COMPARATIVE EVALUATION OF CORROSIVE BEHAVIOR OF FOUR COMMERCIALLY AVAILABLE NICKEL – CHROMIUM ALLOYS IN ARTIFICIAL SALIVA BY CYLCIC POLARISATION TEST

- AN INVITRO STUDY

Dissertation submitted to

THE TAMILNADU Dr .M.G.R MEDICAL UNIVERSITY

In partial fulfillment for the degree of

MASTER OF DENTAL SURGERY

BRANCH VI PROSTHETIC DENTISTRY

MARCH 2007

CERTIFICATE

Certified that the dissertation on the **"COMPARATIVE EVALUATION OF CORROSIVE BEHAVIOR OF FOUR COMMERCIALLY AVAILABLE NICKEL – CHROMUIM ALLOYS IN ARTIFICIAL SALIVA BY CYLCIC POLARISATION TEST- AN INVITRO STUDY"** done by **Dr .Rubina Ibrahim** Post graduation student [MDS], **Branch VI Prosthetic Dentistry** ,Saveetha Dental College and Hospitals ,Chennai submitted to The Tamilnadu Dr.M.G.R Medical University in partial fulfillment for the M.D.S degree examination in March 2007,is a bonafied dissertation work done under my guidance and supervision.

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ACKNOWLEDGEMENT

Firstly I express my gratitude to the Almighty God for His blessings in making things right for me.

I first take an opportunity to express my sincere and heartfelt thanks to my esteemed teacher **Dr.R.Haribabu MDS**, **Professor** and **Head of the Department of Prosthodontics**, Saveetha Dental College and Hospitals, Chennai for his profound knowledge ,sterling encouragement , valuable guidance and constant support during the course of this study and also through out my post graduate course.

I gratefully acknowledge **Prof.N.D.Jayakumar ,Principal**, Saveetha Dental College, **Prof .A.Venkatesan ,Dean** of Dental Faculty , ,Saveetha Dental College **Prof .M.F.Baig Dean** , **Dr. R.Rajagpopal, Vice Chancellor**, Saveetha Dental College **Dr.N.M.Veeraiyan, Chancellor**, Saveetha Institute of Medical and Technical Sciences and for giving me an opportunity to be a part of this esteemed institution.

I would like to thank **Prof Dr.E.G.R Solomon, Prof Dr.H.Annapoorani** and **Prof Dr.Fiaz-ur Rahman** for their encouragement, invaluable guidance and helpful nature during my course.

I would like to extend my deepest gratitude and sincere thanks to my **Guide** and **Associate Professor Dr.Padma Ariga** for her keen surveillance, constant encouragement and timely suggestion and pain staking effort for perfection that helped to complete my dissertation ,through out the course and this study.

I extend my thanks to **Dr.M.Dhanraj MDS, Dr. Jafar Abdulla MDS, Dr.Mohammed Behanam M.D.S** for their timely suggestion through out my post graduation course and in this study.

Special thanks to **Dr. Deepak Nallaswamy** for his fine gesture in providing all facilities, valuable suggestion and guidance during my post graduation course..

I am extremely grateful to **Dr.Rajeswari, PhD**, Prof and Head of the Dept of Analytical Chemistry, Madras University, Guindy Campus for permitting me to utilize the facilities of corrosion study required during this study.

I express my heartfelt thanks to **Dr .Prabhakaran,** Research Scholar, Dept of Analytical Chemistry for his timely help and support in shaping this study.

I thank **Mr.A.K.Mathai,** statistician for helping me meticulously with the analysis of the statistical data.

I thank **Dr. Manimegalai** , chief librarian for providing me required books during the course of this study.

I am also thankful to the **Teaching** and **Non-teaching staffs**, who had been behind the scene have helped me during the course.

I express my intense thanks and indebt ness to my **parents, in-laws**, **sister** and my husband **Mohammed Ibrahim** who gave me the moral support all through my life, timely suggestions and took lots of pain to raise me to this level.

I am extremely thankful to all **Batch mates, seniors, Co-PGS** and **friends** who helped me in every aspect for the completion of this endeavor successfully.

CONTENTS

Dental casting alloys made of high noble alloys, noble alloys and predominantly base metal alloys have been used in the fabrication of fixed and removable prosthetic dental appliances. The advantages of high noble and noble alloys include high resistance to tarnish and corrosion and biocompatibility[1] and the major drawback of these alloys are their increased cost. To overcome these drawback , base metal alloys such as Cobalt-Chromium [Co-Cr] and Nickel –Chromium [Ni-Cr], have been widely used in the fabrication of fixed and removable partial denture frameworks owing to superior physical properties like high modiolus of elasticity , thermal coefficient of thermal expansion matching with porcelain , high melting temperature withstanding high firing temperature without producing cracks[2] and lower cost ,but their resistance to tarnish and corrosion is debatable.

One of the most important factors affecting the choice of dental alloys is its biocompatibility and resistance to tarnish and corrosion. **Tarnish** is defined as a "surface discoloration of the metal or as a slight loss or alteration of the surface finishes or luster"[3]. **Corrosion** defined as "the action, process, or effect of corroding; a product of corroding; the loss of elemental constituents to the adjacent environment"[4]. The metals and alloys which are to be used in oral cavity should withstand the moisture, temperature and pH changes, which occur during the breakdown of foods. Acid or alkaline solutions and certain chemicals and most of the food stuff show pH below 7 which may accelerate the corrosion process [5]. Tarnish is the forerunner of corrosion because a thin film that produces tarnish may in time accumulate compounds that chemically attack the metallic surface[4.]

The corrosion resistance of an alloy is a highly important consideration in the success of prosthesis as it can lead to poor esthetics, compromised physical properties and release of significant amount of corrosion products from the alloys adversely affecting the biocompatibility of an alloy[6]. Corrosion is clearly related to biocompatibility of an alloy.

The release of metallic elements from dental alloys during corrosion into the oral cavity is of continuing concern because of the harmful biologic effects that these elements may have on oral tissues and it is a potential health problem to the dental patient[6, 7, 8, 9]. In sufficient concentration, metals are known to cause toxic, inflammatory, allergic or mutagenic reactions[10]. The elements that are released from dental alloys can be detected in tongue scrapings and in saliva[11] and gingiva adjacent to these alloys [12, 13.] The release of Nickel from some dental alloys may approach the daily dietary intake of these elements[14].

The corrosion property of metals and alloys depends upon their composition, their electrode potential, to stress over the metals and to the surface roughness. In addition, it also depends upon specific characteristics of different oral environment [saliva, dental plaque bacteria, gastric acid reflux] and the acidity, oxidation level, temperature, velocity of mixing and the inhibitors of the media[15, 16, 17].

The corrosion rate of metals and alloys can be determined using various chemical and electrochemical methods[18.] The chemical methods are by the mass loss of corroding metal and alloy, amount of corrosion products in corrosive media, by the amount of gas produced during corrosive reaction. The electrochemical methods are potentiostatic and potentiodyanamic polarization tests in various solutions.

Base metal alloys usually have multiphase structures and contain several metals that are known to be biologically active and hence release of elements may occur that might cause various cytotoxic effects[19,20] .The cytotoxicity is generally assessed by measuring certain type of cellular activity such as morphology, viability, hemolysis of human red blood cells and the membrane status[21]

With the advent of new dental alloys in the market, the biocompatibility of dental materials is of critical concern and the evaluation of corrosive behavior of new dental alloys is highly warranted.

The aim of this study is:

1. To evaluate and compare the corrosive behavior of four commercially available Nickel Chromium alloys with that of a high noble alloy as the control group in artificial saliva by cyclic polarization test.
2. To analyze and authenticate the element released from four commercially available Nickel Chromium alloys by inductively coupled plasma –mass spectrometer.

Fusayamaet al, (1963)[22], had done a study to determine whether the contact of gold with amalgam produced any significant corrosion on those metals by immersion in artificial saliva and chemical reaction were determined by various acids and concluded that contact of gold inlays with amalgam restoration produced silver stains on gold inlays.

J.Brugirard et al, (1973)[23], evaluated the electrochemical behavior of gold dental alloys in artificial saliva by polarization method and concluded that the high carat gold alloys gives a rapid indication of corrosion tendency when placed in artificial saliva with zinc and cadmium seems to be the weakest alloy.

Ronald D. et al , (1977)[21], had done a study to determine the cytotoxic potential of base metal alloys in cell culture medium containing L-929 mouse fibroblast and examined microscopically and concluded that the cultures containing Nickel Chromium powders showed prominent zones of cell lysis and cell alterations.

N.K. Sarkar et al, (1979)[24,] conducted a study to evaluate the corrosion and tarnish resistance of various dental alloys and to characterize the chloride corrosion behavior of low gold casting alloys by Potentiodynamic polarization, Controlled potential polarization, Reverse polarization and concluded that ‘low-gold’ casting alloys are characterized by decreases chloride corrosion resistance.

David C. Wright et al, (1981)[25]had developed a reliable potentiodynamic technique to examine Copper and silver corrosion resistance, Twenty-three composition were formulated from 99.95% pure Au, Ag, and Cu. The samples were prepared and corrosion study was done by potentiodynamic technique and concluded that copper and silver has characteristic potentials for which individual current densities are maximal. Microstructure as well as alloys chemistry plays a major role in determining corrosion resistance.

T.K. Vaidayanathan et al, (1981)[26], studied the invitro corrosion and tarnish of Ag- Pd binary system by polarization in ringers lactate and immersion in .5% in Na2S and concluded that there is wide range of protection and repassivation tendencies when Pd is alloyed with Ag.

D.L.Johnson et al, (1983)[27] had evaluated a wide composition range of commercially available dental; casting alloys containing varying amounts of

Au, Ag, Pd, In, Cu and Zn in relation to corrosion resistant and also to screen the effect of casting temperature and hold time on corrosion resistance by potentiodynamic scans on 11 alloys of varying Ag-Au-Pd content and concluded that high Au alloys are highly corrosion-resistant.

G. Baran et al, (1984)[28], had done a study to determine the oxides formed on four Ni-Cr dental casting alloys in three temperature domains and in two different atmospheres were chemically analyzed using scanning Auger MicroProbe. Distribution of Ni and Cr in the oxide layers varied with the alloy; oxidation in air resulted in apparently thicker oxide then oxidation in a reduce oxygen atmosphere.

L. Niemi et al, (1986)[29], had done a study to determine the interaction between the uppermost surface layers of Ag-Pd-Cu-Au based casting alloys and artificial saliva. Three commercially available Ag-Pd-Cu-Au were taken and potentiodynamic scan was done and the concentration of Ag, Cu and Pd in the artificial saliva solution were determined by atomic absorption spectrometry and concluded that Cu was found to dissolve considerably from the Cu-Pd-rich alloy, with simultaneous enrichment of Pd in the surface layer of the alloy.

R.G. Craig et al, (1990)[30], had determined the cytotoxicity of a series of 29 experimental alloys and six pure was determined with cell culture techniques and succinic dehydrogenase histochemistry Cell culture testing with Balb/c 3T3 cells and cytotoxicity of the alloys and pure metals was evaluated and concluded that Au, Pd and Ti were least cytotoxic, followed by Ag, then Ni and finally Cu.

J.C. Wataha et al, (1991)[31]**,** had done a study to determine the amount of alloys elements released from ten of selected casting alloys under standard cell culture conditions and to relate this composition and microstructure of the alloys which in turn relate to the toxicity of the alloys. The elemental release was done by flame atomic absorption spectrophotometer and concluded that Au, In, and Pd generally did not dissolve into the medium but Ag, Cd, Cu, Ga, Ni and Zn are frequently released.

Vaidyanathan TK et al, (1991)[32]**,** had done a study to investigate the co-relation of micro organism and tarnish of five dental alloy, on exposure to blood and chocolate media with and without inoculated microorganisms and concluded that there is a potential role for the oral microorganisms in inducing clinically observed tarnish of dental alloys. Actinomyces viscosus and periodontal pocket specimens show a similarity in their activity to induce tarnish in base metal-containing dental alloys.

J.C. Wataha et al, (1992)[33], had done a study to determine the in vitro kinetic patterns of release of elements from six types of dental casting alloys and also to determine the effect of the alloy cleaning procedure on the release of the elements.. The elemental releases were analyzed by flame atomic absorption and concluded that cleaning does not change the pattern of release but significantly decrease the quantities of element released. The Augur analysis of alloys surfaces after exposure to medium showed the presence of organic films up to 50nm thick.

Herrmann M et al, (1992)[34], had analyzed the in vitro resistance to fracture of the porcelain-fused-to-metal restoration of one palladium and five base metal alloys by three-point bending test for the crack resistance measurement and concluded that the Corrosive components of the oral environment and the details of firing were of crucial importance for long-term bond stability.

Mjor IA et al, (1993)[35], had done a retrospective survey to assess side effects of alloys used in fixed and removable partial prosthodontics and reported that the gingiva and oral mucosa adjacent to the restorations were normal but showed slight changes in all groups.. The soft tissue reactions were considered to be largely due to factors other than the metal components.

J.C Wataha et al, (1995)[36], studied the correlation and cytotoxicity and elements released by dental casting alloys by atomic absorption spectroscopy and cellular mitochondrial function, and concluded that the high noble alloys released low levels of elements , whereas other alloys released elements higher levels causing cytotoxic effects.

J.C Wataha et al, (1996)[37], stated the biological effects of palladium and said that the ionic form of palladium at sufficiently high concentration has toxic and allergic effect in biologic system. In spite of potential adverse biological effects, the risk of using it is low because of low dissolution rates.

Mulders et al , (1996)[38], conducted a study to determine the effect of composition and casting process on corrosion rates on palladium based alloys and nickel based alloys by electrochemical method and concluded that casting process does not influences the corrosion process but the changes in crystallographic structure effected by change in composition influences the corrosion processes.

J.C Wataha et al, (1996)[39], studied the correlation of elemental release and surface composition and to determine the depth of the effect of the medium on three types of alloys by atomic absorption spectroscopy and auger milling, and concluded that the depth of effect of the medium varied with the alloy and high gold alloy appeared to develop the most stable surface composition which released the lowest levels of elements.

Tomotaka et al , (1996)[40], reported the soft tissue discoloration of marginal gingival, attached gingival and gingival papilla by spectrophotometer and stated that noticeable color difference occurs in marginal gingiva and gingival papilla than attached gingival in patients wearing crowns.

Karl F. Leinfelder et al, (1997)[41], reviewed that gold based alloys were used almost exclusively for most of the time, but they are replaced by base metal alloys merely because of increased cost of gold alloys. The major drawback of base metal alloys are long term risks to patients demonstrating extraoral allergic responses or positive response to nickel patch tests.

J.C Wataha et al, (1998)[42], studied the effect of pH on elemental release from dental casting alloys on three groups of alloys namely high noble, noble, nickel based alloys exposed to acidic environment in 30 mts with ph ranging from 1 to 7 elemental release was measured by atomic absorption in before, during and after exposure and concluded that significant release occurs in nickel based alloys than gold based alloys in acidic environment.

S.P Kedici et al, (1998)[43]**,** conducted a study to find out the corrosion rates, the change of corrosion potentials due to time and to perceive the corrosion tendencies of various alloys used in dentistry namely precious and base metal alloys and their recasts done by scanning electron microscope, Energy dispersive X –ray analyzer system and potentiodynamic methods and concluded that alloys shows ion leakage in corrosive medium. Titanium proved to be corrosive resistant and chromium, nickel and molybdenum are resistant to corrosion but a small variation in composition affects their corrosion resistance.

T.K .Patro et al, (1998)[44], had done a study to determine the corrosive behavior of indigenous Ag-Sn- Cu cast dental alloys in artificial saliva by potentiodynamic method with and without 0.1M lactic acid and concluded that the rate of corrosion of indigenous alloy was higher than Ag –Pd alloy and tendency for repassivation was not seen in artificial saliva in presence of 0.1M lactic acid for indigenous alloys.

G. Schmalz et al, (1998)[45] conducted a study to determine the elemental release in cell-culture medium and their cytotoxicity of these medium extracts were compared with their respective metal salt solutions. elemental release was done by inductively coupled plasma atomic emission spectrometry and cytotoxicity by MTT assay and concluded that the cytotoxicity of culture medium extracts proved to be less toxic when compared to corresponding salt solutions, probably due to limitations of chemical analysis of extracts.

Ozdemir S et al, (1998)[46]conducted a study to determine the corrosion products released from two recast Ni-Cr base metal alloys Wirolloy and Wiron 99 . The release of Ni, Cr and Mo ions from both alloys was measured by using a flame model Atomic Absorption Spectrophotometer and concluded that the number of recasting was found to have negligible effect on surface texture and on the amount of corrosion products released.

J.C Wataha et al, (1998)[47], had done a study to determine the release of elements from eight types of commonly used dental casting alloys into cell-culture medium was measured over a 10-month period by atomic absorption spectrophotometry and concluded that the total mass lost over the 10-month period ranged from < 2 micrograms/cm2 for the Au-Pd alloy to 55 micrograms/cm2 for the Au-Ag-Cu alloy.

Steven K.Nelson et al, (1999)[48], had done a study to evaluate the cytotoxicity and mass release of conditioned and unconditioned alloys by atomic absorption spectroscopy and succinic dehydrogenase activity and reported that the conditioned of casting alloys appeared to be a useful method for predicting the long term cytotoxicity.

J.C .Wataha et al, (1999)[49], had done a study to determine the initial release of elements and cytotoxicity and their subsequent release and cytotoxicity after 1 month with six types of noble alloys by atomic absorption spectrometry and MTT assay and concluded that the initial release was higher in first weeks than subsequent weeks for single phase alloys but for multiple phase alloys shows a steady release and cytotoxicity is complex in nature.

Petra E. Lockwood ,et al, (1999)[50], reported the long term cytotoxicity of 8 dental casting alloys in the mouth in a biological medium containing serum proteins for a period of 10 months and cytotoxicity was measured and compared with initial cytotoxicity and concluded that short term toxicity does not accurately measure the long term cytotoxicity of dental casting alloys.

Shogo Minagi et al , (1999)[51], developed a clinically serviceable method for microsampling of dental casting alloys and collected the ground metal particles by a silicone sampling tube after the surface were ground with a carbide bur. The recovered samples were analyzed by energy dispersive x-ray microanalysis and concluded that this method of sampling is of great benefit to clinicians to patients who have allergic reaction to dental alloys.

John C. Wataha et al, (1999)[52], studied the effect of tooth brushing on the elemental release from dental casting alloys from a Au-Pd alloy, Au- Pd alloy , Pd-Cu-Ga alloy and Ni-Cr alloy placed in a biologic medium for 1 week, then brushed for 30mts at 90strokes in saline and again transferred to biologic medium and elemental release was done by atomic absorption spectroscopy to measure mass loss and concluded that Ni-Cr alloys show increased elemental release than other alloys during brushing.

Steven K. Nelson et al, (1999)[53], conducted a study to determine that preconditioning of alloys have an effect on cytotoxicity from six types of alloys by exposing the alloys to either saline, cell culture medium , bovine serum albumin serum for 72 hours . the elemental release and cytotoxicity was evaluated and concluded that the preconditioning of alloys decreases the cytotoxicity of dental alloys.

Michael D.Roach et al, (2000)[54]**,** determined the electrochemical corrosion behavior of 6 commercial nickel – chromium alloys in as cast and PFM fired / polished states by potentiodynamic polarization and x-ray photoelectron spectroscopy and concluded that the effect of PFM firing and repolishing on Ni- Cr dental casting alloys surface oxides and corrosion properties appear to be alloy dependent.

John C. Wataha et al, (2000)[55], reviewed the biocompatibility of various dental alloys and stated that the dentists should select alloys that release lowest of elements by using high noble or noble alloys with single phase microstrucures and selection can be done by using corrosion and biological data from dental manufactures.

G. Bayramoglu et al, (2000)[56], had done a study to determine the effect of pH on corrosion of dental metals and alloys that have different compositions by electrochemical method and concluded that dissolution occurs in all of the tested pH. The dissolution was moderate in titanium and high in samples containing tin and copper and addition of cobalt and molybdenum improved the corrosion resistance.

Laurent F et al, (2001)[57]**,** conducted a study to compare the corrosion resistance of dental alloys in a solution containing oral bacteria. Actinomyces viscosus (ATCC19246) of two dental alloys (Ni-Cr alloy and gold-based alloy) by electrochemical means in sterile Fusayama artificial saliva (AS), AS enriched with sterile yeast extract (YE) and YE modified by introducing bacteria (AV) and concluded that the absence of oxygen in non-precious alloy, led to an increase in polarization resistance whereas the slight decrease in polarization for the precious alloys by the organic and inorganic metabolites released by bacteria in to the electrolyte.

Desheng Sun et al, (2002)[58], had done a study to compare the in vitro corrosion characteristics of 3 high-palladium alloys and 1 gold-palladium alloy in simulated body fluid and oral environments by Cyclic Polarization and concluded that the corrosion resistance of the 3 high-palladium alloy in simulated body fluid and oral environments were comparable to that of the gold palladium alloy.

Senay Canay et al, (2002)[59], conducted a study to investigate the effect of 10% carbamide peroxide on the electrochemical corrosion of various dental casting alloys used for fixed partial dentures and dental amalgam by polarization method. The material chosed were dental amalgam, noble alloy and base metal alloys and concluded that unpolished amalgam and nickel-chromium alloy samples had the most corrosion rates and the noble alloys had the least.

Ahmad S. AL-Hiyasat et al, (2002)[60], investigated the element release from 7 commercial available dental casting alloy and tested their cytotoxic effects.

The elemental release was done by ICPAES and cytotoxic effects assist on Balb C Fibroblasts using MTT assay and concluded that elemental release is proportional to the conditioning time.

Schmalz G et al, (2002)[61], had done a survey the describe the interactions of dental cast alloys with living tissues and to relate them to clinically adverse local reactions of the oral tissues and concluded that Patients relating oral symptoms to metal restorations should be subjected to a thorough dental and general medical examination in order to exclude non-material related diseases being the cause for their complaints/symptoms.

Shettlemore MG et al, (2002)[62], had done a study to examine dental material degradation product toxicity using the Microtox bacterial bioluminescence assay Polarization was used to produce ionically dissolved (ID) and precipitated corrosion products and concluded that the Microtox is useful for evaluating dental degradation product biocompatibility.

Geurtsen W et al, (2002)[63], reviewed the biocompatibility of dental casting alloys and reported that the Ni-based alloys, such as beryllium-containing Ni alloys, exhibit increased corrosion, specifically at low pH. Further, microparticles are abraded from metallic restorations due to wear. In sufficient quantities, released metal ions-particularly Cu, Ni, Be, and abraded microparticles-can also induce inflammation of the adjacent periodontal tissues and the oral mucosa.

Ahmad S. AL-Hiyasat et al, (2003)[64], investigated the cytotoxicty of a high noble alloy, four Ni-Cr alloy and one Co-Cr alloy and one Cu based alloy by Balb C Fibroblasts using MTT assay and concluded that cytotoxicity of casting alloy was markedly affected by their composition and presence of Cu in the alloy adversely affected cell viability.

Ahmad S. AL-Hiyasat et al, (2003)[65], **i**nvestigated the cytotoxicity of a high noble alloy, four Ni-Cr alloy and one Co-Cr alloy and one Cu based alloy in distilled water by Balb C Fibroblasts using MTT assay and concluded that conditioning of base metal alloy other than those containing Cu for 168hrs in distilled water makes their cytotoxicity level comparable to that of high noble alloy.

Cabrini RL et al, (2003)[66], had done a study to estimate tissue response of the corrosion processes. The quantitative evaluation of the deposits was performed in an MPM-800 (Carl Zeiss)* microscope. The light microscopy images were digitalized and then analyzed employing the DNA-IBAS-Kontron software that allows for the identification and evaluation of cells loaded with corrosion products (objective 20 xs) and concluded that the method proposed serves to quantitatively evaluate, at light microscopy level, the deposition of corrosion products in tissues.

Dong H et al, (2003)[67], studied the corrosion behavior of dental alloys in electrolyzed strong acid water, weak acid water and neutral water using a 7-day immersion test by X-ray microanalysis and concluded that the neutral water appeared the least corrosive to metals among the three types showing equivalent bactericidal activity. Silver alloy showed the greatest surface color change and dissolution of constituents in the strong acid water and the smallest in the neutral water. The release of Au from gold alloy was especially marked in the strong acid water. Co-Cr alloy showed greater corrosion and tarnish resistance in the strong acid water rather than in the weak acid water and the neutral water.

Gulsen Can et al, (2004)[68], designed to determined the cytotoxicty effect of Ni-Cr and Co-Cr on the cytoskeleton in cultured human fibroblasts. The samples were exposed to human fibroblasts for 120hrs and analyzed by 3D laser confocal microscope and concluded that Ni-Cr and Co-Cr dental alloys especially sand blasted forms have detrimental effects on the actin based cytoskeleton.

Denizoglu S et al, (2004)[69], reported the influence of salivary pH on the corrosion of two base-metal alloys. Cobalt-chromium (Co-Cr) and nickel-chromium (Ni-Cr) alloy submerged in artificial saliva of different pH values by flame atomic absorption spectrophotometry and concluded that the pH significantly affected total and Co ion release, but not Ni or Cr ion release. The alloy type did not affect total ion release, but was significant for Cr ion release

Garhammer P et al, (2004)[70], had done a study to examined the metal content of saliva of patients with and without metal restorations. The composition was analyzed using the energy-dispersive X-ray analysis of metal biopsy specimens and Saliva analysis was performed using atomic absorption spectroscopy and concluded that the metals Ag, Cr, Cu, Fe, Ni, and Zn were found in saliva of patients without metal restorations. The metals Ag, Au, Cr, Cu, Fe, Ni, and Zn were identified in saliva of patients with metal restorations being higher in concentration than in control patients.

Ahmad S. AL-Hiyasat et al, (2005)[71], investigated the effect of recasting on the elemental release and the cytotoxicity of five base metal alloys by Balb C Fibroblasts using MTT assay and concluded that Cu content in an alloy increases its cytotoxicity level remarkably, recasting of alloys significantly increase the cytotoxicity level and Co-Cr alloy was adversely affected by recasting than Ni-Cr alloys.

Okazaki y et al, (2005)[72], had done a study to quantify the amount of Ni released from stainless steel and the quantities of Al release in a biologic medium and concluded that Ni and Al release gradually decreased with increasing pH. .

Ren Y et al, (2005)[73], had done a study to determine the Potential harmfulness of nickel in medical metal materials and reported that the nickel ions leaching from stainless steel because of corrosion have the harmfulness of malformation and cancerization besides allergenicity in human body.

Reclaru L et al, (2005)[74], had done a study to determine the corrosion resistance of new generation of Co-Cr alloys enriched with precious metals (Au, Pt, Ru) by electrochemical techniques in two different milieus based on the Fusayama artificial saliva and an electrolyte containing NaCl and concluded that the presence of precious metals can deteriorate the corrosion behaviour of Co-Cr alloys in a significant way.

Ardlin BI et al, (2005)[75]**,** evaluated the irritation potential of (one iron and two cobalt alloys, unalloyed titanium and an experimental titanium-zirconium alloy, and one gold alloy containing copper and zinc) by Static immersion and irritation tests. and concluded that the only the 1 mmol l(-1) Cu(2+) solution was graded as slightly irritating.

Celebi A et al, (2006)[76] **,** had done a study to determine the release of metal ions (Al, Ag, Au, Ca, Cd, Co, Cr, Cu, Mg, Mo, Ni, Pd, Pt, Ti, and Zn) from the commercial gold/platinum (Au/Pt) dental alloy by Inductively coupled plasma atomic emission spectroscopy for 30 days and concluded that the undeclared chromium from Au/Pt dental alloy, or some other element might be responsible for the contact allergy thus far attributed to the gold.

Chen L et al, (2006)[77], conducted a study to determine the effect of porcelain firing cycle on microstructure of 4base metal alloys, and to analyze the changes of their corrosion resistance in the artificial saliva by field emission scanning electron microscopy and energy dispersive spectroscopy and polarization curves and concluded that the procedure of porcelain firing cycle can affect the surface microstructure and increase the corrosion of 4 metal-ceramic alloys.

Sujs et al, (2006)[78,] tested the corrosion behavior of three kinds of dental casting alloys and to investigate the effect of the released metal ions on the DNA damage of dog buccal mucosal cells. The concentration of the released metal ions was measured after the restoration of 2 weeks, 1 month, 2 months and 3 months. The DNA damage of buccal mucosal cells was studied by the method of SCGE and concluded that the noble alloy (gold 58%) is most corrosion resistant of the three alloys and has good biocompatibility. The NiCr and NiCrBe are prone to corrode and have cytotoxicity to cells.

- Aquasil TM Soft putty Regular set - [DENTSPLY ,GERMANY]
- Four commercially available Nickel Chromium alloys
  - Bellabond plus [BEGO and Co, GERMANY]
  - 4 ALL [IVOCLAR VIVADENT technical, U.S.A]
  - Heraenium [HERAEUS KULZER GmbH, GERMANY]
  - Ceramet [LABOLINE S.p.A , EUROPE]
- Composition of these alloys is shown in table I

Table I: Composition of four alloy groups alloys used

Elements	**Bellabond plus**	**4 ALL**	**Heraenium**	**Ceramet**
Ni	65.2	61.4	62.9	62.0
Cr	22.5	25.7	23.0	26.0
Mo	9.5	11.0	10.0	10.0
Si	-	1.5	2.0	1.5
Mn	-	<1.0	-	
Al	-	<1.0	-	
C	-	<1.0	<1.0	

- High Nobel alloy - d- SIGN 98 [IVOCLAR VIVADENT, U.S.A]

Table II: Composition of high noble alloy

Au	**Pt**	**Zn**	**In**	**Ir**	**Others**
85.9	12.1	1.5	<1.0	<1.1	<1.0

- Inlay Wax medium (Gc Corporation Tokoyo, Japan)
- Bellasun (Bego & Co, Germany)
- Iwansons gauge Metal calipers
- Metal trimmers, emery papers, polishing wheels, mantrels and rouge
- Digital ultra sonic cleanser – Uni kleen
- Stereozoom Optical Microscope –UM-1530R-Hongkong
- Electronic balance – Single pan- Dhona-260D
- Vibrant potentiostat / Galvanostat – VSM / CS / 30
- Three unit electrolytic cell
    - Working electrode – specimen
    - Reference electrode – Saturated calomel electrode
    - Counter electrode – Platinum electrode

- **Artificial saliva / Electrolyte - Composition is shown in table III**

Table III: Composition of contents in artificial saliva [79]

Sodium chloride	0.4g
Potassium chloride	1.21g
Sodium dihydrogen phosphate	0.78g
Sodium sulphide	0.005g
Urea	1g
Distilled water	1000ml

- Personal computer with software attached
- Scanning electron microscope –[FEI 200,Quanta ,Japan]
- Inductively coupled plasma –Mass spectrometer-[Agilent 7500 Series, Japan].

Preparation of Samples / Working Electrodes:

A sample die measuring 1cm square and 3mm width was machined in stainless steel [Fig 1, 5]. An addition silicone impression [Aquasil TM Soft putty Regular set - DENTSPLY, GERMANY] was made from the sample die for the preparation of samples [working electrodes]. Wax patterns were fabricated from the impressions using Inlay wax- medium [Fig9]. [GC Corporation, Tokyo, Japan] and invested by phosphate bonded investment. [Bella sun, Bego and Co, Germany**]**[Fig: 6]. Four commercially available Nickel Chromium alloys as shown in[Fig7,8] were used [Four all , Bellabond plus , Ceramet , Heraenium] to cast the four groups of samples using induction casting machine [Fornax , Bego and Co , Germany].

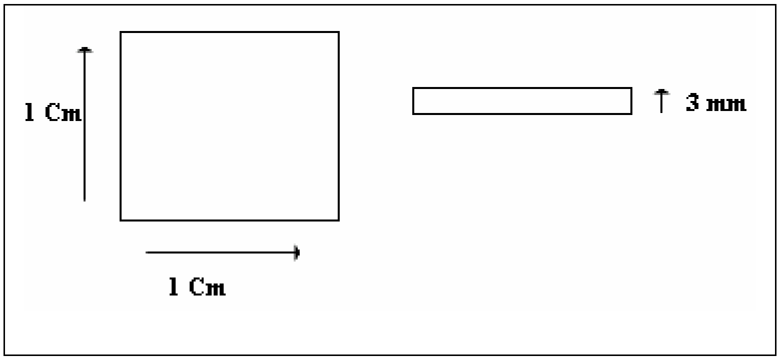

Fig 1: Sample Dimensions

The study groups were given in Table IV:

Table IV: Study groups taken in corrosion study

STUDY GROUPS	ALLOYS USED
GROUP I	**d-SIGN 98** **[CONTROL GROUP]**
GROUPII	**CERAMET**
GROUPIII	**4 ALL**
GROUPIV	**BELLABOND PLUS**
GROUPV	**HERAENIUM**

Samples used for the high noble alloy were of the same dimensions as the base metal alloys used [Fig10].The samples [**working electrodes**] were mechanically polished using different grades of emery papers up to 800 grit papers and subsequently on a rubber polishing wheel and rouge. The samples [working electrodes] were then ultrasonically cleansed in acetone using digital ultrasonic cleanser [Unikleen] [Fig12] and thoroughly washed in distilled water. The surfaces of each samples [working electrodes] were visualized by Stereo zoom Optical Microscope [UM -1530R Hong Kong] [Fig21] to ensure the uniformity of the surface before initiating the corrosion study. [Fig 24-28]

Current density of the samples [working electrodes] per unit-area were obtained by applying lacquer on one side so that only on side with 1cm^2 surface area was exposed on the other side and this formed the working electrode[Fig14].

Electrochemical cell assembly:

The electrochemical studies involving open circuit-time measurements and cyclic polarization measurements were carried out using a three electrode cell assembly system of 500ml capacities, potentiostat and desktop with software attached [Fig 2, 18, 19]. The electrodes are Reference electrode, Counter electrode and Working electrode.

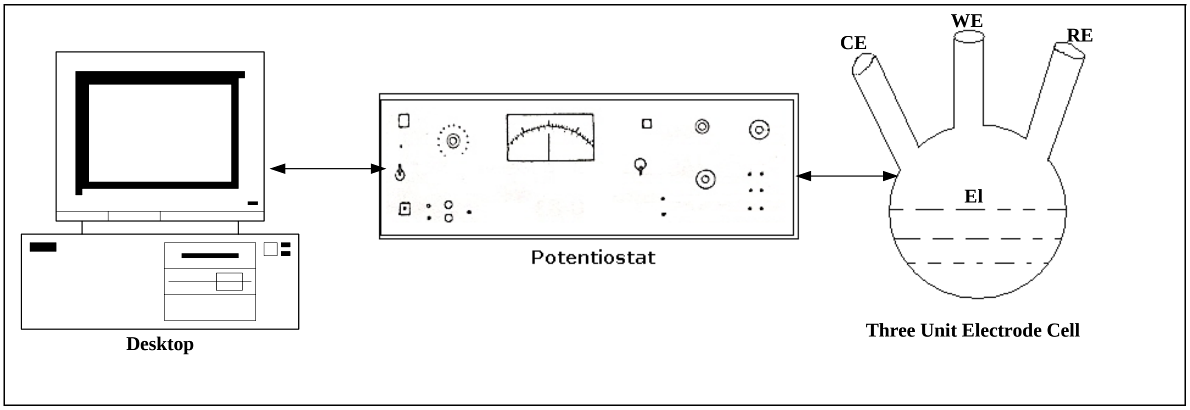

Fig 2: Electrochemical Cell Assembly

CE ➔ Counter electrode

WE ➔ Working electrode or sample

RE ➔ Reference electrode

El ➔ Electrolyte / Artificial saliva

A platinum foil was used as the counter or auxiliary electrode, saturated calomel (SCE) as the reference electrode (connected through Lugin capillary) and the alloy samples as working electrode [Fig 19].

Artificial saliva preparation:

The electrochemical cyclic polarization studies were carried out in artificial saliva [Electrolytes].Artificial saliva is freshly prepared each time for each sample [working electrode][Fig:16] The composition of the artificial saliva used in the present study are given in Table II. They are weighed in Electronic balance – Single pan- Dhona-260D [Fig 17]. It may be noted that there was also a small concentration of sodium sulphide (Na_2s, $9H_2O$) in the solution to make it closer in the composition to oral conditions. The specimens were suspended into the electrolyte (artificial saliva) to a surface area of about 1 $cm^{2.}$

Corrosion study [cyclic polarization study]

Open circuit-time measurement (OCP):

The open circuit potential was monitored for each alloy sample .As soon as the sample were immersed in the electrolyte, the initial potentials of the specimens were noted and monitored as a function of time until a constant potential was reached and referred to as corrosion potential

(**ECorr).** All the alloy specimens were allowed to reach a steady Open circuit potential for a period of 1hr.

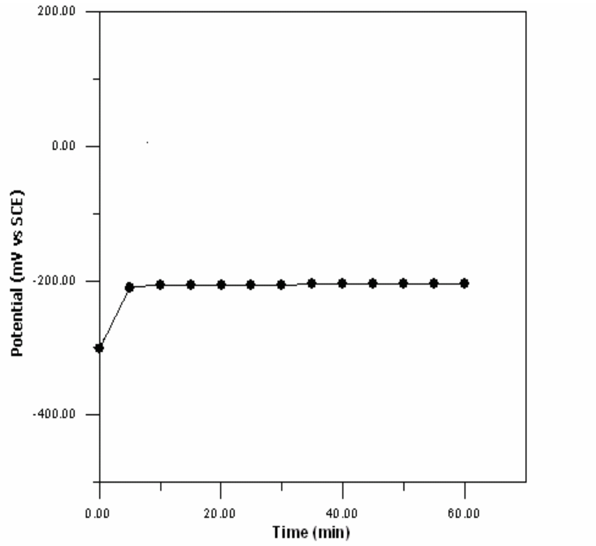

Fig 3: Open circuit potential curve

Cyclic Polarization Measurements:

Cyclic polarizations tests were conducted for each sample [working electrode] with a Vibrant Potentiostat / Galvanostat [Fig: 18] electrochemical interface controlled by commercial software. When the sample attained constant potential or steady state potential (**ECorr),** cyclic polarization was initiated by applying a potential below the corrosion potential (**ECorr)** and increased towards the positive direction at a scan rate of 1mV/cm2. and scan [anodic scan] was continued until the threshold current density of 0.1 µA/cm^2 was reached , during this period the alloy entered the transpassive or pitting region named as Breakdown potential,[**Eb**] and then the scan [cathodic scan] is reversed back to **Ecorr** of the alloy. The potential at which the reverse cathodic scan meets the forward anodic scan is termed the Repassivation potential, [**Ep**.] The current density **[Icorr]** was monitored with respect to the potentials during polarization experiments.

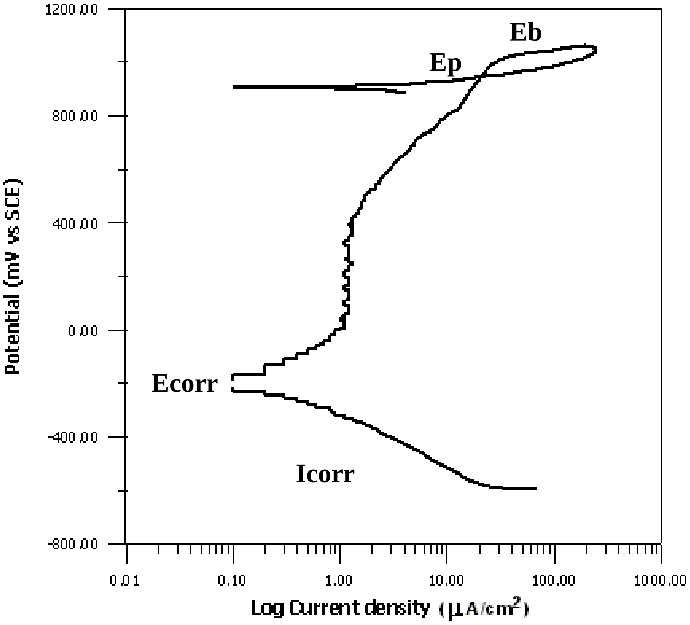

Fig 4: Cyclic polarization curve

The parameters of interest recorded during the cyclic polarization studies are shown in Fig 4:

Corrosion potential, [Ecorr] is the steady state potential of an alloy where oxidation and reduction reaction are in equilibrium.

Breakdown potential, [Eb] is the potential at which the oxide layer of an alloy breaks down.

Repassivation potential, [**Ep]** is the potential at which the reformation of passive oxide layer which helps in corrosion resistance of an alloy.

Corrosion current, [Icorr] determines the corrosion rate of an alloy.

Forty samples of Nickel- Chromium alloys of four groups and five samples of high noble alloy [control group] were used to study the corrosive behavior namely Corrosion potential , Breakdown potential , Repassivation potential and corrosion current .The data obtained were tabulated and statistically analyzed.

Accelerated leaching study –Inductively coupled plasma –Mass spectrometer.

One sample from each alloy was subjected to accelerated leaching study**.** The working electrodes were immersed in artificial saliva and allowed to stabilize at corrosion potential [Ecorr] and breakdown potential [Eb] for a period of one hour in 250 ml of the test solution. At the end of each experiment, the chemical composition of the test solution was analyzed by inductively coupled plasma – mass spectrometer. In ICP –MS, [Fig22] argon gas and plasma are utilized to atomize and ionize the elements in the sample matrix. These resulting ions are then passed through a series of apertures [cones] into a high vacuum analyzer where the isotopes of the elements are identified by their mass to charge ratio. The intensity of specific peaks in the mass spectrum is proportional to the amount of the elemental isotopes present in the original sample.

Scanning Electron Microscopic Study

The surface morphology of the corroded samples was examined under scanning electron microscope at 15kv [Fig23]. The SEM photomicrographs were evaluated at 1000x magnification.

The corrosive behavior of four commercially available Nickel – Chromium alloys in artificial saliva with high noble alloy as the control groups were studied by cyclic polarization test. The parameters that were studied from the corrosion study are corrosion potential [**Ecorr**], Breakdown potential [**Eb**], Repassivation potential [**Ep**] and Corrosion current [**Icorr]**.The data obtained were subjected to statistical analysis.

The significance level between the control group I and among other test groups [II to V] was determined by **Students t independence test**. The significance level between the test groups [II to V] were determined by **one way ANOVA variance tests** and significance level among all the study groups [I to V] were determined by **multiple turkey tests –HSD procedures.**

The study groups were tabulated as follows:

Table IV: Study groups

STUDY GROUPS	ALLOYS USED
GROUP **I**	HIGH NOBLE ALLOY
GROUP **II**	CERAMET
GROUP **III**	4 ALL
GROUP **IV**	BELLABOND PLUS
GROUP **V**	HERAENIUM

Table V, VI, VII, VIII, IX shows the corrosion parameters **[Ecorr], [Eb], [Ep], [Icorr]** values obtained by corrosion study for each study groups.

Table V: Group I [HIGH NOBLE ALLOY]

SNo	Ecorr	Eb	Ep	Icorr
1	-205	1028	939	0.15
2	-217	1015	919	0.17
3	-234	1002	905	0.20
4	-225	1021	934	0.16
5	-209	1019	929	0.16

Table VI: Group II [CERAMET]

SNo	**Ecorr**	**Eb**	**Ep**	**Icorr**
1	-171	743	806	0.29
2	-165	740	801	0.30
3	-154	731	791	0.32
4	-170	739	798	0.30
5	-140	725	784	0.36
6	-162	741	803	0.30
7	-159	738	795	0.30
8	-160	743	806	0.29
9	-169	755	795	0.31
10	-168	729	789	0.33

Table VII: Group III [FOURALL]

SNo	Ecorr	Eb	Ep	Icorr
1	-136	808	840	0.21
2	-117	803	820	0.22
3	-128	798	819	0.22
4	-131	805	825	0.21
5	-102	787	805	0.26
6	-122	792	813	0.24
7	-135	799	825	0.24
8	-144	801	855	0.22
9	-136	808	840	0.21
10	-132	800	838	0.22

Table VIII: Group IV [BELLABOND PLUS]

SNo	Ecorr	Eb	Ep	Icorr
1	-158	686	789	0.44
2	-170	650	770	0.52
3	-134	672	782	0.47
4	-146	681	784	0.46
5	-156	665	765	0.49
6	-165	685	785	0.44
7	-175	659	775	0.51
8	-152	679	780	0.46
9	-156	684	782	0.44
10	-142	662	772	0.49

Table IX: Group V [HAERINIUM]

SNo	Ecorr	Eb	Ep	Icorr
1	-112	730	802	0.32
2	-132	725	791	0.33
3	-124	722	784	0.34
4	-109	727	799	0.33
5	-101	729	800	0.32
6	-134	715	789	0.35
7	-142	719	775	0.38
8	-116	715	795	0.32
9	-105	726	801	0.31
10	-118	730	802	0.32

Table X, XI, XII, XIII, shows the descriptive statistics of four parameters [Ecorr], [Eb], [Ep] and [Icorr] for all the study groups [I to V]

Table X: Corrosion potential [Ecorr]

Groups	Mean ± Standard deviation	Standard Error	Median (Range)
I	-219.5 ±12.1	3.8	-219 [-205 to -242]
II	-161.6 ± 9.4	3.0	-163.5 [-140 to -171]
III	-128.3 ± 12.0	3.8	-131.5 [-102 to -144]
IV	-155.4 ± 12.6	4.0	-156 [-134 to -175]
V	-119.3 ± 13.4	4.3	-117 [-101 to -142]

Table XI: Breakdown Potential, [Eb]

Groups	Mean ± Standard deviation	Standard Error	Median (Range)
I	1016.2 ± 9.5	3.0	1017 [1002 to 1028]
II	738.4 ± 8.5	2.7	739.5 [725 to 755]
III	800.1 ± 6.7	2.1	800.5 [784 to 808]
IV	672.3 ± 12.7	4.0	675.5 [650 to 686]
V	723.8 ± 5.8	1.8	725.5 [715 to 730]

Table XII: Repassivation potential, [Ep]

Groups	Mean ± Standard deviation	Standard Error	Median (Range)
I	924.7 ± 12.5	3.9	925 [905 to 939]
II	796.8 ± 7.4	2.3	796.5 [784 to 806]
III	828.0 ± 15.0	4.8	825 [805 to 855]
IV	778.4 ± 7.6	2.4	781 [765 to 789]
V	793.8 ± 9.0	2.9	797 [775 to 802]

Table XIII: Corrosion current [Icorr]

Groups	Mean ± Standard deviation	Standard Error	Median (Range)
I	0.17 ± 0.02	0.006	0.165 [0.15 to 0.20]
II	0.31 ± 0.02	0.007	0.30 [0.29 to 0.36]
III	0.23 ± 0.02	0.005	0.22 [0.21 to 0.26]
IV	0.47 ± 0.03	0.009	0.465 [0.44 to 0.52]
V	0.33 ± 0.02	0.006	0.325 [0.31 to 0.38]

Table XIV, XV, XVI, Comparison among each study groups for Corrosion potential [Ecorr,] of corrosion study were tabulated separately

Table XIV: Corrosion potential [Ecorr]

Groups	Mean ± Standard deviation	P-value*
I	-219.5 ±12.1	-
II	-161.6 ± 9.4	<0.0001
III	-128.3 ± 12.0	<0.0001
IV	-155.4 ± 12.6	<0.0001
V	-119.3 ± 13.4	<0.0001

In table XIV , The mean absolute Ecorr value in control group [-219 .5 ±12.1] was significantly higher than Group II [-161.8 ±9.4] , Group III [128.3±12.0] , Group IV [-155.4 ±12.6] and in Group V [-119.3± 13.4] [p 0.0001]

Table XV: Corrosion potential [Ecorr]

Groups	Mean ± Standard deviation	P-value$	Significant # Groups at 5% level
II	-161.6 ± 9.4	<0.0001	V vs. II,IV III vs. II ,IV
III	-128.3 ± 12.0	<0.0001	
IV	-155.4 ± 12.6	<0.0001	
V	-119.3 ± 13.4	<0.0001	

In table XV, The mean Ecorr in group V [-119.3 ±13.4] was significantly higher than the mean Ecorr in Group II [-161.8 ±9.4] and in Group IV [-155.4± 12.6] [p 0.05]. However there was no significant difference in mean Ecorr between any other contrasts.

Table XVI: Corrosion potential [Ecorr]

Groups	Mean ± Standard deviation	P-value$	Significant # Groups at 5% level
I	-219.5 ±12.1	-	V vs. I, II, IV
II	-161.6 ± 9.4	<0.0001	
III	-128.3 ± 12.0	<0.0001	
IV	-155.4 ± 12.6	<0.0001	
V	-119.3 ± 13.4	<0.0001	

In table XVI , The mean Ecorr in group V [-119.3 ±13.4] was significantly higher than the mean Ecorr in Group I [-219.5 ±12.1] , Group II [-161.8± 9.4] and in Group IV [-155.4 ±12.6] [p 0.05] However there was no significant difference in mean Ecorr between any other contrasts.

Table XVII, XVIII, XIX, Comparison among each study groups for Breakdown potential [Eb] of corrosion study were tabulated separately

Table XVII: Breakdown Potential, [Eb]

Groups	Mean ± Standard deviation	P-value*
I	1016.2 ± 9.5	-
II	738.4 ± 8.5	<0.0001
III	800.1 ± 6.7	<0.0001
IV	672.3 ± 12.7	<0.0001
V	723.8 ± 5.8	<0.0001

In table XVII, The mean absolute Eb value in control group [1016.2± 9.5] was significantly higher than Group II [738.4 ±8.5] , Group III [800.1± 6.7] , Group IV [672.3± 12.7] and in Group V [723.8± 5.8] [p 0.0001]

Table XVIII: Breakdown Potential, [Eb]

Groups	Mean ± Standard deviation	P-value$	Significant # Groups at 5% level
II	738.4 ± 8.5	<0.0001	III vs. II, IV, V
III	800.1 ± 6.7	<0.0001	
IV	672.3 ± 12.7	<0.0001	
V	723.8 ± 5.8	<0.0001	

In table XVIII, The mean Eb in group III [800.1± 8.5] was significantly higher than the mean Eb in Group II [-738.4 ±8.5], Group IV [672.3 ±12.7] and in Group V [723.8± 5.8] [p 0.05]. However there was no significant difference in mean Eb between any other contrasts.

Table XIX: Breakdown Potential, [Eb]

Groups	Mean ± Standard deviation	P-value$	Significant # Groups at 5% level
I	1016.2 ± 9.5	-	I vs. II, III, IV, V III vs. II, IV, V
II	738.4 ± 8.5	<0.0001	
III	800.1 ± 6.7	<0.0001	
IV	672.3 ± 12.7	<0.0001	
V	723.8 ± 5.8	<0.0001	

In table XIX, The mean Eb in group I [1061.5 ±12.1] was significantly higher than the mean Eb in Group II [-738.4 ±8.5], Group III [-128.3± 12.0], Group IV [672.3 ±12.7] and in Group V [723.8 ±5.8] [p 0.05]. Further, the mean Eb value in Group III [-800.1± 12.0] was significantly higher than the mean Eb value in Group II [-738.4± 8.5], Group IV [672.3± 12.7] and in Group V [723.8± 5.8] [p0.05]. However there was no significant difference in mean Eb between any other contrasts.

Table XX, XXI, XXII, Comparison among each study groups for Repassivation potential [Ep] of corrosion study were tabulated separately

Table XX: Repassivation potential, [Ep]

Groups	Mean ± Standard deviation	P-value*
I	924.7 ± 12.5	-
II	796.8 ± 7.4	<0.0001
III	828.0 ± 15.0	<0.0001
IV	778.4 ± 7.6	<0.0001
V	793.8 ± 9.0	<0.0001

In table XX, The mean absolute Ep value in control group [924.7 ±12.5] was significantly higher than Group II [796.8± 7.4] , Group III [828.0± 15.0] , Group IV [778.4 ±7.6] and in Group V [793.8± 9.0]

Table XXI: Repassivation potential, [Ep]

Groups	Mean ± Standard deviation	P-value$	Significant # Groups at 5% level
II	796.8 ± 7.4	<0.0001	III vs. II, IV, V
III	828.0 ± 15.0	<0.0001	
IV	778.4 ± 7.6	<0.0001	
V	793.8 ± 9.0	<0.0001	

In table XXI, The mean Ep in group III [828.0 ±15.0] was significantly higher than the mean Ep in Group II [796.8± 7.4], Group IV [778.4 ±7.6] and in Group V [793.8± 9.0] [p 0.05].

Table XXII: Repassivation potential, [Ep]

Groups	Mean ± Standard deviation	P-value$	Significant # Groups at 5% level
I	924.7 ± 12.5	-	I vs. II, III, IV, V III vs. II, IV, V
II	796.8 ± 7.4	<0.0001	
III	828.0 ± 15.0	<0.0001	
IV	778.4 ± 7.6	<0.0001	
V	793.8 ± 9.0	<0.0001	

In table XXII, The mean Ep in group I [924.7± 12.5] was significantly higher than the mean Ep in Group II [796.8± 7.4], Group III [828.0 ±15.0], Group IV [778.4± 7.6] and in Group V [793.8 ±9.0] [p 0.05]. Further, the mean Ep value in Group III [828.0± 15.0] was significantly higher than the mean Ep value in Group II [796.8 ±7.4], Group IV [778.4 ±7.6] and in Group V [793.8± 9.0] [p 0.05] However there was no significant difference in mean Ep between any other contrasts.

Table XXIII ,XIV, XV , ,Comparison among each study groups for Corrosion current[Icorr]of corrosion study were tabulated separately

Table XXIII: Corrosion current, [Icorr]

Groups	Mean ± Standard deviation	P-value*
I	0.17 ± 0.02	-
II	0.31 ± 0.02	<0.0001
III	0.23 ± 0.02	<0.0001
IV	0.47 ± 0.03	<0.0001
V	0.33 ± 0.02	<0.0001

In table XXIII, The mean absolute Icorr value in control group [0.17 ±0.02] was significantly lower than Group II [0.31± 0.02], Group III [0.23 ±0.02], Group IV [0.47 ±0.03] and in Group V [0.33± 0.02]

Table XIV: Corrosion current, [Icorr]

Groups	Mean ± Standard deviation	P-value$	Significant # Groups at 5% level
II	0.31 ± 0.02	<0.0001	IV vs. II, III, V
III	0.23 ± 0.02	<0.0001	
IV	0.47 ± 0.03	<0.0001	
V	0.33 ± 0.02	<0.0001	

In table XXIV, The mean Icorr in group IV [0.47 ±0.02] was significantly higher than the mean Icorr in Group II [0.31± 0.02], Group III [0.23± 0.02] and in Group V [0.33± 0.02] [p 0.05]. Further, the mean Icorr value in Group V [0.33± 0.02] was significantly higher than the mean Icorr value in Group III [0.23 ±0.02] [p 0.05]. However there was no significant difference in mean Icorr between any other contrasts.

Table XV: Corrosion current, [Icorr]

Groups	Mean ± Standard deviation	P-value$	Significant # Groups at 5% level
I	0.17 ± 0.02	-	IV vs. I, II, III, V
II	0.31 ± 0.02	<0.0001	
III	0.23 ± 0.02	<0.0001	
IV	0.47 ± 0.03	<0.0001	
V	0.33 ± 0.02	<0.0001	

In table XXV, The mean Icorr in Group IV [0.47± 0.02] was significantly higher than the mean Icorr in Group I [0.17± 0.02], Group II [0.31± 0.02], Group III [0.23± 0.02] and in Group V [0.33± 0.02] [p 0.05]. Further, the mean Icorr Group V [0.33± 0.02] value in was significantly higher than the mean Icorr value in Group III [0.23 ±0.02] [p 0.05]. Further, the mean Icorr of Group II [0.31± 0.02] value in was significantly higher than the mean

Icorr value in Group III [0.23± 0.02] [p 0.05]. However there was no significant difference in mean Icorr between any other contrasts.

The most commonly used alloys in the fabrication of removable and fixed prosthetic appliances are predominantly base metal alloys. They have greatly replaced the expensive high noble alloys, as these alloys possess good mechanical properties and lower cost. The biocompatibility of these alloys is an important consideration for the suitability of a material for use in the oral cavity. Corrosion of an alloy is of fundamental importance to its biocompatibility because the release of elements from the alloy is responsible for adverse biologic effects such as local and systemic toxicity, allergies and mutagenicity[55.]

Metal ceramic restorations and cast partial denture that are largely exposed to conducive oral environment may exhibit greater corrosion tendencies. The concern about the corrosion tendency has greatly intensified in recent years after the introduction of several base metal alloys. With the advent of various new commercially available Nickel Chromium alloys in day to day practice, the scientific evaluation of corrosive behavior during the process of development of new dental alloys is highly recommended and hence the purpose of this study was to evaluate and compare the corrosive behavior of four commercially available Nickel Chromium alloys in simulated oral environment.

An in vitro corrosion study had been chosen to evaluate the corrosive behavior of base metal alloys as they are faster, less expensive and less controversial than studies involving animal and human subjects[24]. The corrosion property of base metal alloys has been determined by various chemical and electrochemical methods[18]. The chemical methods are by the mass loss of corroding metal and alloy, amount of corrosion products in corrosive media and by the amount of gas produced during corrosion reaction. The electrochemical methods are potentiostatic /linear and potentiodyanamic /cyclic polarization tests in various solutions. [19]

The corrosion study was carried out in artificial saliva [79, 80, 81] at room temperature which was used to simulate the oral environment with pH maintained at 6.7 ± 0.4. Sodium chloride was added to this electrolyte to simulate an aggressive electrolyte[82], who reported that electrolytes with high chloride content are much more reactive than natural saliva.

Various parameters such as Corrosive potential **(Ecorr)**, Break down potential (**Eb**), Repassivation potential (**Ep**) and corrosion current densities (**Icorr**) were determined by cyclic polarization test to assess the corrosive behavior of base metal alloys as the mass loss of these alloys were minimal.

C**orrosion potential (Ecorr):**

The initial potentials of the samples were recorded as soon as the sample / working electrode were immersed in the electrolyte. This was monitored until a constant potential was reached and this represents the **corrosion potential [open circuit potential / steady state potential/ reference** potential.] of that particular alloy. It is at this potential that the anodic [oxidation] and cathodic [reduction] reaction of an alloy in the electrolyte solution are in the equilibrium state [83] and at which the corrosion study could be initiated

This potential shifted towards the more positive direction with a greater shift in the beginning and then attained a steady state value after a lapse of 8-10mts in both base metal groups and control group. [Fig 3]. This displacement of corrosion potential towards more positive direction for all alloy samples could be due to the existence of a more protective surface oxide layer[54] as it is the inherent tendency of chromium and molybdenum to provide a passive layer on the surface of the alloy[84].

The mean corrosion potential [Ecorr] value of group I,II,III,IV and V are -219.5 ,-161.5,-128.3,-155.4 and -119.3 respectively[Table X]. The mean corrosion potential [Ecorr] of the noble alloy was significantly higher than the base metal alloy groups, which is mainly attributed to the high inertness of the noble alloy [Table XIV].Within the base metal alloy groups, the mean Corrosion potential [Ecorr] of Group V was significantly higher than the other study groups because of the presence of Silica [2%] which reduces the oxidation of an alloy [Table XV] , which goes in accordance studies done by **Gregory and Lewis**[85,86]and the mean corrosion potential [Ecorr] of Group IV was significant than group I and III because of presence of 1.5% of silica [Table XVI].The electrode potential of the noble alloy is positive ,hence they are hardly soluble. The electrode potential of base metal alloys is negative and hence they tend to dissolve easily and move to ionic state[1].

Nickel-Chromium base metal alloys rely on Chromium and Molybdenum surface oxide layer for resistance to corrosion in the oral environment[54] The oxides layer from the constituents of the alloy are formed because of the rapid oxygen uptake from the atmosphere after polishing [87,88] . According to **Bumgardner**[89], Nickel - Chromium alloys containing 16-27 % chromium develop an adequate protective oxide layer.

Molybdenum also plays an important role in oxide layer formation as described by **Y.C Lu and C.R Clayton** [90,91]Molybdenum oxide layer as MoO_2-was seen in all passive films formed on Molybdenum containing alloys in solutions, as they possess a bipolar film comprising of an extrinsic cation layer and an intrinsic anion layer . This bipolar layer acts as an interfacial barrier. This passive oxide film formed by Nickel, Chromium and Molybdenum by XPS analysis were evaluated by **Suresh**[92] and he concluded that NiO , Ni[OH]2 ,Cr2O3, Cr[OH]3 and Mo4+ are formed on the surface of an alloy for corrosion resistance. The lower rate of corrosion of these alloys were mainly attributed to the presence of Chromium, Molybdenum surface oxide levels.

Breakdown potential [Eb]:

Cyclic polarization tests was initiated by applying a potential below the corrosion potential [Ecorr] and increased towards the positive direction [anodic scan] at a scan rate of 1 mV/s and this anodic scan was continued until threshold current density of .1µA /cm2 was reached. During this period, the alloy enters a transpassive state characterized by pitting indicating a protective oxide layer breakdown [Eb]. At this breakdown potential pits grow faster resulting in greater release of metal ions into the test solutions.

The mean Breakdown potential [Eb] of group I [control group] [1016.2] was significantly higher than other study groups[738.4, 800.1, 672.3 and 723.8] indicating that the high noble alloy demonstrated the highest Eb Value followed by Group III, Group II , Group V and finally Group IV alloys [table XVII].This value is in accordance with **Marek**[82]who concluded that noble alloys exhibit a single phase, uniform homogenization, grain refinement and high thermodynamic stability.

The mean Breakdown potential [Eb] of Group III was significantly higher than other three base metal groups [Table XVIII and XIX]. , because of the presence of 11% Mo, 25% Cr and 1.0 % of manganese which is responsible for increase in Eb value [808Mv]. This finding goes in accordance with **Pourbaix**[93,] **who** reported that a small addition of manganese increases the corrosion resistance of an alloy. It is also reported that the molybdenum plays an important role in grain refinement [1]. Group IV alloy exhibits low breakdown potential [Eb] of 672.3, because of decrease in Molybdenum [9.5%], Chromium [22.5 %] content and absence of manganese in the alloy. The mean Breakdown potential [Eb] of Group II alloy and Group V alloy are almost similar but there is slight increase in Eb value of group II is because of higher content of Chromium [26%] when compared to group V [23%]

Thus it is evident that a high Chromium and molybdenum contents increases the breakdown potential in a positive direction and corrosion current in a negative direction, thereby improving the pitting corrosion resistance of an alloy in oral environment[94], which is in agreement with **Tullmin**[95] who stated that the improvement in breakdown potential occurs even with small addition of molybdenum. **K.Hasimoto**[96] also stated that the Molybdenum could form Mo6+ oxide in passive film thereby blocking the penetration of chloride attack and alternatively decrease the rate of dissolution by formation and retention of Molybdenum ox hydroxide at active areas. **Streicher**[97] has also suggested that the combined addition of chromium and molybdenum to alloys greatly improves their corrosion resistance in chloride solutions with molybdenum interacting synergistically with chromium

Yang et al [84] reported that the presence of molybdenum inhibits the corrosion process through the formation of a molybdenum salt film, which is apparently difficult to break. **Mitrovic**, **Seepanovic and Ives**[98] also studied the electrochemical behavior of molybdenum oxides and concluded that a mixed oxide phase type (Mo4+, Mo6+) could be present in the films on the higher molybdenum containing alloys due to its higher thermodynamic stability.

Repassivation potential [Ep]:

Once the threshold current density of 0.1 µA/cm2 was reached [breakdown potential], then the scan [cathodic scan] is reversed back to Corrosion potential [Ecorr**]** of the alloy. The potential at which the reverse cathodic scan meets the forward anodic scan is termed the Repassivation potential, [**Ep**.]**.**This repassivation potential slows the kinetics of pit growth and hinders the development of new pits.

The mean Repassivation potential, [**Ep**] value of group I [924.7]was significant higher than other four study groups [796.8, 828.0, 778.4 and 793.8] [Table XX].This can be attributed to the presence of gold and platinum which are highly corrosion resistant and posses positive electrode potential indicating their passive activity in electrolyte solutions .

Within the four study groups, the mean Repassivation potential, [**Ep**] value of Group III[828Mv] was significantly higher than other study groups, [Table XXI, XXII] showing little or no hysterisis .This may be due to the higher percentage of molybdenum[11%] in group III. **Wanklyn**[99] stated that the addition of Molybdenum was expected to have a pronounced effect on corrosion resistance than on the mechanical properties. Several authors have studied and suggested that the repassivation potential can be improved by a small addition of molybdenum and the corrosion rate of the base metal alloys decreased as the percentage of Molybdenum increased[100, 101,102].

Corrosion current density [Icorr]:

A point of intersection on a tangent drawn on anodic and cathodic scan determines the amount of current generated during process of corrosion. This corrosion current determined is directly proportional to the rate of corrosion occurring in an alloy.

The mean Corrosion current density [Icorr**]** value of high noble alloy[0.17]was significantly higher than the base metal groups[0.31.,0.23,0.47,0.33] [Table XXIII] .This indicates that high noble alloy are more resistant to corrosion because of its superior mechanical and physical properties. The mean Corrosion current density [Icorr**]** of Group III [0.23] was lower when compared to other three study groups, showing that the Group III is more resistant to corrosion than the other three groups [Table XXIV and XV]. The corrosion current density [Icorr**]** value of Group IV is higher [0.47] indicating that this group is more prone to corrosion.This could be due to the reduced amount of chromium [22.2%] and molybdenum [9.5%] contents in the alloy which play important role in their corrosive behavior even at higher temperatures[103].

Accelerated leaching study:

To analyze the specific ions which were leached during the process of corrosion, accelerated leaching study was done on one sample from each alloy**.** The working electrodes were immersed in artificial saliva and allowed to stabilize at corrosion potential [Ecorr] and breakdown potential [Eb] for a period of one hour in 250 ml of the test solution. At the end of the experiment, the chemical composition of the test solution was analyzed by inductive coupled plasma – mass spectrometer [ICP-MS] .The elemental release data was converted to µg of element released per cm^2 of the exposed alloy surface.

There were no specific ions released during the accelerated leaching study in group I. The results represent the relative stability and nobility of group I alloys. The results of this study confirmed the observation by **Wataha** [7] and **Schalmz**[104] who all stated that the gold and platinum ion release are always below detection limits.

When the base metal alloy groups were subjected to ICP-MS, there were no specific ions in the test solutions at Ecorr .However at Eb [breakdown potential], the average value of Nickel and Chromium ions released were 60-68 $\mu g/cm^2$ and 50-53$\mu g/cm^2$ respectively after one hour and approximately 1440$\mu g/cm^2$ and 1200$\mu g/cm^2$ after 24hrs which may be 3 times greater than the dietary intake of Nickel and Chromium ions which are suggested to be in the range of 400 and 240 µg in about 24 hours[14] .The exaggerated values in this study could be attributed to the testing conditions which were done intentionally to determine the specific ions leached during the study.

The leaching of metal ions from the alloy involves the adsorption of aggressive halide ions at discrete sites on a passive metal surface. This process is a continuous thinning of a passive film until the bare metal surface is reached at the end of the induction period. **Kruger**[105.] .**Hoar and Jacob**[106] suggested the formation of transitional complex by adsorption of halide ions on the surface of the passive film around a lattice cation. Once the complex is formed, it will readily remove the cation from the passive film lattice. Thus thinning of the film occurs at the site where the complex is formed resulting in a stronger anodic field. This field pulls another cation through the thinned site of the film resulting in a soluble complex formation causing traverse of cations from the passive film until the bare metal is reached.

SEM Observation:

The morphology of the pit of corroded alloy samples was evaluated in terms of pit dimensions and depth [107]**.** The surface topography of high noble alloys reveal no pit formation. [Fig29].The pit dimensions of Nickel Chromium alloys indicate their potential to corrosion. The pit size was greater in Group IV [Fig32] indicating that they are more prone to corrosion followed by Group V [Fig33], Group II [Fig30] and Group III [Fig:31] which are least prone to corrosion.

Nickel –chromium alloy are commonly used in day to day practice, but these alloys are prone to corrosion when compared to noble and high noble alloys and hence certain precautions are mandatory to enhance the biological safety of these alloys. Since the release of ions are maximum in the first week after insertion ,the cytotoxicity of Nickel-Chromium alloy could be reduced by preconditioning the alloys in distilled water prior to insertion[53]. They also release more ions significantly when they are subjected to acidic environment [pH-1-4] during intake of acidic foods[42] and tooth brushing[52]. Hence rinsing with water after food intake is suggested to reduce the cytotoxic potential of these alloys. Patient who report with a history of oral symptoms due to metallic restoration such as swelling, pain, redness and lichenoid reactions should be subjected to thorough general and dental examination[63] and an alternative treatment protocol can be recommended. Removable prosthesis should not be cleansed with a hypochlorite solution as they result in greater tendency to corrode[108].

Further studies are needed to determine the cellular toxicity of these alloys in the oral environment and by the introduction of single phase alloys which minimizes corrosion.

One of the most important factors affecting the choice of dental alloys is its biocompatibility and resistance to tarnish and corrosion. Corrosion of an alloy occurs when elements in the alloy ionize .It is a chemical property of an alloy that affects other properties such as esthetics, strength and biocompatibility. The purpose of this study was to evaluate and compare the corrosive behavior of four commercially available nickels – chromium alloy with high noble as the control group.

In this study ,forty samples from four nickel –chromium alloys and five samples from high noble alloy were subjected to cyclic polarization test and its corrosive behavior were evaluated. Four parameters such as Corrosion potential [Ecorr], breakdown potential [Eb], repassivation potential [Ep] and corrosion current [Icorr] were analyzed to evaluate the corrosive behavior. The specific elements released from the alloys were determined by inductive coupled plasma –mass spectrometer.

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

1. High noble alloys are more resistant to corrosion than the base metal alloys
2. Within the base metal alloys study groups, Group III behaves to be more resistant to corrosion followed by Group II, Group V, Group IV

3. Both Nickel and Chromium ions were released during the corrosion process, but the percentage of Nickel ions was relatively higher than Chromium ions.

Base metal alloys are generally used due to their good mechanical properties and low cost however certain precautions should be taken to ensure biological safety of metal ceramic restoration.

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Fig 5:Machined sample die

Fig 6:Armamentarium for sample preparation

Fig 7: Ceramet and Bellabond plus -Nickel – Chromium alloys

Fig 8: Heraenium S and 4 all

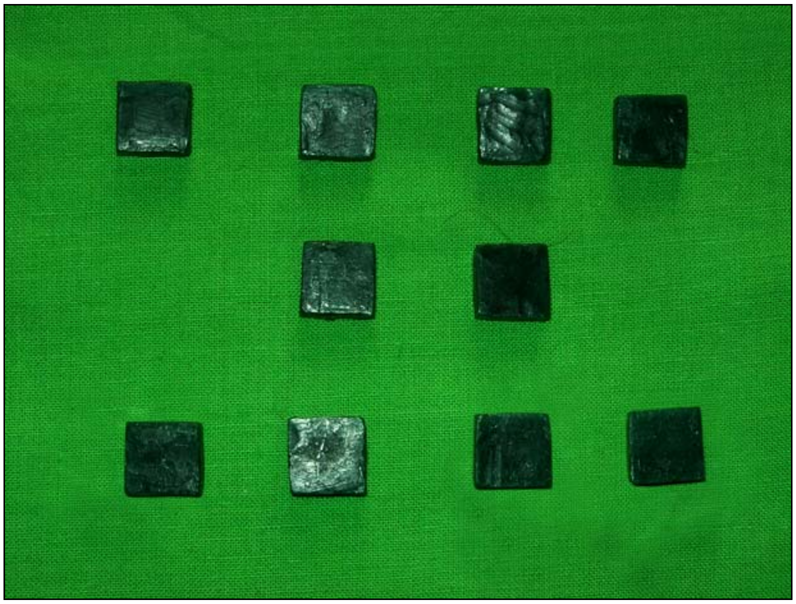

Fig 9: Fabricated wax pattern

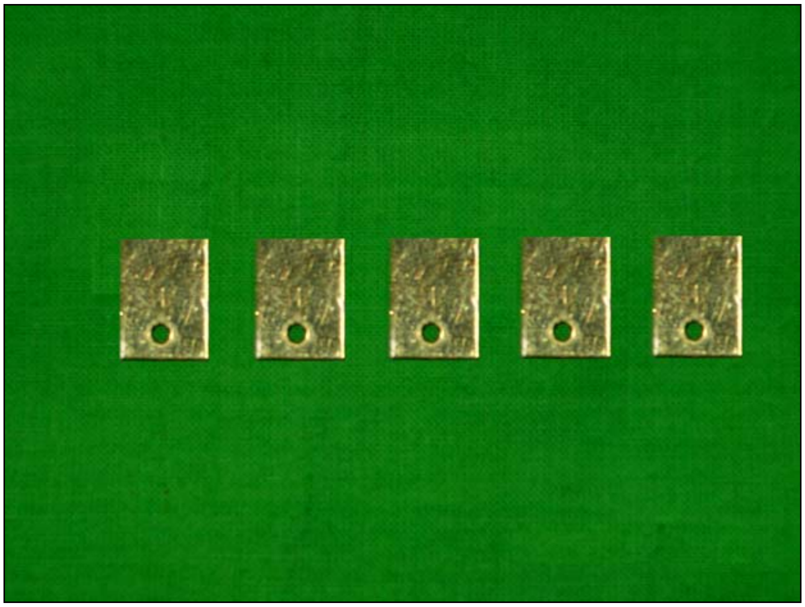

Fig10: High noble alloy samples

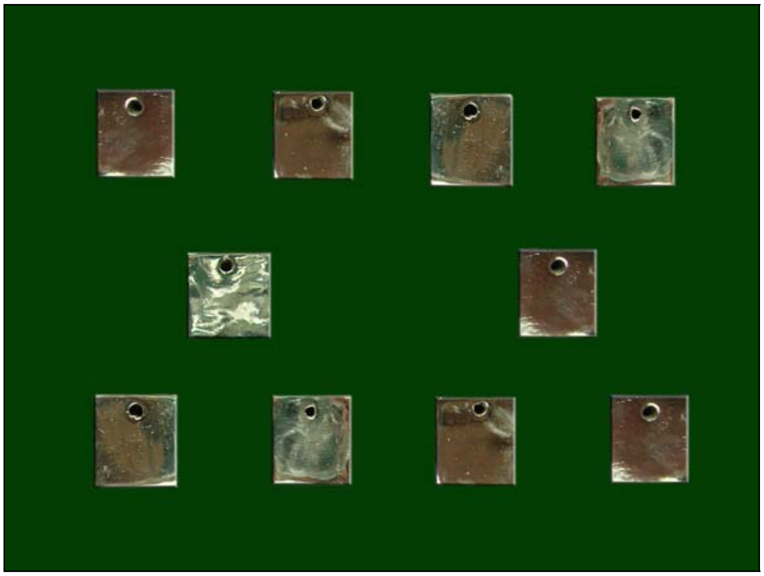

Fig13:Nickel –Chromium alloy samples

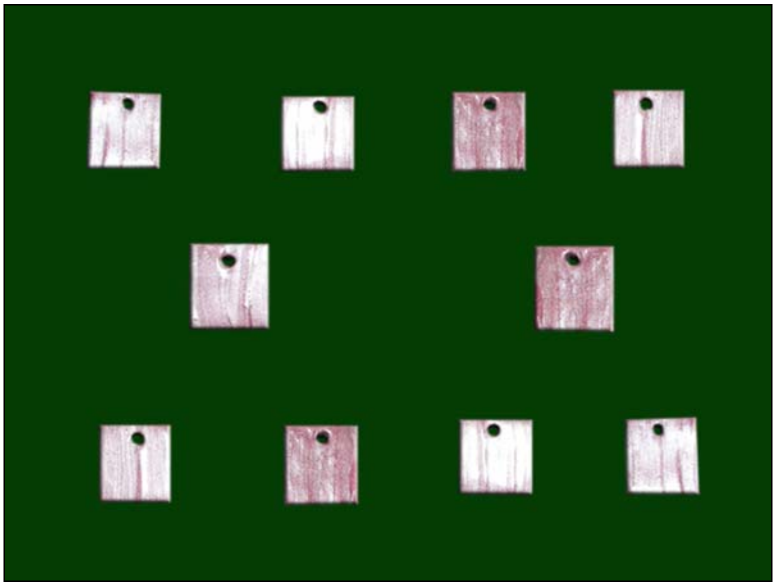

Fig14:Nickel –Chromium alloy samples covered with lacquer

Fig 15:Armamentarium for artificial saliva preparation

Fig16: Prepared Artificial Saliva

Fig 17:Electronic Balance DHONA-260D

Fig 18:Vibrant Potentiostat VSM /CS/30

Fig 19:Three unit Electrode cell

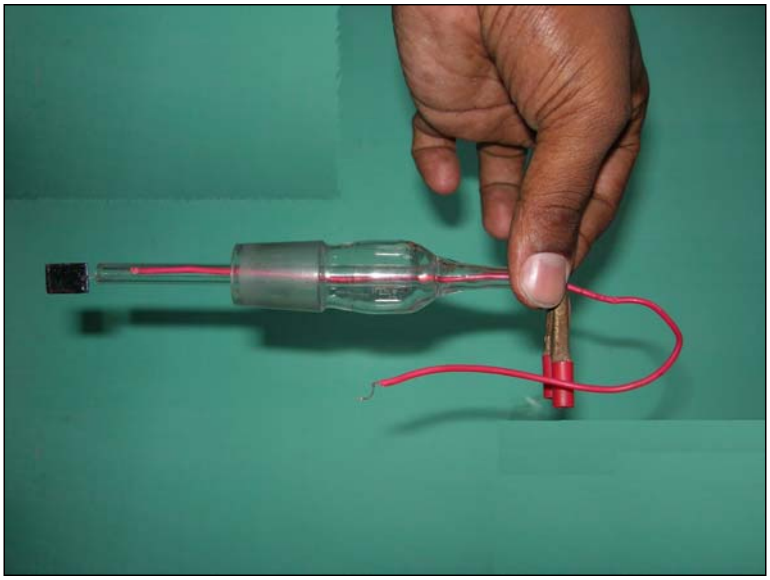

Fig20:Sample / Working electrode

Fig11:Digital ultrasonic cleanser-Unikleen

Fig 12: Samples in Digital ultrasonic cleanser

Fig 21:Stereozoom Optical Microscope –UM-1530R-Hongkong

Fig22: Scanning electron microscope –[FEI 200, Quanta]

Fig23:Inductive coupled plasma mass spectrometer – Agilent 7500

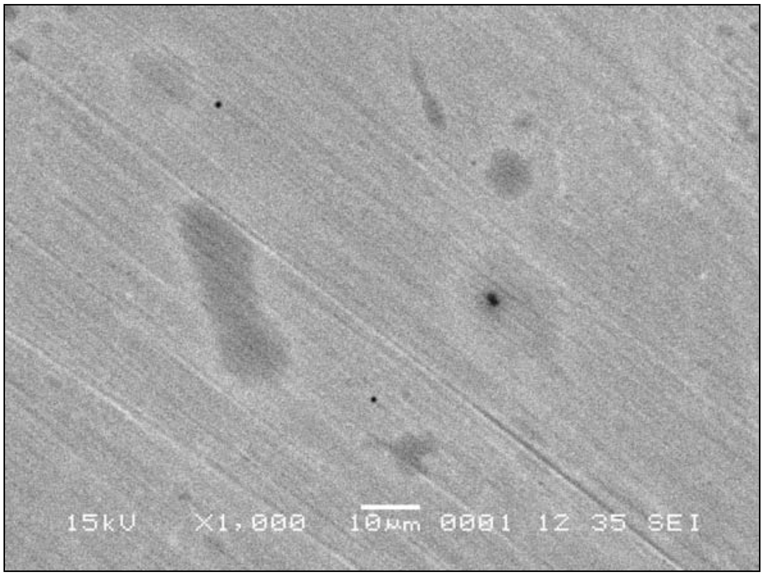

Fig 29:SEM Photomicrograph of Group I

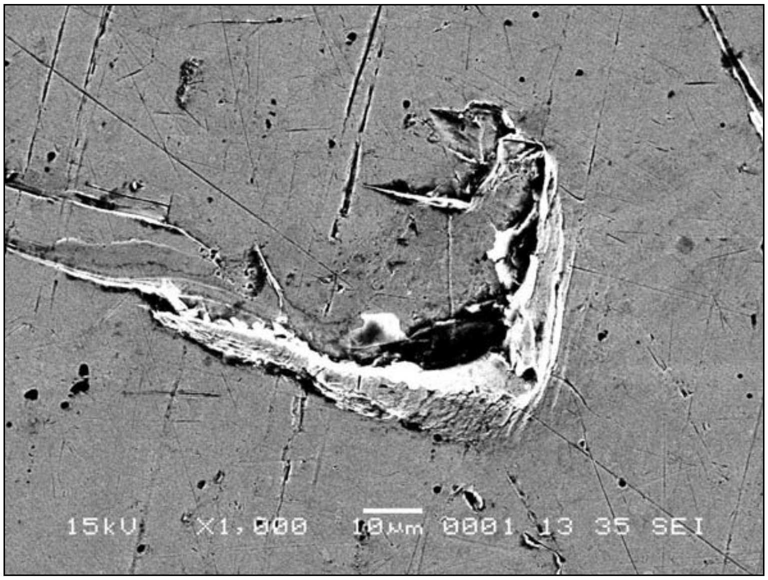

Fig 30 : SEM Photomicrograph of Group II

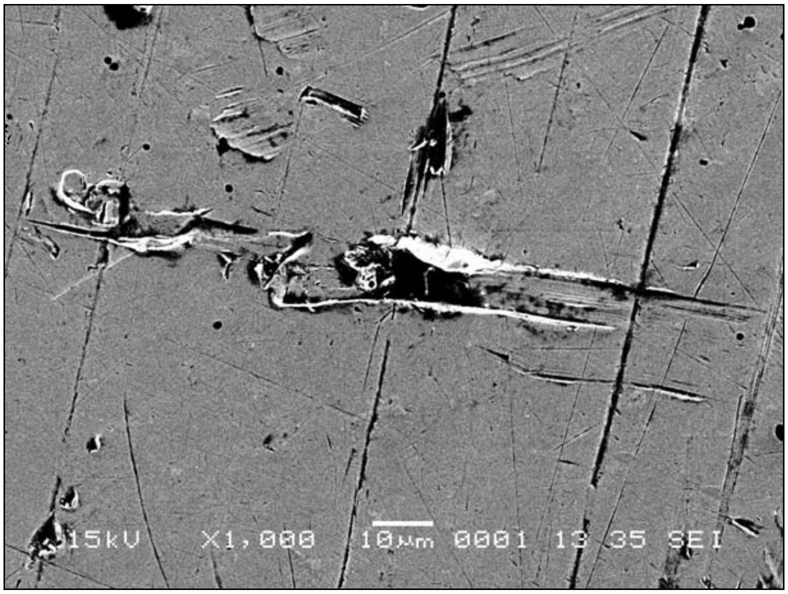

Fig 31 : SEM Photomicrograph of Group III

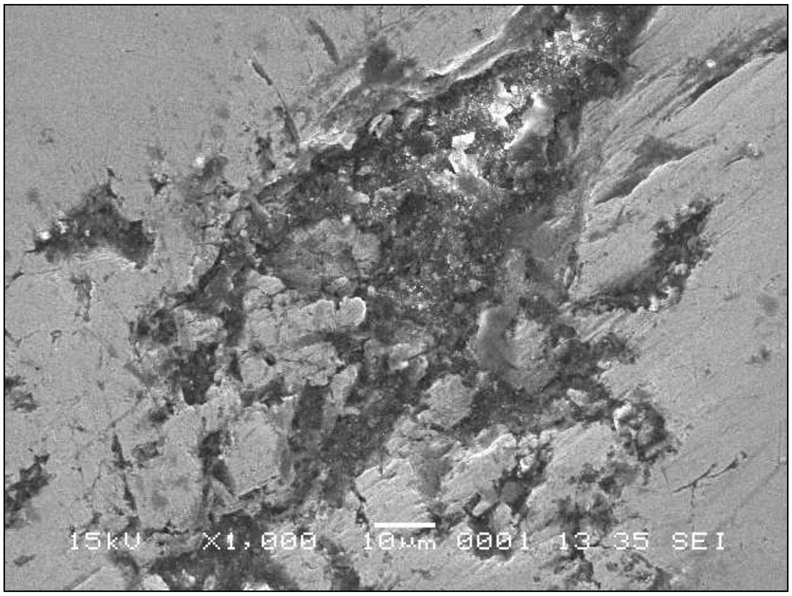

Fig 32 : SEM Photomicrograph of Group IV

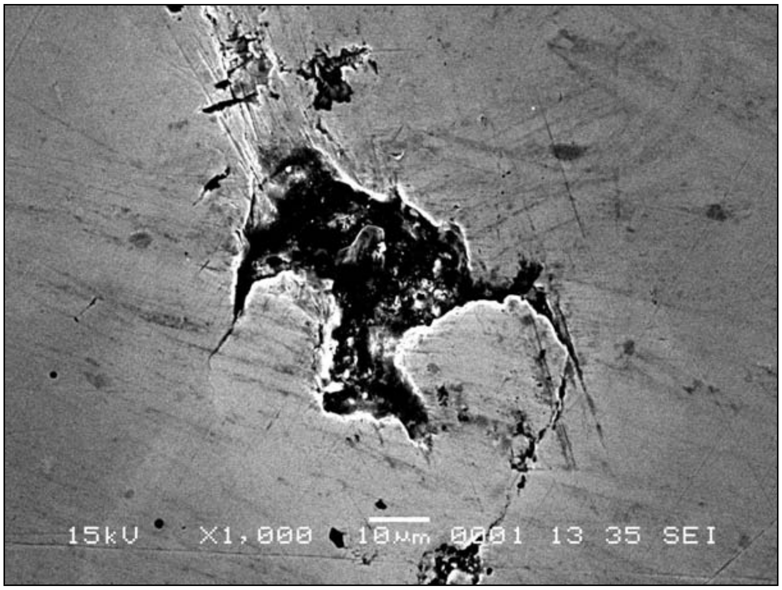

Fig 33 : SEM Photomicrograph of Group V

Fig 24: Optical microscopic view of Group I

Fig 25: Optical microscope view of Group II

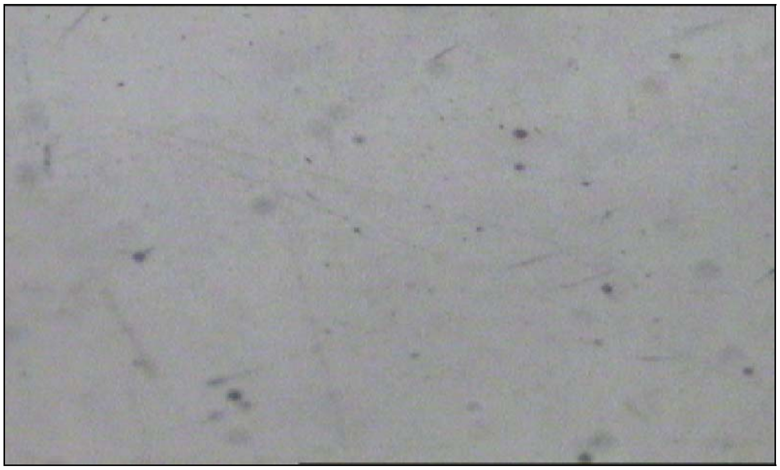

Fig 26 : Optical microscopic view of Group III

Fig 27 :Optical microscopic view of Group IV

Fig 28 :Optical microscopic view of Group V