CORRELATION OF CHEMICAL PROPERTIES OF A-NITI WIRES WITH MECHANICAL PROPERTIES AN IN-VITRO STUDY

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In Partial Fulfilment of the requirements for the Degree of

MASTER OF DENTAL SURGERY

BRANCH – V

ORTHODONTICS AND DENTOFACIAL ORTHOPAEDICS

2016 – 2019

CERTIFICATE

This is to certify that **Dr. R. Durga**, Post Graduate student (2016–2019) in the Department of Orthodontics and Dentofacial Orthopaedics, Tamil Nadu Government Dental College and Hospital, Chennai – 600 003 has done this dissertation titled**"CORRELATION OF CHEMICAL PROPERTIES OF A-NITI WIRES WITH MECHANICAL PROPERTIES – AN INVITRO STUDY"**under my direct guidance and supervision in partial fulfilment of the M.D.S degree examination in May 2019 as per the regulations laid down by The Tamil Nadu Dr. M.G.R. Medical University, Chennai – 600 032 for M.D.S**.,** Orthodontics and Dentofacial orthopaedics (Branch – v) degree examination.

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DECLARATION

I,**Dr. R. Durga** declare that this dissertation titled**"CORRELATION OF CHEMICAL PROPERTIES OF A-Niti WIRES WITH MECHANICAL PROPERTIES – AN INVITRO STUDY"** was done in the Department of Orthodontics, Tamil Nadu Government Dental College & Hospital, Chennai 600 003.I have utilized the facilities provided in the Government Dental College for the study in partial fulfilment of the requirements for the degree of Master of Dental Surgery in the specialty of Orthodontics and Dentofacial Orthopaedics (Branch V) during the course period **2016-2019** under the conceptualization and guidance of my dissertation guide, **Professor Dr. SRIDHAR PREMKUMAR, M.D.S.,**

I declare that no part of the dissertation will be utilized for gaining financial assistance for research or other promotions without obtaining prior permission from the Tamil Nadu Government Dental College & Hospital.

I also declare that no part of this work will be published either in the print or electronic media except with those who have been actively involved in this dissertation work and I firmly affirm that the right to preserve or publish this work rests solely with the prior permission of the Principal, Tamil Nadu Government Dental College & Hospital, Chennai 600 003, but with the vested right that I shall be cited as the author(s).

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I take this opportunity to express my gratitude to my friends andcolleagues for their valuable help and suggestions throughout this study.

TRIPARTITE AGREEMENT

This agreement herein after the “Agreement” is entered into on this day...............January 2020 between the Tamil Nadu Government Dental College and Hospital represented by its Principal having address at Tamilnadu Government Dental college and Hospital, Chennai-03, (hereafter referred to as ,’the college’)

Dr.R.Durga aged 31 years currently studying **M.D.S (Orthodontics)** in Tamilnadu Government Dental College and Hospital (hereinafter referred to as the ‘PG/Research student and -Principal investigator’).

And

Dr. SRIDHAR PREMKUMAR, M.D.S.,aged 52 years working as Professor &Head in theDepartment of Orthodontics and Dentofacial orthopaedics, at the college, having residence address at B-3, Block 2 Jains Ashraya phase III, Arcot Road Virugambakkam, Chennai-92, (herein after referred to as the ‘co-investigator’)

And

Whereas the ‘PG/Research student as part of his curriculum undertakes to researchon **“CORRELATION OF CHEMICAL PROPERTIES OF A-NITI WIRES WITH MECHANICAL PROPERTIES – AN INVITRO STUDY”** for which purpose the PG/co- investigator shall act as principal investigator and the college shall provide the requisite infrastructure based on availability and also provide facility to the PG/Research student as to the extent possible as a Principal-investigator.

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Witness **Student Guide**

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CERTIFICATE – II

This is to certify that this dissertation work titled **"CORRELATION OF CHEMICAL PROPERTIES OF A-NITI WIRES WITH MECHANICAL PROPERTIES – AN INVITRO STUDY"** by the candidate, **DR. R. DURGA,** with Registration Number **241619002** for the award of **MASTER OF DENTAL SURGERY** in the branch of **ORTHODONTICS AND DENTOFACIAL ORTHOPAEDICS (BRANCH V)**. I personally verified the urkund.com website for the purpose of plagiarism check. I found that the uploaded thesis file contains from introduction to conclusion pages and results shows **4%** percentage of plagiarism from in the dissertation.

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LIST OF ABBREVIATIONS

NiTi	NICKEL TITANIUM
SS	STAINLESS STEEL
M	MARTRENSITIC
A	AUSTENITE
TTR	TEMPERATURE TRANSITION RANGE
UTS	ULTIMATE TENSILE STRENGTH
TEM	TRANSMISSION ELECTRON MICROSCOPE
SEM	SCANNING ELECTRON MICROSCOPE
HV	HARDNESS VICKERS
FET	FIELD EFFECT TRANSISTOR
FET	FIELD EFFECT IMAGING
EDS	ENERGY DISPERSION SPECTROSCOPY
EDP	ESSENTIAL DENTAL PRODUCTS
AO	AMERICAN ORTHODONTICS

INTRODUCTION

Ever since Anderson introduced nickel titanium in the orthodontic field in the year 1971[46], it has become an integral part of orthodontic mechanotherapy due to their characteristics of low stiffness, superelesticity, and high reversibility. Nitinol is highly useful in the cases with severe crowding that requires large deflections with light continuous forces[53] . This increases the clinical efficiency by reducing the overall treatment time with fewer activations and attachment which is needed in regular orthodontic treatment mechanics[48].

NiTi alloy exists in two crystalline forms, martensitic (M-NiTi) and Austenitic (A-NiTi) depending on the temperature induced crystallographic transformation which is called temperature transition range (TTR) [1, 6]. The Austenitic phase occurs at low stress, higher temperature above the TTR and has body centered cubic crystal structure (BCC) in ordered fashion. The martensitic NiTi forms at higher stresses and lower temperature below the TTR and have the distorted mono, triclinic and hexagonal structure[1]. The wire is manufactured, shaped at temperature above the TTR and cold drawn and deformed into any configuration below the TTR[47]. The cold drawn wire recovers its original shape completely when heated through TTR. This phenomenon is called shape memory, associated with martensitic to austenitic reversible phase transformation, due to crystallographic twinning at atomic level. An intermediate R-phase with rhombhohedral crystal structure exists between this transformation phases[49, 52]. The addition of third elements like copper and cobalt to the alloy system induces thermo elastic behaviour with martensitic active NiTi which has accurate control of TTR by reducing the hysteresis[1].This type of martensitic-active (M-Active) thermo elastic NiTi archwires helps to align the severe crowding in initial phase of treatment. The phase transformation occurs due to stress, the elasticity is known as superelesticity or pseudo elasticity as a result of austenitic to martensitic not due a change of temperature but to force applied which results in non-elastic, non-linear loading and unloading curve [2-4], this group consists of Chinese[50] and Japanese NiTi.

The presence of trace elements or impurities in the NiTi can have effects of reducing TTR to 220˚C, narrow hysteresis e.g., adding copper[54] and cobalt reduces the hysteresis to about 10-15˚C, niobium can expands the hysteresis over 100˚C and dissolved interstitial elements of small atoms such as 0, N, C disrupts the matrices which affects the mechanical properties of alloy consequently the shape memory and superelesticity behaviour of archwires. Fe, Al, Co, Cr, etc. may be added to increase the stiffness of alloy[1, 5].

The varying clinical performance within A-NiTi groups like Chinese Japanese NiTi[50, 51]might be due to difference in chemical composition and its structure which consequently affects in the mechanical properties. The purpose of the study is to compare the correlation of mechanical properties with chemical properties in four different commercially available A- NiTi wires

AIMS AND OBJECTIVES

AIM OF THE STUDY

The aim of this in-vitro study is to correlate the chemical properties with mechanical properties of four different commercially available A-NiTi wires.

OBJECTIVES OF THE STUDY

- To measure the mechanical properties of widely using four different A-NiTi wires.

  1. Micro hardness
  2. Microstructure
  3. Ultimate tensile strength (UTS)
  4. Percentage elongation

- To assess the quantitative and qualitative chemical properties in each group of A-NiTi.

- Correlating the mechanical properties with chemical properties in each group of A-NiTi

REVIEW OF LITERATURE

J.**Fercec et al (2014)[7].,**studied microstructure observation of six commercially available SMA NITI orthodontic wires with diameter of 0.014 by using transmission electron microscope (TEM) and determined chemical composition using electron dispersion X-ray spectroscopy in stress free conditions and on different regions within the volume to find starting microstructure and it final influence on functional behaviour of each orthodontic wire and concluded that particle size are in range from 0.2 to 5µm approximately. The austenitic phase contains Ti2Ni and/or TiC particles which are crystallized matrix of grain size ranges from 50 to 160 µm

Hunt, Cunningham et al[8](1999), investigated polishing effects on surface hardness and corrosion of four different alloys archwires in stainless steel, beta titanium, cobalt chromium and NiTi. Each type divided into polished group, an industrially polished to provide a uniform surface finish and non polished group and concluded that nickel titanium showed greatest corrosion density in non-polished group when compared to other alloyed archwires. Micro hardness testing of the wire surfaces of each alloy showed no significant work-handed occurred due to polishing.

Deepak Kapoor[21](Johnson Matthey Technol. Rev- 2017), reviewed Nitinol for Medical Applications described due to its properties like biocompatibility, superelasticity and fatigue and kink resistance,. Nitinol is used to manufacture catheter tubes, guide wires, stone retrieval baskets, filters, needles, dental files and archwires and other surgical instruments. As the use becomes more widespread for more than thirty years, the industry is faced to interrogate tests the check the capability of the material. Chemical composition is carefully controlled during melting and recent developments in melting have yielded materials with low inclusion sizes, a key to improving the fatigue life of the material. The performance of the product can be refined by thermo mechanical processing and one can tailor properties to meet functional requirements. Superior concentricity and surface uniformity of tube inner diameter help stent manufacturers increase yields. Products with small form factors are being used for physiological conditions that cannot be tackled with conventional materials. The unique properties of the material make it a desirable choice for product designers whose design ambitions would probably be limited without the versatility of such a material.

B. N. Coelho et al [9](2015) Automatic Vickers Micro hardness Measurement based on Image Analysis thatexplained aboutproblem associated with this technique is the influence of the human operator on the measures performed. Aiming to reduce, or even eliminate, the dispersion of results, this work proposes to design an image analysis-based method for automatic identification of sample surface printed and determination of its dimensions. The data used for its calibration is obtained from tests on samples of steel, cast iron, and aluminium and cooper alloys. The experimental results corroborate the method capability for performing the required measures with good precision and excellent reproducibility.

Daniel Jogaib Fernandez et al[10] (2015),studied about Mechanical Performance of commercially available six Nickel-titanium Archwires Superelastic (SE) and heat-activated (HA) NiTi archwires from two lots of six manufacturers (3M, GAC, Tp, Orthosource, Orthometric and Morelli) were partitioned into eleven groups (n=132) and tested under three-point bending tests and concluded that HA archwires showed better mechanical performance than SE ones, with lower stress level on loading and unloading plateau, greater mechanical hysteresis and thus, lower forces should be delivery to tooth. There was no evidence of shape memory effect behaviour by the HA wires.

Jose Fernando Castanha Henriques et al[11] (2017), Evaluated deflection forces of orthodontic archwires with different ligation types. Stainless steel, conventional nickel-titanium and thermally activated nickel-titanium archwires tied into conventional brackets by a ring-shaped elastomeric ligature (RSEL), an 8-shaped elastomeric ligature (8SEL) and a metal ligature (ML) were tested. Forces were measured with an Instron Universal Testing Machine. lighter forces, the thermally activated nickel-titanium wire with the RSEL are recommended, while the steel wire with the 8SEL or the ML are recommended when larger forces are desired. The ML exhibited the highest force increase with increased deflections, compared with the elastomeric ligatures.

Marcelo Faria da Silva[12], (2016), an in-vitro study aimed to deflection force behaviour of 106 segment of 0.01980.025 NiTi ad 0.01680.025 heat activated nickel titanium orthodontic wires in four commercially available brands. Divided into heat activated group (n=40) and heating-free group (n=40) according to the distal end heat treatment. Deflection analysed with temperature controlled universal testing machine. There were no statistically significant differences between the tested groups with the same size and brand of wire and Concluded that Heat treatment applied to the distal ends of rectangular NiTi archwires does not permanently change the elastic properties of the adjacent portions.

Eng. Aseel Mohammed Ali Hussein[13] (2012), tested mechanical properties of stainless steel and titanium orthodontic wires of size 0.4*0.4 mm, tested modulus of elasticity (E), yield strength (Sy), ultimate tensile strength (UTS), modulus of resilience (Ur).A standard tensile test was performed in a WP 300 universal Material Tester with the gauge length of each specimen was set to 100 mm. The crosshead speed was set to 1mm/min. titanium wire shows a higher elastic material with a large amount of spring back, than stainless steel, with a slightly little brittle nature and this confirms that, for clinical applications, the wires of titanium should be used at the beginning of treatment, when severe misalignments require large activation, and the wires of stainless steel should be used in the later stages of treatment, where applied forces are less significant.

Marco Abdo Gravina et al [13] (2014), compared the qualitative chemical compositions and the surface morphology of fracture regions of eight types of Nickel (Ni) Titanium (Ti) conventional wires, superelastic and heat-activated (GAC, TP, Ormco, Masel, Morelli and Unitek), to the wires with addition of copper (CuNiTi 27oC and 35oC, Ormco) after traction test.

Neeraj Sharma et al[14] (2015) Nitinol has various applications in biomedical, automotive actuators, micro-electromechanical systems (MEMSs), actuators, energy conversion devices and aero-space industries due to its distinctive properties of pseudo-elasticity, bio-compatibility, corrosion resistance and shape memory effect.

Torstein R. Meling[15] (2001), compared the effects of transient alterations in temperature on the elastic responses of superelastic NiTi-based archwires to orthodontic bending during the activation and the deactivation phases. When a superelastic wire is subjected to cold water during its activation phase, the bending force that is exerted drops and remains sub baseline until the wire is heated or deactivated and reactivated. In contrast, the effect of brief cooling is transient when the wire is in its deactivation phase. Brief heating induces a transient increase in wire stiffness when the wire is in its activation phase, but the effect is prolonged and probably permanent as long as the strain remains constant, when the wire is heated during the deactivation.

Hossein Aghili et al[16] (2017) Evaluation of the effect of three mouthwashes on the mechanical properties and surface morphology of several orthodontic wires: An in-vitro study

This study compared the changes seen in mechanical properties and surface morphology of three different orthodontic wires after immersion in mouthwash solutions.

1. All mouthwashes significantly reduced the stiffness of SS wires

2. Fluoride treatment caused a reduction in stiffness of NiTi wires whereas CHX and ZM increased its stiffness

3. Both Fluoride and CHX mouthwashes increased the stiffness of coated wires, but ZM decreased it

4. All mouthwashes i.e. Fluoride, CHX and ZM caused a change in loading and unloading forces and surface morphology of different orthodontic wires, which might have an impact onthe mechanical properties during orthodontic treatment.

Hossein Aghili, Sogra Yasssaei et al [17] (2015) studied Load Deflection Characteristics of Nickel Titanium Initial Archwires. This study assessed and compared the characteristics of commonly used initial orthodontic archwires with their load deflection graphs

1. Load/deflection characteristics of wires vary depending on the bracket type and the deflection amount.

2. The NiTi wires and MSNT wires with all types of brackets and with deflection of 2mm and 4mm, generated the highest and lowest average plateau forces, respectively.

3. By increasing the primary amount of deflection, the force generated by the wire in the plateau phase remained constant while the plateau length increased.

4. There was a greater average plateau force when SLB brackets were used which might be due to lower frictional force between the wire and the bracket.

Nawal Mohammed Dawood et al[18]'Fabrication of Porous NiTi Shape Memory Alloy Objects by Powder Metallurgy for Biomedical Applications, This study evaluated the effects of compacting pressure (150-450) MPa and Copper additions (2.5, 5, 7.5 and10) wt.% on the mechanical properties of NiTi alloys produced by powder metallurgy.

1. When copper with (2.5-10) wt. % is added to NiTi prepared alloy, it reduced the hardness relatively compared to master samples without addition of copper.
2. Hardness test showed an increase in SME properties in the prepared NiTi alloy with the addition of copper.
3. The yield strength (σy), compressive strength (σcom), and modulus of elasticity (E) have raise with increased copper addition to NiTi alloys.
4. The value of E of the porous NiTi ranged from (2. 5 – 8. 8) GPa for samples compacted at 450 MPa which is almost nearer to that of the cortical bone (10-30) GPa and thus the danger of stress shielding after implantation in human body is reduced.

Saba Ammar Mohammed[22]**(2019),**an in vitro study evaluated the roughness of Copper Nickel Titanium arch wires from different brands in dry and wet condition (acidic and neutral artificial saliva).Orthotechnology brand CuNiTi wires exhibited the greatest roughness while Ormco brand exhibited the minimum roughness when compared to other equivalent archwires.

A statistically significant difference existed in surface roughness between groups of archwires from different companies in wet condition while non-significant difference existed between groups of archwires from different companies in dry condition.

Abdul Razzak et al[23] (2015)' studied to show that new heat-activated NiTi wireshad more rough surfaces than superelasticNiTi wires,whereas both types released almost the same tinyamounts of Ni ions in the artificial saliva. NiTi wires surface roughness significantly increased after clinical exposure, whereas the amount of their released Ni ions in the artificial saliva decreased; an increase of surfaceroughness was observed to a greater extent in superelastic NiTi wires compared to the heat-activated ones,whereas the difference between them regarding the decrease of Ni ions release following oral exposure wasnot significant.

Hudgins JJ1[24](1990), the purpose of this study is to quantify permanent deformation after long-term deflection of available nickel-titanium archwires. Nine nickel-titanium, one beta-titanium and one stainless steel archwires, .016 inch round, were deflected into orthodontic brackets of simulated arch form. The nickel-titanium wires exhibited better spring back characteristics and less permanent deformation than the stainless steel and TMA wires. Several wires increased deformation as deflection time increased.

Murilo Gaby Neves et al [25](2016) compared the elastic properties of the load-deflection ratio of orthodontic wires of different lot numbers and the same commercial brand. A total of 40 nickel-titanium (NiTi) wire segments (MorelliOrtodontiaTM - Sorocaba, SP, Brazil), 0.016-in in diameter were used.

There are no changes in the elastic properties of different lots of the same commercial brand; thus, the use of different lots of the orthodontic wires used in this research didnot compromise the final outcomes of the load-deflection ratio.

Zhou et al [26](2012) evaluated the longest fatigue life of Ni-Ti wire under ultrasonic conditions when it was annealed after cold drawing. Proper thermo mechanical treatment increased the fatigue life of Ni-Ti wire due to the adjustment of microstructure in Ni-Ti alloys as well as the work hardening effect. The strengthening effect of heat treatment and cold drawing was attributed to the fine Ti3Ni4 precipitated phase and the high density of dislocations respectively. The SEM results showed smooth fractured faces with dimples existed in the propagation region of the fatigue cracks, indicating the excellent ductility and better resistance to propagation of fatigue cracks of NiTi wires.

Rerhrhayeet al[27] (2014) investigated the impact of acidic and fluoridated environment (in-vitro) on the electrochemical behaviour and the mechanical properties of orthodontic alloys in nickel titanium and in stainless steel wires. For the NiTi archwires, immersion in the fluoridated and acidic medium showed a statistically significant reduction of the Young's modulus (E), the elastic limit (σe) and the maximum tensile load (σm). Similarly, a higher level of released nickel proportionate to the increase in the fluoride concentration and acidity was observed in the immersion solutions.

Wang et al[28] (2012), studied the MAO coating prepared on the NiTi which has been Soaked in a simulated body fluid (SBF) toinvestigatethe biomimetic deposition of apatite on thesurface of the MAO coating on NiTi. It is found that the MAO coating shows an excellent apatite formingability after soaking in a SBF for 14 days.

RoslizaRazali et al[29] (2015), Processed Nickel Titanium (NiTi) alloy from elemental powders of Nickel and Titanium by conventional powder metallurgy process. Two batches offeedstock containing 2 different formulations (1) Ti-50.4 at % Ni and (2) Ti-50.8 at % Ni wereprepared. Results showed that the conventional sintering produced greater amount of the predominant NiTi (B2) phase and minor fraction of martensitic NiTi (B19') phase. All samples exhibited a reversible phasetransformation during heating and cooling. Allsamples exhibited open pores with irregular structure that are suitable for implants which provided the direct in-growth of tissue into the implants

Ines Ben Naceu et al[59] (2014) evaluated the influence of possible intraoral temperature differences on the forces exerted by NiTi orthodontic arch wires with different cross sectional shapes and sizes.

A bending test is simulated to study the force variation of an orthodontic NiTi arch wire when it loaded up to the deflection of 3 mm, for this task one half of the arch wire and the 3 adjacent brackets were modelled. The results showed that the stress required for the martensite transformation increases with the increase of cross-sectional dimensions and temperature. Associated with this increase in stress, the plateau of this transformation becomes steeper. In addition, the area of the mechanical hysteresis, measured as the difference between the forces of the upper and lower plateau, increases.

Abbas Amini et al [60](2013)stated that spherical indentation tests on NiTi SMAs show a significant increase in the hardness with indentation depths. This behaviour is not only opposite to the hardness decrease-depth increase behaviour of NiTi SMAs in indentation tests with sharp tip it is in contrast with the depth insensitivity of the spherical hardness of ordinary materials with no phase transition.

It is further shown that the anomalous behaviour of the spherical hardness in NiTi SMAs can be explained by the elastic contact theory incorporated with the effect of the phase transition phenomenon. In NiTi SMAs, the phase transition weakens the depth dependency of the spherical hardness.

Uzer et al [61] (2016) investigated the biocompatibility of Nickel-Titanium (NiTi) archwires Archwires (undeformed, and bound to brackets on acrylic dental molds) were statically immersed in artificial saliva (AS) for 31 days. Then analysed with the aid of various electron-optical techniques and it was observed that carbon-rich corrosion products formed on both archwire sets upon immersion. The corrosion products preferentially formed at the archwire–bracket contact zones, which is promoted by the high energy of these regions and the micro-cracks brought about by stress assisted corrosion. Moreover, it is suggested that these corrosion products prevented significant Ni or Ti ion release by blocking the micro-cracks, which, otherwise, would have led to enhanced ion release during immersion.

Mauro Dolce et al [62] (2001) analysed the most important outcomes of the torsion tests in terms of three fundamental mechanical quantities: secant stiffness, energy loss and equivalent damping. The SMA specimens were different in shape (wires and bars with different diameter), physical characteristics (alloy composition, thermo mechanical treatment and material phase) and stress mode (tension, torsion, bending and shear). The experimental results showed that SMA bars subjected to torsion, especially the martensitic ones, have great potential for their use in seismic devices due to their considerable energy dissipation capacity and outstanding fatigue resistance

N. H. Faisal et al [63] (2017) studied the mechanisms of Nano scale fatigue of functionally graded TiN/TiNi films multiple-loading cycle Nano indentation and Nano-impact tests. The results were sensitive to the applied load, loading mode (e.g. semistatic, dynamic) and probe geometry. Based on indentation force–depth profiles, depth–time data and post-test surface observations of films, it was concluded that the shape of the indenter is critical to induce localised indentation stress and film failure, and generation of pseudo-elasticity at a lower load range. Finite-element simulation of the elastic loading process indicated that the location of subsurface maximum stress near the interface influences the backward depth deviation type of film failure.

Xiao Ji Li et al[64] (2008) investigated the influence of fluoride on stress corrosion cracking (SCC) of NiTi orthodontic wires using slow strain rate test (SSRT) and scanning electron microscopy (SEM). The results indicated that fluoride significantly accelerated the stress corrosion cracking of NiTi orthodontic wires. The fractographies of NiTi orthodontic wires exhibited striation pattern.

A.Wadood et al [65] (2015) investigated the effects of partial substitution of Ti with Zr and Au with Ag in Ti–Au on phase constitution, phase transformation, and high temperature thermo-mechanical and shape memory properties.

Partial substitution of Ti with Zr in Ti–50Au and Ti–40Au–10Ag was found to improve the thermo-mechanical and shape memory effect. However, partial substitution of Au with Ag in Ti–50Au and Ti–50Au–10Zr was found to have negligible effects.

MATERIALS AND METHODS

MATERIALS

- Samples: 4- commercially available groups of A-NiTi rectangular archwires of size (0.017 * 0.025) with 15 samples in each group.

  Group 1- G &H,franklin, Indiana, US.

  Group 2- Dentaurum, ispringen, Germany.

  Group 3- Essential dental products (EDP), New Delhi, India

  Group 4-American orthodontics (AO)-Sheyboygan, W, USA
- Silicon carbide-emery paper polish (grades 80,120,220,400,600,800,1000)
- Alumina cloth polishing- 1µm size abrasive particles
- Metal etchant – Hydrofluoric acid HF, Nitric acid (HNO3)

EQUIPMENT USED

- Qness 60 A+ universal microhardness testing machine (Salzburg, Austria)
- Polarised electron microscope- carl zeis make - axio lab A1 model (carl ZeissIndia, Bangalore pvt limited)
- Instron tensile testing machine(Norwood, Massachusetts)
- Oxford energy dispersive spectrometer(oxford instruments, US)

OPERATIONAL DEFINITIONS:

1. **Microhardness-**The ability of metal to resist permanent deformation, when load is applied to measure the hardness of the material. Thehardness of the material gauged with instruments using small diamond indenters of particular shape to make the impression called a test load or applied force, which can be at 1-1000 gram force, on the testing material. The micro indentation on the material recorded at microscopic level[30]. Therefore, a lower amount of force is applied relative to the standard measuring instruments, allowing measurement of thinner sheets or smaller test materials that may not respond accurately to measurement conducted with standard instruments. Hardness testing plays a vital role in material testing, quality control and acceptance of components.

2. **Microstructure** -Very small scale surface structure of the material can be revealed under magnification higher than 25X.Thestructure of an alloy viewed after etching, polishing, and observed under a microscope[31]. The structure of the material can be described in terms of microscopic crystal structure. It is used to refer the average position of atoms throughout the structure. It is term used to describe the material in Nano to centimetre length scales. The microstructure can be viewed under wide range microscopes its can differs in large scale when viewed at dissimilar scales length. Therefore, it is important to illustrate the length of scale in every observation. A randomly distributed crystal oriented in different direction indicates underworked and non stressed and not prone to deformation. Lower stress and high ductility indicated if larger crystals observed under microscope.

3. **Ultimate tensile strength (UTS)-** maximum resistance to fracture by the material. Equivalent to the maximum load that can be carried by one square inch of cross-sectional area when load is applied as simple tension. The UTS is usually found by performing a tensile test to measure the maximum stress in uniaxial stress strain test[32]. It is an intensive property and its values not depending on size of the test sample. However it is dependent on other factors such as specimen preparation, surface defects and temperature in which the test carried out.

4. **Percentage elongation-** measurement that captured the amount a material will plastically and elastically deform up to fracture[33]. An increase in gauge length compared to the original length. It is one way to measure and quantify theductility of the material and fracture resistance just before fracture of materials. Test to measure percentage elongation carried out along with tensile test. The gauge records the elongation over a set volume of material in centre of the specimen rather measuring the entire length of entire length of sample before and after tests. The final length of the material compared with its original length to determine the percentage elongation[34].

METHODOLOGY

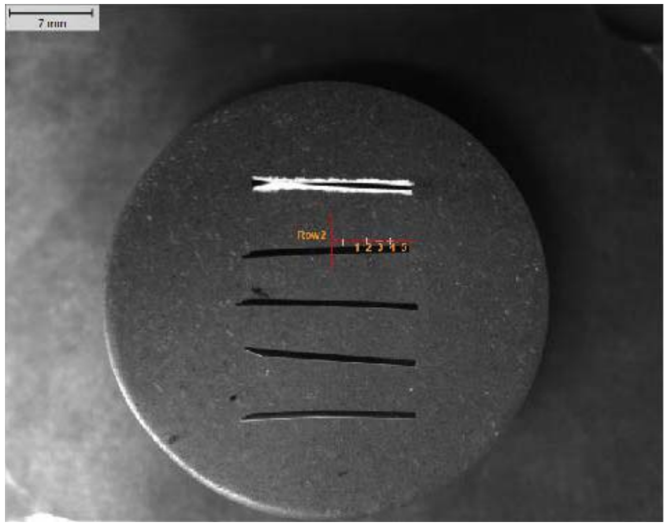
1. MICROHARDNESS

The microhardness of five samples in each group rectangular A-NiTi archwires of dimension (0.075*0.025), using QNESS MICROHARDNESS TESTING MACHINE (Q60A+) attached with automated SOFTWARE QPIX CONTROL[19].

The wire sample placed parallel on the anodized aluminium platform equipped with highly precision optical path and eight fold sample holder which prevent rolling during indentation. The machine precalibrated with Vickers diamond indenter is automated by the operator to respective contour parallel the indenter automated to apply load of 1000 gram. the function of software module system to edge recognition with contact free gauging of indentation depth standard three dimensional auto snap function.

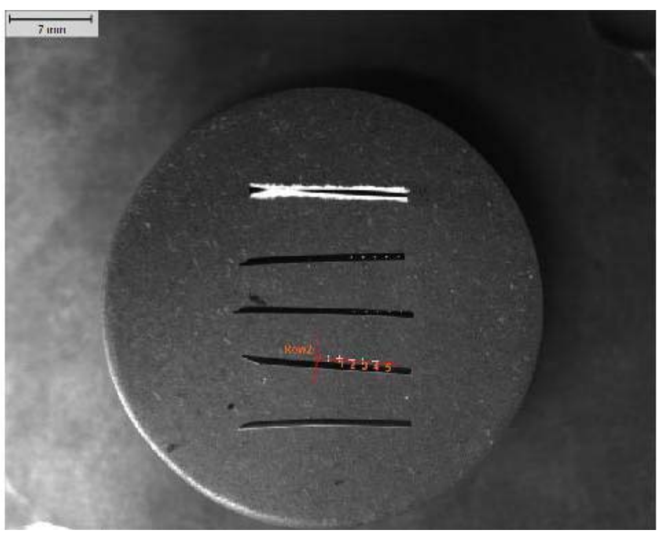
QPIX SOFTWARE CONTROL scan the sample with sample camera which fed into monitor and digital measurement of microhardness obtained.

Qness microhardness testing machine (Q60A+) attached with automated software QPIX control (Figure 1)



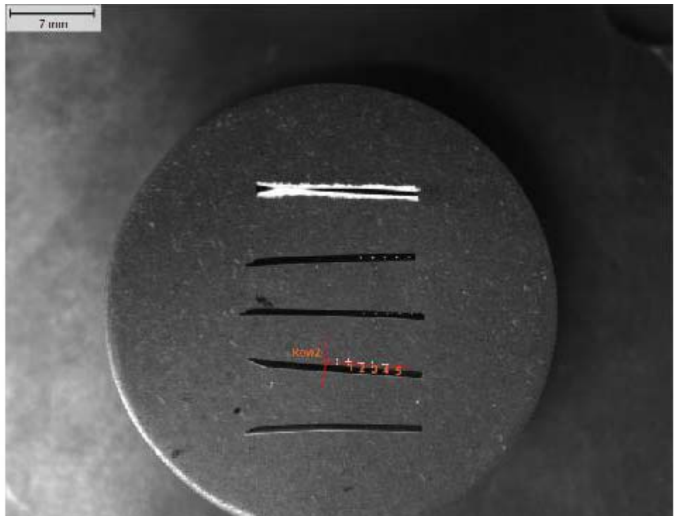
Group 1: G&H

Sample tested in Qnessmicrohardness testing machine and prepared row measurement with 7mm reference and each portion tested. (Figure 2)



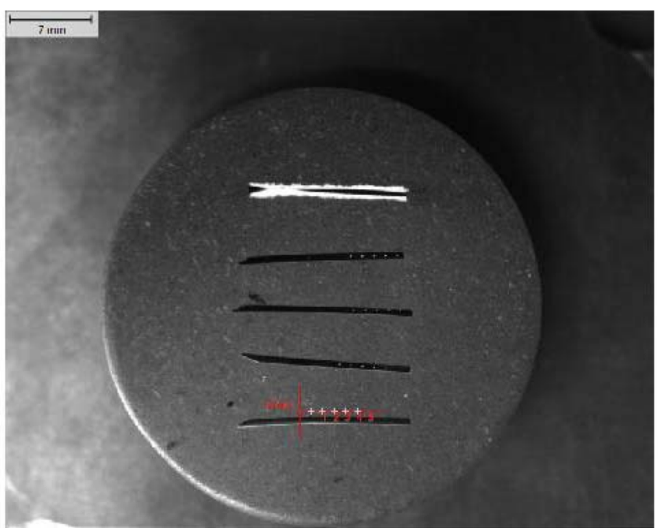
GROUP 2: DENTAURUM:

Sample tested in Qnessmicrohardness testing machine and preparedrow measurement with 7mm reference and each portion tested. (Figure 3)

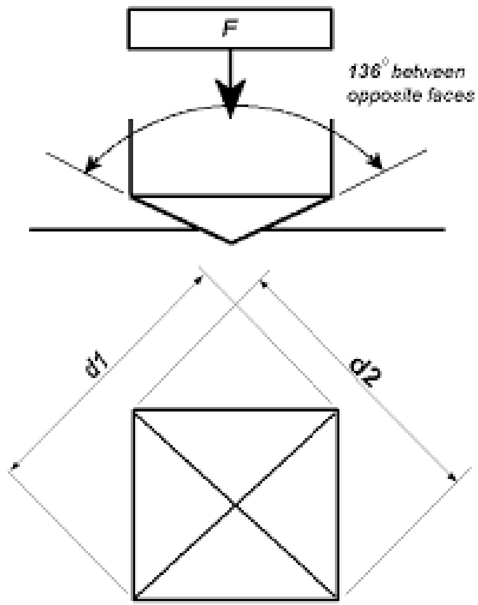


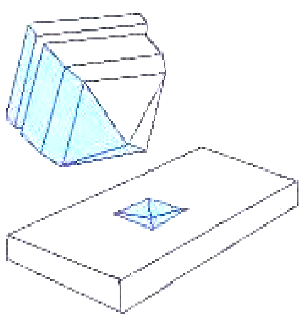
GROUP 3: ESSENTIAL DENTAL PRODUCTS

Sample tested in Qness microhardness testing machine and prepared row measurement with 7mm reference and each portion tested. (Figure 4)

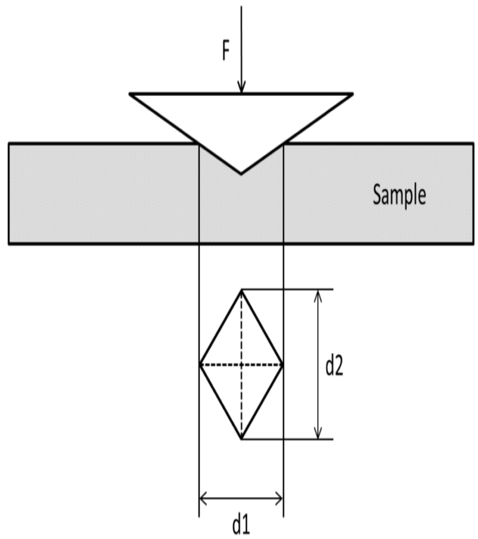


GROUP 4: AMERICAN ORTHODONTICS
sample tested in Qness microhardness testing machine and preparedrow measurement with 7mm reference and each portion tested. (Figure 5)





Schematic diagram of Vickers hardness indenter with profile angles (angle formed between the opposite faces at the vertex of pyramidal indenter)α=136˚±0.5˚ and the tested sample.(Figure 6)



$$HV = 1.854(F/D^2)$$

HV – VICKERS HARDNESS NUMBER,

F- APPLIED LOAD,

D-AREA OF INDENTATION

Schematic diagram showing measurement of hardness value from tested samples (Figure 7)

MICROSTRUCTURE

Surface Preparation of wire

The rectangular NiTi archwires of size 0.017*0.025 in each group, surface preparation done with silicon carbide emery paper with coated abrasives from coarse to fine grades of 80,120,220,400,600,800,1000 followed by polishing with alumina cloth with abrasive particle size-1µm. polished wires treated with chemical etchant composition (1ml hydrofluoric acid HF +3ml of nitric acid HNO3 + 16 ml of water H2O .The microstructure of surface treated A-NiTi archwires tested with carl zeis (axio lab A1)[20] , polarized electron microscope. Magnification 200X.

Carl Zeiss axio lab A1 polarized electron microscopy (Figure 8)

3. ULTIMATE TENSILE STRENGTH (UTS)

The tensile test done by applied pulling or tensile force to an A-NiTi archwires sample and measures the specimen’s response to the stress which determines the breakage strength of material. The ultimate tensile strength is maximum stress that a specimen tolerates during test. UTS is a measure of stress versus strain it may or may not corresponds to the strength of material at break, depending on the material brittle ,ductile or exhibits both properties

The sample length of 100 mm length in each sample group taken and marked 25mm gage length was marked. The sample archwires was conducted at room temperature with 2000kN the Instron universal tensile testing machine using suitable fixture with constant crosshead speed of 0.1mm /min .Tensile load is applied just before failure of the specimen.

The ultimate tensile strength calculated using the following formulas,

$S = F/A$

Formula derivation,

S = BREAKING STRENGTH

F= FORCE

A= AREA

Instron universal tensile testing m machine(Figure 9)

4. PERCENTAGE ELONGATION

The sample length of 100 mm length in each sample group taken and marked 25mm gage length was marked. The percentage elongation test was conducted at room temperature with 2000kN the Instron universal tensile testing machine using suitable fixture with constant crosshead speed of 0.1mm /min .Tensile load is applied just before failure of the specimen.

Final lengthy after breakage of archwires noted and percentage elongation calculated using the formula

*Percentage elongation = change in length - original length *100/ original length*

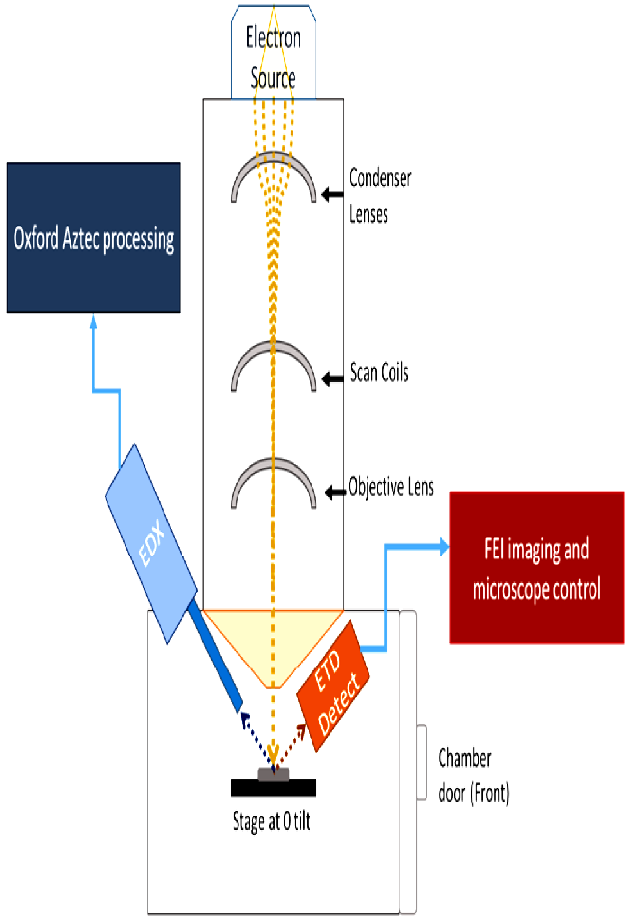
CHEMICAL PROPERTY ANALYSIS

1. QUANTITATIVE AND QUALITATIVE CHEMICAL TEST ANALYSIS

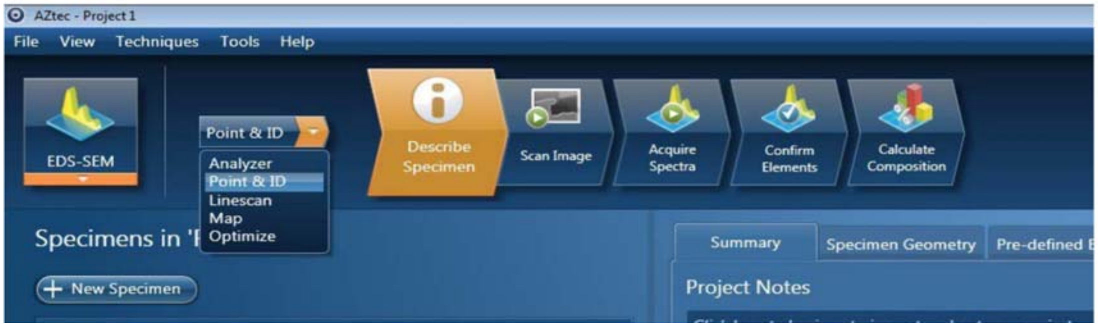
The quantitative chemical composition of all the four group done by the x-ray florescence (XRF) spectroscopy method with Oxford instruments energy-dispersive spectroscopy (EDS) machine ,in which high energy x-rays are used to bombard against the surface of sample from all the groups. The wavelength of emitted secondary radiations which corresponds to the characteristics Individual elements present in the wires. All the samples confirms the presence of nickel and titanium as major elements and trace composition of zinc (zn) noticeably along with other elements like iron (Fe), antimony (Sb), tin (Sn) and manganese (Mn). Electron probe X-ray microanalysis techniques- energy dispersive X-ray spectroscopy (EDS), use the characteristic X-rays generated from a sample bombarded with electrons to identify the elemental constituents comprised in the sample. In this technique generate a spectrum in which the peaks correspond to specific X-ray lines and the elements can be easily identified. Quantitative data can be obtained by comparing peak heights or areas in the unknown with a standard material. Elemental information is obtained by measuring the energy and intensity distribution of x-ray signals generated when SEM focused electron beam interacts with the sample archwires. The elemental information is color-coded and superimposed on different morphological features of the samples imaged by SEM.

An EDS system comprises three basic components that must be designed to work together to achieve optimum results: the X-ray detector or spectrometer, the pulse processor, and the analyser. The Energy Dispersion spectrometer converts the energy of each individual X-ray into a voltage signal of proportional size. This is achieved through a three stage process. Firstly the X-ray is converted into a charge by the ionization of atoms in a semiconductor crystal. Secondly this charge is converted into the voltage signal by the field effect transistor (FET) preamplifier. Finally the voltage signal is input into the pulse processor for measurement. The output from the preamplifier is a voltage 'ramp' where each X-ray appears as a voltage step on the ramp.EDS detectors are designed to convert the X-ray energy into the voltage signal as accurately as possible. At the same time electronic noise must be minimized to allow detection of the lowest X-ray energies.

Software for data analysing and elemental mapping is done by Aztec Energy standard consist of automated analyser, point&ID, mapping, line scan, standardization manager.



Cross sectional image of oxford energy dispersion spectroscopy attached with software to obtain field element image (figure 10)



Point & ID: Quickly analyses composition at a specific point or across a region of interest (figure 11).

Elemental Mapping: Marks the location of elements across a region of interest by overlaying different colours. If there is intense or brighter the colour at a specific location, than higher concentration of that element is present. (Figure 12).

Line profile mapping: Determines the concentration of elements across a user-defined line (figure 13).

STATISTICAL ANALYSIS

Statistical analysis was done by IBM SPSS (IBM Corp. Released 2011. IBM SPSS Statistics for windows, Version 20.0. Armonk, NY: IBM Corp). Mean and SD were used to summarize the continuous data. Initially, the data was checked for normality using Shapiro Wilk test. The data was found to be normal, and thereby it was decided to use parametric tests for further comparisons. For inter group comparison (of parameters like Hardness, Ultimate Tensile Strength and % Elongation) One-Way ANOVA was used. One-way ANOVA was used to find the difference in hardness between the four groups, to find the difference in UTS and % elongation between the four groups. Significance level was fixed as 5% (α=0.05).

Results are discussed under the following five topics:

1. Comparison of hardness between four groups

2. Interpreting microstructure of all four groups

3. Comparison of ultimate tensile strength and percentage elongation between groups

4. Comparison of percentage elongation between the four groups

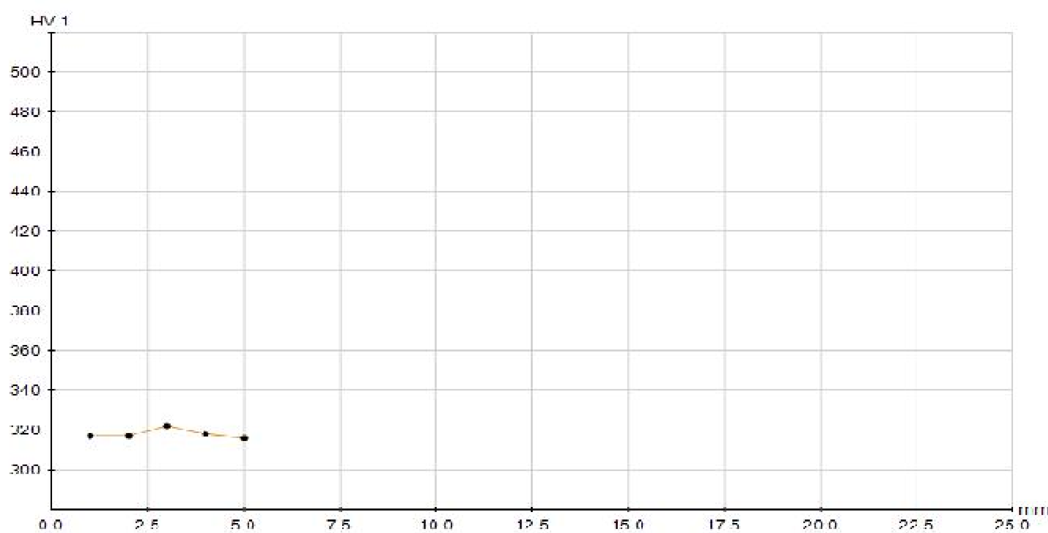
5. Correlating chemical properties with mechanical properties

RESULTS

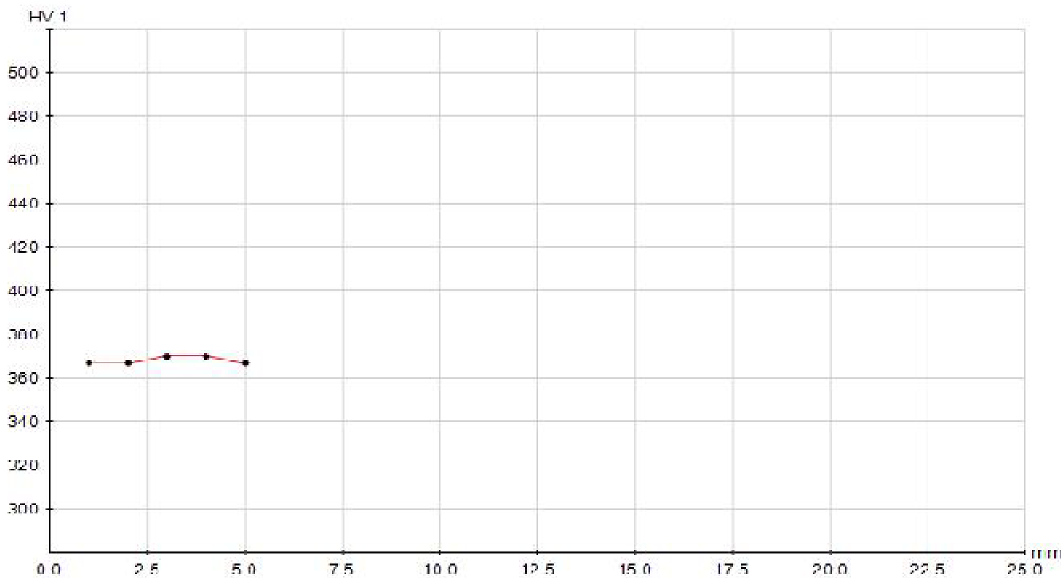
The study done with four group of commercially available rectangular A-NiTi archwires of dimension (0.017* 0.025).five samples in each group tested for microhardness and the mean value taken for correlation with other mechanical analysis. Three samples from each group tested for other mechanical property test analysis like microstructure, ultimate tensile strength, percentage elongation and chemical property analysis.

1**.COMPARISON OF HARDNESS BETWEEN FOUR GROUP**

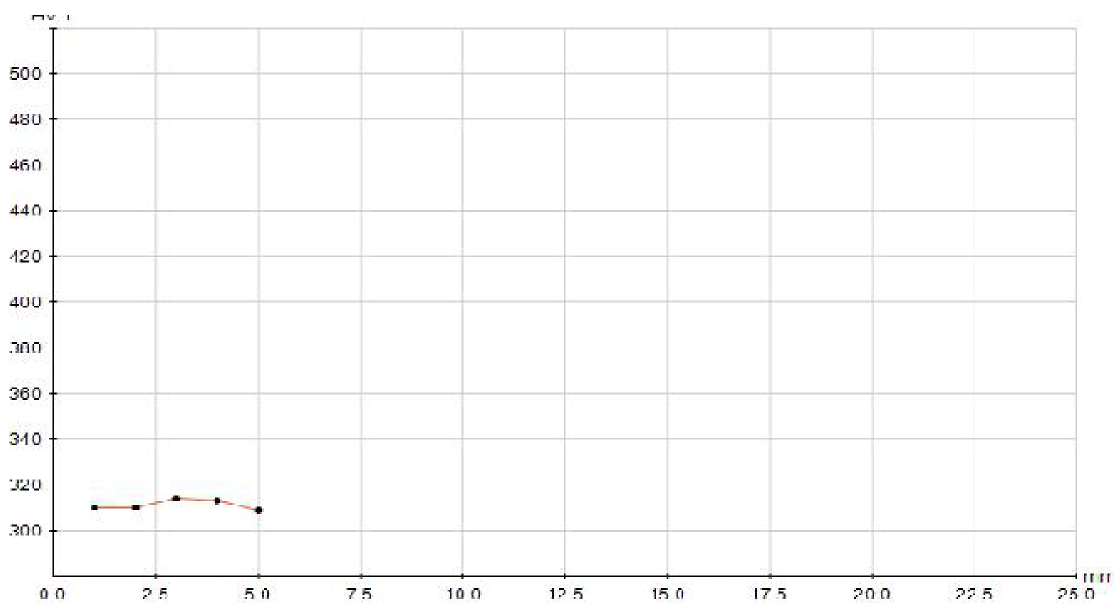
A.GRAPHICAL REPRESENTATION OF HARDNESS VALUE OF FIVE SAMPLES IN EACH GROUP



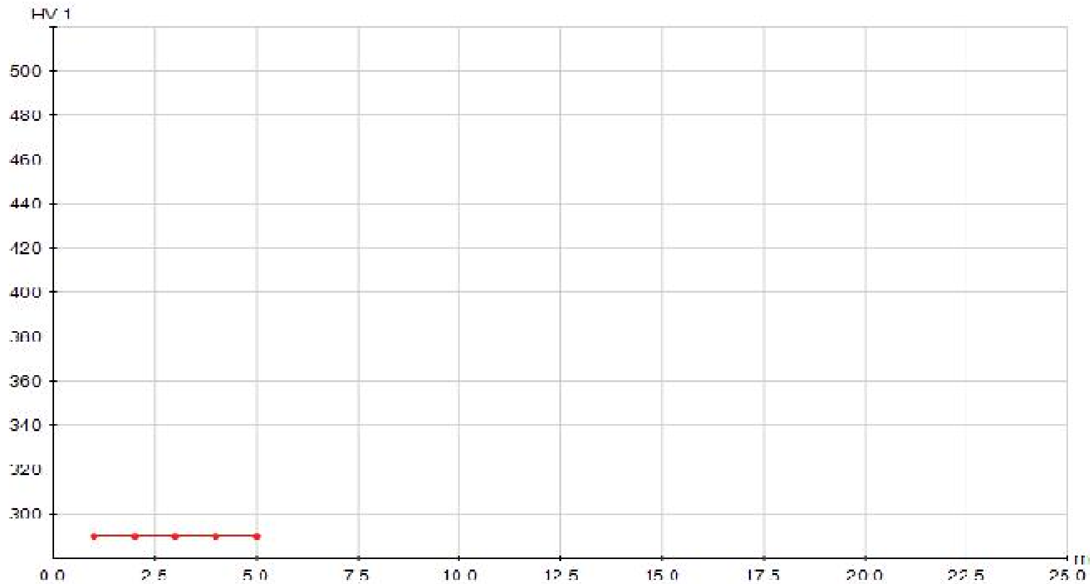
GROUP 1- G&H(HARDNESS VALUE OF FIVE SAMPLES HV1=317. 317,322,318 and 316) Graph 1



GROUP-2: DENTAURUM(HARDNESS VALUE OF FIVE SAMPLES HV1=367, 367, 37,370 and 367) - Graph 2



GROUP-3: ESSENTIALDENTAL PRODUCTS (HARDNESS VALUE OF FIVE SAMPLES HV1=310,310,314,313 and 3090. graph 3



GROUP-4: AMERICAN ORTHODONTICS (HARDNESS VALUE OF FIVE SAMPLES HV1=290,292,291,292 and 290) -Graph 4

B**.<u>STATISTICAL REPRESENTATATION OF HARDNESS BETWEEN THE GROUPS</u>** (table 1)

Group	Mean	SD	SEM	95% of C.I Lower	Upper	df	P VALUE
GH	318.00	2.34	1.04	315.08	320.91	16	0.001
Dentaurum	368.20	1.64	0,734	366.15	370.24	16	0.001
EDP	311.20	2.16	0.969	308.50	313.89	16	0.001
AO	291.00	1.00	0.447	289.75	292.24	16	0.001

BONFERRONI PAIRWISE COMPARISON OF VARIABLES OF MICROHARDNESS (tb: 2)

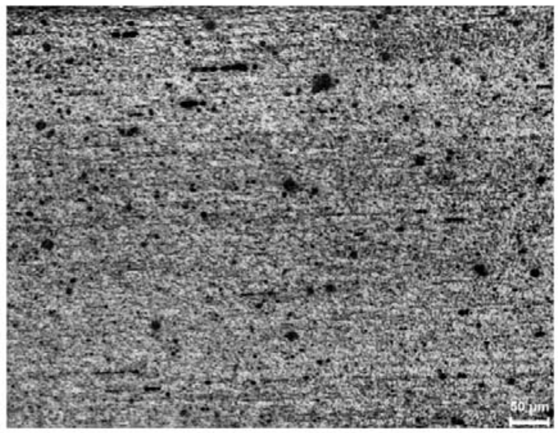
(I)Group	(J) Group	Mean Difference (I-J)	P-Value
GH	Dentaurum	-50.20000*	.000
	EDP	6.80000*	.000
	AO	27.00000*	.000
Dentaurum	GH	50.20000*	.000
	Dentaurum	57.00000*	.000
	EDP	77.20000*	.000
EDP	GH	-6.80000*	.000
	Dentaurum	-57.00000*	.000
	AO	20.200000*	.000
AO	GH	-27.00000*	.000
	Dentaurum	-77.20000*	.000
	EDP	-20.20000*	.000

Test: One-way ANOVA was used to find the difference in hardness between the four groups.

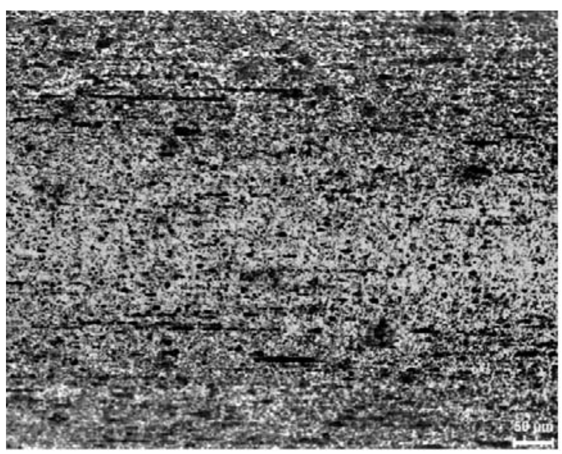
Interpretation

It is seen that there is a significant difference in hardness among the four groups. Post hoc test showed that there is a significant difference in hardness between each of the groups with each other groups in pair wise comparison.

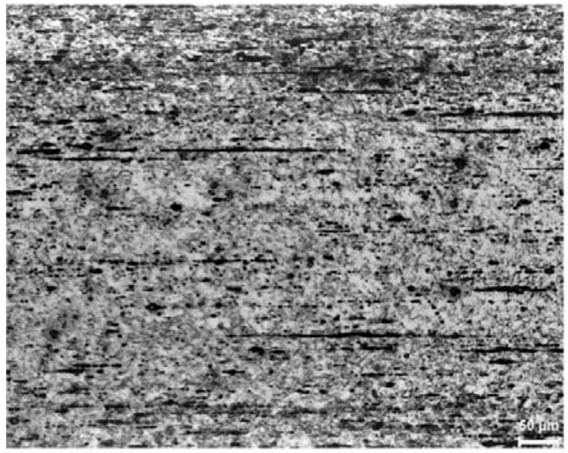
2. INTERPRETING MICROSTRUCTURE OF ALL FOUR GROUPS



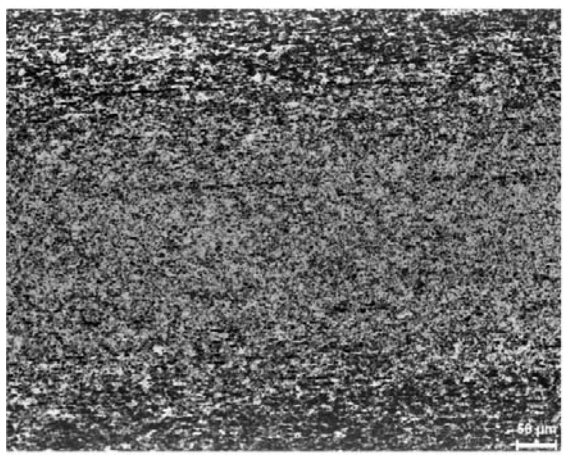
GROUP-1: G&H (fine grain structure in uniform deformation arrangement pattern 200X) **Figure 14**



GROUP-2: DENTAURUM (fine grain structure in differential deformation arrangement pattern 200X) F**igure15**



GROUP-3: ESSENTIAL DENTAL PRODUCTS(coarse grain structure in uniform deformation arrangement pattern 200X) **Figure16**



GROUP-4: AMERICAN ORTHODONTICS (coarse grain structure in differential deformation arrangement pattern 200X) **Figure**17

Inference

All the four samples shows typical wire drawn structure , longitudinal flow pattern observed.

G&H with finer grains and American orthodontics with coarse grains observed .G&H and essential dental products archwires observed with uniform deformation. Dentaurum and American orthodontic archwires observed with differential deformation which was more in boundaries than the core region of archwires.

3. Comparison of ultimate tensile strength (UTS) and percentage elongation between groups (tb: 3)

Variables	Group	mean	S.D	95% of C.I for Lower	Upper	F	P-value
UTS	G&H	1148.00	11.13	1120	1175.66	664.2	0.001
	Dentaurum	1539.66	10.01	1514.78	1564.54		
	EDP	1426.33	15.30	1388.30	1464.36		
	AO	1277.33	8.386	1256.50	1298.16		
PERCENTAGE ELONGATION	G&H	63.65	4.030	27.43	99.86	198.2	0.001
	Dentaurum	18.53	1.209	15.52	21.53		
	EDP	20.26	0.9451	17.91	22.61		
	AO	22.66	2.753	15.82	29.50		

Test: One-way ANOVA was used to find the difference in UTS and % elongation between the four groups.

Inference: It is seen that there is a significant difference in UTS and % elongation among the four groups. Post hoc test showed that there is a significant difference in UTS and % elongation between each of the groups with each other groups in pair wise comparison

<u>Bonferroni pair wise comparison of variables (UTS)-tb: 4</u>

(I)GROUP 2	(J)GROUP 2	Mean difference(I-J)	P-Value
GH	Dentaurum	-391.66667*	.000
	EDP	-278.33333*	.000
	AO	-129.33333*	.000
Dentaurum	GH	391.66667*	.000
	EDP	113.33333*	.000
	AO	262.33333*	.000
EDP	GH	-278.33333*	.000
	Dentaurum	-113.33333*	.000
	AO	149.00000*	.000
AO	G&H	129.33333*	.000
	Dentaurum	-262.33333*	.000
	EDP	-149.00000*	.000

Test: One-way ANOVA was used to find the difference in ultimate tensile strength (UTS) between the four groups.

Interpretation

It is seen that there is a significant difference in ultimate tensile strength (UTS) among the four groups. Post hoc test showed that there is a significant difference in UTS between each of the groups with each other groups in pair wise comparison.

Bonferroni pair wise comparison of variables (% Elongation)-tb: 5

(I)GROUP 2	(J)GROUP 2	Mean difference(I-J)	P-value
GH	Dentaurum	45.11667*	.000
	EDP	43.38333*	.000
	AO	40.98333*	.000
Dentaurum	GH	-45.11667*	.000
	EDP	-1,73333	1.000
	AO	-4.13333	.367
EDP	GH	-43.38833*	.000
	Dentaurum	1.73333	1.000
	AO	-2.40000	1.000
AO	G&H	-40.98333*	.000
	Dentaurum	4.13333	.367
	EDP	2.40000	1.000

Test: One-way ANOVA was used to find the difference in percentage elongation between thefour groups.

Interpretation: It is seen that there is a significant difference in percentage elongation among the four groups. Post hoc test showed that there is a significant difference in percentage elongation of group-1 G&H between other groups in pair wise comparison.

Chemical composition analysis(tb:6)

S.No	Group	Ni	Ti	Zn	Others
1	GH	55.81	43.71	0.24	0.23
2	Dentaurum	55.75	43.86	0.23	0.16
3	EDP	55.62	44.01	0.11	0.26
4	AO	55.80	43.84	0.10	0.26
Significance		**NS**	**NS**	**S**	**S**

The chemical composition showed there is significant and comparable difference present in zinc (zn) and in trace elements composition only among the four groups.

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Correlation of chemical property with Micro hardness (tb: 7)

Group	Correlation coefficient with zinc			Correlation coefficient with trace elements		
	r	**strength**	**significance**	**r**	**strength**	**significance**
G&H	0.778	Strong	Non-significant	0.68	Strong	Significant
Dentaurum	0.612	Strong	Significant	0.81	Very strong	Non-significant
EDP	0.84	Very Strong	Non - significant	0.802	Very strong	Non-significant
AO	0.812	Very strong	Significant	0.82	Very strong*	Significant*

Test: Pearson correlation test to measure the strength and association between composition and hardness.

Inference : Strong correlation present between of zn and trace elements concentration in all the four groups as R-value is closer to 1, significantly due to zinc in group 2 and 4 and trace elements in group 1.

Correlation of chemical properties with UTS(tb:8)

Group	Correlation coefficient with zinc			Correlation coefficient with trace elements		
	r	**strength**	**significance**	**r**	**strength**	**significance**
G&H	0.65	Strong	Non-significant	0.75	Strong	Significant
Dentaurum	0.56	Strong	Significant	0.79	Very strong	Non-significant
EDP	0.67	Strong	Significant	0.825	Very strong	Non-significant
AO	0.712	Strong	Significant	0.912	Very strong	significant

Test: Pearson test to measure the strength and association between composition and ultimate tensile strength (UTS).

Inference: strong correlation present between zinc composition and ultimate tensile strength (UTS) in all the four groups and significant in group 2,3 and 4.

strong correlation present between trace elements composition and ultimate tensile strength (UTS)in all the four groups and significant in group 1 and 4.

Correlation of chemical properties with percentage elongation(tb:9)

Group	Correlation coefficient with zinc			Correlation coefficient with trace elements		
	r	**Strength**	**significance**	**r**	**strength**	**Significance**
G&H	0.75	Strong	Significant	0.75	Moderately Strong	Significant
Dentaurum	0.69	Strong	Non-Significant	0.79	Very strong	Significant
EDP	0.852	Very Strong	Significant	0.825	Very strong	Non-significant
AO	0.78	Strong	Significant	0.912	Very strong	Significant

Test: Pearson correlation test to find the strength and association between chemical composition and percentage elongation.

Inference: strong association present between zinc concentration and percentage elongation in all the four groups, significantly in group 1, 3 and 4.

strong association present between trace elements concentration and percentage elongation in all the four groups , significantly in group 1,2 and 4.

DISCUSSION

Metallic alloys that tend to return to the original shape after large deflections have been appreciated since the 50s. To begin with, they have been studied only for their use in aeronautical engineering, because of their sufficient ductility, later their use was expanded in medicine in the manufacturing of prostheses that replace long bones and in the study of surfaces and biofilms[45]. In Orthodontics, these materials are used in archwires for the alignment of teeth, in the initial stages of treatment, when large deflection is necessary and also because they present a low modulus of elasticity (E) and excellent spring back when compared to other alloys. There is great variability in the amount of stored energy in same cross-section nickel-titanium alloys, available from different manufacturers. Many of them are commercialized as shape memory alloys, while others do not even show the effect of superelesticity and present characteristics of martensitic-stabilized alloys as the alloys originally known as Nitinol (Unitek, Monrovia, CA, USA)[21]. NiTi shape memory alloys have more attention in research field[43] as they are widely used in manufacturing of orthodontic wires devices, stents materials for minimal invasive surgeries as it shows biocompatibility, special functional properties with high mechanical strength,[41] good corrosion, wear and electrical resistance. It can be utilized many times before structural or functional fatigue service life limits[42]. These properties strongly depend on the exact chemical composition, processing history and impurity level[44]. Contaminants like carbon, oxygen can affect the properties of NiTi in greater extent. The penetration of other chemicals occurs during production and processing of the alloy. Nickel and titanium are most commonly manufactured into the NiTi alloy by the process of vacuum induction[45] or arc melting. Segregation is often a problem because there is a relatively wide disparity of melting points. Several remelts are often needed to improve homogeneity of NiTi alloy then made into powders. The process of hot isostatically pressing is used by the manufacturer to form the powders into wires. The wires obtain their final shape by the process of drawing or rolling which may leave scratch marks on the surface[7, 55.]

Mehrabi et all[58,] studied to produces NiTi SMA Parts via powder metallurgy (PM) technique and then deformed the PM parts at different deformation ratio for to obtain high mechanical and Density values. To obtain the green parts for debiding process, water soluble binder system, which is the usable for NiTi SMA Powders, has been tried to be developed. 1200°C sintering temperature has been applied to obtain the red parts. 0% deformation ratio has been applied on red parts to achieve desired density and mechanical process. It is also aimed at achieving possible intermetallics such as Ni4Ti3, Ni3Ti, NiTi and NiTi (B19), which directly affect the shape memory, wanted to obtain with hot deformation. The effect of stress due to hot and room temperature on NiTi particles observed. The first deformation mechanism described is the room temperature deformation mechanism. Five steps of micro structural formation can be identified. The first step is martensitic transformation, the second step is deformation twinning, the third step is dislocation shift, the fourth step is nano crystallization, and the final step is the formation of the amorphous structure[35]. However, when deformation is applied at high temperature, the mechanism will be changed. In deformed parts, the dislocation rate will decrease with temperature rising above 700◦C. The direction of displacement at 800◦C starts sharply into the grain from grain boundaries. This kind of displacement shift activity plays an important role in the plastic deformation of NiTi SMA under compression. Especially under the influence of sufficient thermal motion force, the external displacement mechanism can cross the general grain boundary during hot plastic deformation of NiTi SMA at 800◦C. In the first stage, the dynamic recrystallization mechanism becomes active at high temperatures[39]. The second step is the mechanism of grain growth. The third mechanism begins with grain growth and new intermetallics components[37]. The fourth mechanism is under high stress and increases the formation of high-density sub particles, dislocations and intermetallics components. The final step is the formation of new sub grain, recrystallization and intermetallics. These formations will directly affect the microstructure[36, 38.] In order to obtain high density and high mechanical properties, debiding, sintering and hot deformation processes were applied on the raw parts in order. Main austenite B2NiTi (cubic) and a lenticular disk-like morphology Ni4Ti3 (rhombohedral-space group R3) phases can be obtained after sintering at 1200◦C for 60min. The hot plastic deformation HPD process causes intense lattice distortion and microscopic strain in the sample, and a significant grain refinement outcome arises. By increasing at the 50% deformation rate at 1000◦C obtained fine grain structure. Fine grain structures are increased hardness and density values. Main NiTi phases decrease in deformation rate and increase the formation mainly of Ni3Ti and the other intermetallics reflection as Ni4Ti3 andTi2Ni. The reflection affect the shape memory effects directly in NiTi SMA.

Standard Nitinol material vacuum arch melting and vacuum induced melting contains high numbers of inclusions within the microstructure while vacuum arch melting material contains comparatively few. It is frequently observed that fatigue cracks tend to initiate at inclusions this may also explain why vacuum arch melting Nitinol appears to result in improved fatigue performance. Fewer inclusions (oxides and carbides) appear to increase the strain limit below which fatigue lives greater than 107 cycles may be achieved. Electropolished surfaces tend to result in greater fatigue lives than unpolished for any given strain level. Inclusions frequently lead to pinhole/pit type defects on electropolished surfaces. Extra low inclusion, Nitinol results in fewer surface defects after electropolishing. The improvements in electropolished surface finish for extra low inclusion, Nitinol may reduce fall out during production and therefore may offer greater production yields[57].

A major source of contaminants is the melting crucible and its environment which need to be carefully controlled in order to improve the quality of shape memory effect and mechanical properties under different conditions. The effect of annealing temperature on the shape memory effect is relation to the number and dislocations and the strength of alloy phases. At lower annealing temperature, the dislocation density and the strength are higher, which hinders the reorientation of martensite, so the shape memory effect gets weak. When annealing temperature increased, the reverse effect can happen with increase in pseudo elastic region .it is obvious that the microhardness decreased with increase in annealing temperature.

mayer[59] and Scherngel[60] et al, concluded that addition of tungsten (W) disperoids increases the stability of shape memory effect and increases hardness which required to improve the wear resistance. The processing conditions as material of crucible and mould also can cause effects in mechanical properties. Heating the alloys in ceramic crucible, the amount of second phase particles like Ti2Ni3 and Ti2Ni in NiTi grain boundaries increased and there is in hardness than using a graphite crucible. The moulds also influence the thermal conductivity of NiTi leading to different alloys solidification and its hardness.

In this study, mechanical properties like microhardness, microstructure, ultimate tensile strength and percentage elongation presenting greater importance in clinical performance of archwires in biomechanics were evaluated.

The microhardness analysis test was done using Vickers microhardness test in each group of the sample as it is highly suitable to test microhardness of smaller metals. The highly reliable automated tested machine named Qness microhardness tester is used to test microhardness in this study. Series of square pyramidal shaped indentation marks on sectioned parts of archwires[61] were analyzed by auto snap function to drag the row of test points and the distances of test points diagonals were measured.

The microstructure of grain size and pattern of arrangements were viewed under 200X high resolution. Pre analytical sample surface was prepared to remove the defective surface layers on samples of all the four groups. The microstructure evaluated in this study was similar to the method used by fercec[7] to find the microstructure of nickel and nickel free archwires.

To measure an ultimate tensile test and percentage elongation, it is mandatory to apply a uniaxial low load application without any torsion effects until the sample gets failed. Both tests were done with highly precise and automated universal tensile testing machine. The length of pre and post tested archwire samples were noticed before and after failure of samples to obtain the percentage elongation values and UTS respectively[62].

To find the chemical composition, X-ray florescence (XRF) energy dispersion spectroscopy EDS method was used in this study. It is an integrated "Scios method" the combination of a Scanning Electron Microscope (SEM)and a Focused Ion Beam (FIB) system[63]. The software(AZTEC) was attached with the EDS generated chemical composition data in reference to periodic table. EDS system was used in this study to measure sub-surface (0.3-3um) with a detection limit of ~1 atom%[6]. It elicited the presence of nickel and titanium as major elements and trace composition of zinc (zn) and other elements like iron (Fe), antimony (Sb), tin (Sn) and manganese (Mn) in the four groups.

The microhardness was the highest in group-2(Dentaurum). Statistically significant differences were present among all the four groups according to Bonfferoni pair wise comparison statistical analysis.

The microstructure analysis showed the presence of fine grains structure in group 1(G and H). Fine grain particle size was found to be increased in group-4(American orthodontics), group-2 (Dentaurum)respectively in increasing order and highly coarser particles were found to be associated with group-3 (Essential dental products). All of the four groups showed that longitudinal wire drawn arrangement pattern of grains. Uniform pattern of deformation was observed in group 1 and 3, whereas differential pattern of grain deformation associated with more grain boundaries than core of the archwires samples were observed in group 2 and 4.

One way ANOVA test showed statistically significant differences present between each group with the highest values of UTS and least values of percentage elongation in group-2 (Dentaurum).

Bonferroni pair wise comparison to compare the percentage elongation among the four groups showed that there was statistically significant difference observed only in group-1(G and H), when compared to other individual groups. Group-1 had the highest percentage elongation value which was of greater difference when compared with other groups.

The statistically significant variation in chemical trace elements and zinc among the four groups, with highest zinc content was found in group-1 G and H (0.24%), followed by group -2 (0.23%), group-3 (0.11%) and group 4 (0.10%). The trace elements were the highest in both group 3 and group 4 which had about 0.26%, followed by group-1 G and H which consisted of 0.23% and the least in group-2 Dentaurum with 0.16% of total net composition.

Pearson correlation coefficient tests showed that chemical composition differences in zinc and trace elements had strong correlation with microhardness in all of the four groups as r value is greater than 0.5 and more closer to 1. Strong and significant correlation present between zinc content and microhardness was seen in group-2 Dentaurum and significantly very strong correlation with ‘r’ value of 0.812 was obtained in group-4 American orthodontics. The presence of trace elements represented the significantly strong correlation with microhardness property associated with group-1 G and H.

The presence of zinc content had the strong correlation with UTS in all of the four groups but significantly strong correlation was present in group-3 Essential dental products and group-4 American orthodontics with r values of 0.67 and 0.712, respectively. The trace elements showed statistically strong and significant correlation with UTS in group-1 and significantly very strong correlation with r value of 0.912 in group-4 American orthodontics.

Pearson correlation coefficient test showed significant and strong correlation of the zinc content with percentage elongation in all groups except in group-2 Dentaurum, while the strong and significant correlation of trace elements to the percentage elongation was found in all groups except group-3 essential dental products.

SUMMARY AND CONCLUSION

The present study was done in the department of orthodontics and Dentofacial orthopaedics, Tamilnadu government dental college and hospital, Chennai. The study was conducted in four commercial brands (G and H, Dentaurum, Essential dental products and American orthodontics) of rectangular shaped Nickel titanium archwires of dimension 0.017 X 0.025" .The laboratory tests were done to analyze the mechanical properties of microhardness, microstructure, ultimate tensile strength and percentage elongation along the chemical analysis simultaneously and the correlation of varying chemical composition in individual mechanical behaviour were found.

From the findings observed in this study it can be concluded that

- The microhardness value was the highest in the group-2(Dentaurum) HV1=368 followed by group-1(G and H), group-3(Essential dental products) and group-4(American orthodontics). Trace chemical contents were least in group-2(Dentaurum) was 0.16%, was increased in group-1, 3 and 4 respectively. It may be concluded that the increase in microhardness can be correlated with less amount of trace elements present.
- The ultimate tensile strength (UTS) associated with group-1(G and H) was noticeably least among all followed by group-4(American orthodontics), group-3(Essential dental products) and group-2(Dentaurum). The presence of highest zinc content in group-1(G and H) was the highest (0.24%). It may be concluded that decrease in UTS can be correlated with increase in zinc content.
- The percentage elongation associated with group-1, G and H was noticeably highest among all followed by group-4(American orthodontics), group-3(Essential dental products) and group-2(Dentaurum). The presence of zinc

content in group-1(G ad H) was the highest (0.24%).It may be concluded that increase in percentage elongation can be correlated with increase in zinc content.

- Increased ultimate tensile strength with decrease in percentage elongation in each individual group was found. Both UTS and percentage elongation were found to be inversely proportional to each other in all the four groups. It may be concluded that nickel titanium can have the property of good flexibility.
- The Microstructure of fine grain was present in group-2(Dentaurum) among all the groups. The highest value of UTS (1530 Mpa) was present in this group could be attributed to the presence of fine grains.

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