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Centre for Dental Sciences

Dental Technology Dissertation Project
BSc (Hons) in Dental Technology

The Flexural Strength of Zirconia after immersion in Acidic Drink

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A report submitted in the part fulfilment for the degree of Bachelor of Science (Hons) in Dental Technology

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Declaration page

This dissertation project is original work carried out by solely by the author and it has been accurately referenced and acknowledged, complying with the University of Bolton’s guidelines regarding plagiarism (Student Handbook).

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I would like to extend my gratitude toward Nikolaos Poulis, who educated me throughout this process and believed in me when I didn't believe in myself. Without your support, this Dissertation Project would not have been possible. Thankyou.
Abstract: Acidic beverages can produce erosion of natural dentition in humans when consumed frequently or excessively. The aim of the present study was to investigate the flexural strength of Zirconia after immersion in acidic drink.

Methods: Zirlux® anterior multi (Zirlux®, Zahn Dental, Langden, Germany) Zirconia was tested. Thirty specimens of Zirconia were divided into 3 groups (n=10): specimens of group 1 were used as a control group that were immersed in ionised water for 7 days, specimens of group 2 were immersed in 50 ml of Coca-Cola for 4 days, and specimens of group 3 were immersed in 50 ml of Coca-Cola for 7 days. The flexural strength was measured for each material using an Instron Universal Testing Machine. The Data was subjected to statistical analysis.

Results: After acidic drink immersion, no significant differences were reported between control group 1, group 2 and group 3.

Conclusions: After immersion periods of 4 and 7 days, there was no statistical reduction in flexural strength value due to immersion in acidic drink.

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1.0 Introduction

1.1 Dental Ceramics

In order to meet increasing patient demand, dental ceramics have been developed at a fast rate allowing both patient and provider the choice of material with optimal strength and optical properties, creating a natural, aesthetic and durable restoration (Colombo et al., 2017). Described as non-metallic, inorganic, manmade materials which are produced by heating raw materials to extremely high temperatures under pressure (Rosenblum and Shulman, 1997), these materials are used alone or in-conjunction with other materials to fabricate fixed dental prostheses (Shenoy and Shenoy, 2010). Ideally, a dental prosthesis would replace, as identical as possible, the natural tissues that have been lost through wear, trauma or disease. (Shillingburg et al., 2012).

1.1.1 Metal free restorations

Metallic coronal restorations and porcelain bonded crowns have been used in dentistry for a very long time. Porcelain fused to metal crowns combine the desirable aesthetic component of an all-ceramic restoration together with the outstanding mechanical properties of metal, which can withstand the occlusal forces it is subjected to within the oral environment (Shenoy and Shenoy, 2010). The resounding issue with metallic based restorations is the question of biocompatibility with the human body. Problems with metallic dental restorations have presented as allergies (Stejskal et al., 1999), release of ions into the adjacent gum tissues (Bumgardner and Lucas, 1995), and gingival staining (Arvidson and Wróblewski, 1978; Venclíkova et al., 2007). The many adverse patient reactions, together with advances in research and the search for more aesthetic materials has resulted in the facilitation of all-ceramic (metal free) restorations (Shenoy and Shenoy, 2010; Çötert et al., 2015).
1.1.2 Aesthetics

The main factors that influence the final decision when choosing a dental material are aesthetics and strength of the material in which the prostheses will be manufactured (Daou, 2014). The questions on biocompatibility of metallic restorations and the highly requested aesthetic aspect of dentistry has led to a massive increase in metal free restorations. A multitude of different aesthetic restorative material choices are available with outstanding aesthetic outcome, for the patient to choose from (Schley et al., 2010). These options include enamels with a natural appearance and highly translucent incisal edges that are difficult to distinguish from the original. Although they are beautiful and aesthetically pleasing, these traditional glass, glass reinforced, feldspathic ceramics and even Alumina (Al$_2$O$_3$) ceramics are lacking in the mechanical and physical properties that are required to withstand the large occlusal and masticatory forces that they are subjected to in the posterior region of the mouth (Schley et al., 2010). Several clinical trials in the past have reported failures in this field of Dentistry, however some of these materials did show high aesthetics and good clinical performance in single tooth restorations, there were high failures reported for multiple unit bridges and fixed partial dentures (Kern, 2005).

A review by Schley et al. (2010) made the suggestion in the conclusions, that the best aesthetic outcome for the patient with lasting strength properties would be a Zirconia framework, for strength and longevity, layered with other ceramics for high aesthetic qualities.
1.1.3 Mechanical properties

The importance of high mechanical performance in the oral cavity cannot be underestimated. This is due to varying masticatory forces, averaging between 11-150 Newtons (N) in the anterior region, which can be as high as 200N. Posteriorly these forces can be around 500N, increasing up to 1000N in patients with parafunctional habits, such as bruxism (Hidaka et al., 1999; Yilmaz et al., 2007). When aesthetic dental materials are used to fabricate fixed dental prosthetics, the requirement for the material to be able to withstand these forces is vital for the success of the restoration.

In more recent times, the production of high-strength ceramic restorations without compromising translucency and aesthetics may be solved by facilitating the strength properties of zirconia, but in the case of minimal space or a less than ideal occlusion, the metal-ceramic crown is probable to be around for a sometime yet (McLean, 2001).

1.1.4 Flexural Strength

The many factors considered when choosing which material would be best to ensure the clinical performance and success of a dental restoration include flexural strength. All materials used in dentistry must have a minimum mechanical property requirement in order to function, for example; a material placed to restore a permanent molar must be able to withstand the occlusal forces that it is subjected to over a long period of time. It must be able to endure the concomitant wear and attrition that will inevitably happen.

To ability to cope with these issues is rigorously tested during the research and testing stages of materials before they are marketed. When an outside force is applied to a material during testing, an equal force but opposite in direction is set up with the body of the material. The stress that resists a flexural force is known as the flexural
stress. For brittle materials such as dental ceramics, the flexural strength can be greatly reduced by the presence of surface imperfections or voids, causing stress in much higher concentration levels. These increase in concentrations of stress can cause the material to fracture under less force than if it was in perfect condition, where the force would be more evenly distributed throughout a larger surface area (McCabe, 2008).

The flexural strength of zirconia is an important physical factor and mechanical property to ensure the success of a dental restoration and the overall clinical performance because flexural strength substantially contributes to resistance to fracture (Hiafeng et al, 2015). Together with the impeccable mechanical properties and aesthetic values of zirconia (Kim et al, 2016), it is the strongest ceramic for use in monolithic formed dental protheses (Kim et al, 2016; Zhang and Lawn, 2017).

1.2 Computer Aided Design/Computer Aided Manufacture

1.2.1 History

Computer aided design and computer aided manufacturing (CAD/CAM) has been applied and facilitated in practically every product manufacturing market throughout the world, producing high quantities of high quality, accurate products. This incredible technology has been facilitated in Dentistry since 1987 (Freedman et al., 2007). The development of these varied systems has since enabled dental professionals to manufacture single unit restorations chairside, enabling same day restorations for patients, but CAD/CAM has revolutionised the dental laboratory. Improvements have been made, standardising the processing methods and allowing high precision dental prostheses to be machine processed proficiently and even in the absence of personnel (Tran et al., 2016) for example throughout the night and over weekends. It is becoming commonplace that digital technology is a part of Dentistry. Since its inception, it has
continued to profoundly improve Dentistry and changes at a rapid rate. In exactly the same way that all students know and use the internet in modern study, dental schools will soon abolish the traditional teachings of impression taking and opt to only facilitate the use of intra-oral scanning to record the oral anatomy of patients.

1.2.2 Digital workflow

Significant advances in the use of digital equipment has already infiltrated throughout dental laboratories globally, with the everyday use of cast scanners, digital design and nesting software, laser sintering facilities and an array of milling and 3-dimensional printers (Masri and Driscoll., 2015). When applied to dentistry, a prosthesis is designed using a design software, it is then converted into an STL file. An STL file has different interpretations of what it is, usually described as a Standard Triangle Language, which the machine uses to understand the design requirements and physically manufacture the prosthesis. It is separated into one of two separate and very different manufacturing technologies, Additive Technology (printing) or Subtractive Technology (milling).

1.2.2.1 Subtractive Technology

The subtracting (taking away material) technology known as milling, is the dominant form of digital manufacturing and was the first of its kind. It works by the machine taking very small but precise amounts of material from a preformed puck, until the desired shape is reached. When an STL file is nested into a nesting software, the file is placed within the material blank, holding bars are strategically placed and the figuration is calculated into a CNC file. This creates a tool path for the spindle and cutting tools to manipulate the work piece movement and control the bur speed.
Various materials have been developed for machining in this way from composites, ceramics, many different hybrid variations of the two and of course zirconia. The milling machines subtract the material and shape it using a chipping away method through the use of one or more cutting tools and multiple axis’ to manipulate the material blank, allowing access. The use of between 3 to 5 axis’ are required to remove overhang residual matter around the equator point in which the two milling angles meet. Machines with a higher number of axis have more freedom of movement and are faster. Milling can be performed wet or dry. Wet milling is facilitated where a material may produce high heat due to friction during milling such a metal, but most other materials can be milled dry.

After milling is complete, some post milling treatment is required depending on the material. In the case of ceramics, removal of the holding bars and sintering in a furnace where shrinkage and shade change takes place. In the case of composites, PMMA indirect temporary materials or hybrids, removal of the holding bars and polishing will complete the processing method. The addition of stains or glazes is also possible to personalise the prosthesis.

Advantages of subtractive technology include efficiency, affordable and cost effective once the initial set up costs are met and accuracy. The tool paths and corresponding parameters can be set to ensure reproducibility in the case of a remake requirement. The ease of use also eliminates the room for human error. Material variations are another clear advantage, if a material can withstand the manoeuvre and cutting forces, it can be milled.

Disadvantages include the high cost of initial purchase of the equipment and the time consumption of training and safety measures that must be undertaken when
using milling machinery. Another disadvantage is design limitations, some areas of a design are difficult for the cutting tools to reach, tool paths are limited, and hollows are not always possible (Masri and Driscoll, 2015).

1.2.2.2 Additive Technology

Additive technology is exactly that, the addition of material. It has been referred to in many ways such as layered manufacturing technology, rapid prototyping and more commonly 3D printing. It works by layering very small amount of material and setting that material with light, following a design STL file that has been configured into the language that the intended machine uses to produce shape.

Similar to subtractive technology in a varied amount of different materials can be printed, including resins, polymers and metals. Unlike subtractive technology, additive technology is limitless when it comes to the application of design freedom. Hollows and difficult to mill areas of a design can be printed due to the platform in which the working area is based, with a greater area for holding structures. Other advantages include the simple one stage fabrication possibilities of one piece designs and the ease of use. Disadvantages include the initial cost of machinery, materials are also costly and are quite limited in capability. Although design freedom is a clear advantage, there is also the requirement for the improvement in accuracy. The finishing of prostheses created through additive technology requires more human input than those that are milled using subtractive (milling) technologies (Masri and Driscoll, 2015).

1.2.3 Comparison to Traditional/Analogue

The modern technology of CAD/CAM processing methods has been widely scrutinised and appraised. Some studies have claimed that these relatively new
methods of providing dental restorations and prostheses are superior to those manufactured through traditional methods. These aspects of superiority included clinical durability, optical aesthetic qualities and marginal accuracy (Baroudi and Ibraheem, 2015; Ender et al., 2015; 2016). Patients have also reported an overall higher satisfaction when dental restorations were created using a full digital workflow (Sfondrini et al., 2018).

1.2.4 Application of CAD/CAM sytems

Digital processing methods can be selected by the application of the dental prosthesis and its determining material. The materials include ceramics, composites, waxes to be cast after milling, hybrid materials, temporary materials and metal alloys. Materials can be subtractive machined in two states, hard milling and soft milling. Hard milling is the removal of material to shape it into the prosthesis in its final form. Soft milling is the removal of material to shape it before a finalisation process such as sintering. Advancements in CAD/CAM processing technologies has led to the development of materials that were not previously used in traditional methods. The quick manufacture possibilities using digital processing is growing in popularity along with the use of Zirconia because of desirable optical and outstanding mechanical properties, together with high biocompatibility (Masri and Driscoll, 2015).

1.3 Zirconia

Ceramic dental materials that have been specifically developed in dentistry are called bio-ceramics. The technology developing Zirconia has encouraged a fast advance in metal free dentistry that encourages the choice to eliminate metal and may facilitate high biocompatibility, improved strength and excellent aesthetics (Vagkopoulou et al, 2009).
The strongest dental ceramic, Zirconia (Zhang and Lawn, 2017) is consistently being manufactured to full monolithic form because of the superior mechanical properties displayed when compared to porcelain veneered restorations (Kim et al., 2016). This has also expanded on the possible clinical indications of Zirconia (Beuer et al., 2011; Kim and Kim, 2014).

1.3.1 Development of zirconia

“Zirconium” received its name from the Arabic word “Zargon” meaning “golden in colour”. Martin Heinrich Klaproth accidentally discovered Zirconium dioxide (ZrO₂) was accidentally in Germany, 1789. He was working with positive trials involving the heating of gems (Piconi & Maccauro, 1999). Later, the development of research resulted in the use of zirconium (ZrO₂) for medical purposes was made in 1969 as a new material for hip femoral head replacement instead of titanium (Evans and Heuer, 1980; Hannink et al., 2000). Zirconium has exceptional resistance to corrosion and is a lustrous, transitional metal element. It has a melting point of 1855°C (3371°F) and a boiling point of 4409°C (7968°F). The atomic number 40 on the periodic table belongs to Zirconium, it has an atomic weight of 91.22, density of 6.49 g/cm³ and is ranked as 18th among the element in abundance on earth. It does not only occur in conjunction with silicate oxides or as a free oxide, such as Zirconium Dioxide (ZrO₂) and not in a pure state on its own (Chevalier, 2006; Denry & Kelly, 2007; Kelly & Denry, 2008). Zirconium Dioxide (ZrO₂) is found in Zircon (ZrO) and is white crystalline in appearance (Ossema et al., 2016).

1.3.2 Composition and structures

Of the many different types of zirconia containing products are available, there are only currently three that are used in dentistry (Denry and Kelly, 2008). (Y-TZP)
Yttria-stabilized tetragonal zirconia polycrystal, (Mg-PSZ) magnesium-stabilized zirconia and zirconia toughened alumina (ZTA).

Y-TZP is the most common choice, but it is aesthetically limited by the absence of translucency when compared to feldspathic alternatives, in many cases restricting the material to posterior use (Zhang and Lawn, 2018). More recently, further advancements in the materials used for digital manufacturing processes have led to a more translucent zirconia (Kim et al., 2016).

1.3.3 Transformation toughening mechanisms

Zirconia is polymorphic in nature, meaning there are changes in the structure induced by changes to temperature. It is stable at room temperature in its monoclinic form. Unalloyed, Zirconia primarily functions in three possible states: at room temperature it is monoclinic (M) in form with a prism with parallelepiped sides, until 1170°C, it then becomes tetragonal(T) formed as a straight prism with rectangular sides and is stable between 1170°C and 2170°C, and cubic(C) formed as a straight prism with squared sides is stable above 2370°C (Ebeid et al., 2014). The cubic phase is associated with moderate mechanical properties, the tetragonal phase is associated with improved mechanical properties and the monoclinic phase is associated with lower mechanical properties (Ruiz & Readey, 1996; Burger et al., 1997; Hannink et al., 2000). The tetragonal state of zirconia is ideal for dental application, but it is not stable at room temperature, thus a need to stabilise it through transformation toughening.

As a crack is propagating in ZrO₂ which has metastable tetragonal particles, at the crack tip a stress induced particle transformation occurs. This transforms the particles from tetragonal to monoclinic. Only the particles at the tip of the crack
transform. This particle transformation strengthens the material. The energy from the crack is dissipated, allowing the absorption of forces when the molecules transform phase on impact. The structures from the tetragonal (T) phase to the monoclinic (M) phase, is accompanied by a considerable increase in volume (about 4.5%), causing residual compressive stress on the material, bringing the crack to a stop within the structures and compacting the particles together, the compressive stress resists further crack advancement. Because the volume increase could lead to failure, which is possibly catastrophic to the material (Chevalier et al., 2004), the processing must be managed very carefully in order to achieve toughening. Stopping the return from tetragonal to monoclinic is important to stabilise it at room temperature. This is done by the addition of another compound such as Magnesium Oxide (MgO) or Yttria (Y₂O₃) (Hjerpe et al., 2016; Frary, 2017).

The addition of another compound into the crystalline structures of ZrO₂ such as Yttria (Y₂O₃) will stabilise the tetragonal phase and inhibit the monoclinic transformation to occur. In the case of the addition of Yttria (Y₂O₃) the resulting material is known as Partially Stabilised Zirconia (YTZP), allowing tetragonal zirconia to be stable at room temperature. Resulting in enhancement in fracture toughness from this mechanism as the energy associated with crack propagation is dispersed in both phase transformation and in overcoming stresses due to the volume expansion, known as transformation toughening (Garvie & Nicholson, 1972). The ceramic most commonly toughened in this way is Zirconia, but it is also used to toughen Alumina and others (Frary, 2017). The desirable properties of 3%mol partially Yttrium stabilized Zirconia (3YTZP) used in Dentistry allow the manufacture of prostheses which are able to withstand large occlusal forces. These materials encouraged the use of the
material in restorative Dentistry and implant Dentistry due to higher fracture resistance and flexural strength (Chevalier et al., 2004; Hjerpppe et al., 2016).

1.3.4 Applications and Aesthetics of Zirconia

Zirconia can be applied to a varied amount of dental restorations, but this is dependant on where it is positioned in the oral cavity and the amount of occlusal and masticatory force it is exposed to. Differing types of machinable blocks have been manufactured to be placed in the different areas of the mouth, with different strength properties shown on the manufacturing data sheets. The data provided with the material will state which level of force it can successfully cope with in the oral cavity in Megapascals (MPa), but this is directly in relation to the aesthetic value of the material and the amount of translucency it will display. For example, a highly translucent aesthetic Zirconia, designed for use in the anterior region, ideally for veneers and crowns which will only be subjected to 400 MPa of pressure will be indicated in the manufacturers’ instructions for use as it is adequate in strength values, but it may not be the best aesthetic option.

On the other end of the scale, in the posterior region of the mouth, where forces can reach from 500MPa – 1000MPa (Hidaka et al., 1999; Yilmaz et al., 2007) a Zirconia material designed for this purpose, is ideal for the monolithic contoured crown will withstand impressive compressive forces up to 1400 (MPa), but the aesthetic optical value is compromised when compared to the optimum aesthetic possibilities of the feldspathic, layered crown. Because Zirconia is a classed as a metal free option for the patient, it is an ideal aesthetic option when compared to metallic coronal crowns, or porcelain fused to metal crowns with incredible compressive and flexural strength properties, but it is not the most aesthetically pleasing option once the
naturally aesthetic optical properties of layered feldspathic restorations have been observed (Shahmiri et al., 2018).

When Zirconia is applied to the substructure of a crown or a fixed partial denture (FDP) similar to a porcelain fused to metal crown, the success rates are much higher. This with the added attractive optimum optical characteristics make this the ideal choice for the patient (Schley et al., 2010).

1.4 Acidic Beverages

The irreversible loss of hard enamel dental tissue without the involvement of micro-organisms by a chemical process is known as dental erosion. The causes of this type of destruction to hard tissues of the oral cavity can include intrinsic sources such as frequent vomiting that comes with suffering from an eating disorder, or extrinsic sources such as high consumption of acidic drinks. Many studies through the years have confirmed that these destructive and acidic drinks can substantially damage the tissues of the teeth and the materials used to restore them (Scribante et al., 2019).

Acid erosion is prevalent in children and young adults in many different countries across the globe, in particularly ranging in ages 18-35. This is exacerbated by the consumption of carbonated and energy drinks (Al-Dlaigan et al., 2017; Erdemir et al., 2012). A study by Al-Dlaigan et al. (2001) stated that the most frequently consumed drinks associated with dental erosion contained phosphoric and naturally occurring fruit acids, namely fresh fruit juices and Coca-cola. Coca cola is a commonly consumed carbonated beverage, having PH level of 2.52, is extensively linked to dental erosion (Colombo et al., 2019), which is the functional loss of tooth tissue...
enamel, dentin and cementum, possibly resulting in occlusion alteration and pain (Hengtrakool et al., 2011).

The oral environment has the potential to be a very hostile environment for any dental prosthesis, with the fluctuation of PH levels, temperature and humidity, the addition of acids such as propionic, lactic and acetic acids present in acidic and carbonated beverages. With recurring consumption, these acids can substantially damage the enamel layer of natural teeth and lead to a reduction of the mechanical properties and clinical performance of dental restorations, for example composite or hybrids (Colombo et al., 2019). Studies have also shown that acidic beverages cause the surface degradation of these composite resins, altering the surface roughness in differing degrees dependant on the time spent immersed (Hengtrakool et al., 2011; Poggio et al., 2012).

1.5 Zirconia and Acidic exposure research

Crowns manufactured from Zirconia products are more durable and reliable than every other all-ceramic systems (Xie et al, 2015). Although clinical studies by Sailer et al. (2007) and Triwatana et al. (2012) have reported failures in Y-TZP when applied to extended sub-structures, such as fixed partial dentures. The causes were veneer fracture, restoration dislodgment, abutment tooth fracture and secondary caries (Triwatana et al., 2012). Low temperature degradation (Kelly and Denry, 2007; Giordano and Sabrosa, 2010), and chemical degradation (Egilmez et al., 2014) have also been reported to cause changes in the mechanical properties of Y-TZP adversely. Egilmez et al., (2014) suggested the flexural strength of Zirconia was significantly decreased after being immersed in 4% acetic acid, during chemical degradation testing for 7 days, but this was at 80⁰C. Other studies have also reported Y-TZP corrosion and changes in surface morphology after acid etching testing with nitric acid,
hydrofluoric acid and sulfuric acid for 30 minutes, but again these studies were at high temperatures (Casucci et al., 2009; Xie et al., 2015). These previous studies are acid etching at high temperatures, not room or temperatures of the oral cavity, and the concomitant issues surrounding a warm environment and phosphoric acidic presence that comes with consumption of acidic drinks, such as Coca-cola. During researching this topic, the studies reported significant changes in the mechanical properties in the materials they were testing after exposure to the different acidic substances, whether it was acid etching or immersion in acidic drinks. All papers were investigating materials that included feldspathic ceramics, composites and hybrids, CAD/CAM materials, Zirconia or a combination of materials (Haifeng et al., 2015; Colombo et al., 2017; Poggio et al., 2018; Colombo et al., 2019).

When researching Zirconia on any scientific search engine, thousands of publications are returned that have been published over the last 30 years (Lughì and Sergo, 2010). Since the clinical research on this material seems plentiful, in direct relation to dentistry the use of Zirconia is relatively recent. The prediction of long-term success is limited because some aspects of the material’s behaviour are not fully known by researchers in this field (Kelly and Denry, 2007; Denry and Kelly, 2008; Hisbergues et al., 2009). According to the available literature, there is no study testing the flexural strength of Zirconia after immersion in Coca-Cola drink, as far as we are aware. This justifies the requirement of a new study.

1.6 Aims of Study and Research Hypotheses

The aim of this study was to investigate and evaluate the effect (if any) of phosphoric acid contents of acidic drink on the flexural strength of dental Zirconia after immersion for different time lengths, at an ambient temperature. Based on similar previous studies, the hypothesis being that there will be an adverse effect or reduction
in the flexural strength of the zirconia specimens. Also, that the specimens immersed in the acidic drink for longer, will be more adversely affected.
2.0 Materials and Methods

2.1 Material Selection

The Zirconia used in this study was Zirlux® Anterior Multi (Zirlux®, Zahn Dental, Langden, Germany). The material characteristics can be seen in Figure 2.1.1 and Figure 2.1.2.

![Chemical Composition](image1)

![Physical Characteristics](image2)

2.2 Specimen preparation

The specimen type used for this experiment are within the dimensions specified by the BS EN ISO 6872 (2015) standard for dental ceramics, within the three-point testing section. These dimensions are:

Width = 4mm, Height = 3mm, Length = 35mm (4 x 3 x 35mm)

The specimens were designed using SolidWorks Software (SolidWorks, Solid Solutions, Leamington Spa, UK) and then nested using Millbox software (Millbox, DOF...
Inc, Seoul, South Korea). There was adequate space in each pre-sintered soft state zirconia puck for 13 specimens. One specimen was nested central in the block with a holding bar at each long axis end measuring 2.7mm, no holding bars were place on the front, bottom, top or back planes to ensure a completely smooth surface without imperfections, that may implicate the results. Once one specimen was nested, the other 12 specimens were copies to ensure a uniform and identical nesting conformity. The 2\textsuperscript{nd} and 3\textsuperscript{rd} pucks of specimens were all milled using the same nested STL file, again to ensure identical and uniform specimen results (see Figure 2.2.1).

The specimens were machine milled in the Roland DWX-52D 5axis (DGShape, London, UK) milling machine, as seen in Figure 2.2.3. Sintering was performed using the CDF15/1C furnace (Carbolite Gero, Derbyshire, UK) following the 4-step sintering programme. Once the holding time at 1450\(^\circ\)C was complete, the furnace was switched off, in line with the manufacturer instructions. The furnace was left to completely cool to room temperature to avoid any thermal shock surface cracking.
All specimen samples were milled from 4 pre-sintered zirconia blocks, with corresponding LOT codes and shrinkage factor of 1.255 (see Figure 2.2.4) After sintering, specimens were left to cool fully to room temperature before removing them from the furnace, avoiding any thermal shock differences. The samples were not glazed or polished for any surface treatment to maintain the accuracy of the dimensions of the specimens. The immersion into acidic drink commenced in the original state after sintering.
2.3 Specimen Groups

The specimens were randomly allocated to 3 designated specimen groups:

Group A (control group) – immersed in ionised water for 7 days

Group B – immersed in acidic drink for 3 days

Group C – immersed in acidic drink for 7 days

There were 10 specimens in each group, in order to repeat the three-point flexural strength test 10 times. This is to ensure accurate, reproducible results (See Table 2.3.1)

The brand of acidic drink was original Coca-cola (Coca-cola company, Milan, Italy) that has full sugar content (not diet or zero calorie formula) PH level of 2.52.

<table>
<thead>
<tr>
<th>Group</th>
<th>Immersion time</th>
<th>Immersion Liquid</th>
</tr>
</thead>
<tbody>
<tr>
<td>Control</td>
<td>7 Days</td>
<td>50ml Ionised Water</td>
</tr>
<tr>
<td>Group 2</td>
<td>4 Days</td>
<td>50ml Coca-Cola</td>
</tr>
<tr>
<td>Group 3</td>
<td>7 Days</td>
<td>50ml Coca-Cola</td>
</tr>
</tbody>
</table>

Table 2.3.1 Summarisation of the study groups.

There were 3 groups, the first Group (Control) was not immersed in Coca-cola drink, instead it was immersed in ionised water for the duration of the experiment. This was so there was an original control group of specimens allowing comparison. The other 2 groups (2 and 3) were immersed in Coca-cola for different time lapses, 4 days and 7 days. The drink was not emptied or refreshed at any point during this time.
After each immersion period had lapsed, the specimens were removed from the acidic drink and rinsed. They were kept in ionised water for 4 days until the testing facility was available. They were transferred in plastic pouches to the testing facility.

2.4 Flexural strength testing

Three-point flexural strength testing was performed using the Universal Testing Machine (Instron Model 3699, Norwood, MA, USA). The specimens were subjected to the same type of force, applied at the exact crosshead speed until fracture occurred, as shown in figure 2.2.4. The results were recorded accurately.

The exact amount of force applied in Newtons (N) at the moment before fracture occurred (known as the breaking load) was recorded and the flexural strength in megapascals (MPa) was calculated using the following formula:

\[ \sigma = \frac{3Pl}{2wb^2} \]

Where:

- \( P \) = is the breaking load, in newtons;
- \( l \) = is the test span (centre-to-centre distance between support rollers), in millimetres;
- \( w \) = is the width of the specimen, i.e. the dimension of the side at right angles to the direction of the applied load, in millimetres;
- \( b \) = is the thickness of the specimen

This formula was used to calculate the flexural strength, \( \sigma \), in megapascals and record the mean and standard deviation of the strength data. This is line with the guidelines set out in ISO Standard 6872 Dental Ceramics (2015).
2.5 Statistical analysis

Statistical analysis was conducted to compare the data of each group using the IBM SPSS software (IBM SPSS Statistics 25, IBM, city, country).
3.0 Results
The mean Flexural strength of the three Zirconia Groups are shown in Table 3.1. The standard deviation of each group is displayed.

![Bar chart exhibiting the mean flexural strength in Megapascals (MPa) of three study groups after immersion.]

Table 3.1. Bar chart exhibiting the mean flexural strength in Megapascals (MPa) of three study groups after immersion.

Most of all specimens fractured into two pieces, with a few fracturing into three pieces. None of the specimens shattered completely, so it was possible to record a breaking load for each individual specimen. The mean of each specimen group was calculated using the formula specified in the materials and methods section.
As depicted in Table 3.2, a comparison of results was performed to distinguish statistical differences. After statistical analysis, the results show no significant difference in flexural strength between the Control Group 1 and Group 3. There was a reduction in the flexural strength of Group 2. When comparing the differences between the control group and group 3, there was a slight difference in the flexural strength after 7 days of immersion, but the difference was more than 0.05, considering the difference insignificant.

Table 3.2. The multiple comparisons of the mean flexural strength between the three specimen groups.

<table>
<thead>
<tr>
<th>(i) Zirconia Group</th>
<th>(j) Zirconia Group</th>
<th>Mean Difference (i-j)</th>
<th>Std. Error</th>
<th>Sig</th>
<th>95% Confidence Interval</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>Coke 4 days</td>
<td>30.5854212</td>
<td>26.4201765</td>
<td>.488</td>
<td>-34.921232 - 96.092074</td>
</tr>
<tr>
<td>Coke 7 days</td>
<td>Coke 4 days</td>
<td>-3.1380413</td>
<td>26.4201765</td>
<td>.992</td>
<td>-68.646694 - 62.368612</td>
</tr>
<tr>
<td>Coke 4 days</td>
<td>Coke 7 days</td>
<td>-33.7234625</td>
<td>26.4201765</td>
<td>.420</td>
<td>-99.230115 - 31.783190</td>
</tr>
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Discussion

The flexural strength of Zirconia was three point tested after the immersion in Coca-Cola drink in this study. This resulted in the rejection of the hypothesis that there would be a lower mean flexural strength value after the immersion in Coca-cola for 4 and 7 days. The results showed that there is no statistically significant difference in flexural strength of the Zirconia specimens.

However, there is a reduction in the flexural strength in Group 2, which were immersed in the same acidic drink but for the shorter time period of only 4 days. If the reduction in the mean flexural strength was accompanied by a statistical analysis reading of 0.05 or less, deeming it statistically different, it could be said that the reduction was due to the immersion in the coca cola, but it is not.

The statistical analysis comparison between groups 1/group 3 (0.488) and for group 2/group 3 (0.420) are more than 0.05. This suggests that the reasons for this reduction in flexural strength in group 2 is not due to the acidic drink, it could be a different factor.

The lower mean flexural strength of group 2 is of interest. The reduction in mean flexural strength was not caused by the immersion in the acidic drink, because the mean flexural strength of group 3 is similar to that of the control group. The lower flexural strength of group 2 must be because of another reason, possibly one of the following.

The allocation of specimens into study groups was completed at random. All specimens for this study were created in a uniform and structured manner, with the aim of producing specimens that were as identical as possible. The reliability of the
way the specimens were created is reinforced by the consistency shown in the similar values of standard deviation of all three groups.

After sintering, when inspecting the specimens by eye, there were some cracks and voids on some of the surfaces of specimens that could be seen. Due to cost and time restrictions, it was not possible to repeat the manufacturing process at this time. This could be considered as a limitation of this study. It could be possible that imperfect specimens could have been allocated into group 2, this would explain the lower mean flexural strength value. McCabe (2008) stated that the flexural strength of a material could be significantly reduced by surface imperfections or voids, due to higher concentration levels of stress causing the material to fracture at lower levels or force.

Some past studies of mechanical properties of Zirconia have experimented with specimens that have been surface treated by electro-polishing or glazing. It could be said that because these studies had ensured perfect finished to the surface of the Zirconia being tested, that the results from said studies were more reliable. In the current study, the decision was made to avoid altering the surface of the specimens in any way, to avoid inducing surface cracks that may compromise the flexural strength of the specimens. The idea of testing the specimens in the unaltered milled shape would eliminate the possibility of human error, maintain uniform shape and keep the dimensions accurate and in line with those specified in ISO standard 6872 (2015). In the case of further study or a repeat of this type of study, surface treated specimens of Zirconia may a possible answer to ensuring a more consistent result between comparison groups.

After researching this subject area, it is evident that studies testing mechanical properties of Zirconia after exposure to different acidic factors are few, reducing the
availability of comparison literature. In order to make assumptions of why there was no significant change in the mean flexural strength value between Control Group 1 and Group 3 (7 Days), this current study must be compared to other studies of a similar format of method. The studies may be testing different mechanical properties or different materials entirely. This can only be done by comparing the results and causes of flexural strength reductions in other similar studies.

A study by Poggio et al (2018) evaluated the effects of acidic drinks such as orange juice and coca cola on the Vickers microhardness (VK) of different aesthetic materials including composites, nano-filled composites and hybrid composite-ceramics. The study was similar to the current one, although it was testing the Vickers Microhardness (VK) it was still a study evaluating effects on materials after immersion in Coca-cola for a varied amount of time between 1-7 days. The results showed a significant decrease in the VK for all materials tested between day 1 and day 7. The conclusions specifically stated the disappointing result of the Gradia Direct hybrid composite. This low microhardness value being due to the micro filled nature of the material, as the grain size, type and concentration levels of the fillers in which hybrid composites are comprised, may impact on the overall material resistance to degradation (Poggio et al., 2018). When this is considered in direct relation to the structural composition of Zirconia, studies often find that there is not a statistical change due to the nature of the material and the harder polycrystalline structure when compared to composite resins. This is due to the different chemistry of these two materials.

Another study (Scribante et al., 2019) tested the flexural properties of a material after exposure to acidic drink, but the materials were 9 different variations of composite aesthetic restorative materials. The study had a similar method structure to this current
one where three specimen sub-groups of each material for different time spans spent uninterrupted and immersed in Coca-cola. The control group (group 1), a group 2 (group 2) that were immersed for 1 week in coca-cola and a group 3 (group 3) that were immersed in Coca-cola for 1 month. After removal from the acidic drink, the specimens were tested using the same Instron Universal testing machine as in the present study and were subject to statistical analysis. The results showed a significant reduction in flexural values across all of the materials tested, with the nanofilled composite performing better than the others.

When reviewing the results of the studies by Scribante et al. (2019) and Poggio et al. (2018) in the last paragraph, it can be assumed that there is a pattern emerging. As both of these studies showed a reduction in mechanical property performance of the materials tested, but the Zirconia specimens in this current study showed no significant statistical change in flexural performance after immersion to acidic drink. Based on the previous research discussing the absorption of different materials due to their crystalline structural composition, the assumption is that Zirconia is not absorbing any of the acidic drink thus not allowing it to penetrate the structure of the material or breakdown the crystalline structural matrix of the material surface. Resisting the reduction of the flexural strength and maintaining composition.

Because the Zirlux® Anterior multi Zirconia in this study is created for the manufacture of anterior restorations, where the optical properties are paramount over the requirement for mechanical properties, this Zirconia has a cubic form to enhance the translucency of the restoration. Cubic forms of Zirconia have similar mechanical properties to Lithium Disilicate dental materials, with similar flexural strength withstanding up to 400MPa, thought provoking a suggestion for further study. It would
be interesting to explore the mechanical performance of Lithium Disilicate after exposure to Acidic Drink.

5.0 Conclusions
After 30 Zirconia specimens were immersed in Acidic Drink for 3 days and 7 days, the immersion in Acidic drink had no statistically significant effect on the flexural strength of Zirconia. The flexural strength was not affected by the length of time spent immersed in Coca-cola.

6.0 Reference List


Fig 1. Chemical Composition of Zirlux® Anterior Multi Zirconia, Zirlux Anterior Multi, Multi-Layered Zirconia with more indications and strength than Lithium Disilicate [online] Available at: https://www.zirlux.com/zirlux-anterior-multi/ [Accessed 02 March 2020]

Fig 2. Physical characteristics of Zirlux® Anterior Multi Zirconia, Zirlux Anterior Multi, Multi-Layered Zirconia with more indications and strength than Lithium Disilicate [online] Available at: https://www.zirlux.com/zirlux-anterior-multi/ [Accessed 02 March 2020]


Giordano, R. & Sabrosa, C.E. (2010), Zirconia: material background and clinical application, Compendium of continuing education in dentistry [Online], vol. 31, no. 9, pp. 710 Available at: https://bolton.summon.serialsolutions.com/#!/search/document?ho=t&fvf=ContentType,Book%20Review,l&l=en-UK&q=Zirconia:%20material%20background%20and%20clinical%20application.&id=FETCHMERGED-LOGICAL-p476-6c4762f0e7724bb1b7822ac18c4b88d92fcaabab86151400c109d1f1ce1f06202 [Accessed: 16 February 2020]


