ORIGINAL RESEARCH

Thermogravimetric Characterization of the Microstructure Composition of Polyamide Injection Molded Denture Base Material *vs* Conventional Compression Molded Heat-cured Denture Base Material

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ABSTRACT

Background: Thermoplastic resin polymers are widely used in medicine due to their biostability and hypoallergenic properties, making them a possible alternative to poly-methylmethacrylate (PMMA). The current research examined the microstructure of a rapid injection molding system thermoplastic resin for construction of flexible denture compared with that of heat-cured PMMA.

Materials and methods: A total of 40 disk-shaped specimens (25 mm in diameter and 3 mm in thickness) were prepared and divided into two groups of 20 disks each (group I samples were of thermoplastic acrylic resin while group II was heat-cured PMMA resin).

Results: In group I, thermogravimetric analyzer showed that increasing the temperature up to 169°C resulted in about 1.3% of the material loss, and after that the material remains thermally stable up to 200°C. Group II showed 2.24% weight loss at 171°C, and further weight loss (12.025%) was observed on heating to 230°C. Fourier transform infrared (FTIR) spectrophotometer analysis in the range of 400–4000 cm^{-1} detected the presence of an amine group (N-H) in group I samples and the presence of methylene group attached to inorganic Si as reinforcement filler (Si-CH$_3$).

Conclusion: Thermoplastic resin displayed excellent thermal stability and the absence of residual monomer within the polymerized material, suggesting its suitability for the fabrication dentures.

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INTRODUCTION

Traditionally and for decades, poly-methylmethacrylate (PMMA) has been used as a denture base material due to its many favorable properties including optimum esthetics, low water sorption, and ease of repair.[1] However, PMMA has several limitations. Several documented cases of patients and technicians have hypersensitivity to the material as well as low impact strength that results in fracture of acrylic denture base. Both limitations are attributed to the polymerization process of the material due to residual monomer which is present as bubbles within the material. This results in an allergic reaction and weakens the mechanical strength. This ultimately results in reduced dimensional and color stability as well as water sorption.[2]

Thermoplastic resin polymers are commonly used as biomaterial with a wide range of medical applications including knee, hip, and joint replacement as well as the fabrication of parts and coating for heart pacemakers. The polymerization and processing technologies for these have undergone many advances in the past three decades. Today, thermoplastic monomers are easily prepared by injection molding.[3-5] The most favorable property of thermoplastic resins, such as polyamides (nylons) is their biostability to degradation, whether its oxidative,

hydrolytic, and/or enzymatic degradation. Additionally, these materials are nontoxic and hypoallergenic. As well as being thermally stable, they offer superior physical properties in terms of hardness, wear resistance, and overall physical strength (impact, tensile, and flexural strength). Furthermore, polyamides are characterized with good dimensional stability and optimum modulus of elasticity as well as structural integrity.[6]

Recently polyamides (nylons) were introduced as an alternative to PMMA due to their flexible nature, which offers many advantages including simplifying the design of the removable partial denture (RPD) since the flexible nylon resin will provide stress breaking.[1,7]

Flexible denture base polyamides undergo many modifications to overcome undesirable properties, such as warpage and water sorption as well as difficulty in polishing rough surfaces. Denture base polyamide is a low allergen material with high physical strength, improved water sorption, improved heat and chemical resistance, optimal esthetics, and high flexibility. Its ability to adapt to the constant movement of the removable prosthesis increases patient's comfort when denture is used. This material could be used in the fabrication of performed clasps, metal-free RPD, temporary crowns as well as fixed partial dentures, occlusal splints, implant abutments, orthodontic appliances, among others.[8-11]

Thermoplastic resin used for the construction of flexible denture is a rapid injection molding system that offers some advantages over the traditional compression molded methylmethacrylate (MMA) denture base material. It produces minimal changes in vertical dimension, reduced processing error, improved dimensional stability, better control of polymerization shrinkage, and increased resin density.[12,13] Polyamides are plasticized by thermal processing with no chemical reactions.[1] Several thermoplastic materials are available today for dental use including flexiplast, proflex system, Bio-dentaplast, flexite, valplast, and flexible resin system.[10]

Many polymers yield simple gases on heating, such as water vapor, ammonia, acetic acid, carbon dioxide, and nitrogen and carboxyl group. Increasing temperature is a key to obtain accurate mass percentage, for measurement of material composition, and to test the polymer susceptibility to hydrolysis. Thermal analysis (Thermogravimetric Analyzer, TGA) is a useful and reliable method for studying the decomposition and thermal stability of materials under a variety of conditions. Thermogravimetric analyzer in polymers helps in determining their thermal stability as well as offering compositional analysis of polymers by measuring the weight loss values as a function of temperature. Thermogravimetric curves are referred for the characterization and/or identification of the

analyzed sample as they rely on the unique sequence of the physiochemical reaction which follows heating of the samples and is an inherent property of the samples' molecular structure.[14-16]

Infrared (IR) spectrum is a helpful tool for assessment of the molecular structure and composition of a given substance. Therefore, it is also known as molecular spectrum. When exposing the sample to IR light with continuously changing frequency, irradiation of certain frequencies is absorbed by the molecule and is subject to vibration or rotation, causing the change of dipole moment. Thus, transforming the molecule from normal state to excited state weakens the intensity of the corresponding transmitted light in the absorption region. The IR software is used to obtain the IR spectrum. Infrared offers many advantages as it does not affect the sample under analysis, with its ability for analyzing organic and inorganic compounds of different physical states and exterior forms (solid, liquid, gas, elastic, fibrous, coating, and/or thin film).[17,18]

A large number of standard IR spectrogram for numerous chemical compounds have already been reported and can, therefore, be referred to in spectra analysis, which makes Fourier transform infrared (FTIR) spectroscopy an effective method for the quantitative analysis of polymer materials since it is of high resolution and high scanning speed.[19]

Nowadays, it is very common in modern analysis to combine technology of FTIR with that of TGA to obtain thermogravimetric curve and the IR spectrum of the weight loss material, hence enabling the determination of the real composition of vapor generated in the various weight loss stages and the decomposition process.[17,18]

Hardly any information is available on the thermal stability and the chemical composition of thermoplastic resin material. The aim of this study is the *in vitro* evaluation of thermoplastic resin material using TGA and FTIR to assess its suitability for use as a denture base material compared with the traditionally used PMMA denture base material.

MATERIALS AND METHODS

Sample Preparation

A total of 40 disk-shaped specimens measuring 25 mm in diameter and 3 mm in thickness were prepared. Two groups of samples each containing 20 disks were prepared. Group I was thermoplastic acrylic resin prepared using injection molding technique (Bre.flex polyamide; Bredent, GmbH & Co. KG, Senden, Germany). Group II was heat-cured PMMA resin using the conventional compression molding technique (vertex RS; Dentimex, the Netherlands).

Group I specimens were first prepared using the lost wax technique. Wax disks were prepared (Dental wax; Lordell trading, New South Wales, Australia), then invested in stone and placed in injection molding flask, where eliminated wax was replaced with injected molten thermoplastic material according to the manufacturer's instructions. The injection molding machine was used with the metallic cartridges containing thermoplastic grains heated to plasticize the resin at injection pressure of 720–750 kPa, 220°C for 15 minutes. Once processing was completed, the flask was allowed to bench cool and the specimens were finished and polished.

Group II wax specimens were similarly prepared and invested in stone and then flasked, packed, and processed using heat-cured PMMA according to the manufacturer's instructions. Following deflasking, the specimens were finished and polished.[20,21]

All the specimens were tested *in vitro* for thermal stability using TGA and FTIR.

Thermogravimetric Analysis

For each experiment, 5 mg of each sample from each group was used and placed in a platinum pan inside the TGA (Shimadzu, TGA-50, Kyoto, Japan). Thermogravimetric analyzer measured and recorded the dynamics of sample weight loss with increasing temperature (25–30°C), at a controlled heating rate of 10°C/min. Nitrogen was used as carrier gas of the pyrolysis of the studied samples. Pyrolysis is the chemical decomposition of organic materials by heating in the absence of oxygen. The flow rate of the carrier gas was fixed at 20 ml/min and the periodic calibration of TGA was observed.[22]

Fourier Transform Infrared Spectrophotometer

Following the manufacturer's specification (IR spectrum; Shimadzu, IR spectrophotometer-8400S, Kyoto, Japan), the polymerized specimens of groups I and II were grounded with 10 times its bulk of pure potassium bromide (KBr). The mixture was pressed into a disk using the special mold and the hydraulic press of the apparatus. The disk specimens were placed on the magnetic sheet and the beam was directed onto the sample to be transmitted through the surface of the sample and onto the detector for final measurement. The interferogram signals were measured, digitized, and sent to the computer where Fourier transformation takes place. The final IR spectra were collected on a FTIR using software program IR Solution.

RESULTS

Thermogravimetric Analysis

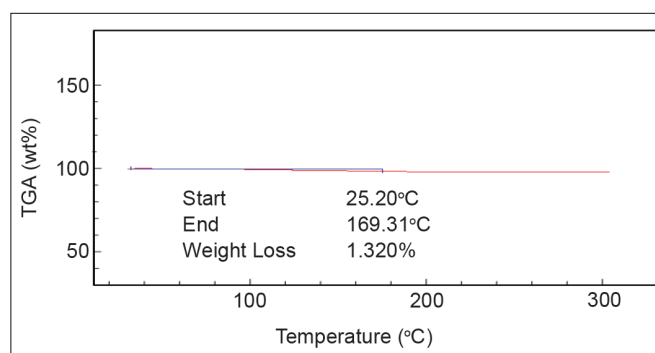Thermal stability of the samples was studied using TGA under nitrogen atmosphere to record the percentage of weight loss as a result of temperature increase. Graph 1 demonstrates the TGA curve for group I. It is clearly shown that increasing the temperature from 25 to 169°C resulted in about 1.3% of the material loss, whereas the material remains thermally stable when the temperature was raised further, displaying no weight loss (from 169 to 200°C). Thermogravimetric analysis results for group II samples are shown Graphs 1 and 2; the material showed 2.24% weight loss at 171°C, and further weight loss was observed with heating to 230°C (about 12.025% weight loss).
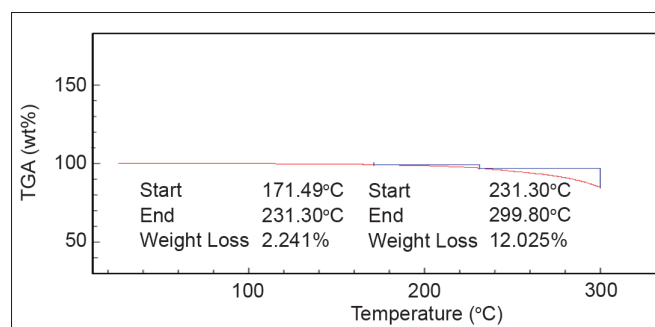
FTIR Analysis

Fourier transform infrared spectrophotometer chemical analysis revealed both the composition and the bonds present within both groups in the range of 400–4000 cm^{-1}, thus identifying the organic function on the surface.

Table 1 and Graph 3 present the FTIR spectra for group I, the resonating peak at 3,458.48 cm^{-1} represents an amine group (NH), while the peak at 1,639.55 cm^{-1} represents the double bond between carbon atoms (C=C) that disappeared in the polymerized spectrum of thermoplastic resin. Other peaks were observed at 646.17 and 1111.03 cm^{-1} and are assigned to C–H (or Cl-acid) and Si-CH$_3$ respectively.

Fourier transform infrared spectrophotometer results for group II are shown in Table 2 and Graph 4. These
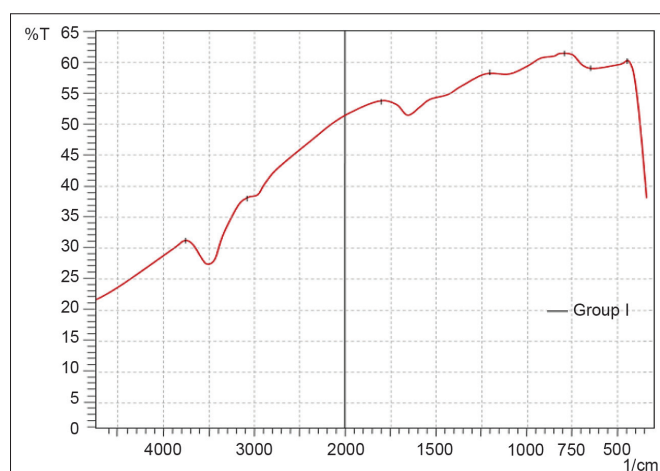

Graph 1: Thermogravimetric analysis curve for samples (group I) represents the relation of temperature (°C) to loss of material weight %

Graph 2: Thermogravimetric analysis curve for samples (group II) represents the relation of temperature (°C) to loss of material weight %

Table 1: Fourier transform infrared spectrophotometer characterization of group I

Peak cm^{-1}	Assignments		Intensity
3,458.48	N–H	Amines	Medium
2,951.19	O–H	Carboxylic acid	Strong and very broad
1,639.55	C≡C	Alkenes	Symmetric, reduced intensity, and strong
1,111.03	Si–CH$_3$	Silicon function	Strong and sharp
646.17	C–H	Alkynes or C–Cl acid chlorides	Deformation stretch, weak to medium

Table 2: Fourier transform infrared spectrophotometer characterization of group II

Peak cm^{-1}	Assignments		Intensity
3,454.62	O–H	Alcohol and phenols	Strong and broad
2,972.40	CH$_3$, CH$_2$ and C-H	Alkanes	Strong
2,605.92	O–H	Carboxylic acids	Very broad
2,384.10	O–H	Carboxylic acids	Strong and very broad
2,017.61	C≡C	Alkynes	Asymmetric stretch
1,734.06	C=O	Aldehydes and ketones	Stretch
1,456.30	CH$_2$	Aldehydes and ketones	Bending
1,190.12	C–O	Alcohol stretch	Medium

Graph 3: Fourier transform infrared spectrophotometer analysis of group I

Graph 4: Fourier transform infrared spectrophotometer analysis of group II
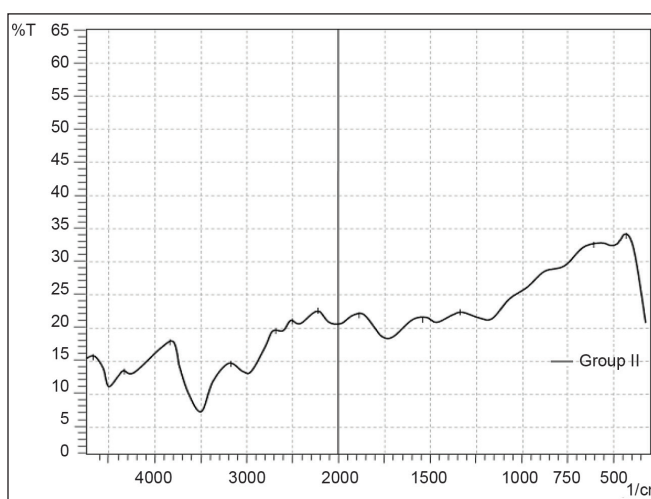
show strong broad resonating peak at 3,454.62 cm^{-1} representative of OH group. The resonating strong peak at 2,972.40 cm^{-1} is that of CH$_3$, CH$_2$ and C–H groups, while the peaks at 2,605.40 and 2,384.10 cm^{-1} are assigned to carboxylic acid O–H. Asymmetric stretch of C≡C alkane peaks is apparent at 2,017.61 cm^{-1}, and stretch of aldehyde and ketone peaks C=O is also demonstrated at 1,734.06 cm^{-1}. Finally, the medium peak at 1.190.12 cm^{-1} is assigned to alcohol C–O.

DISCUSSION

The fabrication of removable prosthesis for the complete and/or partially edentulous patient meets numerous challenges as dentures are subjected to high biting stresses, temperature fluctuation (25–45°C),[23] and pH changes from acidic to alkaline. In addition to this, oral environments are moist and warm and rich in enzymes and bacteria, which can cause the injury to soft tissues in contact with the denture polymers from the toxic leaching or breakdown of the material.[24-26] Designing removable prosthesis also encounters many challenges such as interferences in the edentulous arch, soft tissue and bony undercuts, tilted teeth with or without deranged occlusion; all these factors complicate treatment. Thermo-

plastic acrylic resin materials are supposed to overcome these problems. The disadvantages associated with using heat-cured PMMA are also overcome, since it is completely free of residual monomers which results in a homogenous composition. The theory of thermoplastic resin is based on a physical reaction (heat) and no polymerization reaction. These materials are used with an injection molding system that produces high material density and better surface quality since weighing or mixing problems are eliminated by the preinstalled programs, thus minimizing the risk of error.[7,27]

The current study examined changes in the weight of a sample in relation to changes in temperature using TGA, which quantifies loss of water, plasticizer, solvent, as well as oxidation, decarboxylation, and decomposition. Additionally, TGA provides information regarding relative rates of polymer decomposition and the estimation of the lifetime of polymer at various temperatures.

The current investigation demonstrated that thermoplastic resin material (group I) initially underwent

minute weight loss, which may be attributed to the loss of adsorbed surface water and moisture bound to the surface. The material remained stable afterward, displaying no weight loss and no degradation despite increasing the temperature up to 300°C. Such findings might be attributed to the development of polyamide resin combined with other polymers that led to improvement of water sorption properties and heat and chemical resistance. These were confirmed by FTIR results.[28] Others have reported about decomposition of medical-grade polymers at temperatures above 400°C when using similar analytically conditions (TGA in nitrogen).[6]

Heat-cured PMMA resin (group II) showed TGA weight loss, which may be attributed to crystalline water or due to decomposition of the material at temperatures above 170°C. The liability of heat-cured PMMA (group II) to high water absorption and volumetric changes is well established[17] and was shown to hydrolyze by heat at temperatures above 200°C with signs of oxidation accompanied with the appearance of carbonyl $C=C$ groups.[29] Other studies reported on thermal decomposition of heat-cured PMMA using TGA and reported the start of decomposition at 142–148°C with weight loss lower than 4% and a second step of decomposition at 332–339°C with weight loss lower than 14%. The final step of decomposition was at 369–380°C with a weight loss lower than 50% and complete decomposition at 400–420°C.[30] Others reported decomposition of heat-cured PMMA at 200°C with noted deflection at 250°C, which ends at 400°C. The two peaks indicating decomposition were noted at 266 and 377°C, indicating a two-step decomposition process.[2]

This study utilized FTIR spectrum for chemical analysis of all samples due to its high specificity and sensitivity for detection of the chemical structure as it offers a chemical fingerprint of samples examined according to the absorption peaks that correspond to the frequencies of vibration within the bonds of the atoms that make up the material tested. Infrared spectroscopy can result in a positive and qualitative identification of material tested.[31-33]

Preliminary studies have given few information about the definite or exact composition of some thermoplastic materials (Bre.flex polyamide). Polyamide resins (–$CO(CH_2)(6NH)n(5-NH)$) are produced by direct amidation of a diacid with a diamine. Alternatively, they can be produced by self-amidation of an amino acid. Polyamides are polymers in which the repeating units are held together by amide links. An amide group has the formula (–$CONH_2$) that contains an amide link. Upon polymerization, the ring opens, and the molecules join up in a continuous chain.[34,35]

The tested thermoplastic material (group I) was found to contain an amine group (N–H) not present in PMMA (group II). Additionally, the presence of a strong hydrogen bond is believed to be responsible for the material's melting point through ring-opening polymerization and/or step polymerization. Thus, the monomer disappears early in the reaction before the production of any polymer of sufficient high molecular weight. No catalyst is required for ring-opening polymerization and the amine group is capable of reacting with carboxylic acid to make high molecular weight polymer in addition to the reaction between OH-containing molecules and COOH-containing molecules. Finally, the double bond between two carbon atom ($C=C$) decreased absorbance at frequency 1,639.55 cm^{-1}, indicating polymerization progress in group I which is in agreement with other publications. The detection of (–CH_3) indicates the presence of methylene group in the tested samples (group I) where the organic CH_3 is attached to inorganic Si (Silicon) to act as reinforcement filler (Si–CH_3). It is thought that thermoplastic materials containing methylene group have better moisture resistance and dimensional stability.[36]

Studies have shown that injection molding thermoplastics tested at 210°C and under steam pressure of 250 Psi or 1.7 MPa prevent salt precipitation and are free of residual monomer as the monomer of the thermoplastic resin has a better ability to polymerize due to higher molecular chain motions and neutralization of the immobilization of MMA at high temperature.[12]

The IR spectrum of group II revealed the presence of monomer in heat-cured PMMA.

CONCLUSION

Current standards for prosthetic rehabilitation of aging people to obtain higher quality of life fuel the need for new and improved biomaterials; the excellent thermal stability of polyamides and the absence of residual monomer within the polymerized material make it a suitable option for the fabrication of complete and partial dentures.[37] Injection molded system is a safe nontoxic material that is time saving and thermally stable, capable of resisting color and/or dimensional decomposition in the oral environment during clinical services.

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