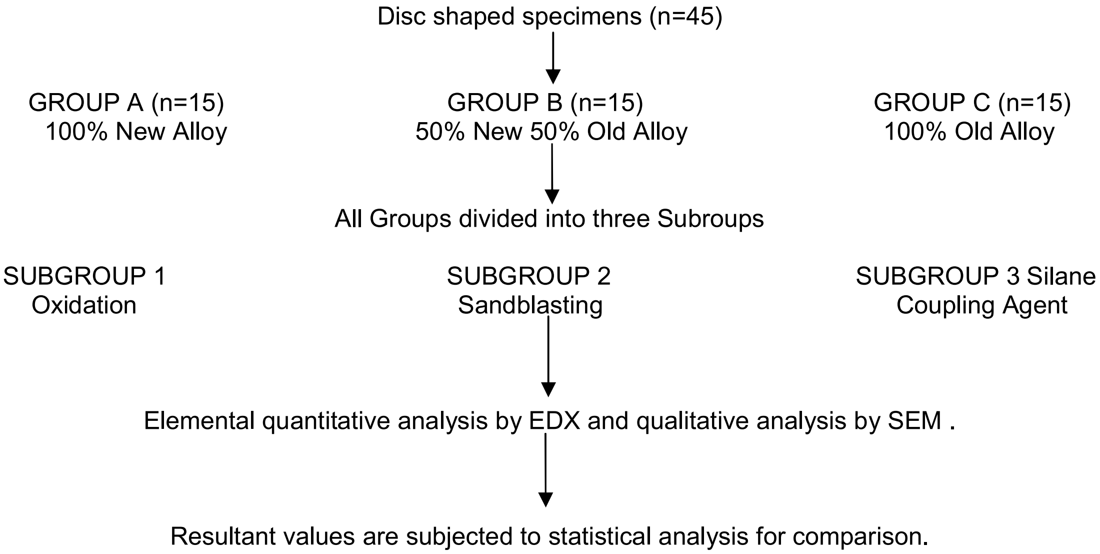

Table 1. Values of EDX with respect to Ni, Cr and Mo

Group	Sample no.	Elements		
		NI (nickel)	CR (chromium)	MO (molybdenum)
100% new alloy oxidation	1	64.89	24.26	10.16
	2	64.92	23.98	10.05
	3	65.32	24.66	10.09
	4	65.26	24.23	10.22
	5	64.99	23.87	10.15
1OO% new alloy sandblasting	1	64.61	23.72	10.39
	2	64.51	23.77	10.43
	3	64.58	23.74	10.39
	4	64.41	23.82	10.49
	5	64.37	23.86	10.49
100% new alloy silane agent	1	64.55	24.02	10.33
	2	64.33	24.14	10.44
	3	64.51	24.02	10.31
	4	64.41	24.02	10.41
	5	64.43	24.04	10.34
50% old 50% new alloy oxidation	1	64.42	24.26	10.15
	2	64.31	24.28	10.17
	3	64.41	24.31	10.01
	4	64.35	24.25	10.02
	5	64.38	24.22	10.05
50 %old 50 % new alloy sandblasting	1	64.45	24.15	10.21
	2	64.43	24.18	10.16
	3	64.38	23.67	10.18
	4	64.39	23.56	10.22
	5	64.45	24.19	10.19
50% old 50% new alloy silane agent	1	64.41	25.1	10.28
	2	64.12	24.81	10.14
	3	64.23	25.12	10.11
	4	64.31	23.89	11.11
	5	64.41	23.78	10.12
100% old alloy oxidation	1	64.31	23.92	10.16
	2	64.13	23.85	10.18
	3	63.82	23.88	10.12
	4	63.78	23.91	10.17
	5	63.56	23.93	10.05
100% old alloy sandblasting	1	64.57	23.41	10.36
	2	63.87	23.23	10.34
	3	63.78	23.13	10.41
	4	64.55	23.32	10.38
	5	63.31	23.35	10.39
100% old alloy silane agent	1	64.59	23.81	10.26
	2	63.88	23.82	10.24
	3	63.46	23.78	10.28
	4	64.57	23.77	10.23
	5	63.56	23.79	10.27

4. DISCUSSION

Dentists and technicians are always conscious of the cost of the materials used in fixed prostheses, and this leads to increased recasting. The reasons for recasting are economic, prevention of wastage of natural resources and environmental protection [5]. *Many investigators have evaluated the effect of repeated use of alloys on the charachteristics of resultant castings.* Most of the investigators studied properties like tensile strength, ultimate tensile strength, percentage elongation, modulus of elasticity, mean yield strength, microstructure, and micro hardness [6,7] The present study was undertaken to evaluate the elemental composition of recast alloy and the findings agreed with the hypothesis that recasting of base metal alloys would change the elemental composition and that mixing new and previous metal can influence the compositional stability of base metal alloys leading to change in concentration major elements like Ni, Cr, Mo and other minor elements which include Cu, Zn and Ag. In this study changes in elemental composition were minimal for castings with new alloy when compared to the recasted alloy. The findings of the present study are contrary to the studies by Tucillo et al who concluded that elemental composition of high gold alloy remained stable during recasting procedures. According to the author most of the clinical failures in castings were attributed due to the tarnish or corrosion when the nobility was decreased or the silver copper ratio was altered [8] Preswood analyzed the chemical composition of Ni-Cr-Be alloy and concluded changes in the amount of Ni, Fe, Mn, Cr, Mo, and Al remained within 0.1%-0.01% after each melting indicating no significant changes in its chemical structure. This could be attributed to the fact that 50 μmm aluminium oxide cleaning was performed and no attempt was made to remove the retained oxide from the buttons and sprues [9].

Table 2. Comparison of three groups (A, B, C) and three subgroups (1, 2, 3) with Ni (nickel) % by two way ANOVA

Sources of variation	Degrees of freedom	Sum of squares	Mean sum of squares	F-value	P-value
Main effects					
Groups	2	3.5835	1.7917	21.4865	0.0001*
Subgroups	2	0.3350	0.1675	2.0086	0.1489
2-way interactions					
Groups x Subgroups	4	0.9601	0.2400	2.8785	0.0363*
Error	36	3.0020	0.0834		
Total	44	7.8806			

p<0.05

Table 3. Comparison of three groups (A, B, C) and three subgroups (1, 2, 3) with Cr (Cromium) % by two way ANOVA

Sources of variation	Degrees of freedom	Sum of squares	Mean sum of squares	F-value	P-value
Main effects					
Groups	2	2.6521	1.3260	18.6463	0.0001*
Subgroups	2	2.0313	1.0157	14.2820	0.0001*
2-way interactions					
Groups x Subgroups	4	0.3527	0.0882	1.2398	0.3115
Error	36	2.5602	0.0711		
Total	44	7.5963			

p<0.05

Table 4. Comparison of three groups (A, B, C) and three subgroups (1, 2, 3) with Mo (Molybdenum) % of elements in by two way ANOVA

Sources of variation	Degrees of freedom	Sum of squares	Mean sum of squares	F-value	P-value
Main effects					
Groups	2	0.0824	0.0412	1.8129	0.1778
Subgroups	2	0.4560	0.2280	10.0379	0.0003*
2-way interactions					
Groups x Subgroups	4	0.1273	0.0318	1.4011	0.2533
Error	36	0.8176	0.0227		
Total	44	1.4832			

p<0.05

Table 5. Pair wise comparisons of three groups (A, B, C) and three subgroups (1, 2, 3) with % of elements in NI by Tukeys multiple posthoc procedures

Groups with Subgroups	**Group A with Subgrp 1**	**Group A with Subgrp 2**	**Group A with Subgrp 3**	**Group B with Subgrp 1**	**Group B with Subgrp 2**	**Group B with Subgrp 3**	**Group C with Subgrp 1**	**Group C with Subgrp 2**	**Group C with Subgrp 3**
Mean	65.08	64.50	64.45	64.37	64.42	64.30	63.92	64.02	64.01
SD	0.20	0.10	0.09	0.05	0.03	0.12	0.30	0.54	0.54
Group A with Subgrp 1	-								
Group A with Subgrp 2	p=0.0666	-							
Group A with Subgrp 3	p=0.0346*	p=0.9999	-						
Group B with Subgrp 1	p=0.0125*	p=0.9989	p=0.9999	-					
Group B with Subgrp 2	p=0.0242*	p=0.9999	p=0.9999	p=0.9999	-				
Group B with Subgrp 3	p=0.0039*	p=0.9713	p=0.9955	p=0.9999	p=0.9988	-			
Group C with Subgrp 1	p=0.0001*	p=0.0700	p=0.1279	p=0.2723	p=0.1707	p=0.5154	-		
Group C with Subgrp 2	p=0.0002*	p=0.2107	p=0.3387	p=0.5791	p=0.4199	p=0.8326	p=0.9998	-	
Group C with Subgrp 3	p=0.0002*	p=0.2022	p=0.3270	p=0.5649	p=0.4069	p=0.8218	p=0.9999	p=0.9999	-

**p<0.05*

Table 6. Pair wise comparisons of three groups (A, B, C) and three subgroups (1, 2, 3) with % of elements in Cr by Tukeys multiple posthoc procedures

Groups with Subgroups	Group A with Subgrp 1	Group A with Subgrp 2	Group A with Subgrp 3	Gourp B with Subgrp 1	Gourp B with Subgrp 2	Group B with Subgrp 3	Group C with Subgrp 1	Group C with Subgrp 2	Gourp C with Subgrp 3
Mean	24.20	23.78	24.05	24.26	23.95	24.54	23.90	23.29	23.79
SD	0.31	0.06	0.05	0.03	0.31	0.66	0.03	0.11	0.02
Group A with Subgrp 1	-								
Group A with Subgrp 2	p=0.2758	-							
Group A with Subgrp 3	p=0.9916	p=0.8105	-						
Group B with Subgrp 1	p=0.9999	p=0.1339	p=0.9306	-					
Group B with Subgrp 2	p=0.8563	p=0.9839	p=0.9996	p=0.6427	-				
Group B with Subgrp 3	p=0.5430	p=0.0021*	p=0.1183	p=0.7788	p=0.0306*	-			
Group C with Subgrp 1	p=0.6877	p=0.9987	p=0.9923	p=0.4454	p=0.9999	p=0.0138*	-		
Group C with Subgrp 2	p=0.0003*	p=0.1153	p=0.0021*	p=0.0002*	p=0.0101*	p=0.0001*	p=0.0227*	-	
Grp C with Subgrp 3	p=0.3110	p=0.9999	p=0.8455	p=0.1549	p=0.9900	p=0.0026*	p=0.9994	p=0.0989	-

**p<0.05*

Hespi et al. measured physical properties of cr-co alloys and recommended metal could be used for four generations without significant differences [10]. Bauer concluded that scrap produced in dental labs can be reused as this metal is melted under controlled conditions [11]. Walczack concluded that microstructure of recasting was similar to brand new castings with no inclusions which could originate from charge contamination of investment materials [12].

Nelson et al. [13] demonstrated no remarkable degenerative change in Ni-Cr alloy recasting for 10 generations and combining used metal with new metal and recasting 100 times demonstrated no remarkable degenerative changes in physical properties, microstructure, or clinical characteristics. The author stated strict adherence to clean techniques was essential to minimize contamination and inclusions that adversely affect the physical properties. The point of attachment between the sprue and wax pattern should be rounded, flared and properly contoured.

Baran stated that easily oxidized elements diffuse to the surface of the casting and combine with oxygen because of which metal near the surface undergoes change in composition. This change may affect the stability of secondary phases and may cause them to dissolve. Hence the surface condition of the alloy affects the oxidation products [4].

Al-Hiyasat also showed that release of elements increased significantly in relation to the percentage of recast material used in the casting procedure in turn leading to increase in cytotoxicity [14].

In the present study the concentration of Ni decreased in recast alloy and sandblasting decreased the concentration of Cr. The Mo content on the surface oxide layers for these Ni-Cr alloys after heat treatment was almost negligible which could be ascribed to the volatility of Mo oxides (mainly as MoO3). The findings of the present study are in accordance with Phillips who indicated that mixing new and previously cast metal influenced the compositional stability of a Ni-Cr base metal alloy, leading to changes in the amount of base metals (Ni and Cr) and other major and minor elements (Mo, Si and Mn etc.) [1]. Ayad concluded that mixing new and previously cast metal can influence the compositional stability of a type 3 gold alloy. The author also concluded that small decrease or increase in the concentration of elements probably would have no clinical significant effect on the mechanical properties or corrosion resistance of the alloys [15].

In this study, changes in elemental concentrations were generally minimal, given that the practical accuracy of values determined by EDAX. A nonhomogeneous distribution of alloy elements along the specimen surface may have influenced the results. Changing oxidation conditions can influence the type of oxide that is formed. Oxidation in air instead of vacuum can lead to thick oxide layers.

McLean recommended at least 50% new alloy be included in copings for metal ceramic restorations [16]. Dental laboratories reusing casting alloys with random amounts of new metal additions is contraindicated. Good restoration requires certain optimum properties which should remain constant in oral environment. Hence recasting should not be done at the expense of the properties of the alloy.

The small decrease in the concentration of Ni and Cr probably would have no clinically significant effect on the mechanical properties or corrosion resistance of the cast alloys. To monitor the reuse of alloys, laboratories should institute effective approaches to metal handling and accounting. Further means to facilitate reclamation of the used alloy in an efficient, expeditious, and economic manner by the manufacturer would encourage laboratories to return the large, unmanageable buttons that tend to accumulate.

5. CONCLUSION

From the present study following conclusions can be drawn.-

Ni Cr Base metal alloys can be used for recasting provided strict adherence to clean casting techniques were to be carried out.

The recurrent casting of alloys may cause surface impairment and compositional deviations in alloys that may further induce organic hazards during their clinical uses.

The very low change ratio of the elements affect the physical and biological properties of the alloy. These changes may have effect on the bond strength of different veneering materials.

COMPETING INTERESTS

Authors have declared that no competing interests exist.

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