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ARTICLE

THERMAL EXPANSION AND CORROSION RESISTANCE OF COBALT-CHROMIUM ALLOYS FABRICATED BY CONTEMPORARY MANUFACTURING PROCESSES. AN *IN VITRO* STUDY.

Expansión térmica y resistencia a la corrosión de aleaciones de cobalto-cromo fabricadas mediante procesos de abricación contemporáneos: un estudio *in vitro.*

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ABSTRACT:

In Purpose: The fabrication technique can influence the mechanical properties of Cobalt-Chromium (Co-Cr) dental alloys. Hence, the present study aims to determine the corrosion resistance and thermal expansion of alloys manufactured us ing three contemporary techniques.

Material and Methods: A total of nine specimens of Co-Cr alloy were prepared according to ISO 22674 by each one of the three manufacturing processes (three in each process); conventional casting, direct metal laser sintering (DMLS) and milling (MIL). All these specimens were tested for coefficient of thermal expansion and corrosion resistance. The data was tabulated and analyzed statistically.

Results: The difference in the thermal expansion of alloys fabricated using three techniques was non-significant at almost all the temperatures from 50ºC to 950ºC ($p>0.05$), except 450ºC and 600°C. The polarization resistance of specimens manufactured using the conventional method was more compared to DMLS and MIL at pH 5 (Conventional>MIL>DMLS) ($p<0.001$).

Conclusion: The thermal expansion behavior of alloys manufactured using the three selected techniques were similar, whereas, at acidic pH, the corrosion resistance of conventional and MIL were better than the DMLS.

KEYWORDS:

Chromium alloys; corrosion; temperature; surface properties; lasers; dental alloys.

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RESUMEN:

Antecedentes: La técnica de fabricación puede influir en las propiedades mecánicas de las aleaciones dentales de cobalto-cromo (Co-Cr). Por lo tanto, el presente estudio tiene como objetivo determinar la resistencia a la corrosión y la expansión térmica de aleaciones fabricadas con tres técnicas contemporáneas.

Material y Métodos: Se prepararon un total de nueve probetas de aleación de Co-Cr según ISO 22674 por cada uno de los tres procesos de fabricación (tres en cada proceso); fundición convencional, sinterización directa de metal por láser (DMLS) y fresado (MIL). Todos estos especímenes fueron probados para determinar el coeficiente de expansión térmica y la resistencia a la corrosión. Los datos fueron tabulados y analizados estadísticamente.

Resultados: La diferencia en la dilatación térmica de las aleaciones fabricadas con las tres técnicas no fue significativa en casi todas las temperaturas desde 50ºC hasta 950ºC (p>0,05), excepto 450ºC y 600ºC. La resistencia a la polarización de las muestras fabricadas con el método convencional fue mayor en comparación con DMLS y MIL a pH 5 (Convencional>MIL>DMLS) (p<0,001).

Conclusión: El comportamiento de expansión térmica de las aleaciones fabricadas con las tres técnicas seleccionadas fue similar, mientras que, a pH ácido, la resistencia a la corrosión de la convencional y la MIL fue mejor que la de la DMLS.

PALABRAS CLAVE:

Aleaciones de Cromo; corrosión; temperatura; propiedades de superficie; lasers; aleaciones dentales.

INTRODUCTION.

In Prosthodontics, cobalt-chromium (Co-Cr) alloys have been widely used for the fabrication of removable and fixed restorations.[1-5] The use of base metal alloys is of great significance, despite the development of all ceramic alternatives. The Co-Cr alloys form an inexpensive alternative to the traditional gold alloys, as these are durable, rigid with high strength, modulus of elasticity and degree of corrosion resistance facilitating the dental use.[1,6-7] Additionally, these alloys do not require the addition of precious metal. The Co-Cr alloys are biocompatible, as the nickel and beryllium, which are allergic and carcinogenic, do not form the elemental composition of this material.[3,5,8,9]

These alloys are processed in many ways; the commonly employed method is the traditional casting using the lost wax technique. Over time, computer-aided design/computer-assisted manufacturing (CAD/CAM) has revolutionized the den-tal manufacturing processes.

The computerized processing minimizes the flaws possible in manual casting.[5,10,11] Subtractive and additive manufacturing are the two approaches based on computer processing.[3,12] One of the additive manufacturing techniques, assisted by CAD/CAM technology, is the Direct Metal Laser Sintering (DMLS) wherein fine layers of metal powder will be fused using a high-power focused laser beam.[8,12-14]

The subtractive technique, based on CAD/CAM technology, is milling (MIL) in which the alloy block is milled for the desired shape. These manufacturing techniques influence the microstructure of the restoration, as reported in the literature,[1,15] because of the alterations in chemical composition and mechanical properties.

However, there is only limited data on the mechanical properties of Co-Cr alloys manufactured by these techniques. In the literature, there are studies which have assessed properties like proof strength, elon-gation after fracture, and young's modulus of cobalt chromium alloys.[3,16-18]

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The influence of the manufacturing technique on the mechanical properties, ion release, toxicity of released elements and surface roughness has been considered in recent studies on cobalt chromium.[19-23]

However, the parameters like corrosion resistance and thermal expansion though important in the long-term survival of restorations have not been considered till now. Hence, the present study was planned to assess the thermal expansion and corrosion resistance of Co-Cr alloy fabricated using DMLS and MIL and compared these with the conventional casting alloy.

MATERIALS AND METHODS.

A total of nine specimens (three in each process) of Co-Cr alloy were prepared according to ISO 22674 by each one of the three manufacturing processes; conventional casting, direct metal laser sintering (DMLS), and milling (MIL).

The specimens were dumbbell-shaped, the dimensions of which are demonstrated in Figure 1. For each technique, the corresponding Co-Cr alloy material was used, and the elemental composition (Table 1).

Fabrication of Co-Cr specimen using the conventional method

The specimens were fabricated by the traditional casting using Co-Cr pellets (BEGO company®, Germany). Prefabricated wax patterns (BEGO company®, Germany) were invested with phosphate bonded investment material (Wirovest®, BEGO, Germany), and the mold was preheated at 910°C (degree Celsius) and cast with alloy at 1450°C using induction casting machine (Fornax® T company). The mold was left to cool down to room temperature, and the specimens were then divested and cleaned by sandblasting with alumina particles of size 110µm.

Fabrication of Co-Cr specimens using DMLS technique

The wax pattern was scanned with the Ceramill scanner (Ceramill® Map 200+, Germany).

The specimens were fabricated from commercially available Co-Cr powder and a binder containing soft blanks (Ceramill Sintron®, Germany). Then, laser sintering was done for the dumbbell-shaped specimen using a computer-aided manufacturing machine (Ceramill Therm 3, Germany).

Fabrication of Co-Cr specimens using milling technique

A digital scanner (Medit Hybrid Dental Scanner, Itero®, South Korea) was used to scan the prefabricated wax pattern. Cobalt-Chromium alloy blocks were milled for the desired shape using the milling unit (VHF K4 milling machine).

All these specimens were tested for coefficient of thermal expansion and corrosion resistance.

Assessment of coefficient of thermal expansion

The coefficient of thermal expansion was tested using a dilatometer (connecting rod type). The furnace was set to reach from room temperature to 950 ˚C at a rate of 10 ˚C per minute. The readings of dial gauge were recorded for each 50 ˚C interval. The percentage of linear thermal expansion was obtained from the following equation:

Linear thermal expansion=Change in length ÷ (Original length×100%)

Change in length= Reading of the dial gauge

Assessment of corrosion resistance

The corrosion resistance was tested using an electrochemical potentiostat. The methodology, as reported by Zeng *et al.*,[9] was used to test the corrosion resistance of metals.

The pH value of 5.0 and 7.5 was measured with a glass electrode connected to a pH meter. All these specimens were stored in artificial saliva consisting of NaCl (400mg/l), KCl (400mg/l), CaCl2-2H2O (795mg/l), NaH2PO4 (690mg/l), KSCN (300mg/l), NaS.9H20 (5mg/l) and urea (1000mg/l) which was chosen as the electrolyte to simulate clinical conditions. The corrosion current and polarization resistance were determined from the acquired polarization curves.

Statistical analysis

The statistical analysis was carried out using SPSS 17.0 version for Windows (Chicago, Ill, USA). The normality of the data was analyzed using the Shapiro-Wilk test, and the level of significance was

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set at 0.05 level. The difference in the linear thermal expansion of the Co-Cr alloys among the three fabrication techniques was analyzed using One-way ANOVA and Bonferroni tests. The difference in the polarization resistance and corrosion current of Co-Cr alloys among the three fabrication techniques was also analyzed using One-way ANOVA and Bonferroni tests.

RESULTS.

Thermal expansion

The linear thermal expansion of Co-Cr alloys fabricated using DMLS, MIL, and conventional techniques are represented in Figure 2. The mean linear thermal expansion values were more in the alloy fabricated using conventional methods compared to DMLS and MIL at the considered

Figure 1. Dimensions of the dumbbell shaped specimen used in the present study.

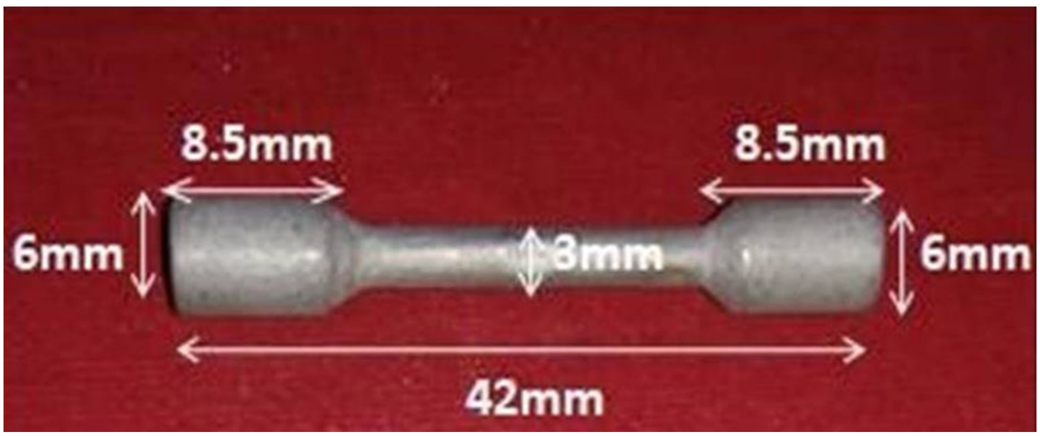

Figure 2. Linear thermal expansion of cobalt chromium alloys fabricated using different techniques.

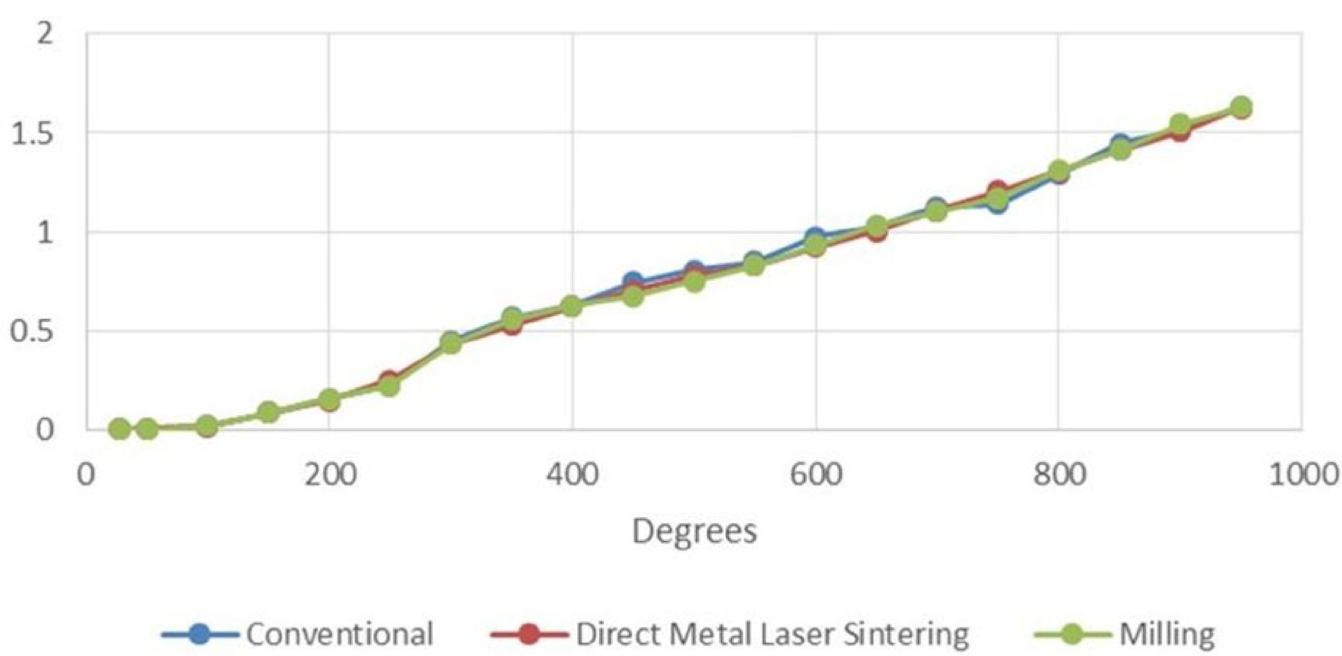

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Figure 3. Polarization resistance of cobalt chromium alloys fabricated using different techniques, at pH 5.0 and 7.5

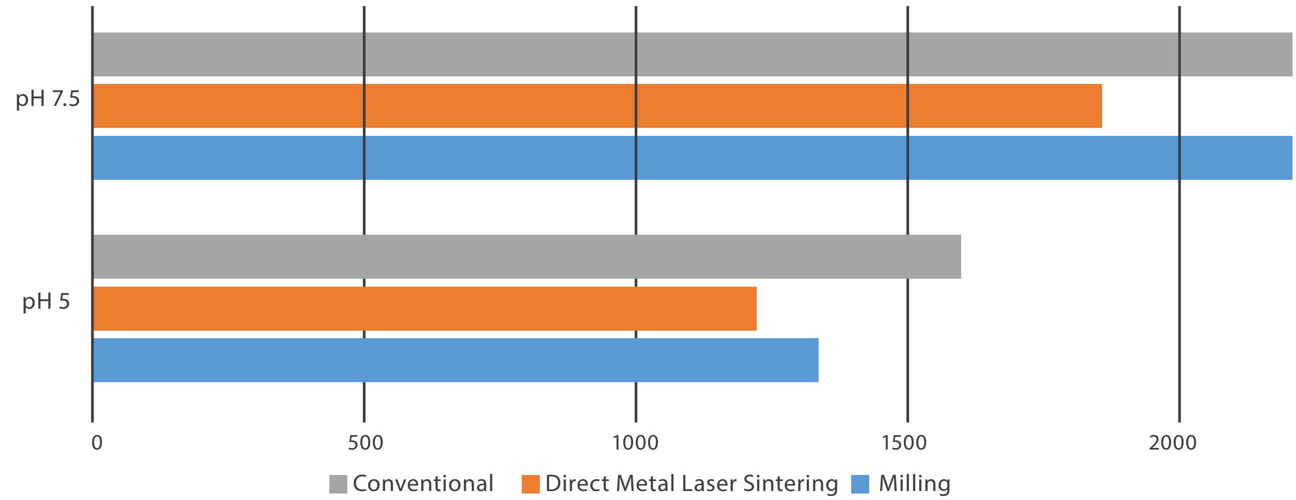

Figure 4. Corrosion current of cobalt chromium alloys fabricated using different techniques, at pH 5.0 and 7.5.

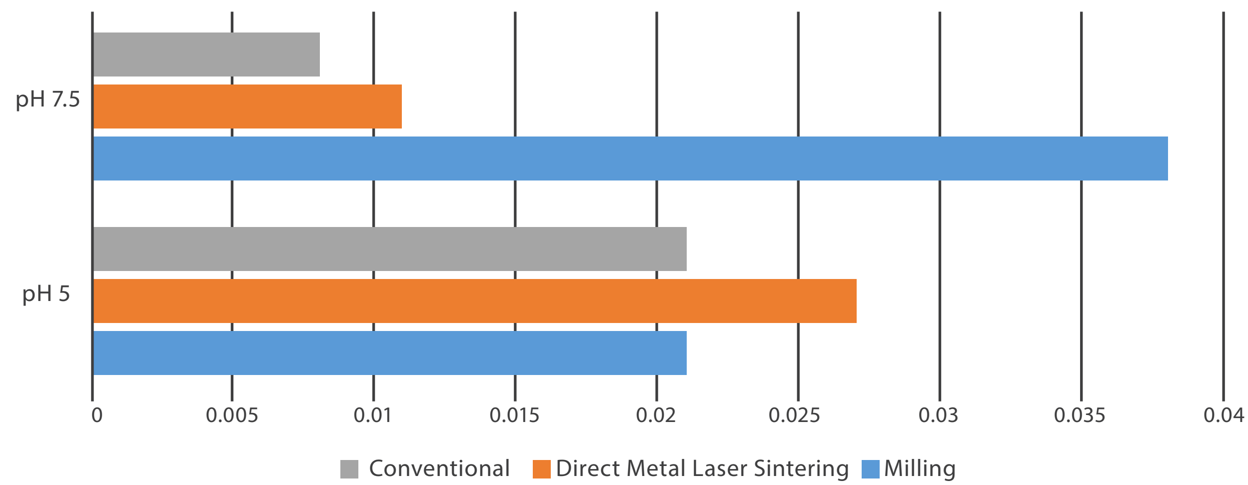

Table 1. Elemental composition of the Cobalt-Chromium alloy materials used in the present study.

Direct Metal Laser Sintering	MILLING	Conventional
Co-66%	Co-63%	Co-64%
Cr-28%	Cr-28%	Cr-28.5%
Mo-5%	W-3%	Mo-5%
Si<1	Nb-4%	Si-1%
Fe<1	V-N/A	Mn-1.0c
Mn<1	Mo-<1	
Further element	Si-1%	
C<0.1	Fe-<1	
Organic binder		

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Table 2. Linear thermal expansion of the Cobalt-Chromium alloys fabricated using contemporary processes.

Temperature (in °C)	DMLS (Mean±SD)	MIL (Mean±SD)	Conventional (Mean±SD)	*p*-value	Temperature (in °C)	DMLS (Mean±SD)	MIL (Mean±SD)	Conventional (Mean±SD)	*p*-value
27	0.00±0.00	0.00±0.00	0.00±0.00	---	500	0.776±0.025	0.749±0.012	0.803±0.034	0.1^{NS}
50	0.002±0.001	0.001±0.001	0.002±0.001	0.58^{NS}	550	0.829±0.016	0.830±0.013	0.845±0.011	0.34^{NS}
100	0.014±0.003	0.016±0.006	0.018±0.004	0.65^{NS}	600	0.925±0.022	0.930±0.017	0.974±0.017	0.03*
150	0.084±0.007	0.085±0.008	0.083±0.009	0.93^{NS}	650	1.001±0.004	1.030±0.016	1.025±0.015	0.07^{NS}
200	0.147±0.005	0.154±0.010	0.155±0.007	0.46^{NS}	700	1.106±0.030	1.101±0.034	1.125±0.034	0.66^{NS}
250	0.249±0.022	0.219±0.029	0.224±0.025	0.38^{NS}	750	1.206±0.032	1.172±0.052	1.141±0.041	0.25^{NS}
300	0.432±0.007	0.432±0.014	0.447±0.011	0.23^{NS}	800	1.306±0.052	1.307±0.057	1.291±0.056	0.93^{NS}
350	0.529±0.013	0.556±0.025	0.563±0.023	0.19^{NS}	850	1.413±0.027	1.413±0.044	1.448±0.037	0.46^{NS}
400	0.623±0.012	0.627±0.020	0.628±0.014	0.91^{NS}	900	1.507±0.035	1.544±0.052	1.511±0.044	0.57^{NS}
450	0.701±0.015	0.674±0.009	0.744±0.022	0.005**	950	1.624±0.016	1.631±0.031	1.632±0.050	0.96^{NS}

Post hoc test - *p*-value Bonferroni test

Temperature - 450 (in ºC)	DMLS *versus* MIL	DMLS *versus* Conventional	MIL *versus* Conventional	Temperature - 600 (in ºC)	DMLS *versus* MIL	DMLS *versus* Conventional	MIL *versus* Conventional
0.26NS	0.06^{NS}	**0.006****		1NS	**0.05***	0.08^{NS}	

°C: Degree Celsius. **DMLS:** Direct Metal Laser Sintering. **MIL:** Milling. **NS:** Non-significant. **SD:** Standard Deviation. *****: Level of significance 0.05. ******: Level of significance 0.01; #: One-way ANOVA

Table 3. Polarization resistance and corrosion current of the Cobalt-Chromium alloys fabricated using contemporary processes.

Polarization resistance (KΩcm³)		DMLS (Mean±SD)	MIL (Mean±SD)	Conventional (Mean±SD)	*p*-value
	pH-5	1227±17.58	1344.33±7.1	1605.67±23.54	≤0.001***
	pH-7.5	1863.7±7.1	2185.33±7.23	2217.33±49.44	≤0.001***
Post hoc test $	pH-5		DMLS *versus* MIL		0.001***
			DMLS *versus* Conventional		≤0.001***
			MIL *versus* Conventional		≤0.001***
	pH-7.5		DMLS *versus* Conventional		≤0.001***
			MIL *versus* Conventional		0.68^{NS}
Corrosion current (μAcm^{-2})		**DMLS (Mean±SD)**	**MIL (Mean±SD)**	**Conventional (Mean±SD)**	***p*-value**
	pH-5	0.0272±0.001	0.0207±0.001	0.0207±0.001	≤0.001***
	pH-7.5	0.011±0.001	0.038±0.05	0.008±0.0008	0.43NS
Post hoc test $	pH-5		DMLS *versus* MIL		≤0.001***
			DMLS *versus* Conventional		≤0.001***
			MIL *versus* Conventional		≤0.001***
	pH-7.5		DMLS *versus* MIL		0.87NS
			DMLS *versus* Conventional		1NS
			MIL *versus* Conventional		0.76NS

pH: Potential of hydrogen. **DMLS:** Direct Metal Laser Sintering. **MIL:** Milling. **NS:** Non-significant. **SD:** Standard Deviation. *****: Level of significance 0.05. *******: Level of significance 0.001. **#:** One-way ANOVA. **$:** Bonferroni test.

temperatures (Table 2). However, the difference in the thermal expansion of alloys manufactured using three techniques was non-significant at almost all the temperatures from 50°C to 950°C, except 450°C and 600°C.

When the post hoc comparisons at these temperatures were observed, only the difference between MIL and conventional techniques was significant at 450°C with MIL showing less thermal expansion. On the other hand, at 600°C, the difference was substantial between DMLS and conventional with conventional showing higher values.

Polarization resistance and corrosion current

The polarization resistance of Co-Cr alloys fabricated using DMLS, MIL, and conventional techniques are represented in Figure 3. The mean polarization resistance of alloy made using a conventional method was more similar to DMLS and MIL at pH 5.0 (Conventional>MIL>DMLS). The difference in the resistance among the groups at this pH was statistically significant ($p \leq 0.001$).

When the post hoc comparisons were observed, the difference between DMLS and MIL, DMLS, and conventional, as well as MIL and conventional, were statistically significant. The mean polarization resistance of alloy fabricated using the conventional technique was more compared to DMLS and MIL at pH 7.5 (Conventional>MIL>DMLS). The difference in the resistance among the groups at this pH was statistically significant ($p \leq 0.001$). When the post hoc comparisons were observed, the difference between DMLS and MIL, DMLS, and conventional were statistically significant; on the other hand, the difference between MIL and conventional was non-significant. (Table 3)

The corrosion current of Co-Cr alloys fabricated using DMLS, MIL, and conventional techniques are also represented in Figure 4. The corrosion current of alloy made using the DMLS technique was more compared to MIL and conventional at pH 5.0 (DMLS>MIL>Conventional). The difference between the three groups was statistically significant ($p \leq 0.001$).

When the post hoc comparisons were observed, the difference between DMLS and MIL, DMLS, and conventional, as well as MIL and conventional, were statistically significant ($p \leq 0.001$). The corrosion current of alloy fabricated using MIL technique was more compared to DMLS and conventional at pH 7.5 (MIL>DMLS>Conventional). The difference between the three groups was not statistically significant ($p=0.43$). (Table 3)

DISCUSSION.

The prosthodontic laboratory work is intensive and demands experience. For improving the sophistication and precision, computer-aided design/ computer-aided manufacture (CAD/CAM) has been introduced.[24] The usage of this tech-nology is increasing day by day and has replaced the manual laboratory steps.[25]

The computer technology is playing an important role and is offering new possibilities for the production of prosthodontic castings and frameworks. Two CAD/CAM procedures considered in the present study are the MIL and DMLS. For MIL, blanks manufactured under standardized industrial conditions will be employed.[26-30] Hence, the advantage of this procedure is the possibility of avoiding flaws and porosities that can degrade the quality of the casting.[31,32] The DMLS is another CAD/CAM technique that involves sintering using a high energy laser beam. The milling of the soft metal, as done in the DMLS group, is reported to have several advantages over conventional casting, concerning elongation, proof strength making the material more robust.[8,33]

Metal alloys are employed for porcelain fused to metal prostheses. These metal alloys should exhibit the strength and toughness required for veneering the porcelain.[34] The dental alloy should also be bio-functional and biocompatible. The base metal alloys are preferable to noble alloys, because of its elastic modulus, fracture hardness, and low cost; but, their susceptibility to corrosion limits the use of the base metal alloys to corrosion. The properties of the metals depend not only on

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the elemental composition but also on the changes that the alloy undergoes during the fabrication procedure. The microstructure and the physical properties of the alloy can be influenced by the fabrication method of the alloy. Also, during the process, the alloys change the micro-structural phases, grain size and surface morphology, which in turn can influence the mechanical properties.[16]

Any alloy in the oral atmosphere is susceptible to continuous corrosion process, the products released from which can cause a wide range of symptoms from local tissue reactions to carcinogenicity and mutagenicity.[35,36] The corrosion resistance of an alloy depends on the presence of a surface oxide layer, which acts as a barrier to electron flow between the surface of the alloy and the electrolyte.[37,38] The polarization resistance characterizes the protection ability of the oxide film on the alloy surface. The effective *in vitro* approach to evaluate the corrosion behavior is the electrochemical tests.

As the heat treatment influences the corrosion behavior of an alloy, it has been suggested in the literature to evaluate the corrosion behavior after the ceramic firing process.[9] The effect of heat treatment has been attributed to the difference in the passive oxide films on the alloy surface, which is responsible for the resistance to corrosion of alloys. It has also been reported that pH conditions can influence the corrosion process.[9] Hence, an acidic pH of 5 was selected to understand metal behavior, which was compared to an alkaline pH of 7.5. A study was done to know the corrosion resistance of alloy manufactured using selective laser melting has selected a pH of 2.5, apart from 5.9 As this is far below the pH of normal saliva, in the present study only pH of 5.0 was selected and compared to an alkaline pH of 7.5.

The polarization resistance and corrosion current represent the ability of an alloy to resist corrosion, and they are inversely proportional.[9] The maximum anodic current density in the oral cavity is 2μAcm^{-2}.[39] The highest current value in the present study was 0.038μAcm^{-2}, and the lowest was 0.0082μAcm^{-2}. The lower values of the current indicate that all the alloys irrespective of manufacturing technique can resist corrosion degradation. The polarization resistance required for the material to be bio-functional in the oral cavity is 103KΩcm^3.[9]

The mean polarization resistance of the alloys in the present study is 1392KΩcm^3 at pH 5, which is higher than the required values. The values reported implying the ability of the alloys to combat the corrosion due to temperature change and the acidic atmosphere. Therefore, the alloys manufactured using conventional/MIL/DMLS are adequate for clinical use.

The thermal expansion and contraction of an alloy depend on the melting temperature of the alloy. In order to withstand the fusing temperature of the porcelain, the dental alloy should have a high melting temperature. However, the drawback of the high melting temperature is that there can be more significant thermal contraction because of the extended cooling to the room temperature.[4]

Therefore, it is essential to determine the coefficient of thermal expansion of alloys used for porcelain fusing. The present findings highlight the fact that there was no influence of the manufacturing technique on the coefficient of thermal expansion of the alloy selected. Thus, there cannot be any influence of the manufacturing technique on the metal-ceramic bond. Similar findings have been reported in a study done to investigate the fracture strength of porcelain fused metal crowns based on Co-Cr produced using the techniques, conventional casting, CAD/CAM milling, and CAD/CAM DMLS. However, the strength was in the order, milled followed by conventional and then DMLS specimens.[40]

Based on the methodology followed in the present study, the coefficient of thermal expansion and corrosion resistance of the Co-Cr alloys fabricated using DMLS, MIL, and conventional casting are within the clinical range. However, further studies on the microstructure and chemical composition of the surface layer are needed to complete the metallurgic properties of these alloys manufactured using different techniques. Even studies on ion release and x

CONCLUSION.

Within the limitations of the present *in vitro* study, the following conclusions can be drawn:

-There was no significant difference in the thermal expansion among the three Co-Cr alloys in the considered temperature range of 27°C to 950°C, except 450°C and 600°C.

-At 450°C, there was a significant difference between MIL and conventional groups, whereas, at 600°C, there was a substantial difference between MIL and conventional, with conventional showing more expansion.

-There was a significant difference in the polarization resistance among the three Co-Cr alloys in both the test solutions (pH 5.0 and pH7.5).

-At pH 5.0, the polarization resistance was in the order, conventional>MIL>DMLS, whereas at pH 7.5, the polarization resistance was in the order, conventional> DMLS>MIL.

-There was a significant difference in the corrosion current among the three Co-Cr alloys at pH 5.0, conventional=MIL<DMLS.

Hence, the thermal expansion behavior of alloys manufactured using the three selected techniques were similar. In contrast, at acidic pH, the corrosion resistance of conventional and MIL were better than the DMLS.

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The authors declare that they have no conflict of interest.

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Concept and Design: Vinnakota DN.

Acquisition of data: Balaji B.

Analysis and interpretation of data: Vinnakota DN.

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