A

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"Fabrication and Wear Analysis of Particulate Filled Glass Ionomer Cement Mixed Dental Composites."

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CERTIFICATE

This is to certify that the dissertation report entitled **“Fabrication and wear analysis of particulate filled glass ionomer cement mixed dental composites.”** prepared by **UTTAM KUMAR MEENA (ID 2013PPE5128)** in partial fulfillment for the award of degree of **Master of Technology** in **Production Engineering** of Malaviya National Institute of Technology Jaipur is a bonafide research work carried out by him under my supervision and guidance.

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CANDIDATE'S DECLARATION

I hereby certify that following work which is being presented in the dissertation entitled **"Fabrication and wear analysis of particulate filled glass ionomer cement mixed dental composites**" in the partial fulfillment of requirement for award of the degree of Master of technology (M.Tech.) and submitted in **Department of Mechanical Engineering** of Malaviya National Institute of Technology Jaipur is an authentic record of my own work carried out by me during a period from July 2014 to June 2015 under the supervision of **Dr. AMAR PATNAIK,** Assistant Professor, Department of Mechanical Engineering, Malaviya National Institute of Technology Jaipur.

The matter presented in this dissertation embodies the result my own work and studies carried out and has not been submitted anywhere else.

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ABSTRACT

Glass ionomer cements (GICs) are clinically attractive filling materials often employed in the field of dentistry as restorative and luting materials. Glass ionomer cements show great potential as low cost, minimally invasive dental restorative materials. However, their use is limited by relatively poor mechanical properties, especially fracture toughness. One possibility for improving their fracture toughness is through the addition of nanoparticles. TiO_2 and SiO_2 nanoparticles with substitution levels ranging from 0wt% to 9wt% have been prepared and added to commercially available glass ionomer cement to examine the effect of the nanoparticles on the cement mechanical properties. The nanoparticles were incorporated into commercial glass ionomer powder and were characterized using Fourier transform infrared, Thermogrametric Analysis and Dynamic mechanical analysis. Preliminary tests have been carried to assess the effect of these particles on the hardness and wear of hand mixed cement. A significant increase was found for hardness and wear although there appeared to be a slight trend of increasing compressive strength upto 6wt% nanoparticle addition after that the hardness started decreasing. The composites having 6wt% TiO_2 shows the highest value of microhardness and wear was less than the other the particulate filled .The composites results indicate that the mixing procedure must be carefully controlled to obtain the desired mechanical effect without degrading the working properties of the cement. It was concluded that glass ionomer cements containing nanoparticles are promising restorative dental materials with both improved mechanical properties and improved bonding strength to dentin.

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CHAPTER-1

INTRODUCTION

BACKGROUND AND MOTIVATION

1.1 Dental materials

The dental materials are material especially made, designed for the use in the odontology. A dental restoration or the stuffed dental sound a dental restorative material used to restore the function, integrity and morphology of the structure of the absent tooth. Structural loss typically results from caries or external trauma. It is also lost intentionally during tooth preparation to improve the aesthetics or the physical integrity of the intended restorative material. Dental restoration also refers to the replacement of the structure of the missing tooth which is supported by dental implants [1]. There are many different types of dental material and their characteristics vary according to his purpose. Examples include the dressings, dental restorations (fillings, crowns, bridges), materials (to be used in root canal therapy), print materials, prosthetic materials (prosthetics), dental implants, and many others.

1.2 Restorative materials

The restorative materials are used to you to replace the loss of the structure of the tooth, in general due to the tooth decay (dental cavities), but also clothes of the tooth and dental trauma. In other occasions, such materials can be used by cosmetic targets to change the aspect of the teeth of an individual [2]. There are many challenges to the physical properties of the ideal dental restorative material. The goal of research and development in materials is to develop the ideal restorative material [5]. The restorative material ideal would be identical to the structure of the natural tooth in force, accession and appearance. The properties of an ideal filler material can be divided into four categories: physical properties, biocompatibility, aesthetics and application.

- Necessary physical properties include low thermal conductivity and expansion, resistance to different categories of forces and wear and abrasion and resistance to chemical erosion. There must also be a good strength of adhesion to the tooth.

Breaker everyday forces and conditions must be resisted without material fatigue [3].

- Biocompatibility refers to how well the material coexists with the biological balance of the body's systems and the tooth. Because landfills are in close contact with mucous, tooth, and pulp, biocompatibility is very important. Common problems with some dental materials flows include the output of the material chemistry, irritation of pulp and less commonly allergy. Some by-products of the chemical reactions during different stages of the hardening material have to be considered [3].
- Preferably, the filling materials should correspond to the surrounding tooth structure in shadow, translucency and texture.
- The dental operators need materials that are easy to manipulate and to form, where the chemistry of any reaction that has to happen is conceivable or controllable [4].

Dental restorations can be divided into two broad types: direct restorations and indirect restorations.

1.3 Direct restorations

This skill implies placing a soft or malleable filling in the prepared tooth and I increase the tooth before the material puts itself strongly. The advantage of direct restorations consists of the fact that in general put themselves quickly and can be placed in an alone procedure. Since it is needed that the material puts itself while in the contact with the tooth, the limited energy can be spent to the tooth of the process of adjustment without damaging it. Where the force needs from itself, especially how the fillings become bigger, the indirect restorations can be the best option in some cases [3].

1.4 Indirect restorations

This skill of the manufacture is the restoration out of the mouth using the dental impressions of the prepared tooth. The common indirect restorations include onlays, crowns, bridges and sheets. In general a dental mechanic makes the indirect restoration of files that the dentist has provided with the prepared tooth. The completed restoration is joined in general permanently with a dental cement. Often it is done in two visits separated to the dentist. The common indirect restorations are done using the gold or the ceramics [3].

While the indirect restoration is being prepared, a provisory/temporary restoration is sometimes used to cover the prepared part of the tooth, which can help maintain the surrounding dental tissues.

1.5 Materials used in dental restoration

(a) Precious metallic alloys

- Gold (high purity: 99.7%)
- Gold alloys (with high gold content)
- Gold-platina alloy
- Silver-palladium alloy
- Base metallic alloys
- Cobalt-chrome alloy
- Nickel-chrome alloy

(b) Amalgam

Silver amalgam (Consists of mercury (50%), silver (~22-32%), tin (~14%), copper (~8%), and other trace metals). The dental Amalgam is used extensively due to the facility of making the plastic material of rigid direct fillings completed in the alone appointment, strongly acceptable, hardness, corrosion and properties of toxicity. It is more indulgent than preparation and skill that compound resins used with this target. It is principally used now for later teeth. Although the mercury in the cured amalgam is not available like the free mercury, the worry of its toxicity has existed from the amalgam invention like a dental material. The amalgam is still used extensionally in many parts of the world due to its profitability, top force and longevity. Nevertheless, the metallic color is not esthetically agreeable and the alternatives of colors of the tooth arise continuously with more and more comparable properties. Due to the toxicity known about the mercury of the element, there is some controversy on the use of amalgams. The Swedish government prohibited the use of the amalgam of mercury in the June, 2009.Research has shown that, while amalgam use is controversial and may increase mercury levels in the human body, these levels are below safety threshold levels established by the WHO and the EPA. Nevertheless, there are certain subpopulations that, due to the inherited genetic changeability they exhibit the sensibility opposite to mercury levels further down that these levels of the threshold. These particular individuals can experience adverse effects caused by the restoration of the amalgam. These include defects of the innumerable nerves, principally caused by the process of harmed neurotransmitter [6].

(c) Tooth-colored

The dental compounds also are called white fillings used in direct fillings. The crowns and inlays can be done in the laboratory of dental compounds. These materials are similar to used in direct fillings and they are of color of the tooth. Its force and durability are not so high as a china or metallic restorations and they are more inclined to wear and bleaching [13].

(d) Composite resin

Dental composites, also called "white fillings", are a group of restorative materials used in dentistry. As with other composite materials, a dental composite typically consists of a resin-based matrix, such as a bisphenol A-glycidyl methacrylate (BISMA) resin like urethane dimethacrylate (UDMA), and an inorganic filler such as silica. Compositions vary widely, with proprietary mixes of resins forming the matrix, as well as engineered filler glasses and glass ceramics [11]. The filler gives the composite wear resistance and translucency. A coupling agent such as silane is used to enhance the bond between these two components. An initiator package begins the polymerization reaction of the resins when external energy (light/heat, etc.) is applied.

After tooth preparation, a thin primer or bonding agent is used. Modern photo-polymerized composites are applied and cured in relatively thin layers as determined by their opacity .After some curing, the final surface will be shaped and polished.

(e) Porcelain (ceramics)

Full-porcelain dental materials include Dental porcelain (porcelain meaning a high-firing-temperature ceramic), other ceramics, sintered-glass materials, and glass-ceramics as indirect fillings and crowns or metal-free "jacket crowns". They are also used as in-lays, on-lays, and aesthetic veneers. A veneer is a very thin shell of porcelain that can replace or cover part of the enamel of the tooth. Full-porcelain restorations are particularly desirable because their color and translucency mimic natural tooth enamel [8].Another type is known as porcelain-fused-to-metal, which is used to provide strength to a crown or bridge. These restorations are very strong, durable and resistant to wear, because the combination of porcelain and metal creates a stronger restoration than porcelain used alone.Cast metals and porcelain-on-metal are currently the standard material for crowns and bridges. The demand for full ceramic solutions, however, continues to grow.

(f) Other historical fillings

Filled with lead were used in the eighteenth century, but became unpopular in the nineteenth century due to its softness. This was before that lead poisoning was understood. According to Civil War era American dental manuals of the nineteenth century, had already used the metal fillings in the early nineteenth century, made of lead, gold, tin, platinum, silver, aluminum, or amalgam. A pellet was filmed a little larger than the cavity, which is condensed into place with instruments, then formed and polished in the patient's mouth. The stuffing is often "high", with the condensation final - "settling down" - that occurs while the patient chewing food. Gold Leaf was the most popular and recommended: fill material during the Civil War.in and amalgam were also popular due to lower cost, but were held in lower regard.

One survey of dental practices in the mid-19th century catalogued dental fillings found in the remains of seven Confederate soldiers from the U.S. Civil War; they were made of:

Gold foil: Preferred because of its durability and safety.

Platinum: Was rarely used because it was too hard, inflexible and difficult to form into foil.

Aluminum: A material which failed because of its lack of malleability but has been added to some amalgams.

Tin and iron: Believed to have been a very popular filling material during the Civil War. Tin foil was recommended when a cheaper material than gold was requested by the patient, however tin wore down rapidly and even if it could be replaced cheaply and quickly, there was a concern, specifically from Harris, that it would oxide in the mouth and thus cause a recurrence of caries. Due to the blackening, tin was only recommended for posterior teeth.

Thorium: Radioactivity was unknown at that time, and the dentist probably thought he was working with tin.

Lead and tungsten mixture, probably coming from shotgun pellets. Lead was rarely used in the 19th century, it is soft and quickly worn down by mastication, and had known harmful health effects.

1.6 Glass Ionomer Cement

Cement glass ionomer (GIC) is a class of materials used in dentistry as filling materials and plugging cements. These materials are based on the reaction of acid powder and polyalkenoic silicate glass. These materials of tooth color were introduced in 1972 for use as restorative materials for anterior teeth (particularly for eroded areas). As that chemically bond to the hard dental tissues and release of fluoride for a relatively long period, today applications of GIC have expanded. The desirable properties of the ionomer cements of glass makes them useful materials in the restoration of carious lesions in areas of low voltage as small and smooth surface cavities earlier proximal in primary teeth [22]. Results from clinical studies also support the use of conventional glass ionomer restorations in primary molars. They need not be put in layer by layer, like in composite fillings [25].

The concept of using "intelligent" materials in the odontology has attracted a lot of attention in the last years. Cements of conventional glass (GI) have a big number of applications in odontology. They are biocompatible with the dental pulp to a certain extent. Clinically, this material was used initially like a biomaterial to replace the bony textiles lost in the human body. A glass ionomer cement (GIC) is a dental restorative material used in dentistry for dental fillings and luting cements. Glass ionomer cement (GIC) was institute to the dental field in the 1970s by Wilson and Kent. These materials are water-based cements, and are also known as polyalkenoate cements. The cement was comprise of ion-leachable fluoroaluminosilicate glass in a solution of polyacrylic acid that sets through an acid-base reaction. GIC combines the advantages of translucency and fluoride release from silicate cement, and the advantages of biocompatibility and advantageous adhesive properties from poly carboxylate cement [12]. As they bond chemically to dental hard tissues and release fluoride for a relatively long period, modern day applications of GICs have expanded. The profitable properties of the cements of glass ionomer turn into useful materials in the injuries restoration caries in areas of low tension like in childrens and smooth surface previous cavities proximity in primary teeth. Results of clinical studies do not support the conventional use of restorations of glass ionomer or reinforced with metal in primary molars, due to the biggest stress charges occlusal. Nevertheless, use of glass ionomer in molars is common like the cementation, choke or to base the materials can be used in restorations of the temporary term to intermediate in children and adults, particularly in the difficult cases and dental compromised and for the medically awkward and aged patients [30]. These materials have several benefits when it is used like a material direct restorer. These include liberation of fluorine and absorption; chemical adherence to dental structure and visage of similar coefficients of thermal expansion to the dental structure. The main disadvantage of the GIC is in its mechanical properties of low value compared to other materials restoratives available they have done efforts to themselves to improve the material and its mechanical properties increase. Although the strongest and more esthetic materials with improved direction characteristics now are available of resistance and hardness there are still vital problems.

1.7 Chemical classification

Glass is a non-crystalline material typically containing silicon. An ionomer is a polymer that comprises of both electrically neutral repeating units and a fraction of ionized units covalently bonded to the polymer backbone as pendant moieties [16].

GICs are commonly classified into five major types:

a. Conventional glass ionomer cements (low- and high-viscosity)
b. Resin modified glass ionomer cements (conventional with addition of HEMA)
c. Hybrid ionomer cements (also known as dual-cured glass ionomer cements)
d. Tri-cure glass ionomer cements

e. Metal-reinforced glass ionomer cements

(a) Conventional glass ionomer cements

They are derived from aqueous polyalkenoic acid such as polyacrylic acid and a glass component that is usually a fluoroaluminosilicate. When the powder and liquid are mixed together, an acid-base reaction occurs.

(b) The low/high viscosity distinction

"High-viscosity GIC" materials are known to share a higher powder—liquid ratio (>3:1) in comparison to "low-viscosity GICs". However, this material characteristic alone may not suffice for definition, as the mere increase of powder to liquid ratio renders the resulting material unsatisfactory for handling abilities. However, these adverse effects were not reported for glass ionomer labeled as "viscous" in comparison with "low viscosity" GIC. On the other hand, a higher success rate was observed for restorations placed with Ionomer Cements "viscous" glass that glass ionomer cements "low viscosity" when both types of GIC were compared with conventional amalgam restorations. On the basis of such evidence, it seems reasonable to assume that the ICG labelled "viscous" share other features so far not disclosed/unknown that a higher liquid proportion of dust,that clinically superior to GIC labeled "of low viscosity". The "low-viscosity"/"high-viscosity" GIC distinction can thus currently not be accepted on basis of characteristics in GIC chemistry, alone, but primarily on clinical outcomes achieved [17].

(c) Resin modified glass ionomer cements

Resin modified glass ionomer cements are conventional glass ionomer cements with addition of HEMA and photoinitiators.

(d) Hybrid ionomer cements or resin-modified glass ionomers or dual-cured GI

These combine a reaction acid-base the traditional glass ionomer with a reaction of polymerization of amine peroxide self-curing. These curing systems have been developed by adding polymerizable methacrylate groups functional with a photo initiator to the formulation. These materials undergo a reaction acid-base ionomer as curing by photo-initiation and self cure of methacrylate carbon double bonds;

or, in other words, acid-base reactions are complemented by a second polymerization of resin (usually) initiated a process of curing or this reason they are also called Dual-Cured GIC. Developed in 1992, the resin-modified glass ionomer cements in their simplest form are glass ionomer cements that contain a small quantity of a water-soluble, polymerizable resin component. More complex materials have been developed by modifying the polyalkenoic acid with side chains that could polymerize by light-curing mechanisms in the presence of photo initiators, but they remain glass ionomer cements by their ability to set by means of the acid-base reaction [18].

(e) Tri-cure glass ionomer cements

Some systems have also incorporated a reaction of amine-peroxide tertiary chemical cure to polymerize the double bonds of methacrylate with the photo-iniciation and acid-base Ionic reaction. These materials are known as Ionomer Cements vitreo tri - cure. The component of curing cements tri chemical - cure has proved to have a significant effect on their overall strength. By photo-initiated cements cannot be used in cases of opaque structures such as metal substrates. Usually resin-modified glass ionomer cements have a release much lower fluoride than conventional glass ionomer materials.

(f) Metal-reinforced glass ionomer cements or cermets

Ionomer cements of glass reinforced with metal were introduced in 1977. The powder is added to the alloy of silver amalgam to conventional materials increases the physical strength of cement and always radiodensity. Subsequently, were annealed silver particle on the glass, and a series of products then appeared where it had been fixed the content of the amalgam alloy level intended to produce optimum mechanical properties for cement glass cermet. Today, these materials are regarded as old-fashioned, as the conventional glass ionomers cements have comparable physical properties and aesthetic much better.

1.8 Composition and preparation

GIC powder

The powder is an acid-soluble calcium fluoroaluminosilicate glass indistinguishable to that of silicate but with a higher alumina-silicate ratio that increases its reactivity with liquid. The fluoride portion acts as a “ceramic flux”. Barium, Lanthanum, Strontium or Zinc Oxide additives provide radiopacity. The raw materials are fused to form a uniform glass by heating them to temperatures of 1100°C to 1500°C. The glass is ground into a powder having particles into a powder in the range of 15 to 50 µm. usual percentages of the raw materials are [7]:

- Silica 41.9%
- Alumina 28.6%
- Aluminium Fluoride 1.6%
- Calcium Fluoride 15.7%
- Sodium Fluoride 9.3%
- Aluminium Phosphate 3.8%

Functions of different components:-

a. Alumina ($Al_2 O_3$)

- Increase opacity

b. Silica (SiO_2)

- Increase Translucency

c. Fluoride

- Decrease fusion t°

- Anticariogenecity

- Increase translucency

- Increase working time

- Increase strength

d. Calcium fluoride ($Ca F_2$)

- Increase opacity

- Acts as flux

e. Aluminium phosphates

- Decrease melting to
- Increase translucency

f. Cryolite (Na3 Al F6)

- Increase translucency
- Acts as flux

g. Na+, K+, Ca+2, Sr+3

- Include high reactivity of glass with polyacid

Al2 O3: SiO2 ratio is crucial and should be > 1:2 for cement formation to occur. Cement formation will occur only when there will there be sufficient replacement of Si by Al to render the network susceptible to acid attack [9].

The glass can be modified by several ways to improve the physical properties of the cement.

a. Ca can be replaced by Sr, Ba or La to give a R/O glass

b. Washing glasses with organic acids to remove surface concentration of Ca which will help prolong WT

c. Corundum, Rutile, Baddelyte disperse phases can be added to increase flexural strength

d. Metals, resins, fibers are added to increase the strength

1.9 Glass Ionomer Liquid

Originally, the liquid for GIC was an aqueous solution of PAA in a concentration of about 50%. This was quite viscous and tended to gel over time. Thus, PAA was copolymerized with other acids such as itaconic, maleic and tricarboxylic acids. This polyelectrolytic liquid of GIC is, thus, also called as polyalkenoic acids. Recently polyvinyl phosphoric acid has also been introduced to this system. A typical liquid of GIC contains 40-55% of 2:1 polyacrylic: itaconic acid co- polymer and water. The basic functions of these co–polymers include: - the co- polymeric acids are more irregularly arranged than the homo polymer. This reduces H- bonding between acid molecules and reduces degree of gelling - decrease the viscosity - reduce tendency for gelation, hence, improves storage. - Increase the reactivity of liquid .The rest of the liquid comprises of water, which is an important constituent of GIC. It is the reaction medium and helps in hydrating the matrix [10].

Additives in liquid are:-

a. Tartaric acid

- Increases WT
- Increases translucency
- Improves manipubality
- Increases strength

b. Polyphosphates: extends Working Time.

c. Metal oxides: accelerates Setting Time.

Advantages

a. Glass ionomer can be placed in cavities without any need for bonding agents.

b. They are not subject to the contraction and the exit of the mike, as the link mechanism is a reaction acid - base and not a polymerization reaction. GIC does not suffer big dimensional changes in a humid ambience in answer to the heat or to the cold and turns out to be resulted from heating only in the movement of the water inside the structure of the material. These exhibit the contraction in a dry ambience to top temperature to 50˚C that is similar to the behavior of the dentin.

c.Glass Ionomer contains and liberates fluorine, which is important for the injuries prevention caries. Also, since ionomer of glass they liberate its fluoride, they can be "recharged" by the use of fluoride that contains teeth pasta. Therefore, it can be used like a treatment form for patients who are in high caries risk. New formulations of glass ionomer that contain resins photopolimerizables can achieve a major esthetic result, but not liberate fluoride as well as ionomer of glass conventional.

Disadvantages

The biggest disadvantage is the lack of adequate strength and hardness. In an attempt to improve the mechanical properties of the conventional GI, ionomer resin modified have been marketed. GIC is generally weak after the adjustment and they are not stable in water. However, become stronger with the progression of reactions and become more resistant to moisture. Generations: the objective is the regeneration of the tissues and use of biomaterials in the form of a powder or solution is to induce local tissue repair. These bioactive materials release chemical agents in the form of dissolved ions or growth factors such as bone morphogenic protein, which stimulates activate cells [25].

Glass ionomers are about as expensive as composite resin. The fillings do not wear as well as composite resin fillings. Still, they are generally considered good materials to use for root caries and for sealants.

1.10 Thesis Outline

The remainder of this thesis is organized as follows:

Chapter 2: Includes a literature review designed to provide a summary of the base of knowledge already available involving the issues of interest. It presents the research works on glass ionomer cement as well as particulate reinforced dental composites by various investigators.

Chapter3: Includes detailed descriptions of the raw materials, test procedures, and design of experiments methodology. It presents the details of fabrication and characterization of the composites under investigation.

Chapter 4: Presents the physical and mechanical properties of the composites under study.

Chapter 5: Wear analysis of particulate mixed glass ionomer cement

Chapter 6: Conclusion and future scope

CHAPTER-2

LITERATURE REVIEW

The purpose of literature review is to provide background information on the issues to be considered in this thesis and to emphasize the relevance of the present study. This treatise embraces various aspects of dental composites with a special reference to mechanical and wear characteristics. The topics include brief review:

2.1 Historical overview of dental materials

The odontology has the exclusive discrimination for the use of the widest diversity of biomaterials. The biomaterials can be divided in two main classes: biomaterial natural and synthetic Ratner et al [47]. The natural biomaterials are extracted from textile of the plant and animal. These materials are useful in the field of biomimetic and they are used for corresponding and familiar implantations to the textiles of the body. These materials also do not raise problems of the toxicity often confronted by made materials [59]. On the other hand, that can carry particular protein binding sites and other biochemical signals that can help in healing of the tissues or integration, for example, collagen from the bones, tendons, ligaments and skin. Biomaterials are natural: corals, chitin that can be extracted from different sources, such as insects and crustaceans, keratin that is derived from cellulose hair and isolated from plants. However natural biomaterials can be protected from the problems of immunogenicity. In addition, some natural polymers tend to distort or decompose at temperatures below its melting point. This severely limits their fabrication into implants of different sizes and shapes (ASMI, 2003)[1].

2.1.1 Study of metal for dental applications

The metals are of the class of the synthetic biomaterials that they have functional properties, included the force, hardness and malleability and are used commonly in the medicine and the odontology. They are easy to make of a usable form. The metals are very rigid and resistant with a high module of elasticity and butcher. An application of the alloys and special manufacture method process provides a wide range of alternatives in the selection of the best metals for a specific use [54]. The metal alloys have received particular attention as dental and orthopedic implants since these materials are well received by the body, partially due to the comparatively nonreactive surfaces Ratner et al

[47]. The metals are used extensively like structural components for the restoration or the substitution of the structure of the tooth. In particular, they are used in the later region of the mouth where the metallic color is not repulsive. Also, they are used of a more conservative form to replace parts of a tooth. When the part of the tooth to be replaced is inside the summits, it called inlay. When one or several summits are comprised in the restoration, but the finished crown is not replaced, it is called onlays. In general, the remaining teeth used as supports for metallic bridges that span the empty spaces to fill in the arch. These bridges are permanently fixed on the teeth with dental cement. Due to the fact that they replace only a part of the dentition, they are often called fixed partials [47]. The Titanium and alloys of titanium have developed like the dental alloys that are used by china. The Titanium is much a reagent with oxygen and it has a very high melting point. Therefore, special smelting device is necessary for the manufacture of the dental prostheses of titanium [19]. Although titanium has superior biocompatibility, the extra processing and cost required to cast this metal has restricted its use as a dental restorative material [54].

2.1.2 Study of composites for dental applications

Composites are compounds of two or more distinctly different materials with properties superior or intermediate to those of the distinctive components or constituents [11]. Dental composites offer practically limitless possibilities for the development of replacement materials because it is characteristically superior to and lower in cost than dental ceramic and metal alloys Ruddell et al. [46]. Generally they contain compounds hard and similar to glazing of particles of filling that are surrounded with the second material hard disk union of the counterfoil the filling particles. The rudeness of the particles of filling changes of big rocks that microscopically thin dust of forms different from fibres balls in flakes. The material of the counterfoil begins like a pasta or liquid that begins when they are activated, hardening or by means of the addition of a catalyst (that can be mixed by the filling particles), or for water addition or another dissolver to facilitate the chemical reactions. What it is possible to touch in a mold before it hardens. Bis-GMA quartz/silica filler and Poly methyl methacrylate (PMMA)-glass fillers are the most successful composite biomaterials used as restorative material or dental cements in dentistry today [43]. The success is due to the filler particles being coated with a silane coupling agent that comprises a methacrylic group able to copolymerize with the matrix to ensure bonding between the filler and the matrix. From the importance to compounds there are the properties and the fraction of the volume of the respective phases, namely the continuous phase (the counterfoil) and the dispersed one. The compounds are generally classified based on the form or the nature of the dispersed phase, for example, reinforced particle, or the fibre reinforced compounds [12]. The fillings are distinguished by size of the particle and distribution, geometry and surface area Harper et al. [23] illustrated that the smallest particles obtain the best intensification of properties compared with big materials.

2.1.3 Study of polymers for dental applications

The polymers are principally organic substances that constitute an additional class of synthetic biomaterials. There are included big molecules of repeating structural units, called monomers. The materials of the polymer can be divided in inorganic and organic types. At present, the synthetic polymers are used practically in almost every industry and area of the life. In the odontology the polymers are used extensionally like glues and lubricants, as well as structural components Allcoc et al.[3]. With the dental work, the polymers are used exhaustively like temporary restorative materials for alone restoration and bridges to be taken while the metallic restoration or of imperishable ceramics it is being constructed in the laboratory. They are used like the adhesive reagent to improve the link between diverse materials and structures of the tooth or like sealants of the present of cracks and holes in the surfaces occlusal of permanent teeth [24].

2.1.4 Study of ceramics for dental applications

The ceramics materials are synthetic or natural materials that can be applied to the odontology and to have the aptitude to imitate natural teeth. The main components of dental ceramics and china are the quartz, the feldspar and the kaolin. In addition to this, the modifier of crystal and the pigments (metallic oxides) are complemented to the china for the color [14]. From 1996 these materials have been used to make intarsia, veneers and crowns, as linings in the metal substrates, and just when the dental bridges, which can be done completely of the ceramics of high force [2]. Ceramic restorations are extensively made by building up the accurate aesthetic combinations of prefired, pigmented particles, and then refiring under vacuum to sinter them together and eliminate voids [3]. Porcelain is a particular type of ceramic that has been used extensively in dentistry. The first use of the dental porcelain of the dentures, and later, of the porcelain crowns called "porcelain jacket crowns" were developed to restore teeth that has little or no occlusion. Ceramics are generally very brittle materials, and, therefore, cannot be bent or deformed in any area without cracks and a braking system. Powders are produced by melting oxide in high temperature furnaces. Most coloring agents used in dentistry are metallic oxides. Its inclusion in appropriate indices allows ceramist to produce almost any color imaginable. This feature also provides the dentist with the ability to adapt almost any color of the teeth with the aesthetic results that are unattainable with other materials [55]. Porcelain is fundamentally made from white clay, quartz and feldspar. The components are pulverized, mixed, formed into shape and baked Kahler et al.[25].

2.2 Study of fillers for dental applications

The type of fillers considerably influences the properties of composite restoratives. The com Fillings effect on the mechanical properties and marginal fracture, strength degradation, cyclic fatigue, wear resistance) and physical properties (shrinkage, contraction stress/tension, color stability) of composite repair has been widely investigated Braga et al. [11]. the influence of methacrylate, fill resin Matrix type load fill and properties of materials have been widely informed [15]. However, the properties of each inorganic built-in in the organic matrix are not fully understood. Characterization of fillers in composites modern restorative is complex because there are many different types of filling, morphologies and distributions of size [9].Composite restoratives are classified according to the type of fillers used Lutz et al. [36]. This classification, in spite of being nearly twenty years, remains valid for the modern dental composites materials. At present, macrofilled compounds with an average particle size ranges between 5 µm and 30 µm are used less frequently due to the poor aesthetics. The landfills are characterized by using different analytical techniques, the average particle size, and the chemical composition. According to [16], an extremely small nano-particle provides high opacity in unpigmented dental composites, which allows investigators to prepare a spacious variety of shades and opacities of dental composites and thus provide highly aesthetic restorations. In addition spherically shaped nano particles provide superior polishability Rüttermann et al.[49]. As the monomer systems do not change much from case to case, composite restoratives has been commonly classified according to the types of filler added Lutz et al.[36]. Macro fillers are larger ground quartz or glass particles. The filler particles that were used in the first dental composites had diameters ranging from 20 to 40 µm and mainly consist of quartz [42]. Because it is so hard, quartz is difficult to grind into fine particles. These macro filled composites are resultantly used less frequently because of aesthetic concerns and the difficulty of achieving a smooth surface. Recent composites contain ground particles that are in the range of micron particle size or even smaller, such as pyrogenic silica. Inorganic particles of nanometer size, such as silica, SiO2, (Kim et al. [29]; Leprince et al.[32], tantalum, Ta2O5, Schulz et al.[50], titania, TiO2, (Schulz et al., [50]; Khaled et al. [28] and zirconia ZrO2 Amirouche et al.[4] have been introduced into the synthesis of inorganic-polymer composite materials in the last five years. Due to their ideal mechanical properties such as flexural strength, hardness, low abrasion and high radiopacity, nano materials are extensively used for reinforcement in dental resin composites Wang et al., [55]. In modern dental composites, the current trend to reduce filler size whilst aiming to improve the filler loading, seeks to optimise the resultant mechano-physical properties and clinical performance [52]. The introduction of so-called nanofilled and nanohybrid materials, therefore, appears to be a logical continuation of this trend. A number of dental material manufacturers have marketed these composites as the advancement of dental materials into the field of nanotechnology Mitra et al.[38]. A nanomaterial possesses components and/or structural features, such as fibers or particles, with at least one dimension in the range of 1 to 100 nm and subsequently demonstrates novel and distinct properties [13]. Researchers and manufacturers are still debating and speculating whether nano dental composites exhibit improved mechano-physical properties compared to existing dental composites Curtis et al.[14]. “Nano” is not a recognized classification. It is interesting to note that, despite the hype and extensive marketing campaigns by manufacturers, there is little difference in the particle size between the fillers of nano and the fillers of nanohybrid dental composites Curtis et al.[14].diagrammatic illustration of the progress of dental composites employed by restorative dentistry and classified according to the size of the filler particl Beun et al. [9] conducted an extensive investigation comparing the flexural strength, elastic modulus and Vickers' micro hardness of several nanofillers with general and micro filler dental composites materials. The study determined that the nanofillers, Filtek™ Supreme (3M ESPE) and Grandio™ (Voco), exhibit exceptional flexural strengths, surface hardness values and elastic modulus compared to the other dental composites materials tested, with the exception of Filtek™ Z100 (3M ESPE). Subsequently, both nanofillers materials were advisable for posterior and anterior placement Beun et al. [9].

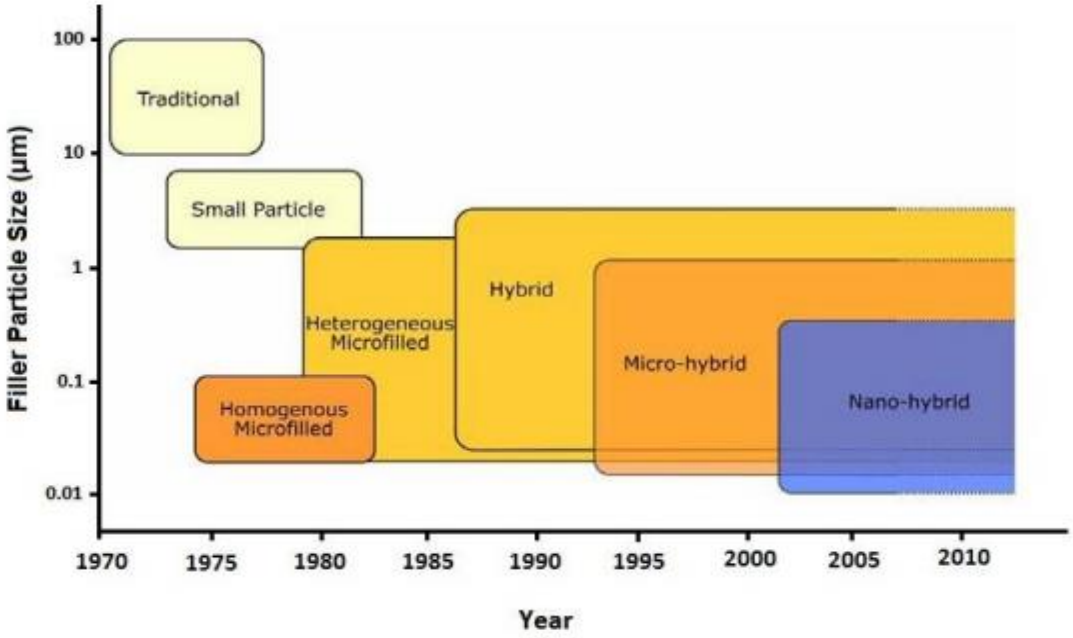

Fig. 2.2 Representation of The Development of Dental Composites [39]

The addition of even small amounts of nano sized silica particles has been recognized to improve the mechanical properties. Tian et al. [52] indicated that the addition of 1 to 2.5wt% of nanosized fibrillar silica to a Bis-GMA/TEGDMA resin significantly improved the flexural strengths (128 and 130 MPa) compared with conventionally dental composites materials, (110 and 120 MPa respectively). This was thought to occur as a result of the reinforcing effect of highly separated and uniformly distributed nano fibrillar silica, whilst the formation of agglomerates of fibrillar silica may debilitate the resulting material Tian et al. [52].

The wear resistance of the composites in the oral cavity depends on the space available between filler particles providing protection against food bolus. The presence of smaller filler particles reduces inter-particle spacing in the composite, improving its wear resistance Bayne et al.[10]. More studies on the implementation of the particles of nano size range as fillers in polymeric composites are carried out. The presence of nanoparticles improves mechanical properties of materials, due to its high specific surface area volume relationship. The use of nanoparticles can give the material with better characteristics, due to the interfacial interactions, resulting in remarkable properties Laine et al. [35]; Wetzel et al. [56]. The nanoparticles have a more uniform distribution of fill in low viscosity materials, such as bonding agents, which restricts "filler", that is to say it is filled with rich, the regions within the matrix Wilson et al. [57].The incorporation of nanosized filler in bonding agents also produced a more homogeneous bond to the tooth/bonding agent interface as filler penetrates the dentine tubules to reinforce the hybrid zone Breschi et al. [12]. Another phenomenon that contributes to the aesthetics of the composite materials of filler is that these translucent materials as a result of the small size of the filler particles combine homogeneously distributed. This occurs when the particle size is smaller than the wavelength of the incident light (400 to 700 nm), the resulting dispersion coefficient is reduced allowing the light to pass through the composite materials without deflection at the interface between the resin matrix and the inclusions, such as filler particles and porosity of hollow [33]. Nanofillers are filler particles smaller than 100 nm. Pyrogenic or fumed silica fillers are often used as filler material in dental composite resins. It is difficult to achieve high landfill using only nanofillers (because of the excessive large surface), and hybrid composite, consisting of a mixture of microsize (about 1 μm in diameter) and nanosize, therefore, normally used. As a result of the increased burden of fillers such as mixing the compounds provide a relatively smooth surface and better mechanical properties Anusavice [9]. One of the most important characteristics of nanoparticles is its very large surface area, causing a very strong thickening effect. In other words, the viscosity of the paste of such composites is much higher than those filled with larger particles at the same filling level [42]. Filling volume fraction must decrease in order of dentists to prepare a manageable dough. However, it can be expected that the modulus of elasticity and resistance is reduced and the contraction would be higher, boosting the rate of defects or what is not suitable for restorations. It is also expected that it will have a greater degree of agglomeration of nanosize particles [42].

2.2.1 Study of nano titanium dioxide for dental applications

There is great interest in the TiO_2 since it has many potential applications, such as chemical sensors, as an anti-fog glass coating material and cleaning and as biomedical material. TiO_2 is also cost-effective and chemically stable, with good optical properties, thermal stability, high refractive index and absorption of visible light deficiency. Several studies have shown that TiO_2 can act as a bioactive material offering strong interfacial adhesion to living tissues Roether et al.[45] . Using nano-TiO_2 particles as fillers in epoxy, some researchers have found that its presence can overcome the drawbacks of traditional toughness, like glass and rubber beads by improving the stiffness, strength and toughness of the epoxy without sacrificing its thermo-mechanical properties Li et al [33]. Nano-TiO_2 particles are very fine and agglomerate easily in practical applications. They are particularly difficult to disperse in organic solvent Zhou et al. [61]. Some surface treatment of the nano-TiO_2 particles is required to overcome this problem. Inorganic fillers in dental composites are normally treated with organo silane in order to improve the bond strength to the resin matrix and increase the service life of the composite [17]. An additional benefit of using this substance is the improved homogeneous dispersion of silanated filler particles throughout the matrix.

2.2.2 Study of nano silicon dioxide for dental applications

SiO_2 is a crystalline metal that occurs widely as sand, quartz and many other minerals and is used to produce a diversity of materials, particularly concrete and glass. Natural of silicone is not to reinforce and has been used as a filler, only to reduce the cost. The reinforce and synthetic today have particle sizes as small as the black coal, in addition to a highly reactive surface [8]. Important natural varieties are silica (crystalline), silica (microcrystalline) and silica diatomaceous (fossil origin). Types of synthetic silica are pyrogenic, precipitated, hydrogels and aerogels. Of this diversity, precipitated silica and pyrogenic (fumed) silica are being used for elastomer reinforcement [8]. The reaction products are satisfied directly after coming out of the burner. Pyrogenic silica is too expensive [8]. Precipitated silica is silicon dioxide, including about 10 to 14% water, with particle size in the range of 1 to 40 nm. They are reinforcing fillers giving composites tear resistance, high tensile strength, and hardness and abrasion resistance and are used in the manufacture of translucent, coloured products and other mechanical rubber goods. Pyrogenic or Fumed silica is silicon dioxide containing less than 2% integrated water. These silica are highly reinforcing fillers of very small particle size, giving tear resistance, good tensile strength and abrasion resistance especially to silicone rubbers [10].

2.3 Study of glass ionomer cement for dental applications

A new era in the world of dentistry has started since the introduction of the Nano-ionomer (GIC) cement in 1972 by Wilson and Kent, which have been considered as a main restorative measure that adhere to tooth structure [42], has an antibacterial activity [44], negligible dimensional changes, release fluoride and can be used in different situations clinical as cementation of indirect restorations, lining, basing and filling Davidson et.al [15]. Use a material ideal for restoration should be adhesive, tooth color, resistant to wear, non-toxic, biocompatible tissue Cramer et al. [20]. Unfortunately, the use of the protection of geographical indications as a restorative material is limited to areas of low chewing forces due to its low mechanical properties, which were also affected by the powder and liquid mixture ratio of this material Kleverlan CJ et al. [26]. The use of nanotechnology greatly increases the reactive surface of the filling material, which in turn, leads to a better reaction. An additive organic carbon chain, which is fully biocompatible, also adds to the glass carbomer to provide the material with greater strength and a greater transparency Kovarik H et al[27]. Although clinical trials would provide the ultimate evidence of clinical performance of dental restorations, preliminary and safety studies on dental materials should be conducted in vitro [14]. The most common and useful mechanical properties for characterizing the GICs are compressive strength (CS), flexural strength (FS), diametric tensile strength (DTS), fracture toughness (FT), microhardness (Knoop (KHN) or Vickers (VHN)), wear rate (WR), fatigue and creep [15–25].The advantage of GIC’s over other dental cements include good adhesion to bone, stability in an aqueous environment, lack of exothermic polymerization and good biocompatibility Kovarik et al.[27].

Chapter summary

This chapter has provided an exhaustive review of research works on particulate reinforced dental composites reported by various investigators. The literature survey presented above reveals the following knowledge gap that helped to set the objectives of this research work:

- Though much work has been reported on various wear characteristics of metals, alloys and homogeneous materials, comparatively less has been reported on the dental wear performance of composites and in fact no study has been found particularly on particulate reinforced composites.
- A possibility that the incorporation of particles in composites could provide a synergism in terms of improved wear resistance and mechanical properties has not been adequately addressed so far and there is inadequate data available about phenomena behind the modified wear behavior due to the addition of particulate fillers to the filler reinforced composites.
- As far as wear study of composites is concerned, no specific theoretical model based.

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CHAPTER-3
MATERIALS AND METHODS

In this chapter, the materials selection, preparation, fabrication, and testing methods are described. The raw materials include the inorganic fillers, resin monomers and chemical additives. The mechanical testing methods include methods to determine micro-hardness, wear resistance. The material characterization methods include FTIR, TGA and DMA. The methodology related to the design of experiment technique based on Taguchi method is also part of this thesis

3.1 Materials

3.1.1 Matrix Material

GIC POWDER

The powder (supplied by unique dental lab and materials, Jaipur India) is an acid-soluble calcium fluoroaluminosilicate glass indistinguishable to that of silicate but with a higher alumina-silicate ratio that increases its reactivity with liquid. The fluoride portion acts as a “ceramic flux”. Barium, Lanthanum, Strontium or Zinc Oxide additives provide radiopacity. The raw materials are fused to form a uniform glass by heating them to temperatures of 1100°C to 1500°C. The glass is ground into a powder having particles into a powder in the range of 15 to 50 µm. usual percentages of the raw materials are:

- Silica 41.9%
- Alumina 28.6%
- Aluminium Fluoride 1.6%
- Calcium Fluoride 15.7%
- Sodium Fluoride 9.3%
- Aluminium Phosphate 3.8%

Functions of different components:-

a. Alumina ($Al_2 O_3$)

- Increase opacity

b. Silica (SiO_2)

- Increase Translucency

c. Fluoride

- Decrease fusion t°
- Anticariogenecity
- Increase translucency
- Increase working time
- Increase strength

d. Calcium fluoride ($Ca F_2$)

- Increase opacity
- Acts as flux

e. Aluminium phosphates

- Decrease melting to
- Increase translucency

f. Cryolite ($Na_3 Al F_6$)

- Increase translucency
- Acts as flux

g. Na+, K+, Ca+2, Sr+3

- Include high reactivity of glass with polyacid.

$Al_2 O_3$: SiO_2 ratio is crucial and should be > 1:2 for cement formation to occur. Cement formation will occur only when there will there be sufficient replacement of Si by Al to render the network susceptible to acid attack.

The glass can be modified by several ways to improve the physical properties of the cement.

a. Ca can be replaced by Sr, Ba or La to give a R/O glass.

b. Washing glasses with organic acids to remove surface concentration of Ca which will help prolong WT.

c. Corundum, Rutile, Baddelyte disperse phases can be added to increase flexural strength.

d. Metals, resins, fibers are added to increase the strength

Glass Ionomer Liquid

Originally, the liquid for GIC (supplied by Unique dental lab and materials, Jaipur India was an aqueous solution of PAA in a concentration of about 50%. This was quite viscous and tended to gel over time. Thus, PAA was copolymerized with other acids such as itaconic, maleic and tricarboxylic acids. This polyelectrolytic liquid of GIC is, thus, also called as polyalkenoic acids. Recently polyvinyl phosphoric acid has also been introduced to this system. A typical liquid of GIC contains 40-55% of 2:1 polyacrylic: itaconic acid co- polymer and water. The basic functions of these co–polymers include: - the co- polymeric acids are more irregularly arranged than the homo polymer. This reduces H- bonding between acid molecules and reduces degree of gelling - decrease the viscosity - reduce tendency for gelation, hence, improves storage. - Increase the reactivity of liquid .The rest of the liquid comprises of water, which is an important constituent of GIC. It is the reaction medium and helps in hydrating the matrix.

Additives in liquid are:-
a. Tartaric acid
- Increases WT
- Increases translucency
- Improves manipubality
- Increases strength
b. Polyphosphates: extends Working Time.
c. Metal oxides: accelerates Setting Time.

Fig 3.1 Glass Ionomer Cement used for testing

3.2 Filler Materials

3.2.1 SILICON DIOXIDE NANOPARTICLE (Sio$_2$)

Nanoparticles of silicon dioxide, also known as silica nanoparticles or nanosilica particles, are the basis for a large number of biomedical research because of its stability, low toxicity and its capacity to be functionalized with a range of molecules and polymers. Nanosilica (supplied by sarthak sales, Jaipur) filler is a typical filler used in dental composites, which has gained popularity in spite of criticism and predicted failures. One of the most important skills used to prepare Nanosilica fillings is the process the sol - gel. Synthesized nanofillings using this method leak out to have a spherical form with a narrow distribution of size that is useful for dental applications. The extremely small nanoparticles size provides low visual opaqueness in composites dental not pigments, which it allows to the investigators to prepare a big variety of tones and opaqicity of dental composites and this way to provide highly esthetic restorations. Also, particles of spherical form Nanosilica provide polished Superior and tend to distribute more uniformly than the particles of irregular form, which leads to the mechanical properties progress mechanical effort.

3.2.2 TITANIUM DIOXIDE NANOPARTICLE (TiO_2)

Titanium dioxide (TiO_2) nanoparticles (supplied by Sarthak Sales Jaipur, India) have been demonstrated to be an effective multifunctional material . As the particle size decreases, especially <50 nm, they exhibit photo-induced activities that originate from the semiconductor band gap of TiO_2 NPs. With their energy higher than the band gap, photons can generate electron–hole pairs whose energy may be applied (1) electrically (solar cells) (2) chemically (photocatalytic activities including killing bacteria and viruses) or (3) by changing the hydrophilicity of the particle surface and resulting in super hydrophilicity. This energy is expressed chemically via free radicals ($HO\bullet$) following a redox reaction mechanism. The photoactivities mentioned above can be efficiently activated over time by easily accessible resources (such as light). TiO_2 NPs also have excellent mechanical properties, for example, the elastic modulus of TiO_2 NPs is approximately 230 GPa, and it is inexpensive with titanium being the fourth most abundant metal on earth, following aluminum, iron and magnesium. The only ones photoactivities of NPs of TiO2 and its top mechanical properties do one of the ideal additives to improve the yield of the materials. NPs of TiO2 has been used as additively in dental materials so that it coincides with the opaque properties of the teeth. In odontology, strong and durable dental adhesives are vital for the long-term success composed of resin used to restore the dental caries. Adhesive dental bond resin composed for the structure of the tooth and debit to support a hard oral ambience and to support charges occlusal of bitting, grinding and chewing. The major components in dental adhesives are dimethacrylate derivatives (dental resins) with/without fillers. Besides high bond strength to tooth and composites, good mechanical properties of the dental resins including modulus and hardness are also essential for success of the dental restoration.

3.3 Sample Preparation

The glass ionomer powder was mixed with the fillers in different proportions (0%,3%,6% and9%). Firstly, for the preparation of sample having 0% filler content 2gram of ionomer powder was weighed and then it was mixed at the room temperature with the liquid in definite proportion prescribed by the supplier on chemically treated paper. For the specimen preparation the mixture was kept into the cylindrical glass tubes of 5mm diameter and 25mm length for wear test. The mixture get hardened itself in 5minutes without any light curing at the room temperature and after 10 minute the glass tube is broken down to get the sample out. Similar process was done for the remaining samples having different composition of filler like for the sample having 3% filler content .3% of 2gram .006gram of nanoparticles and 1.994gram of glass ionomer powder was mixed and then the same process was used as for making 0%filler sample. Samples having 3%, 6%and9% TiO2 and SiO2 nanoparticles was prepared and then rubbed on the grit paper to get the sample required for testing.

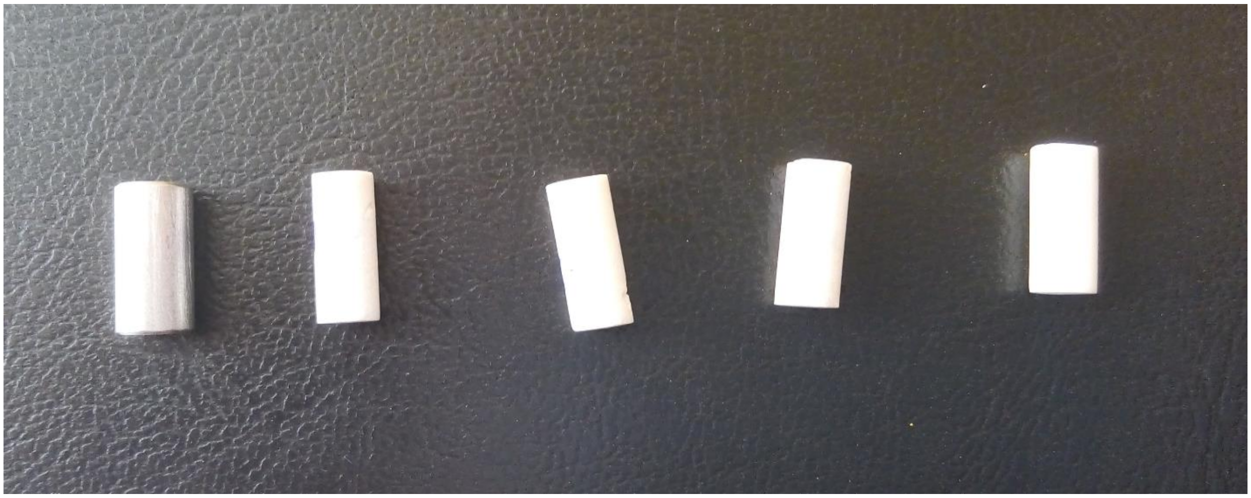

Fig. 3.3.1 Sample for wear and DMA test

For the micro-hardness test samples of circular disc type was made. After mixing the powder and liquid it was transformed into the circular shape and then it was pressed between two flat plates of glass.

Fig 3.3.2   Sample for micro-hardness test

3.4 Physical, Thermal and Mechanical Characterizations

3.4.1 Micro-hardness

Micro-hardness measurement is done using a Micro-hardness tester in Metallurgy Department MNIT Jaipur. A diamond indenter, in the form of a right pyramid with a square base and an angle136˚ between opposite faces, is forced into the material under a load F. The two diagonals X and Y of the indentation left on the surface of the material after removal of the load are measured and their arithmetic mean L is calculated. In the present study, the load considered F = 5N and it had been applied for 15seconds .Vickers hardness number is calculated using the following equation:-

$$H_{V}=0.1889\frac{F}{L} \tag{3.1}$$

and

$$L=\frac{X+Y}{2} \tag{3.2}$$

Where, F is the applied load (N), L is the diagonal of square impression (mm), X is the horizontal length (mm) and Y is the vertical length (mm).

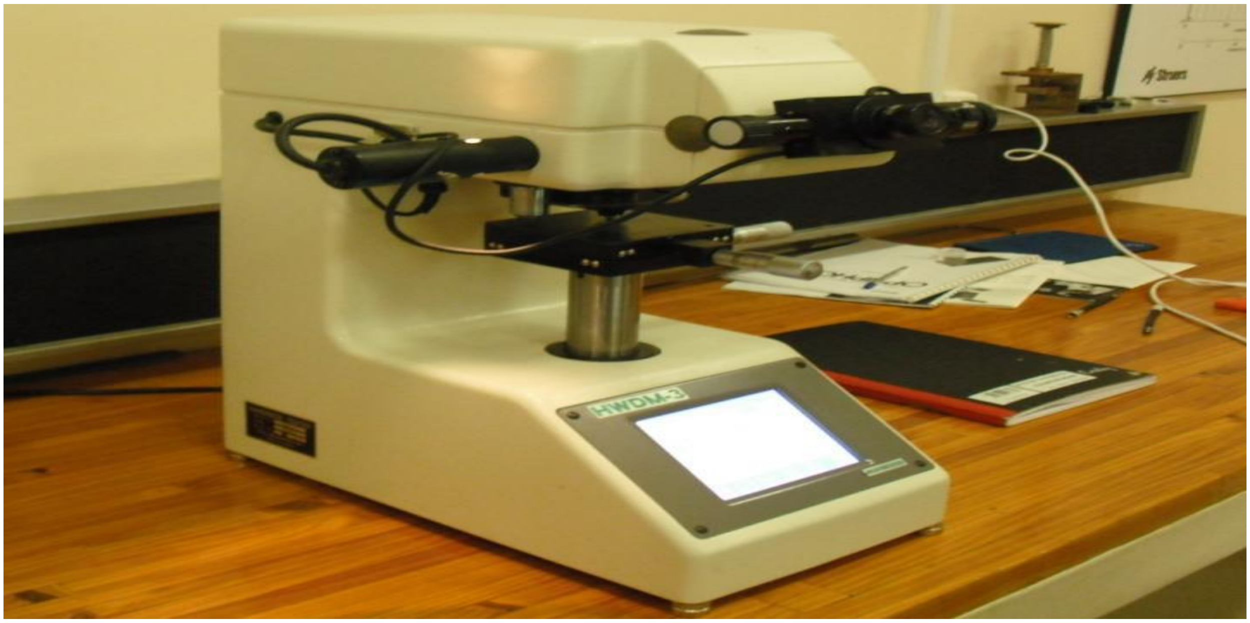

Fig 3.4.1 Apparatus used to determine the Vickers hardness number (VHN)

3.4.2 Fourier transforms infrared spectroscopy (FTIR)

FTIR was used to characterize glass ionomer cement to establish the chemical structure of the GIC after adding nano-particles. A spectrum™ 100 Optica (Perkin–Elmer, USA) instrument, shown in Figure 3.3a, was used at the material research centre at the mnit Jaipur. The following experimental conditions applied: resolution of 2 cm-1 throughout the spectral range (4500 to 400 cm-1) and scanning speed of 0.50 cm s-1.

Fig 3.4.2 FTIR instrument

3.4.3 Thermo-gravimetric Analysis (TGA)

TGA was done at the material research Centre at the mnit Jaipur. Thermogravimetric analysis (TGA) is an analytical technique used to determine a material's thermal stability and its fraction of volatile components by monitoring the weight change that occurs as a specimen is heated. TGA can provide information about physical phenomena, such as second-order phase transitions, including vaporization, sublimation, absorption, adsorption, and desorption. Likewise, TGA can provide information about chemical phenomena including chemisorption, desolation (especially dehydration), decomposition, and solid-gas reactions (e.g., oxidation or reduction). The measurement is normally carried out in air or in an inert atmosphere, such as Helium or Argon, and the weight is recorded as a function of increasing temperature. The maximum temperature is selected so that the specimen weight is stable at the end of the experiment, implying that all chemical reactions are completed (i.e., all of the carbon is burnt off leaving behind metal oxides). This approach provides two important numerical pieces of information: ash content (residual mass,) and oxidation temperature (To).Common applications of TGA are (1) materials characterization through analysis of characteristic decomposition patterns, (2) studies of degradation mechanisms and reaction kinetics, (3) determination of organic content in a sample, and (4) determination of inorganic (e.g. ash) content in a sample, which may be useful for corroborating predicted material structures or simply used as a chemical analysis. It is an especially useful technique for the study of polymeric materials, including thermoplastics, thermosets, elastomers, composites, plastic films, fibers, coatings and paints.

Fig 3.4.3 TGA INSTRUMENT

3.4.4 Dynamic Mechanical Analysis

Dynamic mechanical analysis (abbreviated DMA, also known as dynamic mechanical spectroscopy) is a technique used to study and characterize materials. It is most useful for studying the viscoelastic behavior of polymers. A sinusoidal stress is applied and the strain in the material is measured, allowing one to determine the complex modulus. The temperature of the sample or the frequency of the stress are often varied, leading to variations in the complex modulus; this approach can be used to locate the glass transition temperature of the material, as well as to identify transitions corresponding to other molecular motions. This test was also carried out at the material research center at the mnit Jaipur.

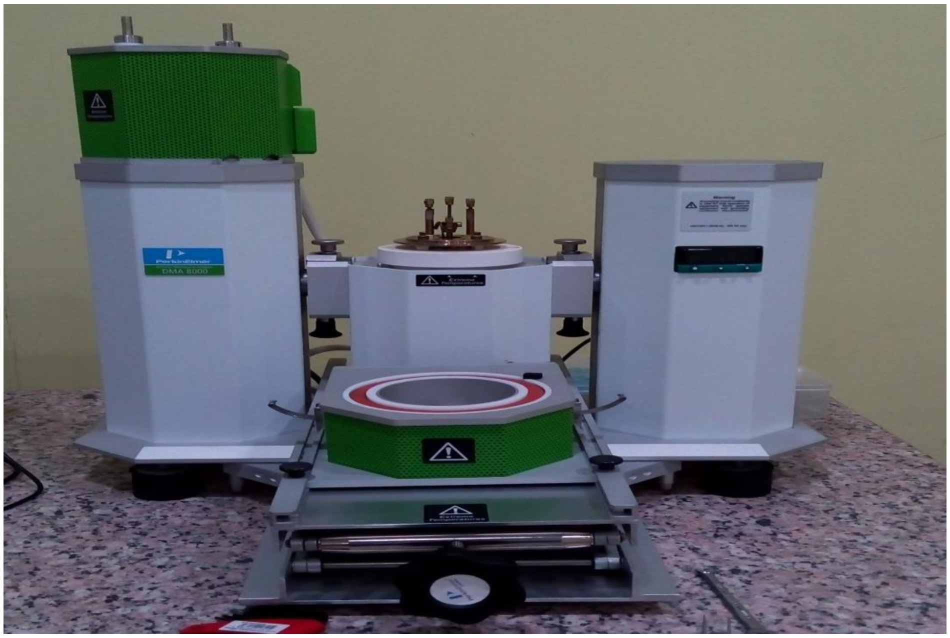

Fig 3.4.4 DMA instrument

3.5 Wear Analysis

Wear test has been done on dental wear simulator at center of research advance tribology in mnit Jaipur. The dental wear simulator is used for evaluating the wear properties of combination of materials that are being considered for use as bearing surfaces of human total joint prostheses. Test can be conducted for all types of prostheses wear applications, like linear reciprocating wear for evaluating the friction and wear of combinations of materials that are used for hinged knee, hinged joints trunnion bearings etc. profile 8 wear motion for test method on combination of materials used for hip replacement .Linear motion delamination wear applications for materials used for tibial replacement etc. in addition circular motion and elliptical motion for other applications. The test rig has four stations simultaneous movements on test rig the top specimen is stationary pressing over the bottom specimen with certain load, while the bottom specimen is under motion. The load is applied on each station by individual pneumatic cylinders controlled by the inlet air pressure from MPPES. The bottom specimen movement is by servo-motor moving the X-Y stage on LM guides, the motion is controlled by PLC controller.

3.6 Process optimization and Taguchi method

Statistical methods are commonly used to improve the quality of a product or process. Such methods enable the user to define and study the effect of every single condition possible in an experiment where numerous factors are involved. Dental wear simulation is such a process in which a number of control factors collectively determine the performance output i.e. the wear rate. Hence, in the present work a statistical technique called Taguchi method is used to optimize the process parameters leading to minimum wear of the dental composites under study.

The Taguchi method is a commonly adopted approach for optimizing design parameters. The method is originally proposed as a means of improving the quality of products through the application of statistical and engineering concepts. Since experimental procedures are generally expensive and time consuming, the need to satisfy the design objectives with the least number of tests is clearly an important requirement. For the elaboration of experiments plan the method of Taguchi for three factors at four levels is used, being understood by levels taken by the factors. In Table 3.5 is indicated the factors to be studied and the assignment of the corresponding levels. The array chosen was the L16 (4^3) which has 16 rows corresponding to the number of tests with 3 columns at four levels, as shown in Table 3 the factors and the interactions are assigned to the columns. In practice, these factors can be assigned arbitrarily to any of the arrays columns, provided that all combinations are included. After assigning appropriate level settings, the S/N analysis (S/N: signal to-noise ratio) is needed to evaluate experiment results. In S/N analysis, the greater the S/N, the better the experimental results:

$$\eta = -10 \log (\text{M.S.D.}) \tag{3.3}$$

where M.S.D. is the mean-square deviation for the output characteristic (specific wear rate). As mentioned earlier, there are three categories of quality characteristics, i.e. lower-the-better, higher-the-better, and nominal-the-better. To obtain optimal performance, lower-the-better characteristic for wear rate must be taken. Furthermore, a statistical analysis of variance (ANOVA) is performed to identify the process parameters that are statistically significant. With the S/N and ANOVA analyses, the optimal combination of the process parameters can be predicted to a useful level of accuracy. Finally, a confirmation experiment is conducted to verify the optimal process parameters obtained from the parameter design.

Table 3.5 Level in taguchi

LEVEL	FILLER(A) wt%	NO. OF CYCLE(B)	LOAD(C) N
1	0	1000	40
2	3	2000	50
3	6	3000	60
4	9	4000	70

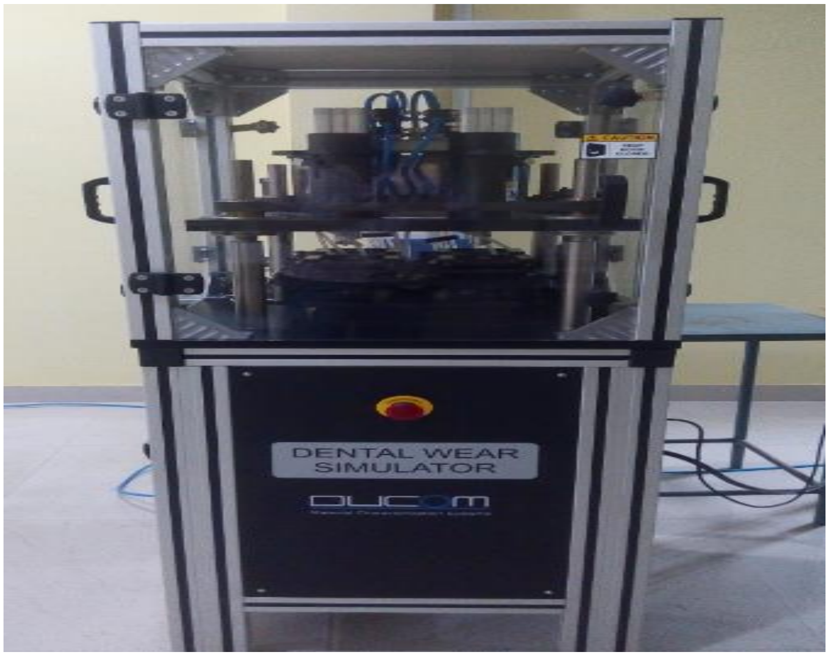

Fig 3.5 DENTAL WEAR SIMULATOR

Chapter summary

This chapter has provided:

- The descriptions of materials used in the experiments
- The details of fabrication and characterization of the composites
- The description of wear test
- An explanation of the Taguchi experimental design.

The next chapter presents the physical and mechanical properties of the dental composites under study.

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CHAPTER-4

Physical, Thermal And Mechanical Characterization Of The Composites

This chapter presents the physical and mechanical properties of the nanoparticles mixed GIC Composites prepared for this work. The interpretation of the results and the comparison among various composite samples are also presented.

4.1 Micro-hardness Testing

The variation of composite micro-hardness with the weight fraction of nanoparticles is shown in the chart below. It is thus seen that with the increase in fiber content in the composite, the hardness improves although the increment is marginal.

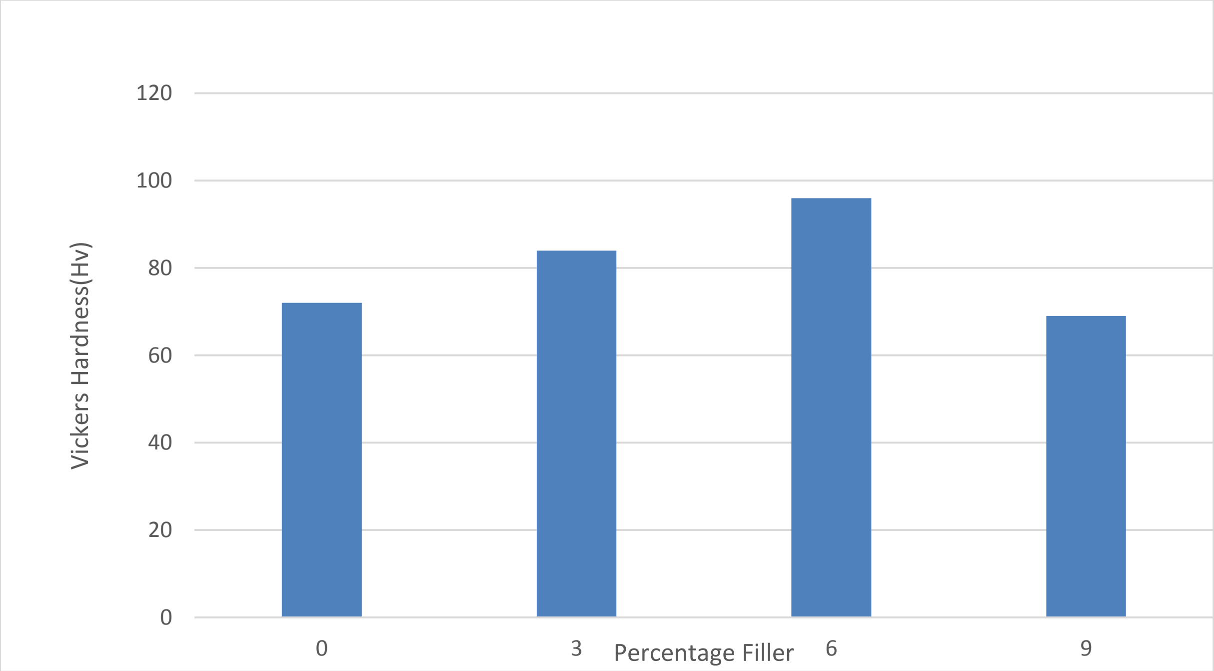

Fig. 4.1.1 Micro-hardness of TiO_2 incorporated filler

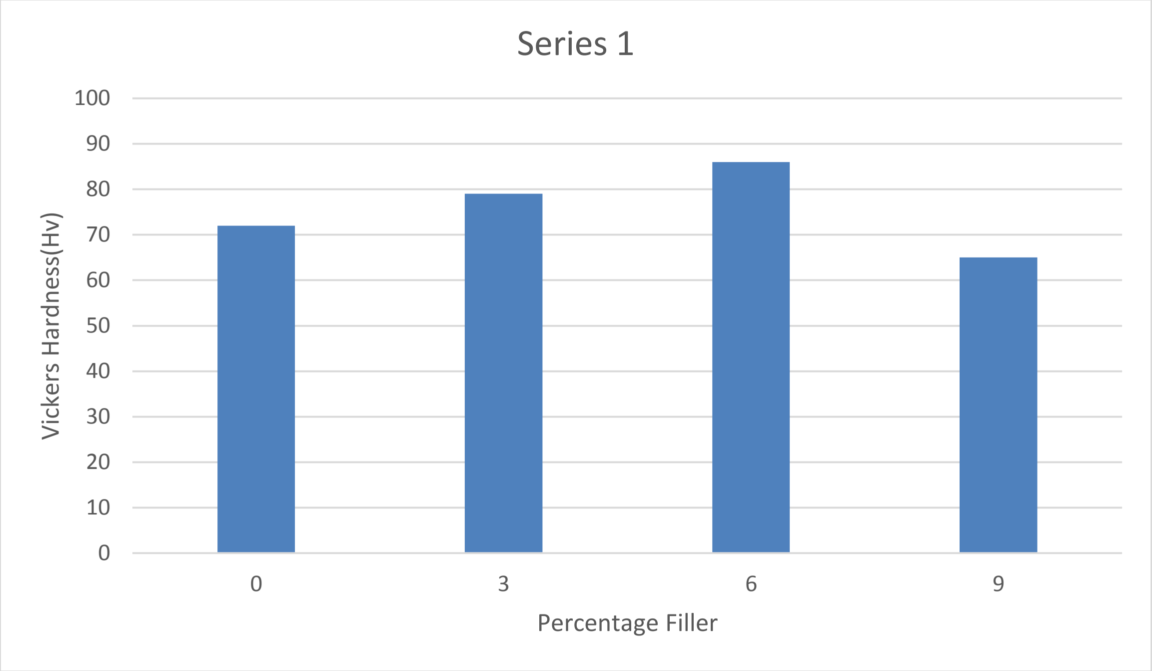

Fig. 4.1.2 Micro-harness of SiO_2incorparated filler

Previous studies show that increasing the filler size and content improves the mechanical properties of the material. From the above result it is found that the hardness of composite increases with the increase in percentage of nanoparticles. The dental composite having 6wt% of filler content shows the highest value of micro-hardness. For the composite containing 6% TiO_2 value of hardness is 96Hv which is the highest and after that sample having 6% SiO_2 has the highest value of 86Hv. But from the above trend it is seen that after a certain amount of filler content the value of hardness starts decreasing which shows that there is a limit upto which we can add the nanoparticles. This can be due to a reason that when the filler content increases the binding forces between the ionomer cement and the polyalkenoic acid started decreasing and thus the strength of the composites started decreasing due to less intermolecular bonding. Khaled et al. [28] reported that the cement reinforced with 1 wt.% functionalized TiO2 nano tubes demonstrated considerably superior hardness values, about 73% higher than that of control cement. Therefore, the increase in the powder concentration from 5 to 10 wt.% would not only expose no positive contribution to generate improved mechanical properties, but also has pose detrimental effects to degrade mechanical properties of the specimens.

4.2 FTIR (FOURIER TRANSFORM INFRARED SPECTROSCOPY)

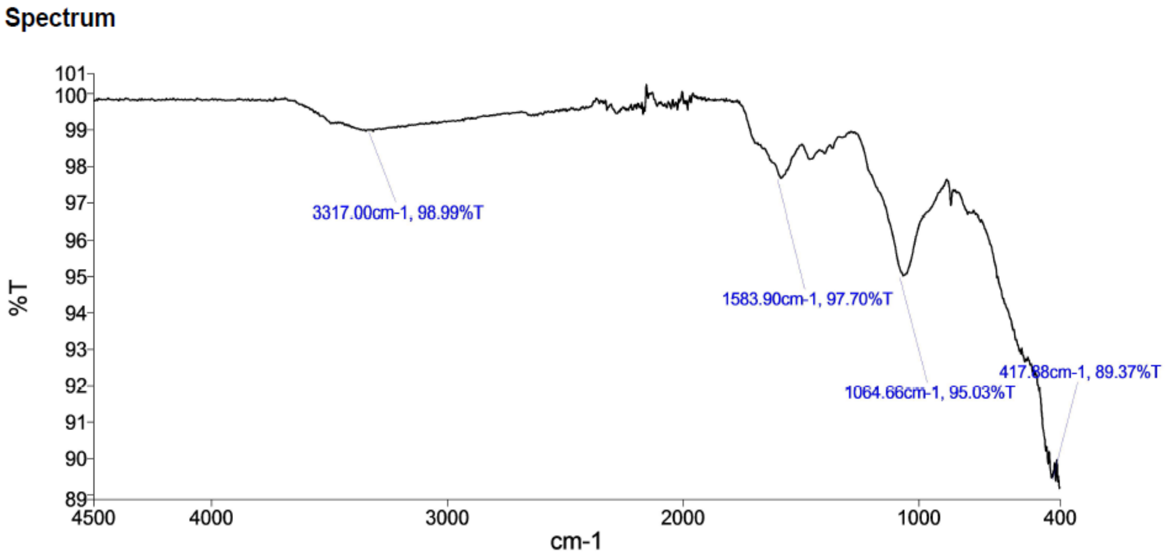

Fig. 4.2.1 FTIR for 0%filler

The highest transmittance is, for 0% which seemed to suggest that it was the most chemically stable material. At the wave numbers from about 900 to 1100 cm^{-1}, the graph of composite showed a drop (dip) in transmittance. The drop in that region represents a decrease in the PN group bonding. The FTIR results for all specimens showed a decreased transmittance at different wave numbers as the times increased. The characteristic apatite bands at wave number 860 cm−1 and 430 cm−1corresponding to structural OH− and $PO^4{}_3$ respectively were clearly seen in GIC composites [42].Wilson et al says that the polyacrylate aliphatic C=C, carbonyl C=O, P–H and C–H stretching bands were observed in composites. Decreased absorbance pattern of amorphous Si band at 1000 cm−1 was observed suggesting the shift towards semi crystalline nature and molecular interaction of calcium with alumino silicate. FTIR results are consistent with, showing a small amount of carbonate and no other impurities. The two large peak groups at 1580 and 1080 cm-1 are mainly due to PO3-4 with a contribution from OH- at 633 cm-1.

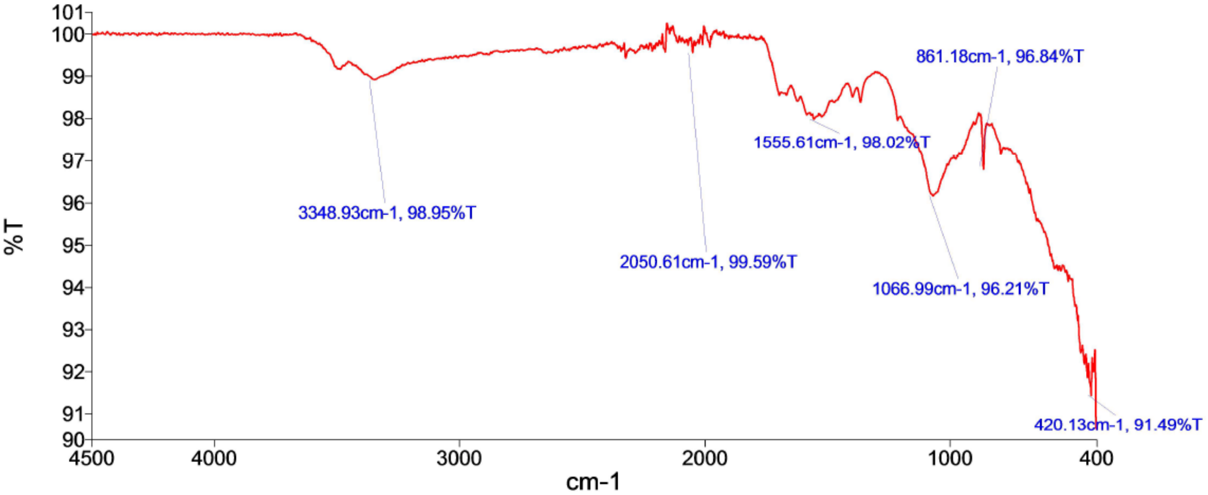

Fig. 4.2.2 FTIR for 3%filler

The highest transmittance were observed 98.95% at a wave no. 3348cm-1 which less than that of owt% filler. This means unstability of the composite have started increasing. Lee et al. reported methacrylate skeletal vibrations at wave number 1060–860 cm-1 and –CH3 alkane, C–H asymmetrical deformation vibrations (2050 cm-1); a very strong peak characteristic of the aliphatic CaC moiety (1555 cm-1) and carbonyl CaO (1730 cm-1) occurred.

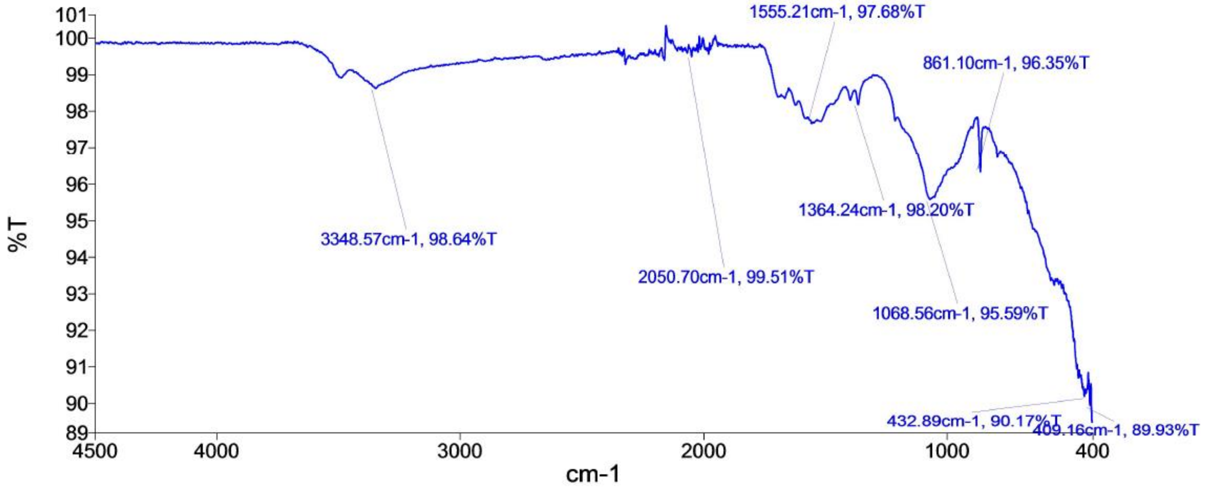

Fig. 4.2.3 FTIR for 6% filler

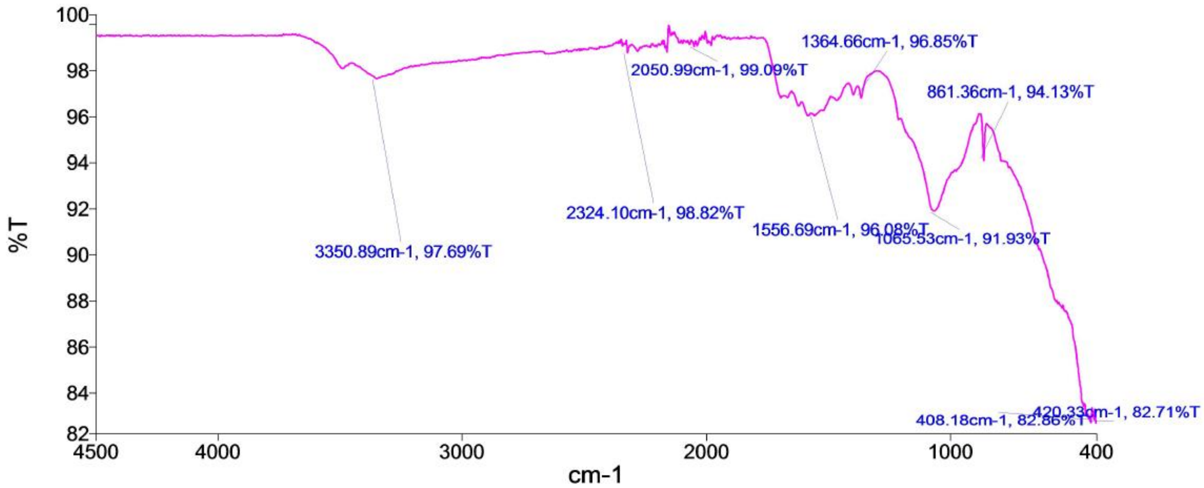

Fig. 4.2.4 FTIR for 9% filler

The result of FTIR spectroscopy is almost same for the different filler percent there is slight shift between the values. The FTIR examinations showed that all of the materials examined showed spectral changes and were not chemically stable. All of the materials showed chemical changes in the whole spectra with a decrease in transmission. At the wave numbers from about 900 to 1000 cm, the graph of composite showed a drop (dip) in transmittance. The drop in that region represents a decrease in the PN group bonding. The FTIR results for all specimens showed a decreased transmittance at different wave numbers as the times increased. The highest transmittance is, for 0% which seemed to suggest that it was the most chemically stable material.

The characteristic apatite bands at wave number 860 cm−1 and 430 cm−1corresponding to structural OH− and $PO^4{}_3$ respectively were clearly seen in GIC composites [42].Wilson et al says that the polyacrylate aliphatic C=C, carbonyl C=O, P–H and C–H stretching bands were observed in composites. Decreased absorbance pattern of amorphous Si band at 1000 cm−1 was observed suggesting the shift towards semi crystalline nature and molecular interaction of calcium with alumino silicate. The P–NH2 and Si–O bands showed a similar pattern to that of amorphous Si in composite cements Nicolsen et al [54]. FTIR results are consistent with, showing a small amount of carbonate and no other impurities. The two large peak groups at 1580 and 1080 cm-1 are mainly due to PO3-4 with a contribution from OH- at 633 cm-1 in Fig 4.4.1 that reduces intensity and shifts to higher wavelengths with increasing F substitution. These groups, along with the OH peak at 3348 and smaller phosphate peaks at 471 and 2000cm-1 constitute the characteristic spectrum of HA [55, 56]. The peaks between 640 and 745 cm-1 in fluoride containing samples are attributed to the effect of OH-F interactions on the OH bond [28]. The broad peaks at 1640 and 3450 cm-1 can be attributed to absorbed or combined H2O [39] and small carbonate peaks are evident at 1400-1550 cm-1 and 870 cm-1 [39, 41, 59]

4.3 TGA (Thermo-gravimetric Analysis)

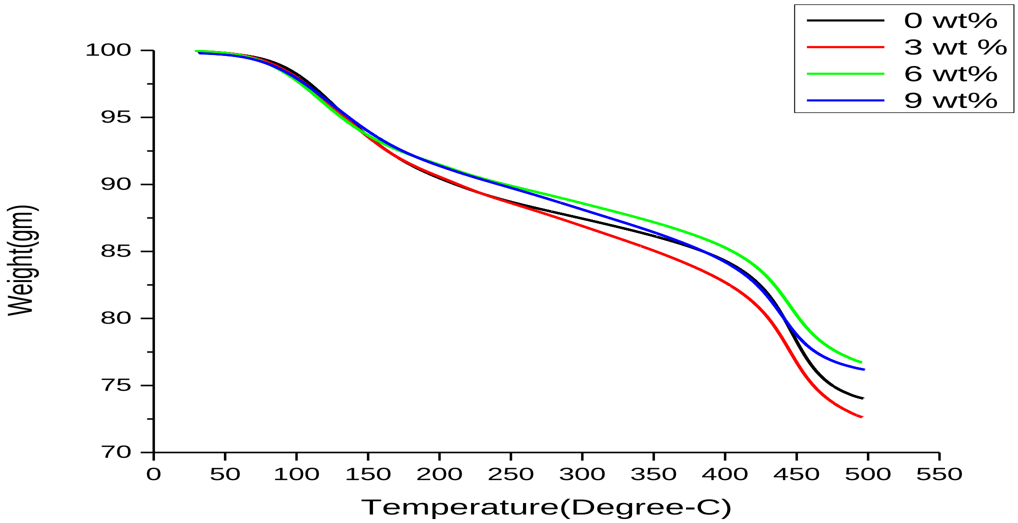

Fig. 4.3 Weight % vs. Temperature

Thermal analysis is an important technique in the characterization of polymeric materials. During the fabrication of new products from polymers, knowledge of the thermal stability of their components is essential. Thermo gravimetric data provide the different stages of thermal breakdown, weight of the materials in each stage. The degradation studies of materials are usually intended to provide a data base for industrial practices in order to ascertain the working conditions that could prevent the degradation of the materials. TGA was done in an inert atmosphere of nitrogen simulating a pyrolysis process, due to the high volatile matter content of these materials. Weight loss was studied under different temperatures by TGA. Perkin Elmer STA 600 analyzer was used to measure and record mass and temperature changes versus temperature over a simulated pyrolysis process. The system was connected to a personal computer for data recording and analysis. TGA was done at the heating rate (5 ˚C/min) from 30˚ to 500˚ C. Nitrogen was used as purge gas to keep an inert atmosphere based on nitrogen in the pyrolysis zone (Li et al., 2005), cutting down unwanted oxidation conditions of the sample. Samples were placed in a platinum pan avoiding the contact with both sides of the oven. The purge gas utilized was 99.995% pure nitrogen, and the total purge gas flow rate was set at 140 ml/min, which was found to be suitable for transferring acceptable quantities of the species generated from the condensed phase to the diagnostic systems. Previously to TGA, temperature, weight and platform calibrations were carried out. Nevertheless, samples were air dried and blended in order to achieve homogeneity, i.e. samples of around 40 mg with particles sizes ranging between 0.1 and 0.2 mm were placed in the analyzer for each experimental run. Fig. 4.3 shows TGA curves for the different %composition of nanoparticles. The plot shows that the thermal decomposition occurred in two steps. TGA plot confirms that only there is 5 % weight loss in the temperature range of 30 °C to 150 °C. The weight loss in this temperature range is attributed to the elimination of moisture and bound water gradually from composite materials. The second phase weight loss with highest decomposition rate is associated with scission of weak bonds and unzipping of highly strained cross links resulting in the formation of straight chains. The onset of decomposition is around 150 °C, and the second stage of degradation temperature range is 150˚C - 450 °C. The weight loss % in this temperature range is minimum for 6 % filled composite while it is highest for 9% filled composite. But for neat composite sample it comes to be slightly lower in value than the 3 % filled composite sample. Weight loss is less for 6 wt% TiO_2 filled dental composite compared to the unfilled sample. This may be due to the fact that filler is preventing the movement of molecular chains of resin at higher temperature, thus minimizing the weakening of the interface between filler and matrix. The first step corresponds to materials, whose thermal degradation began at about 100˚ C and was essentially completed by approximately 250˚ C, presenting a peak at 200˚ C. The second step may correspond to a mixture of filler materials, whose thermal degradation started at 300˚ C and ended at 500˚ C, showing a peak at 400˚ C. The residue obtained was of 20% of the sample, which may consist of ash and unburned material.

4.4 DMA (Dynamic mechanical analysis)

Visco-elastic materials like polymers and their composites, mutually stores part of the energy as potential energy (represented by storage modulus or elastic component) while other part might be dissipated as heat energy within the material (represented by loss modulus or viscous component that theoretically correspondence to the toughness of the composites) when dynamically deformed during test . Such material at any dynamic conditions of load/temperature, possess both elastic and viscous components. At lower temperature regime, owing to limited molecular mobility the material has stable mechanical behavior i.e. molecular bond strength and adhesion (between resin/fiber) is high and satisfactory hence causing higher elastic response or storage modulus and low loss modulus. As the temperature approaches to elevated regime phase transformation form rigid state to rubbery state take place due to segmental slippage thereby enhancing the viscous component in comparison to storage modulus. The steeply decrease in E' (Storage modulus) and E" (loss modulus) may be because of softening of organic matter at higher temperature. The ratio of loss modulus to storage modulus is termed as loss-tangent (Tan δ) factor; it is dimensionless and represents mechanical damping property/capacity of the material. Dynamic Mechanical Analysis (DMA) was conducted in a nitrogen atmosphere at a fixed frequency of 1 Hz, heating rate of 5˚C/min, a temperature range of 20˚C to 200˚C and a strain of 1% on cylindrical samples with approximate dimensions of 7mm length and 4mm diameter using a Perkin Elmer dynamic mechanical analyzer, DMA 8000compression test. For the composites, dynamic mechanical properties such as storage modulus (E'), loss modulus (E'') and loss tangent (Tan δ) are shows in Figure 4.4.1 Figure 4.4.3 demonstrates the variation of storage modulus (E') against temperature and it is observed that storage modulus (E') remained negligibly affected in the temperature range of 20˚C–60˚C. On further increasing the temperature, the E' suffered a sharp decay specifically in the temperature range of 60˚C–120˚Cfollowed by eventual thermo-mechanical failure of the composites above a temperature of120˚C.Quantitatively the storage modulus (E') of 9wt% filler composite remained at 1100 MPa, whereas that of 3wt% and 6wt% composites showed a magnitude well above and below at 1100 MPa and 900 MPa, respectively, in the temperature range of 20˚C– 60˚C before any decay.

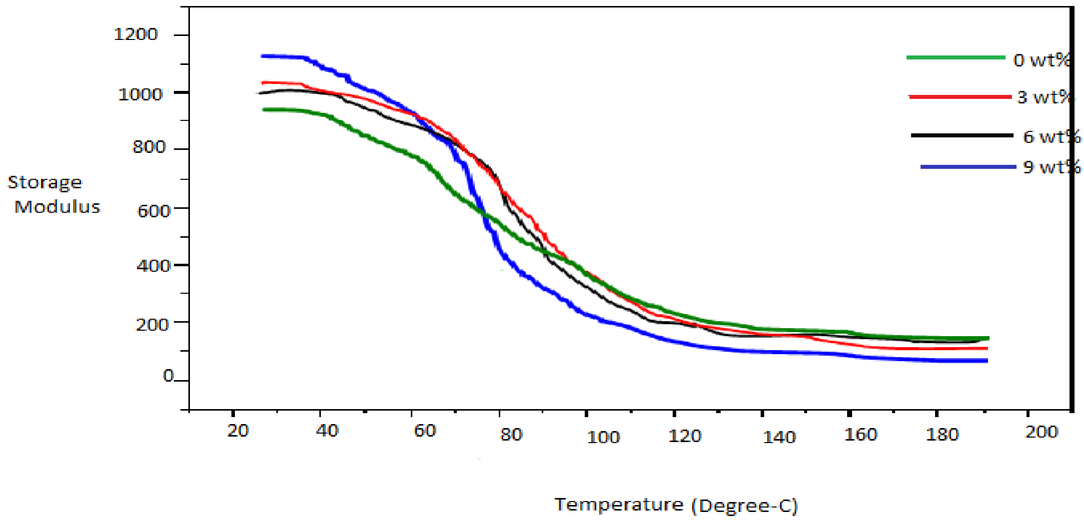

Fig. 4.4.1 Storage Modulus(E') vs Temperature

However, the mode of E' decay has found to be very similar in 3% and 6% composites. Whereas, the nature of modulus decay in 9% composite has beenfound to be partially inhibited with reference to the effect of temperature, i.e., the E' of composite 9% has been observed to be

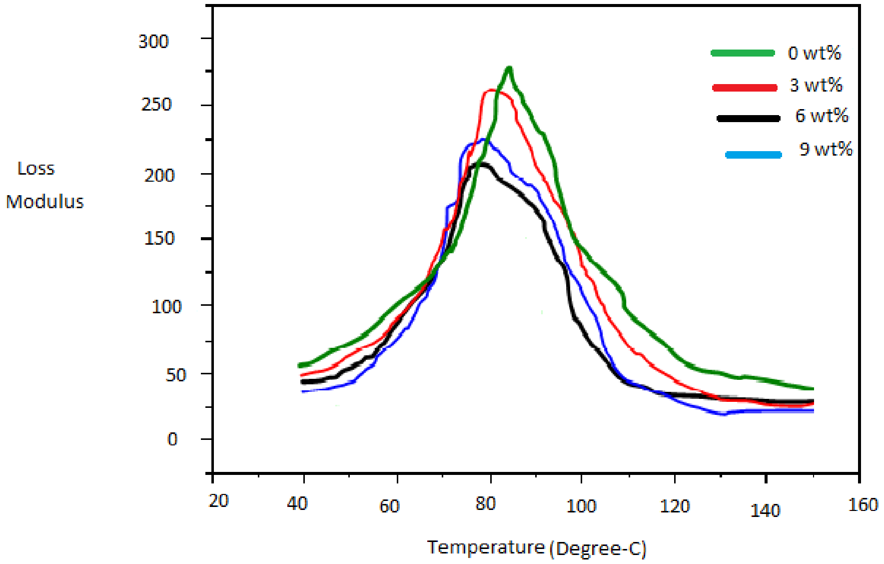

Fig 4.4.2 Loss Modulus(E'') vs Temperature

The peak of the loss modulus curve denotes the glass transition temperature (Tg). Figure denotes that the glass transition temperature increases slightly up to 6 % of the filler content while 9 % composite samples show minimum value of (Tg) equal to 80 °C. The value of Tg is found to be maximum for the 6 % filler content and it is equal to 90 °C.

The loss modulus (E’’) of composites is observed to decrease along withthe addition of filler content as shown in Figure 4.4.2.The addition of filler content (3 wt.% to 9 wt.%) shows increasing effect on the peak temperature of loss modulus (E’’), which indicates that the addition of filler content affect the peak temperature of composites significantly.It is observed that the peak temperature corresponding to maximum value of loss modulus (E’’)shifted uniformly in a decreasing order of 3% > 6% > 9% .Theoretically, the temperature corresponding to the maximum value of E’’ offers a tentative idea regarding the primary transition temperature of the composite.

Figure 4.4.3 presents the tan δ (damping factor) curves of the particulate filled composites. It is found that the 0% and 3% composites showed a slightly higher damping than 6% and 9% composite, owing to the visco-elastic energy dissipated due matrix material and filler friction. The 0%composites exhibit a damping peak at values close to 90˚C, whereas the molecular mechanism of this relaxation in 9% composite is quite different.

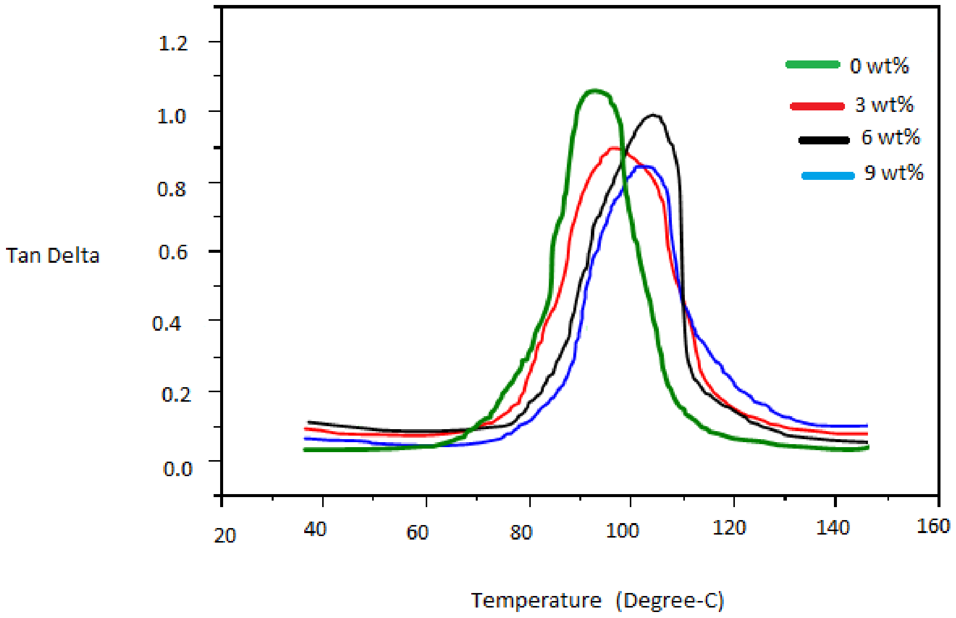

Fig. 4.4.3 Tan Delta vs. Temperature

From Figure 4.4.3 is also obvious that the addition of particulates lowers the peak position of tan δ. Contrary to the observations from Figure 4.4.2 showing E’’ versus temperature, the primary transition temperatures of the composites, as observed from tan δ values [Figure4.4.3], have been observed to undergo a nominal shift to higher temperatures with the increase in particulate wt% in the composites. This inevitably indicates that the composites tend to be less damping resistant with the increase in the filler content.

Chapter summary

This chapter summarizes that:

- Successful fabrication of dental composites with reinforcement of conventional fillers like: TiO_2 and SiO_2 is possible.
- Incorporation of these fillers modifies the hardness and modulus properties of composite.

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CHAPTER-5

Study on Wear Characteristics of Dental Composite

Wear is a common phenomenon that occurs when two or more surfaces subjected to slip or slide movements when a load is applied. Six mechanisms of abrasive wear, adhesive, fatigue, erosive, corrosive and wear due to abrasion. Abrasive wear occurs when a hard surface slides against a smoother surface and results in the loss of the structure of the smoother surface. When an Adhesive wear roughness on a cold surface is of welded to the other surface as a result of friction and leads to the movement of material from one surface to another. Fatigue wear occurs as a result of subsurface crack propagation under dynamic loading. Erosive wear occurs when a material loses particles in an erosive environment pressure. Corrosive wear is defined as the loss of the corrosion products of a material as it skims the surface opposite. Finally, fretting wear occurs when the sliding surfaces each other slowly and for long periods of time under load. Wear Resistance is an important property for all restorative dental materials. That demonstrates the ability of the material to resist the milling and force that is applied from an opposing tooth, at the same time that maintains its restore form and function. Several wear simulation and testing machines have been used in this field. One of the most used machines is wear simulator on dental wear simulator machine. Exposure of GICS several unique properties, however, low resistance to wear makes them suitable to be used in areas of high tension. Efforts have been made to improve the material and increase their mechanical properties. Several methods have been used in an attempt to improve the properties. There is no evidence as to whether the implementation of a nano-fill glass ionomer cement, improve the wear resistance and hardness of high resistance. The main objective of this study was to measure the wear resistance of a glass ionomer high resistance dental composites nano-filling. The different parameters established during the load test, profile size, shape, pattern, and the cycles. Two body wear is calculated on the basis of the amount of mass loss in different conditions and cycles of artificial saliva.

5.1 Steady State Wear Rate of the Composite

After considering all the aspects which the wear and the experiments presented in the introduction, the selected was proof of wear of reciprocity, with contact geometry plane sphere. This type of evidence has been selected first of all because of the similarity with the natural movement of the mouth, and secondly also because it allows variations in the amplitude of movement, normal load and test environment. The ability to assess the wear of materials in contact is also an important advantage of this type of evidence Wear test had been conducted on dental wear simulator. The reciprocating test was performed to determine the wear resistance of the chosen composite material and to evaluate the mass loss. This technique comprises a sliding contact of a spherical body in reciprocating motion against a flat specimen of composite material. During the chewing process of human beings, the magnitude of masticatory force in the oral cavity ranges from 3 to 36N. In first one the load has been varied keeping the number of cycles constant and the amount of mass loss after the cycle had been completed is calculated. The graph have been plotted between the load and mass loss. The samples were weighed before starting the test and then mounted on the machine. Test were conducted under the artificial saliva four samples at a time. The load was varied from 40N to 70N and keeping the cycle constant (2000). The mass loss for various filler concentration under different load conditions had been calculated by subtracting the mass of the sample after the completion of test from mass before the test.

5.1.1 Steady state wear rate under constant cycles

The table below shows the values of difference in weight after test performed and the graph had been plotted between mass loss and the load applied. As it seen from data that the wear rate of TiO_2 filled composite increases with the increase in load for a constant number of cycle. Jorgensen et al. suggested that decreasing the size of particles, space between them and increasing the volume fraction of filler, are the key factors to improve the wear resistance of materials. The authors have considered that filler particles situated very closely to each other can protect a softer resin matrix from abrasives, what results in the reduced wear of material.

Table 5.1.1 Mass loss for TiO2 filled composite when cycle constant

Composition Load	0wt%	3wt%	6wt%	9wt%
40N	0.029	0.018	0.0.11	0.023
50N	0.037	0.023	0.016	0.030
60N	0.43	0.031	0.022	0.037
70N	0.49	0.039	0.026	0.044

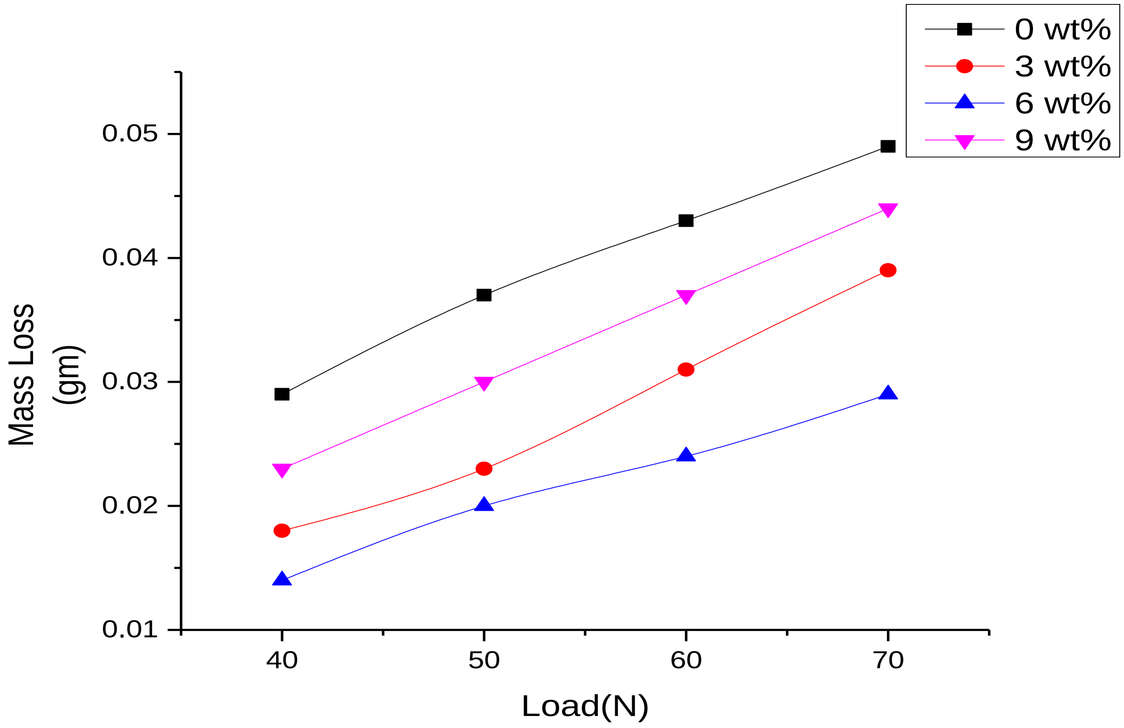

Fig. 5.1.1 Mass loss vs Load applied for TiO_2 under constant cycle

Nagarajan et al. reported that the wear volumes of the medium filled composites are nearly an order of magnitude smaller than that of the highly filled composite. The influence of the hardness in the wear resistance of the composites is much higher in the tests with saliva that relation agrees with Archard equation thus the hardness is more important to enhance wear resistance for contacts without abrasive particles. As it is seen from above data that the values of wear of the composite increase generally with the normal load under constant number of cycles (2000). Thus, different amounts of wear were generated as a function of the test conditions. The mean values of the mass loss for the four medium filled composites (0wt%, 3wt%, 6wt% and 9wt%) are shown in Fig. 5.1.1 as a function of load. Note an increase in the mass loss as the load is increased from 40N to 70 N.

Table 5.1.2 Mass loss for SiO_2

Composition Load	0wt%	3wt%	6wt%	9wt%
40N	0.029	0.019	0.0.12	0.024
50N	0.037	0.022	0.018	0.029
60N	0.43	0.025	0.023	0.037
70N	0.49	0.032	0.027	0.042

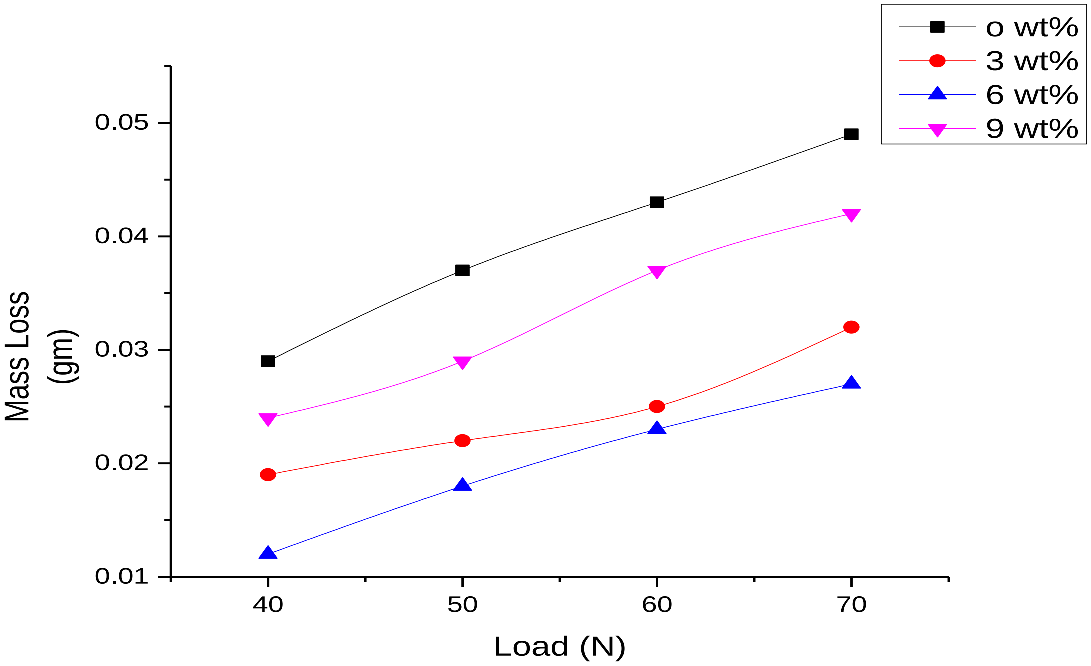

Fig. 5.1.2 Mass loss vs Load applied for SiO_2

The relationship between wear volume and the load is similar for the all four composites. The mass loss of the nanoparticles filled composites are nearly an order of magnitude smaller than that of the 0wt% filled composite. The differences in mass loss between each medium filled composite and the highly filled composite are statistically significant at all loads. The amount of mass loss of specimens having 3wt% and 6wt% filler concentration were significantly lower than the specimen having 0wt% and 9wt% composition. The composite having 6wt% filler content shows the least amount of mass loss during wear test. The value of mass loss decreases upto 6wt% but for the composite containing 9wt% filler again there is an increase in mass loss value. When we further increases filler content after 6wt% the mixture become less viscous and its setting time also increases. This can be due to a reason that filler started occupying more space between the mixture and reduces the binding forces between glass ionomer cement and liquid and hence reduces the bonding strength. From the above data we can say that the specimen having 6wt% SiO_2 filler shows the least amount of mass loss.

5.1.2 Steady state wear under constant load

Table 5.1.3 Mass loss for TiO_2 filled composite when load constant

Composition Cycle	0wt%	3wt%	6wt%	9wt%
1000	0.014	0.009	0.006	0.011
2000	0.029	0.018	0.014	0.023
3000	0.035	0.029	0.026	0.034
4000	0.044	0.037	0.034	0.042

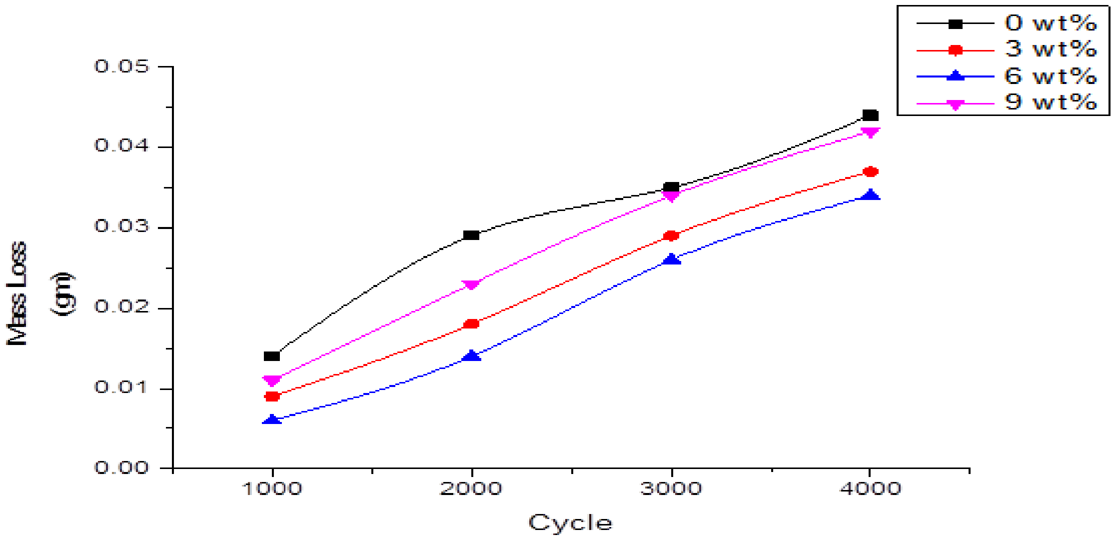

Fig. 5.1.3 Mass loss vs cycle for TiO_2 under constant load

As the number of cycle increases the wear rate also increases under constant loading condition. The composite containing 6wt% filler shows the minimum weight loss. But increasing the filler content after 6wt% the weight again started decreasing. Especially particle size distribution, composition homogeneity and the participation of the largest filler particles, dominate the tribological behaviour of composite materials [4].

Table 5.1.4 Mass loss for SiO_2 filled composite when load constant

Composition Cycle	0 wt%	3wt%	6wt%	9wt%
1000	0.014	0.010	0.008	0.012
2000	0.029	0.019	0.017	0.024
3000	0.035	0.030	0.029	0.037
400	0.044	0.042	0.039	0.049

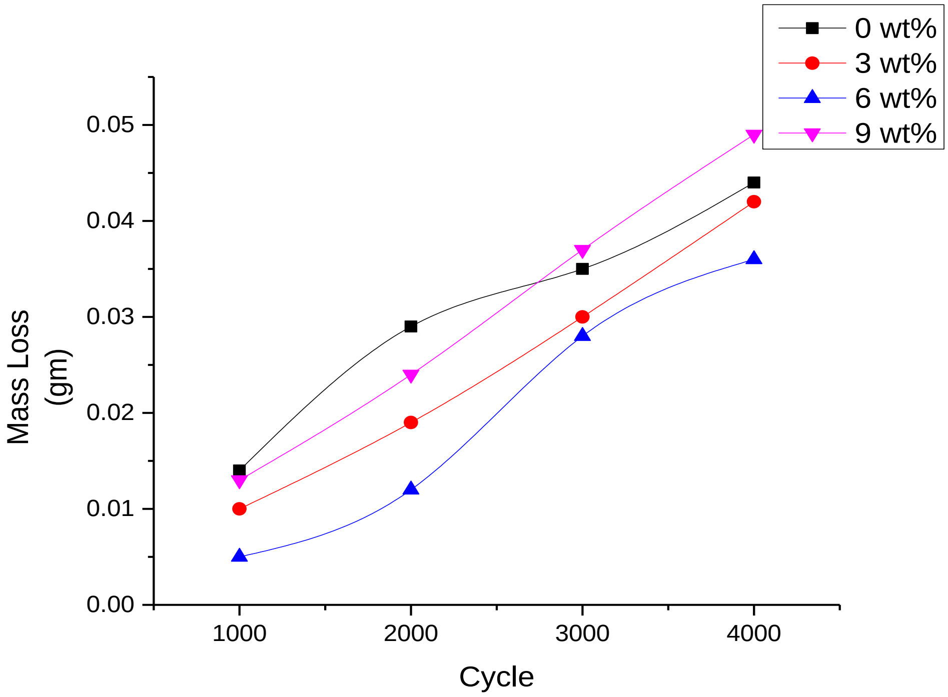

Fig 5.1.4 Mass loss vs cycle for SiO_2

5.2 Taguchi Analysis

The aim of this experiment is to determine the highest possible Signal-to-noise ratio for the parameters under study. A high value of S/N ratio implies that signal is much higher than random effects of noise factors. The wear rates of TiO_2/SiO_2 reinforced dental composites under various test conditions are given in Table 5.2.1. The experimental observations are transformed into a signal-to noise (S/N) ratios. In Table 5.2.1, the last column represents S/N ratio of the wear rate of TiO_2 filled composite. The overall mean for the S/N ratio of the wear rate is found to be 30.28634 dB. The analysis is made using the popular software specifically used for design of experiment applications known as MINITAB 17. In the main effect of control factors on wear rate plot if the line for a particular factor is near horizontal, then the factor has no significant effect. On the other hand, a factor for which the line has the highest inclination will have the most significant effect. The S/N ratio response is given in Fig 5.2.2 from which it can be concluded that among all the factors Applied load is the most significant factor followed by filler content, and while the number of cycles has the least or negligible significance on wear rate of these TiO_2 and SiO_2 filled dental composites. Effect of individual control factors for signal to noise ratio shown in Fig. 4 also leads to the conclusion that factor combination of A3, B1 and C1 gives minimum wear rate.

Table 5.2.1 S/N ratio for TiO_2 filled dental composite

S.No	Filler contents wt% (A)	No of cycle (B)	Load(N) C	Wear rate(gm/gm)	S/N Ratio(db)
1	0	1000	40	0.014	37.0774
2	0	2000	50	0.037	28.6360
3	0	3000	60	0.052	25.6799
4	0	4000	70	0.060	24.4370
5	3	2000	40	0.018	34.8945
6	3	1000	50	0.019	34.4249
7	3	4000	60	0.043	27.3306
8	3	3000	70	0.049	26.1961
9	6	3000	40	0.026	31.7005
10	6	4000	50	0.042	27.5350
11	6	1000	60	0.011	39.1721
12	6	2000	70	0.029	30.7520
13	9	4000	40	0.042	27.5350
14	9	3000	50	0.039	28.1787
15	9	2000	60	0.037	28.6360
16	9	1000	70	0.024	32.3958

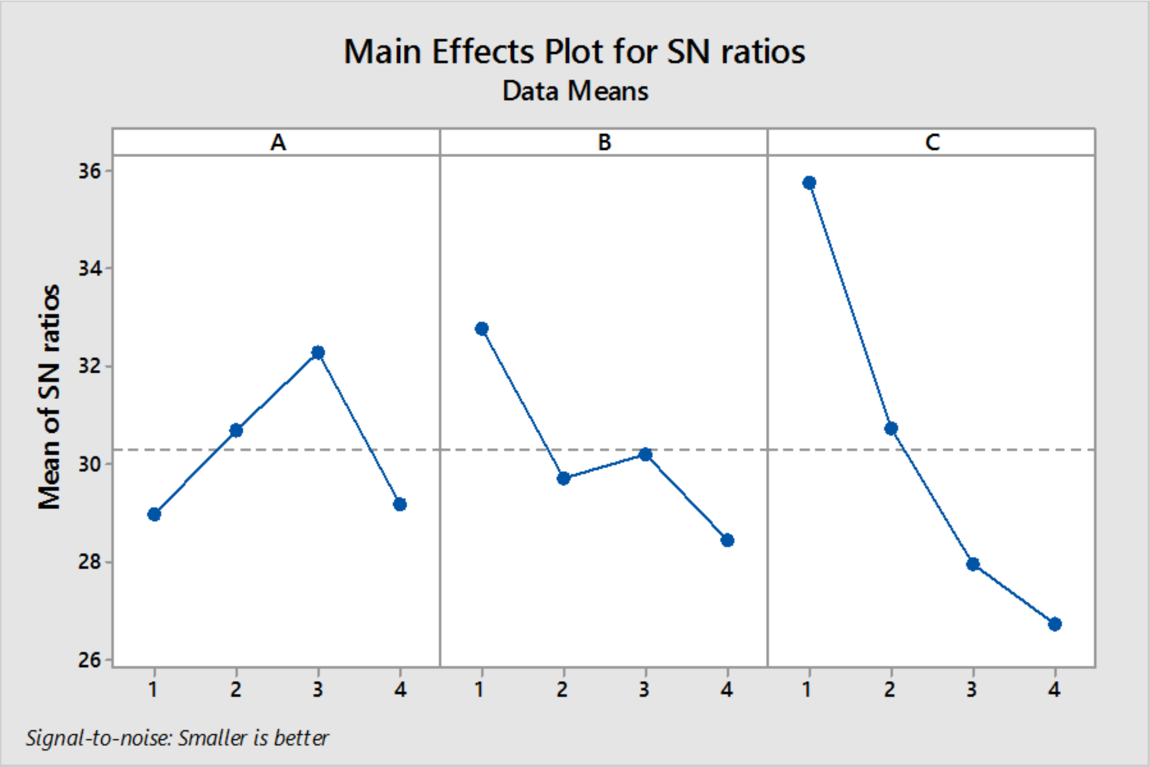

Fig. 5.2.1 Effect of control factor on wear of TiO_2 mixed composite

Figure 5.2.1 clearly shows that weight loss is highly sensitive to load (factor C). Concrete visualization of impact of various factors and their interactions can be easily made with the help analysis of means as shown in the response table (Table 5.2.1). The last row indicates the order of significance of factors and their interactions. It can be observed that the load, factor C (Rank 1), filler content, factor A (rank 2) and no. of cycle, factor B (rank 3) have great influence on weight loss. Before setting the optimal levels of factors for minimum weight loss, the interaction effects of factors need to be analyzed. As such, the interactions do not have much influence in comparison to factors as evident from the response table. For the composite having SiO_2 filler somehow similar results were obtain from the taguchi analysis shown in fig 5.2.2. The overall mean for the S/N ratio of the wear rate of SiO_2 is found to be 32.15606 dB. Here also the load was most affecting factor leading filler content and last one number of cycles. So from the data analyzed from taguchi we can say that the composite containing TiO_2 filler is better than the composite having SiO_2 filler and 6% is the better amount for obtaining good mechanical properties and wear result.

Table 5.2.2 S/N ratio for SiO_2 filled dental composite

S.No	Filler contents wt% (A)	No of cycle (B)	Load(N) C	Wear rate(gm/gm)	S/N Ratio(db)
1	0	1000	40	0.014	37.0774
2	0	2000	50	0.037	28.6360
3	0	3000	60	0.052	25.6799
4	0	4000	70	0.060	24.4370
5	3	2000	40	0.019	34.4249
6	3	1000	50	0.020	33.9794
7	3	4000	60	0.062	24.1522
8	3	3000	70	0.042	27.5350
9	6	3000	40	0.020	33.9794
10	6	4000	50	0.044	27.1309
11	6	1000	60	0.013	37.7211
12	6	2000	70	0.027	31.3727
13	9	4000	40	0.049	26.1961
14	9	3000	50	0.047	26.5580
15	9	2000	60	0.036	28.8739
16	9	1000	70	0.034	29.3704

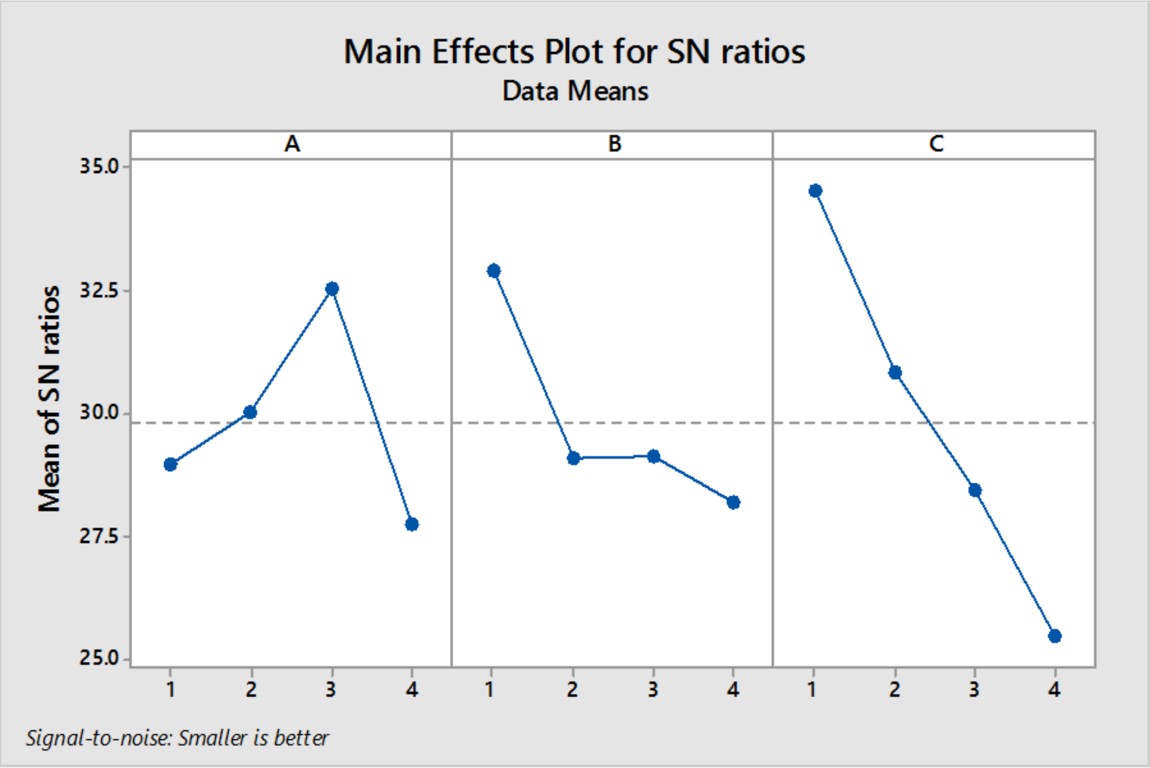

Fig. 5.2.2 Effect of control factors on SiO_2 Filled composite

The S/N ratio response is given in Fig 5.2.2 from which it can be concluded that among all the factors Applied load is the most significant factor followed by filler content, and while the number of cycles has the least or negligible significance on wear rate of these SiO_2 filled dental composites. Effect of individual control factors for signal to noise ratio shown in Fig. 5.2.2 also leads to the conclusion that factor combination of A3, B1 and C1 gives minimum wear rate. This conclude that the load of 40N,6wt% filler content and 1000cycles are good for working condition. The overall mean for the S/N ratio of the wear rate of SiO_2 is found to be 32.15606 dB. Here also the load was most affecting factor leading filler content and last one number of cycles. So from the data analyzed from taguchi we can say that the composite containing TiO_2 filler is better than the composite having SiO_2 filler and 6% is the better amount for obtaining good mechanical properties and wear result.

Chapter summary:

Based on the research presented in this chapter the following conclusions are drawn:

a. Nanoparticulate filled dental composites consisting of glass ionomer cement filled with particulate fillers such as TiO_2 and SiO_2 possess fairly good potential for application in wear environment.
b. Wear characteristics of these composites can be successfully analyzed using Taguchi experimental design scheme.

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CHAPTER-6
CONCLUSION AND FUTURE SCOPE

The addition of 9%wt. of SiO2 and TiO2 concentration has not been observed to result in significant variation in the value of micro-hardness and compressive strength. While increasing the amount of TiO2 and SiO2 nanoparticles from 0 to 6 wt.% has led to a moderate increment of hardness value than the specimens containing 9 wt.% filler content. TiO2 have demonstrated more highlighted enhancement in the value of micro-hardness as well as a higher storage modulus values compared to the others. Likewise, one can hardly find any remarkable improvement in micro-hardness and wear rate of GIC samples, resulted from the addition of TiO2 fraction from 0 to 6 wt.% though, these parameters display an obvious rise in 9 wt.% TiO2 and SiO2 glass ionomer cement specimens. Reduction in strength of the cement caused by the addition of extra nanoparticles indicates that there is a noticeable interference of the nanoparticles with the normal glass-ionomer reaction. In contrast, the increasing trend for the values of the above mechanical properties was not observed to be sustained trough increasing the amount of TiO2 and SiO2 powder to 9wt.%, The low hardness and wear strengths values of 9% titania and silica samples are due to the poor interfacial bonding between nanoparticles and the matrix. In other words, because of the very small particle size and large surface area of the nanoparticles; there may be inadequate ionomer to hold the large amount of nano particles (9 wt.%) effectively. Though, as titania and silica nano particle is the harder phase compared to GIC's powder and polymer, increasing nanoparticles to 9wt.% has led to the enhancement of microhardness values. Therefore, the increase in the powder concentration from 6 to 9 wt.% would not only expose no positive contribution to generate improved mechanical properties, but also has pose detrimental effects to degrade mechanical properties of the specimens. As a result, the optimum as well as the maximum amount for the introduction of TiO2 and SiO2 nano powders were determined to be 6wt.% concentration. The TiO2 -GIC composites demonstrated a fairly higher microhardness values than SiO2-GIC composites, restricting from the attributes presented by titania for deriving higher microhardness as well as mechanical strength as a harder phase compared to silica particles. Furthermore, significant differences in mechanical properties of SiO2/TiO2-GICs can be observed, and the composites fabricated by both SiO2 and TiO2 nano powders have displayed the highest mechanical strength as well as microhardness values. The microhardness of the TiO2-GICs is about 50% higher than that of commercially available glass ionomer cement. In addition, the wear of TiO2-GICs are nearby 44% less than those of GIC respectively. Similarly, it seems that good interfacial bonding as well as crosslinking between TiO2 nano particles and the cement matrix, resulted from the formation of titania salt bridges among trivalent aluminum ions in the glass as well as titanium ions from the nano powders, provides the final strength of TiO2-GICs. Thereby, this may present another reason for the increase of mechanical properties in GICs as a result of applying TiO2 nano powders.

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